

Under the Paperwork Reduction Act of 1995, no persons are required to respond to a collection of information unless it displays a valid OMB control number.

| | | |
|--|---|--|
| TRANSMITTAL LETTER TO THE UNITED STATES DESIGNATED/ELECTED OFFICE (DO/EO/US) CONCERNING A SUBMISSION UNDER 35 U.S.C. 371 | | Attorney Docket No. GBL0007/US U.S. Application No. (if known, see 37 CFR 1.5) |
| International Application No. PCT/GB2016/051688 | International Filing Date June 8, 2016 | Priority Date Claimed June 9, 2015 |
| Title of Invention COATINGS | | |
| First Named Inventor Stephen Richard COULSON | | |
| Applicant herewith submits to the United States Designated/Elected Office (DO/EO/US) the following items and other information. | | |
| 1. <input checked="" type="checkbox"/> This is an express request to begin national examination procedures (35 U.S.C. 371(f)). NOTE: The express request under 35 U.S.C. 371(f) will not be effective unless the requirements under 35 U.S.C. 371(c)(1), (2), and (4) for payment of the basic national fee, copy of the International Application and English translation thereof (if required), and the oath or declaration of the inventor(s) have been received. | | |
| 2. <input checked="" type="checkbox"/> A copy of the International Application (35 U.S.C. 371(c)(2)) is attached hereto (not required if the International Application was previously communicated by the International Bureau or was filed in the United States Receiving Office (RO/US)). | | |
| 3. An English language translation of the International Application (35 U.S.C. 371(c)(2)) | | |
| a. <input type="checkbox"/> is attached hereto. | | |
| b. <input type="checkbox"/> has been previously submitted under 35 U.S.C. 154(d)(4). | | |
| 4. An oath or declaration of the inventor(s) (35 U.S.C. 371(c)(4)) | | |
| a. <input type="checkbox"/> is attached. | | |
| b. <input type="checkbox"/> was previously filed in the international phase under PCT Rule 4.17(iv). | | |
| Items 5 to 8 below concern amendments made in the international phase. | | |
| <u>PCT Article 19 and 34 amendments</u> | | |
| 5. <input type="checkbox"/> Amendments to the claims under PCT Article 19 are attached (not required if communicated by the International Bureau) (35 U.S.C. 371(c)(3)). | | |
| 6. <input type="checkbox"/> English translation of the PCT Article 19 amendment is attached (35 U.S.C. 371(c)(3)). | | |
| 7. <input type="checkbox"/> English translation of annexes (Article 19 and/or 34 amendments only) of the International Preliminary Examination Report is attached (35 U.S.C. 371(c)(5)). | | |
| <u>Cancellation of amendments made in the international phase</u> | | |
| 8a. <input type="checkbox"/> Do not enter the amendment made in the international phase under PCT Article 19. | | |
| 8b. <input type="checkbox"/> Do not enter the amendment made in the international phase under PCT Article 34. | | |
| NOTE: A proper amendment made in English under Article 19 or 34 will be entered in the U.S. national phase application absent a clear instruction from applicant not to enter the amendment(s). | | |
| The following items 9 to 17 concern a document(s) or information included. | | |
| 9. <input checked="" type="checkbox"/> An Information Disclosure Statement under 37 CFR 1.97 and 1.98. | | |
| 10. <input type="checkbox"/> A preliminary amendment. | | |
| 11. <input checked="" type="checkbox"/> An Application Data Sheet under 37 CFR 1.76. | | |
| 12. <input type="checkbox"/> A substitute specification. NOTE: A substitute specification cannot include claims. See 37 CFR 1.125(b). | | |
| 13. <input checked="" type="checkbox"/> A power of attorney and/or change of address letter. | | |
| 14. <input type="checkbox"/> A computer-readable form of the sequence listing in accordance with PCT Rule 13ter.3 and 37 CFR 1.821-1.825. | | |
| 15. <input type="checkbox"/> Assignment papers (cover sheet and document(s)). Name of Assignee: _____ | | |
| 16. <input type="checkbox"/> 37 CFR 3.73(c) Statement (when there is an Assignee). | | |

This collection of information is required by 37 CFR 1.414 and 1.491-1.492. The information is required to obtain or retain a benefit by the public, which is to file (and by the USPTO to process) an application. Confidentiality is governed by 35 U.S.C. 122 and 37 CFR 1.11 and 1.14. This collection is estimated to take 15 minutes to complete, including gathering, preparing, and submitting the completed application form to the USPTO. Time will vary depending upon the individual case. Any comments on the amount of time you require to complete this form and/or suggestions for reducing this burden should be sent to the Chief Information Officer, U.S. Patent and Trademark Office, U.S. Department of Commerce, P.O. Box 1450, Alexandria, VA 22313-1450. DO NOT SEND FEES OR COMPLETED FORMS TO THIS ADDRESS. SEND TO: Mail Stop PCT, Commissioner for Patents, P.O. Box 1450, Alexandria, VA 22313-1450.

Favored Ex. 1028

Ex. 1028 - Favored - p. 1

Under the Paperwork Reduction Act of 1995, no persons are required to respond to a collection of information unless it displays a valid OMB control number.

| | | | | | |
|---|--------------|---|---------|--|---------------------|
| U.S. APPLN. No. (if known – see 37 CFR 1.5) | | INTERNATIONAL APPLICATION No. PCT/GB2016/051688 | | ATTORNEY DOCKET No. GBL0007/US | |
| 17. <input type="checkbox"/> Other items or information: | | | | | |
| The following fees have been submitted. | | | | CALCULATIONS | PTO USE ONLY |
| 18. <input checked="" type="checkbox"/> Basic national fee (37 CFR 1.492(a)) \$280 | | | | \$ 280 | |
| 19. <input checked="" type="checkbox"/> Examination fee (37 CFR 1.492(c)) If the written opinion prepared by ISA/US or the international preliminary examination report prepared by IPEA/US indicates all claims satisfy provisions of PCT Article 33(1)-(4) \$0 All other situations \$720 | | | | \$ 720 | |
| 20. <input checked="" type="checkbox"/> Search fee (37 CFR 1.492(b)) If the written opinion prepared by ISA/US or the international preliminary examination report prepared by IPEA/US indicates all claims satisfy provisions of PCT Article 33(1)-(4) \$0 Search fee (37 CFR 1.445(a)(2)) has been paid on the international application to the USPTO as an International Searching Authority \$120 International Search Report prepared by an ISA other than the US and provided to the Office or previously communicated to the US by the IB \$480 All other situations \$600 | | | | \$ 480 | |
| TOTAL OF 18, 19, and 20 = | | | | \$ 1,480 | |
| <input type="checkbox"/> Additional fee for specification and drawings filed in paper over 100 sheets (excluding sequence listing in compliance with 37 CFR 1.821(c) or (e) in an electronic medium or computer program listing in an electronic medium) (37 CFR 1.492(j)). Fee for each additional 50 sheets of paper or fraction thereof \$400 | | | | | |
| Total Sheets | Extra Sheets | Number of each addition 50 or fraction thereof (round up to a whole number) | RATE | | |
| - 100 = | / 50 = | | x \$400 | \$ | |
| Surcharge of \$140.00 for furnishing any of the search fee, examination fee, or the oath or declaration after the date of commencement of the national stage (37 CFR 1.492(h)). | | | | \$ 140 | |
| CLAIMS | NUMBER FILED | NUMBER EXTRA | RATE | | |
| Total claims | - 20 = | | x \$80 | \$ | |
| Independent claims | - 3 = | | x \$420 | \$ | |
| MULTIPLE DEPENDENT CLAIM(S) (if applicable) | | | + \$780 | \$ | |
| Processing fee of \$140.00 for furnishing the English translation later than 30 months from the earliest claimed priority date (37 CFR 1.492(i)). | | | | + | \$ |
| TOTAL OF ABOVE CALCULATIONS = | | | | \$ 1,620 | |
| <input type="checkbox"/> Applicant asserts small entity status. See 37 CFR 1.27. Fees above are reduced by 1/3. | | | | | |
| <input type="checkbox"/> Applicant certifies micro entity status. See 37 CFR 1.29. Fees above are reduced by 3/4. Applicant must attach form PTO/SB/15A or B or equivalent. | | | | | |
| TOTAL NATIONAL FEE = | | | | \$ 1,620 | |
| Fee for recording the enclosed assignment (37 CFR 1.21(h)). The assignment must be accompanied by an appropriate cover sheet (37 CFR 3.28, 3.31). \$40.00 per property. | | | | + | \$ |
| TOTAL FEES ENCLOSED = | | | | \$ 1,620 | |
| | | | | Amount to be refunded: | \$ |
| | | | | Amount to be charged: | \$ |

Under the Paperwork Reduction Act of 1995, no persons are required to respond to a collection of information unless it displays a valid OMB control number.

- a. ☐ A check in the amount of \$ _____ to cover the above fees is enclosed.
- b. ☐ Please charge my Deposit Account No. _____ in the amount of \$ _____ to cover the above fees.
- c. ☒ The Director is hereby authorized to charge additional fees which may be required, or credit any overpayment, to Deposit Account No. 50-1775 as follows:
- i. ☐ any required fee.
- ii. ☒ any required fee except for excess claims fees required under 37 CFR 1.492(d) and (e) and multiple dependent claim fee required under 37 CFR 1.492(f).
- d. ☒ Fees are to be charged to a credit card. **WARNING:** Information on this form may become public. Credit card information should not be included on this form. Provide credit card information and authorization on PTO-2038. The PTO-2038 should only be mailed or faxed to the USPTO. However, when paying the basic national fee, the PTO-2038 may NOT be faxed to the USPTO.

ADVISORY: If filing by EFS-Web, do NOT attach the PTO-2038 form as a PDF along with your EFS-Web submission. Please be advised that this is not recommended and by doing so your **credit card information may be displayed via PAIR**. To protect your information, it is recommended to pay fees online by using the electronic payment method.

NOTE: Where an appropriate time limit under 37 CFR 1.495 has not been met, a petition to revive (37 CFR 1.137(a) or (b)) must be filed and granted to restore the International Application to pending status.

Statement under 37 CFR 1.55 or 1.78 for AIA (First Inventor to File) Transition Applications

- ☐ This application (1) claims priority to or the benefit of an application filed before March 16, 2013, and (2) also contains, or contained at any time, a claim to a claimed invention that has an effective filing date on or after March 16, 2013.

NOTE 1: By providing this statement under 37 CFR 1.55 or 1.78, this application, with a filing date on or after March 16, 2013, will be examined under the first inventor to file provisions of the AIA.

NOTE 2: A U.S. national stage application may not claim priority to the international application of which it is the national phase. The filing date of a U.S. national stage application is the international filing date. See 35 U.S.C. 363.

Correspondence Address

- ☒ The address associated with Customer Number: 33072 OR ☐ Correspondence address below

| | | | | | |
|---------|--|-------|--|-----------|--|
| Name | | | | | |
| Address | | | | | |
| City | | State | | Zip Code | |
| Country | | | | Telephone | |
| Email | | | | | |

| | | | |
|-------------------|---|-----------------------------------|------------------|
| Signature |  | Date | December 8, 2017 |
| Name (Print/Type) | Paul J. Parins | Registration No. (Attorney/Agent) | 54,358 |

Under the Paperwork Reduction Act of 1995, no persons are required to respond to a collection of information unless it contains a valid OMB control number.

| | | | |
|---|----------|------------------------|------------|
| Application Data Sheet 37 CFR 1.76 | | Attorney Docket Number | GBL0007/US |
| | | Application Number | |
| Title of Invention | COATINGS | | |
| <p>The application data sheet is part of the provisional or nonprovisional application for which it is being submitted. The following form contains the bibliographic data arranged in a format specified by the United States Patent and Trademark Office as outlined in 37 CFR 1.76.</p> <p>This document may be completed electronically and submitted to the Office in electronic format using the Electronic Filing System (EFS) or the document may be printed and included in a paper filed application.</p> | | | |

Secrecy Order 37 CFR 5.2:

| | |
|--------------------------|---|
| <input type="checkbox"/> | Portions or all of the application associated with this Application Data Sheet may fall under a Secrecy Order pursuant to 37 CFR 5.2 (Paper filers only. Applications that fall under Secrecy Order may not be filed electronically.) |
|--------------------------|---|

Inventor Information:

| | | | | | |
|---|--|---|--------------------|---------------|------------------------|
| Inventor 1 | | | | | Remove |
| Legal Name | | | | | |
| Prefix | Given Name | Middle Name | Family Name | Suffix | |
| | Stephen | Richard | COULSON | | |
| Residence Information (Select One) <input type="radio"/> US Residency <input checked="" type="radio"/> Non US Residency <input type="radio"/> Active US Military Service | | | | | |
| City | Abingdon | Country of Residence¹ | GB | | |
| Mailing Address of Inventor: | | | | | |
| Address 1 | c/o P2i Ltd 9-12 North Central, 127 Olympic Avenue | | | | |
| Address 2 | Milton Park | | | | |
| City | Abingdon | State/Province | | | |
| Postal Code | OX14 4SA | Country¹ | GB | | |
| Inventor 2 | | | | | Remove |
| Legal Name | | | | | |
| Prefix | Given Name | Middle Name | Family Name | Suffix | |
| | Delwyn | | EVANS | | |
| Residence Information (Select One) <input type="radio"/> US Residency <input checked="" type="radio"/> Non US Residency <input type="radio"/> Active US Military Service | | | | | |
| City | Abingdon | Country of Residence¹ | GB | | |
| Mailing Address of Inventor: | | | | | |
| Address 1 | c/o P2i Ltd 9-12 North Central, 127 Olympic Avenue | | | | |
| Address 2 | Milton Park | | | | |
| City | Abingdon | State/Province | | | |
| Postal Code | OX14 4SA | Country¹ | GB | | |
| Inventor 3 | | | | | Remove |
| Legal Name | | | | | |

Under the Paperwork Reduction Act of 1995, no persons are required to respond to a collection of information unless it contains a valid OMB control number.

| | | | |
|---|----------|------------------------|------------|
| Application Data Sheet 37 CFR 1.76 | | Attorney Docket Number | GBL0007/US |
| | | Application Number | |
| Title of Invention | COATINGS | | |

| | | | | |
|---|-------------------|--------------------|--------------------|---------------|
| Prefix | Given Name | Middle Name | Family Name | Suffix |
| | Angeliki | | SIOKOU | |
| Residence Information (Select One) <input type="radio"/> US Residency <input checked="" type="radio"/> Non US Residency <input type="radio"/> Active US Military Service | | | | |

| | | | |
|-------------|----------|---|----|
| City | Abingdon | Country of Residenceⁱ | GB |
|-------------|----------|---|----|

Mailing Address of Inventor:

| | | | |
|--------------------|--|----------------------------|----|
| Address 1 | c/o P2i Ltd 9-12 North Central, 127 Olympic Avenue | | |
| Address 2 | Milton Park | | |
| City | Abingdon | State/Province | |
| Postal Code | OX14 4SA | Countryⁱ | GB |

| | |
|-------------------|---------------------------------------|
| Inventor 4 | <input type="button" value="Remove"/> |
| Legal Name | |

| | | | | |
|---|-------------------|--------------------|--------------------|---------------|
| Prefix | Given Name | Middle Name | Family Name | Suffix |
| | Clive | | TELFORD | |
| Residence Information (Select One) <input type="radio"/> US Residency <input checked="" type="radio"/> Non US Residency <input type="radio"/> Active US Military Service | | | | |

| | | | |
|-------------|----------|---|----|
| City | Abingdon | Country of Residenceⁱ | GB |
|-------------|----------|---|----|

Mailing Address of Inventor:

| | | | |
|--------------------|--|----------------------------|----|
| Address 1 | c/o P2i Ltd 9-12 North Central, 127 Olympic Avenue | | |
| Address 2 | Milton Park | | |
| City | Abingdon | State/Province | |
| Postal Code | OX14 4SA | Countryⁱ | GB |

All Inventors Must Be Listed - Additional Inventor Information blocks may be generated within this form by selecting the **Add** button.

Correspondence Information:

Enter either Customer Number or complete the Correspondence Information section below.
For further information see 37 CFR 1.33(a).

☐ An Address is being provided for the correspondence information of this application.

| | | | |
|------------------------|-------|--|---|
| Customer Number | 33072 | | |
| Email Address | | <input type="button" value="Add Email"/> | <input type="button" value="Remove Email"/> |

Under the Paperwork Reduction Act of 1995, no persons are required to respond to a collection of information unless it contains a valid OMB control number.

| | | | |
|---|----------|------------------------|------------|
| Application Data Sheet 37 CFR 1.76 | | Attorney Docket Number | GBL0007/US |
| | | Application Number | |
| Title of Invention | COATINGS | | |

Application Information:

| | | | |
|---|----------------|---|--------------------------|
| Title of the Invention | COATINGS | | |
| Attorney Docket Number | GBL0007/US | Small Entity Status Claimed | <input type="checkbox"/> |
| Application Type | Nonprovisional | | |
| Subject Matter | Utility | | |
| Total Number of Drawing Sheets (if any) | 6 | Suggested Figure for Publication (if any) | |

Filing By Reference:

Only complete this section when filing an application by reference under 35 U.S.C. 111(c) and 37 CFR 1.57(a). Do not complete this section if application papers including a specification and any drawings are being filed. Any domestic benefit or foreign priority information must be provided in the appropriate section(s) below (i.e., "Domestic Benefit/National Stage Information" and "Foreign Priority Information").

For the purposes of a filing date under 37 CFR 1.53(b), the description and any drawings of the present application are replaced by this reference to the previously filed application, subject to conditions and requirements of 37 CFR 1.57(a).

| | | |
|--|--------------------------|--|
| Application number of the previously filed application | Filing date (YYYY-MM-DD) | Intellectual Property Authority or Country |
| | | |

Publication Information:

| |
|--|
| <input type="checkbox"/> Request Early Publication (Fee required at time of Request 37 CFR 1.219) |
| <input type="checkbox"/> Request Not to Publish. I hereby request that the attached application not be published under 35 U.S.C. 122(b) and certify that the invention disclosed in the attached application has not and will not be the subject of an application filed in another country, or under a multilateral international agreement, that requires publication at eighteen months after filing. |

Representative Information:

| | | | |
|---|-------|--|--|
| Representative information should be provided for all practitioners having a power of attorney in the application. Providing this information in the Application Data Sheet does not constitute a power of attorney in the application (see 37 CFR 1.32). Either enter Customer Number or complete the Representative Name section below. If both sections are completed the customer Number will be used for the Representative Information during processing. | | | |
| Please Select One: <input checked="" type="radio"/> Customer Number <input type="radio"/> US Patent Practitioner <input type="radio"/> Limited Recognition (37 CFR 11.9) | | | |
| Customer Number | 33072 | | |

| | | | |
|---|----------|------------------------|------------|
| Application Data Sheet 37 CFR 1.76 | | Attorney Docket Number | GBL0007/US |
| | | Application Number | |
| Title of Invention | COATINGS | | |

Domestic Benefit/National Stage Information:

This section allows for the applicant to either claim benefit under 35 U.S.C. 119(e), 120, 121, 365(c), or 386(c) or indicate National Stage entry from a PCT application. Providing benefit claim information in the Application Data Sheet constitutes the specific reference required by 35 U.S.C. 119(e) or 120, and 37 CFR 1.78.

When referring to the current application, please leave the "Application Number" field blank.

| | | | |
|---|------------------------|--------------------------|------------------------------------|
| Prior Application Status | Pending | Remove | |
| Application Number | Continuity Type | Prior Application Number | Filing or 371(c) Date (YYYY-MM-DD) |
| | a 371 of international | PCT/GB2016/051688 | 2016-06-08 |
| Additional Domestic Benefit/National Stage Data may be generated within this form by selecting the Add button. | | | |

Foreign Priority Information:

This section allows for the applicant to claim priority to a foreign application. Providing this information in the application data sheet constitutes the claim for priority as required by 35 U.S.C. 119(b) and 37 CFR 1.55. When priority is claimed to a foreign application that is eligible for retrieval under the priority document exchange program (PDX)ⁱ the information will be used by the Office to automatically attempt retrieval pursuant to 37 CFR 1.55(i)(1) and (2). Under the PDX program, applicant bears the ultimate responsibility for ensuring that a copy of the foreign application is received by the Office from the participating foreign intellectual property office, or a certified copy of the foreign priority application is filed, within the time period specified in 37 CFR 1.55(g)(1).

| | | | |
|--|----------------------|--------------------------|--|
| | | | Remove |
| Application Number | Country ⁱ | Filing Date (YYYY-MM-DD) | Access Code ⁱ (if applicable) |
| 15386017.6 | EP | 2015-06-09 | |
| Additional Foreign Priority Data may be generated within this form by selecting the Add button. | | | |

Statement under 37 CFR 1.55 or 1.78 for AIA (First Inventor to File) Transition Applications

| |
|--|
| <p>This application (1) claims priority to or the benefit of an application filed before March 16, 2013 and (2) also contains, or contained at any time, a claim to a claimed invention that has an effective filing date on or after March 16, 2013.</p> <p><input type="checkbox"/> NOTE: By providing this statement under 37 CFR 1.55 or 1.78, this application, with a filing date on or after March 16, 2013, will be examined under the first inventor to file provisions of the AIA.</p> |
|--|

| | | | |
|---|----------|------------------------|------------|
| Application Data Sheet 37 CFR 1.76 | | Attorney Docket Number | GBL0007/US |
| | | Application Number | |
| Title of Invention | COATINGS | | |

Authorization or Opt-Out of Authorization to Permit Access:

When this Application Data Sheet is properly signed and filed with the application, applicant has provided written authority to permit a participating foreign intellectual property (IP) office access to the instant application-as-filed (see paragraph A in subsection 1 below) and the European Patent Office (EPO) access to any search results from the instant application (see paragraph B in subsection 1 below).

Should applicant choose not to provide an authorization identified in subsection 1 below, applicant **must opt-out** of the authorization by checking the corresponding box A or B or both in subsection 2 below.

NOTE: This section of the Application Data Sheet is **ONLY** reviewed and processed with the **INITIAL** filing of an application. After the initial filing of an application, an Application Data Sheet cannot be used to provide or rescind authorization for access by a foreign IP office(s). Instead, Form PTO/SB/39 or PTO/SB/69 must be used as appropriate.

1. Authorization to Permit Access by a Foreign Intellectual Property Office(s)

A. Priority Document Exchange (PDX) - Unless box A in subsection 2 (opt-out of authorization) is checked, the undersigned hereby **grants the USPTO authority** to provide the European Patent Office (EPO), the Japan Patent Office (JPO), the Korean Intellectual Property Office (KIPO), the State Intellectual Property Office of the People's Republic of China (SIPO), the World Intellectual Property Organization (WIPO), and any other foreign intellectual property office participating with the USPTO in a bilateral or multilateral priority document exchange agreement in which a foreign application claiming priority to the instant patent application is filed, access to: (1) the instant patent application-as-filed and its related bibliographic data, (2) any foreign or domestic application to which priority or benefit is claimed by the instant application and its related bibliographic data, and (3) the date of filing of this Authorization. See 37 CFR 1.14(h)(1).

B. Search Results from U.S. Application to EPO - Unless box B in subsection 2 (opt-out of authorization) is checked, the undersigned hereby **grants the USPTO authority** to provide the EPO access to the bibliographic data and search results from the instant patent application when a European patent application claiming priority to the instant patent application is filed. See 37 CFR 1.14(h)(2).

The applicant is reminded that the EPO's Rule 141(1) EPC (European Patent Convention) requires applicants to submit a copy of search results from the instant application without delay in a European patent application that claims priority to the instant application.

2. Opt-Out of Authorizations to Permit Access by a Foreign Intellectual Property Office(s)

☐ A. Applicant **DOES NOT** authorize the USPTO to permit a participating foreign IP office access to the instant application-as-filed. If this box is checked, the USPTO will not be providing a participating foreign IP office with any documents and information identified in subsection 1A above.

☐ B. Applicant **DOES NOT** authorize the USPTO to transmit to the EPO any search results from the instant patent application. If this box is checked, the USPTO will not be providing the EPO with search results from the instant application.

NOTE: Once the application has published or is otherwise publicly available, the USPTO may provide access to the application in accordance with 37 CFR 1.14.

| | | | |
|---|----------|------------------------|------------|
| Application Data Sheet 37 CFR 1.76 | | Attorney Docket Number | GBL0007/US |
| | | Application Number | |
| Title of Invention | COATINGS | | |

Applicant Information:

| | | | |
|---|--|--|----------|
| Providing assignment information in this section does not substitute for compliance with any requirement of part 3 of Title 37 of CFR to have an assignment recorded by the Office. | | | |
| Applicant 1 | | | |
| <p>If the applicant is the inventor (or the remaining joint inventor or inventors under 37 CFR 1.45), this section should not be completed. The information to be provided in this section is the name and address of the legal representative who is the applicant under 37 CFR 1.43; or the name and address of the assignee, person to whom the inventor is under an obligation to assign the invention, or person who otherwise shows sufficient proprietary interest in the matter who is the applicant under 37 CFR 1.46. If the applicant is an applicant under 37 CFR 1.46 (assignee, person to whom the inventor is obligated to assign, or person who otherwise shows sufficient proprietary interest) together with one or more joint inventors, then the joint inventor or inventors who are also the applicant should be identified in this section.</p> | | | |
| <input type="button" value="Clear"/> | | | |
| <input type="radio"/> Assignee | | <input type="radio"/> Legal Representative under 35 U.S.C. 117 | |
| | | <input type="radio"/> Joint Inventor | |
| <input checked="" type="radio"/> Person to whom the inventor is obligated to assign. | | <input type="radio"/> Person who shows sufficient proprietary interest | |
| If applicant is the legal representative, indicate the authority to file the patent application, the inventor is: | | | |
| | | | |
| Name of the Deceased or Legally Incapacitated Inventor: <input type="text"/> | | | |
| If the Applicant is an Organization check here. <input checked="" type="checkbox"/> | | | |
| Organization Name | P2i Ltd | | |
| Mailing Address Information For Applicant: | | | |
| Address 1 | 9-12 North Central, 127 Olympic Avenue | | |
| Address 2 | Milton Park | | |
| City | Abingdon | State/Province | |
| Country | GB | Postal Code | OX14 4SA |
| Phone Number | | Fax Number | |
| Email Address | | | |
| Additional Applicant Data may be generated within this form by selecting the Add button. | | | |

Assignee Information including Non-Applicant Assignee Information:

Providing assignment information in this section does not substitute for compliance with any requirement of part 3 of Title 37 of CFR to have an assignment recorded by the Office.

Under the Paperwork Reduction Act of 1995, no persons are required to respond to a collection of information unless it contains a valid OMB control number.

| | | | |
|---|----------|------------------------|------------|
| Application Data Sheet 37 CFR 1.76 | | Attorney Docket Number | GBL0007/US |
| | | Application Number | |
| Title of Invention | COATINGS | | |

Assignee 1

Complete this section if assignee information, including non-applicant assignee information, is desired to be included on the patent application publication. An assignee-applicant identified in the "Applicant Information" section will appear on the patent application publication as an applicant. For an assignee-applicant, complete this section only if identification as an assignee is also desired on the patent application publication.

If the Assignee or Non-Applicant Assignee is an Organization check here. ☐

| | | | | |
|--------|------------|-------------|-------------|--------|
| Prefix | Given Name | Middle Name | Family Name | Suffix |
| | | | | |

Mailing Address Information For Assignee including Non-Applicant Assignee:

| | | | |
|----------------------|--|----------------|--|
| Address 1 | | | |
| Address 2 | | | |
| City | | State/Province | |
| Country ¹ | | Postal Code | |
| Phone Number | | Fax Number | |
| Email Address | | | |


Additional Assignee or Non-Applicant Assignee Data may be generated within this form by selecting the Add button.

Signature:

NOTE: This Application Data Sheet must be signed in accordance with 37 CFR 1.33(b). However, if this Application Data Sheet is submitted with the INITIAL filing of the application and either box A or B is not checked in subsection 2 of the "Authorization or Opt-Out of Authorization to Permit Access" section, then this form must also be signed in accordance with 37 CFR 1.14(c).

This Application Data Sheet must be signed by a patent practitioner if one or more of the applicants is a juristic entity (e.g., corporation or association). If the applicant is two or more joint inventors, this form must be signed by a patent practitioner, all joint inventors who are the applicant, or one or more joint inventor-applicants who have been given power of attorney (e.g., see USPTO Form PTO/AIA/81) on behalf of all joint inventor-applicants.

See 37 CFR 1.4(d) for the manner of making signatures and certifications.

| | | | | | |
|------------|---|-----------|-------------------|---------------------|--------|
| Signature |  | | Date (YYYY-MM-DD) | 2017-12-08 | |
| First Name | Paul J. | Last Name | Parins | Registration Number | 54,358 |

Additional Signature may be generated within this form by selecting the Add button.

Under the Paperwork Reduction Act of 1995, no persons are required to respond to a collection of information unless it contains a valid OMB control number.

| | | | |
|---|----------|------------------------|------------|
| Application Data Sheet 37 CFR 1.76 | | Attorney Docket Number | GBL0007/US |
| | | Application Number | |
| Title of Invention | COATINGS | | |

This collection of information is required by 37 CFR 1.76. The information is required to obtain or retain a benefit by the public which is to file (and by the USPTO to process) an application. Confidentiality is governed by 35 U.S.C. 122 and 37 CFR 1.14. This collection is estimated to take 23 minutes to complete, including gathering, preparing, and submitting the completed application data sheet form to the USPTO. Time will vary depending upon the individual case. Any comments on the amount of time you require to complete this form and/or suggestions for reducing this burden, should be sent to the Chief Information Officer, U.S. Patent and Trademark Office, U.S. Department of Commerce, P.O. Box 1450, Alexandria, VA 22313-1450. DO NOT SEND FEES OR COMPLETED FORMS TO THIS ADDRESS. **SEND TO: Commissioner for Patents, P.O. Box 1450, Alexandria, VA 22313-1450.**

| | | | |
|---|------------------------|-------------------------|--|
| INFORMATION DISCLOSURE STATEMENT BY APPLICANT (Not for submission under 37 CFR 1.99) | Application Number | | |
| | Filing Date | | |
| | First Named Inventor | Stephen Richard COULSON | |
| | Art Unit | | |
| | Examiner Name | | |
| | Attorney Docket Number | GBL0007/US | |

U.S.PATENTS

| Examiner Initial* | Cite No | Patent Number | Kind Code ¹ | Issue Date | Name of Patentee or Applicant of cited Document | Pages, Columns, Lines where Relevant Passages or Relevant Figures Appear |
|-------------------|---------|---------------|------------------------|------------|---|--|
| | 1 | | | | | |

If you wish to add additional U.S. Patent citation information please click the Add button.

U.S.PATENT APPLICATION PUBLICATIONS

| Examiner Initial* | Cite No | Publication Number | Kind Code ¹ | Publication Date | Name of Patentee or Applicant of cited Document | Pages, Columns, Lines where Relevant Passages or Relevant Figures Appear |
|-------------------|---------|--------------------|------------------------|------------------|---|--|
| | 1 | | | | | |

If you wish to add additional U.S. Published Application citation information please click the Add button.

FOREIGN PATENT DOCUMENTS

| Examiner Initial* | Cite No | Foreign Document Number ³ | Country Code ²ⁱ | Kind Code ⁴ | Publication Date | Name of Patentee or Applicant of cited Document | Pages, Columns, Lines where Relevant Passages or Relevant Figures Appear | T ⁵ |
|-------------------|---------|--------------------------------------|----------------------------|------------------------|------------------|---|--|--------------------------|
| | 1 | 98/58117 | WO | A1 | 1998-12-23 | THE SECRETARY OF STATE FOR DEFENCE | | <input type="checkbox"/> |
| | 2 | 2003/097245 | WO | A2 | 2003-11-27 | SURFACE INNOVATIONS LIMITED | | <input type="checkbox"/> |
| | 3 | 03/101621 | WO | A2 | 2003-12-11 | SURFACE INNOVATIONS LIMITED | | <input type="checkbox"/> |

**INFORMATION DISCLOSURE
STATEMENT BY APPLICANT**
(Not for submission under 37 CFR 1.99)

| | | |
|------------------------|-------------------------|--|
| Application Number | | |
| Filing Date | | |
| First Named Inventor | Stephen Richard COULSON | |
| Art Unit | | |
| Examiner Name | | |
| Attorney Docket Number | GBL0007/US | |

| | | | | | | | | |
|--|---|-------------|----|----|------------|---------------|--|--------------------------|
| | 4 | 2007/083122 | WO | A1 | 2007-07-26 | P2I LTD | | <input type="checkbox"/> |
| | 5 | 0834352 | EP | A1 | 1998-04-08 | CIBA-GEIGY AG | | <input type="checkbox"/> |
| | 6 | 2014/155099 | WO | A1 | 2014-10-02 | SEMBLANT LTD | | <input type="checkbox"/> |
| | 7 | 2015/128627 | WO | A1 | 2015-09-03 | P2I LTD | | <input type="checkbox"/> |
| | 8 | 2016/198855 | WO | A1 | 2016-12-15 | P2I LTD | | <input type="checkbox"/> |
| | 9 | 2016/198856 | WO | A1 | 2016-12-15 | P2I LTD | | <input type="checkbox"/> |

If you wish to add additional Foreign Patent Document citation information please click the Add button

NON-PATENT LITERATURE DOCUMENTS

| Examiner Initials* | Cite No | Include name of the author (in CAPITAL LETTERS), title of the article (when appropriate), title of the item (book, magazine, journal, serial, symposium, catalog, etc), date, pages(s), volume-issue number(s), publisher, city and/or country where published. | T ⁵ |
|--------------------|---------|---|--------------------------|
| | 1 | International Search Report and Written Opinion for International Application No. PCT/GB2016/051688, mailed November 8, 2016 (9 pages) | <input type="checkbox"/> |

If you wish to add additional non-patent literature document citation information please click the Add button

EXAMINER SIGNATURE

| | | | |
|--------------------|--|-----------------|--|
| Examiner Signature | | Date Considered | |
|--------------------|--|-----------------|--|

*EXAMINER: Initial if reference considered, whether or not citation is in conformance with MPEP 609. Draw line through a citation if not in conformance and not considered. Include copy of this form with next communication to applicant.

**INFORMATION DISCLOSURE
STATEMENT BY APPLICANT**
(Not for submission under 37 CFR 1.99)

| | |
|------------------------|-------------------------|
| Application Number | |
| Filing Date | |
| First Named Inventor | Stephen Richard COULSON |
| Art Unit | |
| Examiner Name | |
| Attorney Docket Number | GBL0007/US |

¹ See Kind Codes of USPTO Patent Documents at www.USPTO.GOV or MPEP 901.04. ² Enter office that issued the document, by the two-letter code (WIPO Standard ST.3). ³ For Japanese patent documents, the indication of the year of the reign of the Emperor must precede the serial number of the patent document. ⁴ Kind of document by the appropriate symbols as indicated on the document under WIPO Standard ST.16 if possible. ⁵ Applicant is to place a check mark here if English language translation is attached.

**INFORMATION DISCLOSURE
STATEMENT BY APPLICANT**
(Not for submission under 37 CFR 1.99)

| | |
|------------------------|-------------------------|
| Application Number | |
| Filing Date | |
| First Named Inventor | Stephen Richard COULSON |
| Art Unit | |
| Examiner Name | |
| Attorney Docket Number | GBL0007/US |

CERTIFICATION STATEMENT

Please see 37 CFR 1.97 and 1.98 to make the appropriate selection(s):

- ☐ That each item of information contained in the information disclosure statement was first cited in any communication from a foreign patent office in a counterpart foreign application not more than three months prior to the filing of the information disclosure statement. See 37 CFR 1.97(e)(1).

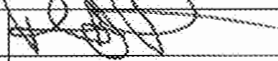
OR

- ☐ That no item of information contained in the information disclosure statement was cited in a communication from a foreign patent office in a counterpart foreign application, and, to the knowledge of the person signing the certification after making reasonable inquiry, no item of information contained in the information disclosure statement was known to any individual designated in 37 CFR 1.56(c) more than three months prior to the filing of the information disclosure statement. See 37 CFR 1.97(e)(2).

- ☐ See attached certification statement.
- ☐ The fee set forth in 37 CFR 1.17 (p) has been submitted herewith.
- ☒ A certification statement is not submitted herewith.

SIGNATURE

A signature of the applicant or representative is required in accordance with CFR 1.33, 10.18. Please see CFR 1.4(d) for the form of the signature.

| | | | |
|------------|---|---------------------|------------|
| Signature |  | Date (YYYY-MM-DD) | 2017-12-08 |
| Name/Print | Paul J. Parins | Registration Number | 54,358 |

This collection of information is required by 37 CFR 1.97 and 1.98. The information is required to obtain or retain a benefit by the public which is to file (and by the USPTO to process) an application. Confidentiality is governed by 35 U.S.C. 122 and 37 CFR 1.14. This collection is estimated to take 1 hour to complete, including gathering, preparing and submitting the completed application form to the USPTO. Time will vary depending upon the individual case. Any comments on the amount of time you require to complete this form and/or suggestions for reducing this burden, should be sent to the Chief Information Officer, U.S. Patent and Trademark Office, U.S. Department of Commerce, P.O. Box 1450, Alexandria, VA 22313-1450. DO NOT SEND FEES OR COMPLETED FORMS TO THIS ADDRESS. **SEND TO: Commissioner for Patents, P.O. Box 1450, Alexandria, VA 22313-1450.**

(51) International Patent Classification:
C09D 5/00 (2006.01)(21) International Application Number:
PCT/GB2016/051688(22) International Filing Date:
8 June 2016 (08.06.2016)

(25) Filing Language: English

(26) Publication Language: English

(30) Priority Data:
15386017.6 9 June 2015 (09.06.2015) EP(71) Applicant: **P2i LTD** [GB/GB]; 9-12 North Central, 127 Olympic Avenue, Milton Park, Abingdon Oxfordshire OX14 4SA (GB).(72) Inventors: **COULSON, Stephen Richard**; c/o P2i Ltd, 9-12 North Central, 127 Olympic Avenue, Milton Park, Abingdon Oxfordshire OX14 4SA (GB). **EVANS, Delwyn**; c/o P2i Ltd, 9-12 North Central, 127 Olympic Avenue, Milton Park, Abingdon Oxfordshire OX14 4SA (GB). **SIKOU, Angeliki**; c/o P2i Ltd, 9-12 North Central, 127 Olympic Avenue, Milton Park, Abingdon Oxfordshire OX14 4SA (GB). **TELFORD, Clive**; c/o P2i Ltd, 9-12 North Central, 127 Olympic Avenue, Milton Park, Abingdon Oxfordshire OX14 4SA (GB).(74) Agent: **BLAKE, Michael**; Greaves Brewster LLP, Copa House, Station Road, Cheddar Somerset BS27 3AH (GB).

(81) Designated States (unless otherwise indicated, for every kind of national protection available): AE, AG, AL, AM, AO, AT, AU, AZ, BA, BB, BG, BH, BN, BR, BW, BY, BZ, CA, CH, CL, CN, CO, CR, CU, CZ, DE, DK, DM, DO, DZ, EC, EE, EG, ES, FI, GB, GD, GE, GH, GM, GT, HN, HR, HU, ID, IL, IN, IR, IS, JP, KE, KG, KN, KP, KR, KZ, LA, LC, LK, LR, LS, LU, LY, MA, MD, ME, MG, MK, MN, MW, MX, MY, MZ, NA, NG, NI, NO, NZ, OM, PA, PE, PG, PH, PL, PT, QA, RO, RS, RU, RW, SA, SC, SD, SE, SG, SK, SL, SM, ST, SV, SY, TH, TJ, TM, TN, TR, TT, TZ, UA, UG, US, UZ, VC, VN, ZA, ZM, ZW.

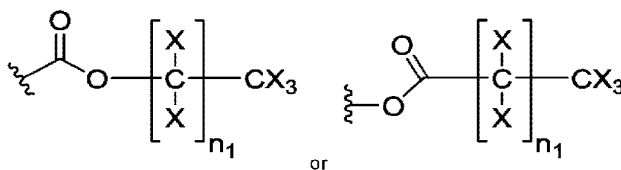
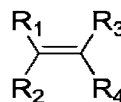
(84) Designated States (unless otherwise indicated, for every kind of regional protection available): ARIPO (BW, GH, GM, KE, LR, LS, MW, MZ, NA, RW, SD, SL, ST, SZ, TZ, UG, ZM, ZW), Eurasian (AM, AZ, BY, KG, KZ, RU, TJ, TM), European (AL, AT, BE, BG, CH, CY, CZ, DE, DK, EE, ES, FI, FR, GB, GR, HR, HU, IE, IS, IT, LT, LU, LV, MC, MK, MT, NL, NO, PL, PT, RO, RS, SE, SI, SK, SM, TR), OAPI (BF, BJ, CF, CG, CI, CM, GA, GN, GQ, GW, KM, ML, MR, NE, SN, TD, TG).

Published:

— with international search report (Art. 21(3))

[Continued on next page]

(54) Title: COATINGS



(57) Abstract: The present invention provides an electronic or electrical device or component thereof comprising a cross-linked polymeric coating on a surface of the electronic or electrical device or component thereof; wherein the cross-linked polymeric coating is obtainable by exposing the electronic or electrical device or component thereof to a plasma comprising a monomer compound and a crosslinking reagent for a period of time sufficient to allow formation of the cross-linked polymeric coating on a surface thereof, wherein the monomer compound has the following formula: where R_1 , R_2 and R_4 are each independently selected from hydrogen, optionally substituted branched or straight chain C_1 - C_6 alkyl or halo alkyl or aryl optionally substituted by halo, and R_3 is selected from: or where each X is independently selected from hydrogen, a halogen, optionally substituted branched or straight chain C_1 - C_6 alkyl, halo alkyl or aryl optionally substituted by halo; and n_1 is an integer from 2 to 27; and wherein the crosslinking reagent comprises two or more unsaturated bonds attached by means of one or more linker moieties and has a boiling point at standard pressure of less than 500°C.



- *before the expiration of the time limit for amending the claims and to be republished in the event of receipt of amendments (Rule 48.2(h))*

Coatings

Field of the invention

This invention relates to protective coatings. Specifically, the present invention relates to protective coatings for electronic or electrical devices and components thereof, and methods of forming such coatings. The coatings can protect by being hydrophobic and so resist the ingress of water-based liquid into electronic devices, or they can protect by forming a barrier coating and so provide electrical resistance between the electrical parts of the phone and water based liquid.

Background of the invention

Monounsaturated monomers are used to make barrier coatings using plasma polymerisation processes (see co-pending application).

The perfluoroalkyl chain monomers are also used for generating hydrophobic surfaces from pulsed plasma deposition processes (see WO 9858117 A1).

The power of the plasma initiated polymerisation affects the nature of the polymer produced. The higher average energy inputs of continuous wave plasmas lead to more fragmentation of the monomer, and so the polymer loses structural properties of the monomer. In the case of 1H,1H,2H,2H- perfluorodecyl acrylate (PFAC8), there's less retention of the perfluoroalkyl chain and the contact angle of the surface coating is compromised. Higher plasma energies also lead to more crosslinking. For the lower average energy inputs of pulsed plasmas, there's better retention of monomer structure and less crosslinking. The greater retention of the perfluoro chain under low energy, pulsed plasma conditions leads to best levels of contact angles for the surface coating.

When the perfluoroalkyl chains have eight or more fluorinated carbons (long chain), the polymer made from the monomer has a crystalline structure. When the perfluoroalkyl chains have less than eight fluorinated carbons, the resulting polymer is amorphous and so can be unstable in the presence of water (see "Molecular Aggregation Structure and Surface Properties of Poly(fluoroalkyl acrylate) Thin Films, *Macromolecules*, 2005, vol 38, p5699-5705)

When long chain perfluoroalkyl polymers are produced by high average power (continuous wave or CW) or low average power (pulsed wave or PW) plasmas, then because of the crystalline structure of the long chains, the polymers are non-stick to the touch and stable in the presence of water.

However, the feel and water stability of shorter chain polymer coatings is affected by the plasma power levels used. For example, when PFAC6 (1H,1H,2H,2H-perfluorooctyl acrylate) is polymerised in low power plasma conditions, the resulting polymer coating can have several disadvantages. For example the coating can cause water drops to spread out a little (slump), be marked by the presence of a water drop on its surface, have a tacky feel, or can be easily smeared (for example on substrates of silicon wafer and ABS plastic).

By increasing the power of the plasma used for polymerisation, the polymer becomes more crosslinked and becomes more resistant to smearing. However, increasing the power has the concomitant effect of decreasing the water contact angle through more monomer fragmentation (as described above). Figure 1 shows the effect of increasing the power to monomer flow ratio for CW plasmas in a 125 litre chamber: at a ratio of 4 W/ μ l/min, the water contact angle is ~85-95 degrees and the coating is tack-free. However, as the ratio drops, the contact angle increases and the occurrence of tacky/smudgy coatings increases too. Figure 2 shows the same effect for pulsed plasma conditions. These results show that the process window for producing tack and smudge-free coatings have a limited plasma processing range and the final coating has a compromised water contact angle.

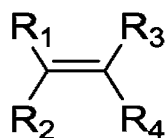
Accordingly, it is an aim to solve one or more of the above-mentioned problems with the prior art coatings.

Statement of Invention

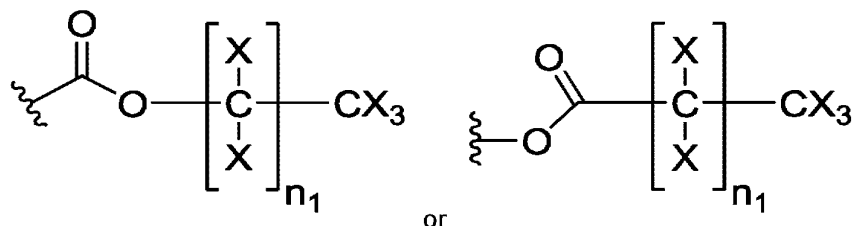
An aspect of the present invention provides an electronic or electrical device or component thereof comprising a protective cross-linked polymeric coating on a surface of the electronic or electrical device or component thereof;

wherein the protective cross-linked polymeric coating is obtainable by exposing the electronic or electrical device or component thereof to a plasma comprising a monomer compound and a crosslinking reagent for a period of time sufficient to allow formation of the protective cross-linked polymeric coating on a surface thereof,

wherein the monomer compound has the following formula:



where R_1 , R_2 and R_4 are each independently selected from hydrogen, optionally substituted branched or straight chain C_1 - C_6 alkyl or halo alkyl or aryl optionally substituted by halo, and R_3 is selected from:



where each X is independently selected from hydrogen, a halogen, optionally substituted branched or straight chain C₁-C₈ alkyl, halo alkyl or aryl optionally substituted by halo; and n₁ is an integer from 1 to 27; and wherein the crosslinking reagent comprises two or more unsaturated bonds attached by means of one or more linker moieties and has a boiling point of less than 500 °C at standard pressure.

High levels of polymer crosslinking (formerly only achievable with high average power continuous wave plasmas) can be achieved by adding a crosslinking molecule to the monomer to produce a cross-linked co-polymer. This has the advantage of increasing the plasma processing range so stable coatings can now be produced in low average energy pulse plasma conditions.

The high degree of retention of the hydrophobic monomer structures from the low energy pulsed plasma gives coatings of the co-polymer a good hydrophobic coating (as evidenced by water contact angles) and a coating that is not sticky or smeary to the touch. Thus, these properties make it suitable for the coating of electronic devices to prevent the ingress of water-based liquids from accidental damage. Such coatings typically have a thickness between 1 and 100 nm, preferably 1 and 50 nm.

The coating may be described as a liquid repellant layer, typically a water repellant layer. The coating may repel water, an aqueous liquid such as rainwater, or oil.

Alternatively, or in addition to providing a liquid repellent layer, the coating may be a physical barrier layer.

The coating may be a physical barrier i.e. it provides a physical barrier to mass and electron transport.

The physical barrier layer restricts diffusion of water, oxygen and ions. When the coating is a physical barrier it typically has a thickness of greater than 50 nm.

The coating is a protective layer i.e. it prevents damage by contact with water or other liquids. The coating may provide protective functionality by forming a liquid repellent layer and/or a physical barrier layer.

The coating is preferably substantially pin-hole free to enable it to provide a physical barrier. Preferably $\Delta Z/d < 0.15$, where ΔZ is the average height variation on an AFM line scan in nm (as shown in Figure 3) and d is coating thickness in nm.

The value of $\Delta Z/d$ tells us to what extent defects/voids on the surface of the coating extend into the coating, i.e. the percentage value of the depth of defect over total coating thickness. For example, $\Delta Z/d = 0.15$ means that the voids on the surface only extend down to 15% of the coating thickness. A coating with a $\Delta Z/d < 0.15$ is defined herein as being substantially pinhole free. If voids are bigger than this, the desired functionality is unlikely to be achieved.

The coating is preferably conformal, which means that it takes the 3D shape of the electronic or electrical device or component thereof and covers substantially an entire surface of the device. This has the advantage of ensuring that the coating has sufficient thickness to give optimal functionality over an entire surface of the device or component. The meaning of the term “covers substantially an entire surface” will depend to some extent on the type of surface to be covered. For example, for some components, it may be necessary for there to be complete coverage of the surface in order for the component to function after submersion in water. However, for other components or housings, small gaps in coverage may be tolerated.

The coating may have a thickness of 50 to 10,000nm, optionally 50 to 8000nm, 100 to 5000nm, preferably 250nm – 5000nm, most preferably 250nm – 2000nm.

The coating may be electrically insulating and be sufficiently compliant that electrical connectors can be joined to the electronic or electrical device or component thereof and an electrical connection made between the electrical connectors and electronic or electrical device or component thereof without the requirement to first remove the coating. In this case, the force exerted by the electrical connector on the coating is sufficient to alter the structure of or even break through the coating local to the electrical connector, allowing the electrical connection to be made. Electrical connectors can typically be joined to the electronic or electrical device or component in this way for coating thicknesses of under 5000nm, and for high performance coatings below 2000nm.

The electronic or electrical device or component thereof can typically withstand immersion in up to 1m of water for over 30 minutes without failure or corrosion whilst power applied to the electronic or electrical device or component. The effectiveness of the coating can be determined by measuring its electrical resistance at a fixed voltage when submerged in water for a set time period; for example, applying a voltage of 8V to the coating of a device which is submerged in water for 13 minutes. Coatings having a resistance value of 1×10^7 Ohms or higher in this test are effective barrier coatings and the coated electronic or electrical device or component thereof will pass successfully an IPX7 test. The IPX7 test is the Ingress Protection Marking which classifies and rates the degree of protection provided against water. In the IPX7 test for phones, the device is immersed in water under defined conditions of pressure and time (up to 1 m of submersion) for a duration of 30 minutes. The device has to be powered on during testing and functional after 24 hrs.

In one embodiment, the coating is electrically insulating and has a thickness of less than 1 micron and wherein a force of 5- 20g applied to the coating using a round probe with 1 mm diameter allows an electrical connection to be made with the electronic or electrical device or component thereof in the local area where the force has been applied.

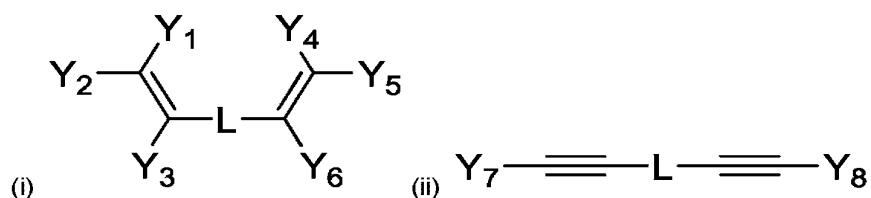
In another embodiment, the coating is electrically insulating and has a thickness of 1-2.5 microns and wherein a force of 20-100g applied to the coating using a round probe with 1 mm diameter allows an electrical connection to be made in the local area of the coating where the force has been applied.

The coating may have a higher density than that of the corresponding monomer from which it is formed. For example, the increase in density may be at least 0.1 g/cm^3 . The increase in density is explained by the highly crosslinked coating. The high density of the coating improves the barrier properties of the coating.

The coating may form a surface defined by a static water contact angle (WCA) of at least 70° . A coating with a WCA of at least 90° is a liquid repellent, typically a water repellent layer. For fluorinated polymers, the coating may have a static water contact angle of at least 100° . The contact angle of a liquid on a solid substrate gives an indication of the surface energy which in turn illustrates the substrate's liquid repellence. Contact angles were measured on a VCA Optima contact angle analyser, using $3 \mu\text{l}$ droplets of deionised water at room temperature.

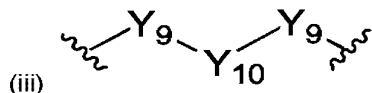
Crosslinker

A basic requirement for the cross-linking reagent is the presence two or more unsaturated bonds, for example, $-\text{C}=\text{C}-$ or alkyne groups. The unsaturated bonds are attached by one or more linker moieties. The linker moiety is not especially limited provided it joins together the two or more unsaturated bonds. The cross-linking reagent must have a boiling point of less than 500°C , preferably from -10 to 300°C , optionally 180 to 270°C , most preferably 205 to 260°C at standard pressure. It is a preference, but not a requirement, that the cross-linking reagent is not unduly hazardous for use in plasma processing, that is to say it can be used in a production environment where low levels of vapour do not present any significant health and safety issues (e.g. strongly oxidising, explosive, toxic, or has an offensive odour (e.g. stench reagent)). The crosslinking reagent preferably has one of the following structures:



where Y_1 , Y_2 , Y_3 , Y_4 , Y_5 , Y_6 , Y_7 and Y_8 are each independently selected from hydrogen, optionally substituted cyclic, branched or straight chain $\text{C}_1\text{--C}_8$ alkyl or aryl; and L is a linker moiety.

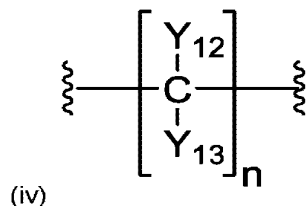
Most preferably L has the following formula:



where each Y_9 is independently selected from, a bond, $-\text{O}-$, $-\text{O}-\text{C}(\text{O})-$, $-\text{C}(\text{O})-\text{O}-$, $-\text{Y}_{11}-\text{O}-\text{C}(\text{O})-$, $-\text{C}(\text{O})-\text{O}-\text{Y}_{11}-$, $-\text{OY}_{11}-$, and $-\text{Y}_{11}\text{O}-$, where Y_{11} is an optionally substituted cyclic, branched or straight chain $\text{C}_1\text{--C}_8$ alkylene; and

Y_{10} is selected from optionally substituted cyclic, branched or straight chain C_1 - C_8 alkylene and a siloxane containing group.

In one most preferred embodiment Y_{10} has the following formula:



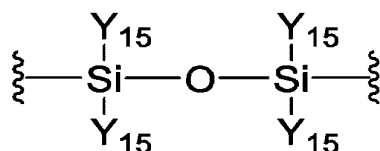
wherein each Y_{12} and Y_{13} is independently selected from H, halo, optionally substituted cyclic, branched or straight chain C_1 - C_8 alkyl, or $-OY_{14}$, where Y_{14} is selected from optionally substituted branched or straight chain C_1 - C_8 alkyl or alkenyl, and n is an integer from 1 to 10.

Optionally, each Y_{12} is H and each Y_{13} is H, i.e. Y_{10} is a linear alkylene chain. For this embodiment Y_9 is preferably a vinyl ester or vinyl ether group.

Optionally, each Y_{12} is fluoro and each Y_{13} is fluoro, i.e. Y_{10} is a linear perfluoroalkylene chain.

Typically, n is from 4 to 6.

In another embodiment Y_{10} has the following formula:

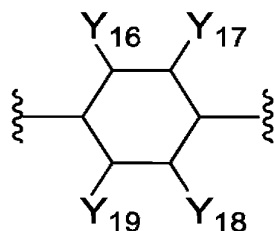


(v)

wherein each Y_{15} is independently selected from optionally substituted branched or straight chain C_1 - C_8 alkyl.

In one embodiment each Y_{15} is methyl, and each Y_9 is a bond.

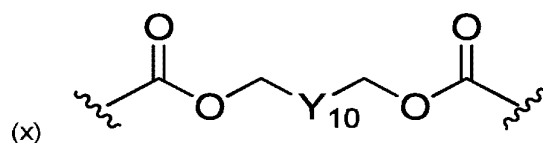
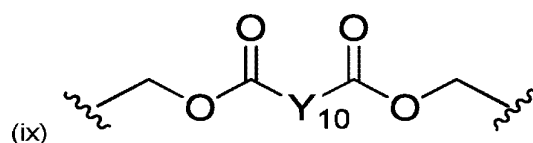
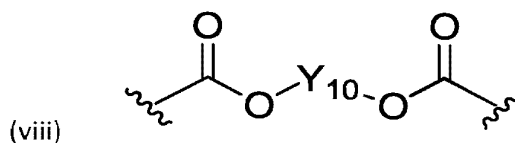
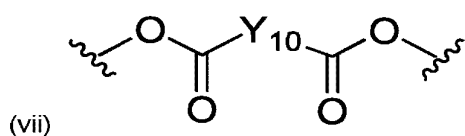
In a further embodiment Y_{10} has the following formula:



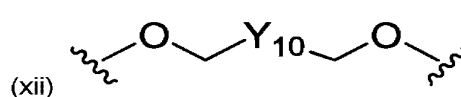
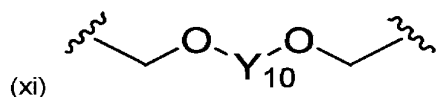
(vi)

wherein Y_{16} to Y_{19} are each independently selected from H and optionally substituted branched or straight chain C_1 - C_8 alkyl or alkenyl. A preferred alkenyl group is vinyl. Optionally Y_{18} is H or vinyl, and Y_{16} , Y_{17} and Y_{19} are each H. In one embodiment each of Y_{16} to Y_{19} is H. In another embodiment Y_{18} is vinyl, and Y_{16} , Y_{17} and Y_{19} are each H.

In preferable embodiments for compound (i), L has one of the following structures:



In another embodiment for compound (i) L has one of the following structures:



For L according structure (vii), Y_{10} is preferably an alkylene chain or a cycloalkylene, such as those shown in structures iv) and vi) above. The alkylene chain may be a straight chain alkylene chain. When Y_{10} is a cycloalkylene, cyclohexylene is preferred, most preferably 1,4 cyclohexylene.

For L according to structure (viii), Y₁₀ is preferably structure (iv), i.e. an alkylene or fluoroalkylene chain.

For L according to structure (ix), Y₁₀ is preferably cycloalkylene, such as the cyclohexylene according to structure (vi).

For L according to structure (x), Y₁₀ is preferably structure (iv) wherein each Y₁₂ and Y₁₃ is F, i.e. a perfluoroalkylene chain.

For L according to structure (xi) or structure (xii) Y₁₀ is preferably alkylene or cycloalkylene. Optionally the alkylene or cycloalkylene may be substituted with one or more vinyl groups or alkenyl ether groups, preferably one or more vinyl ether groups.

When each Y₉ is a bond, each Y₁₀ may be any of structures (iv), (v) and (vi). Preferably Y₁₀ is a straight chain alkylene such that the crosslinking reagent is a diene, such as a heptadiene, octadiene, or nonadiene, most preferably 1,7-octadiene.

When each Y₉ is O, each Y₁₀ is preferably a branched or straight chain C₁-C₈ alkylene, preferably a straight chain alkylene, most preferably a C₄ straight chain alkylene, i.e the crosslinking reagent is 1,4-butanediol divinyl ether

It will be understood that each Y₉ group can be combined with any other Y₉ group and Y₁₀ group to form the crosslinking reagent.

The skilled person will be aware of possible substituents for each of the cyclic, branched or straight chain C₁-C₈ alkylene groups mentioned above. The alkylene groups may be substituted at one or more positions by a suitable chemical group. Halo substituents are preferred, with fluoro substituents most preferred. Each C₁-C₈ alkylene group may be a C₁-C₃, C₂-C₆, or C₆-C₈ alkylene group.

Particularly preferred embodiments of the crosslinker have alkyl chains for Y₁₀ and vinyl ester or vinyl ether groups on either side.

A particularly preferred cross-linking reagent is divinyl adipate. (a divinyl ester)

Another preferred cross-linking reagent is 1,4-butanediol divinyl ether (a divinyl ether)

For the most preferred embodiments the crosslinking reagent is selected from Divinyl Adipate (DVA), 1,4 Butanediol divinyl ether (BDVE), 1,4 Cyclohexanedimethanol divinyl ether (CDDE), 1,7 -Octadiene (17OD), 1,2,4 -Trivinylcyclohexane (TVCH), 1,3-Divinyltetramethyldisiloxane (DVTMDS), Diallyl 1,4-cyclohexanedicarboxylate (DCHD), 1,6-Divinylperfluorohexane (DVPFH), 1H,1H,6H,6H-Perfluorohexanediol diacrylate (PFHDA) and glyoxal bis (diallyl acetal) (GBDA).

For the alkyne crosslinking reagents according to compound (ii), L is preferably selected from a branched or straight chain C₁-C₈ alkylene or an ether group. L may be a C₃, C₄, C₅, or C₆ alkylene,

preferably a straight chain alkylene. Chemical structures for the particularly preferred crosslinking reagents are set out below in Table 1:

| | |
|---|--|
| Divinyl Adipate (DVA) | |
| 1,4 Butanediol divinyl ether (BDVE) | |
| 1,4 Cyclohexanedimethanol divinyl ether (CDDE) | |
| 1,7 -Octadiene (17OD) | |
| 1,2,4 -Trivinylcyclohexane (TVCH) | |
| 1,3- Divinyltetramethyldisiloxane (DVTMDS) | |
| Diallyl 1,4- cyclohexanedicarboxylate (DCHD) | |
| 1,6-Divinylperfluorohexane (DVPFH) | |
| 1H,1H,6H,6H- Perfluorohexanediol diacrylate (PFHDA) | |
| GBDA | |


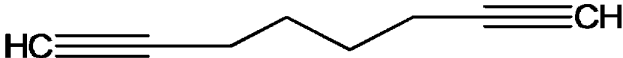
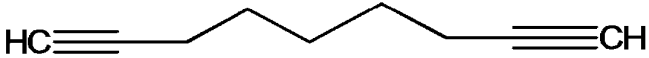
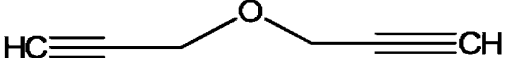
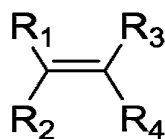
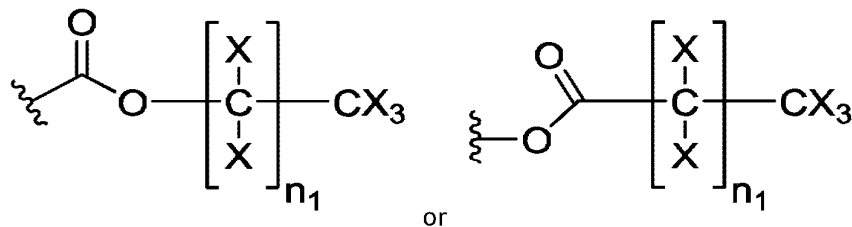
| | |
|-----------------|--|
| 1,6-heptadiyne |  |
| 1,7-heptadiyne |  |
| 1,8-heptadiyne |  |
| Propargyl ether |  |

Table 1*Monomer Compound*

The monomer compound has the following formula:

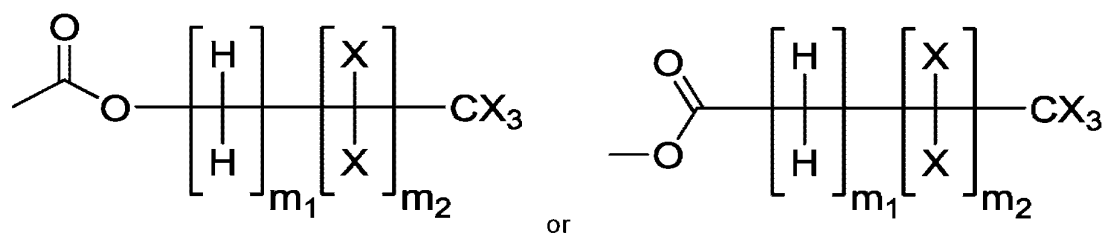


where R_1 , R_2 and R_4 are each independently selected from hydrogen, optionally substituted branched or straight chain C_1 - C_6 alkyl or halo alkyl or aryl optionally substituted by halo, and R_3 is selected from:



where each X is independently selected from hydrogen, a halogen, optionally substituted branched or straight chain C_1 - C_6 alkyl, halo alkyl or aryl optionally substituted by halo; and n_1 is an integer from 1 to 27. Preferably, n_1 is from 1 to 12. Optionally n_1 is from 4 to 12, optionally 6 to 8.

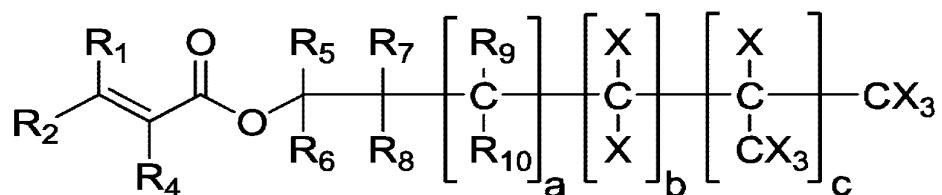
In a preferred embodiment R_3 is selected from:



where m_1 is an integer from 0 to 13 and each X is independently selected from hydrogen, a halogen, optionally substituted branched or straight chain C_1 - C_6 alkyl, halo alkyl or aryl optionally substituted by halo; and m_2 is an integer from 2 to 14;

In a particularly preferred embodiment the monomer is a compound of formula I(a):

I(a)

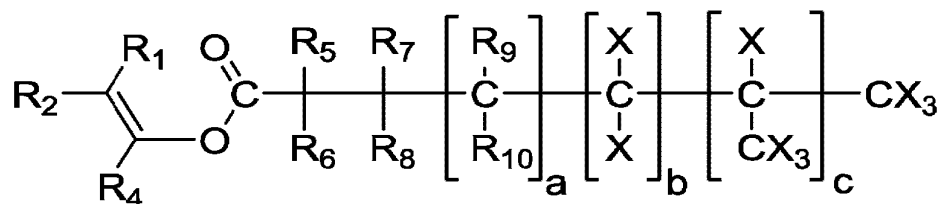


wherein each of R_1 , R_2 , R_4 , and R_5 to R_{10} is independently selected from hydrogen or an optionally substituted C_1 - C_6 branched or straight chain alkyl group; each X is independently selected from hydrogen or halogen; a is from 0-10; b is from 2 to 14; and c is 0 or 1;

; or

the monomer is a compound of formula I(b):

I(b)



wherein each of R_1 , R_2 , R_4 , and R_5 to R_{10} is independently selected from hydrogen or an optionally substituted C_1 - C_6 branched or straight chain alkyl group; each X is independently selected from hydrogen or halogen; a is from 0-10; b is from 2 to 14; and c is 0 or 1.

The halogen may be chlorine or bromine, but is preferably fluorine for compliance with RoHS regulations (Restriction of Hazardous Substances).

a is from 0 to 10, preferably from 0 to 6, optionally 2 to 4, most preferably 0 or 1. b is from 2 to 14, optionally from 2 to 10, preferably 3 to 7.

Each of R₁, R₂, R₄, and R₅ to R₁₀ is independently selected from hydrogen or a C₁-C₈ branched or straight chain alkyl group. The alkyl group may be substituted or unsubstituted, saturated or unsaturated. When the alkyl group is substituted, the location or type of the substituent is not especially limited provided the resultant polymer provides an appropriate liquid repellent and/or barrier layer. The skilled person would be aware of suitable substituents. If the alkyl group is substituted, a preferred substituent is halo, i.e. any of R₁, R₂, R₄, and R₅ to R₁₀ may be haloalkyl, preferably fluoroalkyl. Other possible substituents may be hydroxyl or amine groups. If the alkyl group is unsaturated it may comprise one or more alkene or alkyne groups.

Each of R₁, R₂, R₄, and R₅ to R₁₀ may be independently selected from hydrogen, methyl, ethyl, n-propyl, isopropyl, n-butyl, isobutyl, tert-butyl, sec-butyl, n-pentyl, neopentyl, n-hexyl, isohexyl, and 3-methylpentyl.

In a preferred embodiment each of R₁, R₂, R₄, and R₅ to R₁₀ is independently selected from hydrogen or methyl.

In a preferred embodiment, a and c are each independently 0 or 1; and b is from 3 to 7.

In one preferred embodiment each X is H. In an alternative preferred embodiment each X is F.

Optionally R₁ and R₂ are both hydrogen.

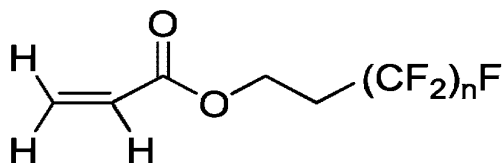
Optionally R₄ is hydrogen or methyl. Preferably R₁ and R₂ are both hydrogen and R₄ is hydrogen or methyl.

Optionally R₉ is hydrogen and R₁₀ is branched or straight chain C₁-C₆ alkyl group. In a preferred embodiment R₁₀ is methyl.

In one embodiment each of R₅ to R₈ is hydrogen.

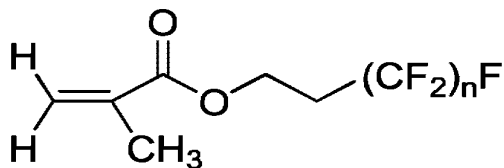
In one embodiment each of R₁, R₂, R₄, and R₅ to R₁₀ is hydrogen, each X is H, a=0 and c=0.

In a particularly preferred embodiment the monomer compound has the following formula:



where n is from 2 to 10.

In another preferred embodiment the monomer compound has the following formula:

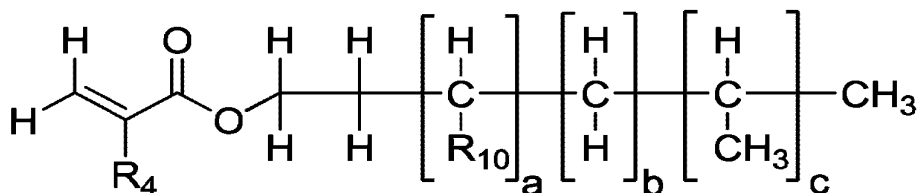


where n is from 2 to 10.

The monomer compound may be selected from 1H,1H,2H,2H-perfluorohexyl acrylate (PFAC4), 1H,1H,2H,2H-perfluorooctyl acrylate (PFAC6), 1H,1H,2H,2H-perfluorodecyl acrylate (PFAC8) and 1H,1H,2H,2H-perfluorododecyl acrylate (PFAC10).

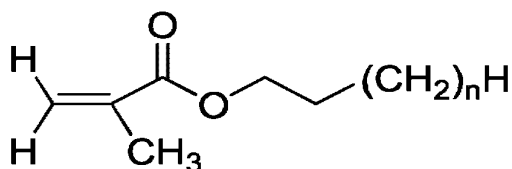
The monomer compound may be selected from 1H,1H,2H,2H-perfluorohexyl methacrylate (PFMAC4), 1H,1H,2H,2H-perfluorooctyl methacrylate (PFMAC6) and 1H,1H,2H,2H-perfluorodecyl methacrylate (PFMAC8).

The monomer compound of formula I(a) may have the following formula:



wherein a and c are each independently 0 or 1, b = 3-7 and n is 4 to 10, where n = a + b + c + 1.

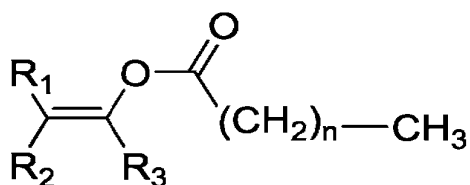
The monomer compound of formula I(a) may have the following formula:



where n is 2 to 12.

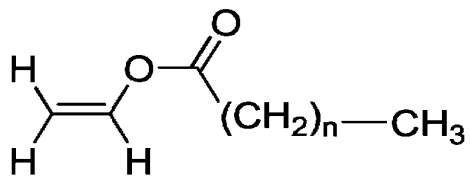
The monomer compound may be selected from ethyl hexyl acrylate, hexyl acrylate, decyl acrylate, lauryl dodecyl acrylate and iso decyl acrylate.

The monomer may have the following formula:



where n is from 4 to 14.

The monomer may have the following formula:

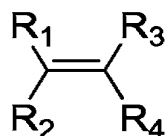


where n is from 4 to 14.

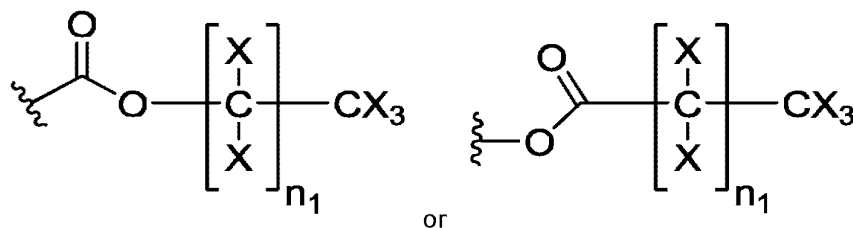
In an additional aspect, the present invention provides a method for treating an electronic device or component thereof comprising:

exposing the electronic or electrical device or component thereof to a plasma comprising a monomer compound and a crosslinking reagent for a period of time sufficient to allow a protective cross-linked polymeric coating to form on a surface of the electronic or electrical device or component thereof;

wherein the monomer compound has the following formula:



where R_1 , R_2 and R_4 are each independently selected from hydrogen, optionally substituted branched or straight chain C_1 - C_6 alkyl or halo alkyl or aryl optionally substituted by halo, and R_3 is selected from:



where each X is independently selected from hydrogen, optionally substituted branched or straight chain C_1 - C_6 alkyl, halo alkyl or aryl optionally substituted by halo; and n_1 is an integer from 1 to 27; and wherein the crosslinking reagent comprises two or more unsaturated bonds and has a boiling point of less than 500 °C at standard pressure.

The monomer compound and the crosslinking reagent utilised in the method are described in further detail above.

Typically the electronic or electrical device or component thereof is placed in a plasma deposition chamber, a glow discharge is ignited within said chamber, and a voltage applied as a pulsed field. .

Preferably the applied voltage is at a power of from 30 to 800W. Optionally the voltage is pulsed in a sequence in which the ratio of the time on/time off is in the range of from 0.001 to 1, optionally 0.002 to 0.5. For example, time on may be 10-500 μ s, preferably 35-45 μ s, or 30-40 μ s, such as about 36 μ s and time off may be from 0.1 to 30 ms, preferably 0.1 to 20 ms, optionally from 5 to 15 ms, for example 6ms. Time on may be 35 μ s, 40 μ s, 45 μ s. Time off may be 0.1, 1, 2, 3, 6, 8, 10, 15, 20, 25 or 30 ms.

The term pulsed may mean that the plasma cycles between a state of no (or substantially no) plasma emission (off-state) and a state where a particular amount of plasma is emitted (on-state).

Alternatively, pulsed may mean that there is continuous emission of plasma but that the amount of plasma cycles between an upper limit (on-state) and lower limit (off-state).

In a further embodiment, the invention resides in a method of forming a coating on an electronic or electrical device or component thereof as defined above, the method comprising: exposing said substrate in a chamber to a plasma comprising a monomer compound, preferably a continuous plasma, for a sufficient period of time to allow a protective polymeric coating to form on the substrate,

wherein during exposure of the substrate the continuous plasma has a power density of at least 2W/litre, preferably 20W/litre.

Optionally the voltage is applied as a pulsed field for a period of from 30 seconds to 90 minutes. Optionally the voltage is applied as a pulsed field for from 5 to 60 minutes. Optionally in a preliminary step, a continuous power plasma is applied to the electronic or electrical device or component thereof. The preliminary step may be conducted in the presence of an inert gas.

Before the crosslinker and/or the monomer compound enter the deposition chamber each may be in the form of a gas, liquid or a solid (for example a powder) at room temperature. However, it is the preferred that the crosslinking reagent and the monomer compound are both liquid at room temperature, and most preferably that the monomer and crosslinker liquids are miscible.

The monomer compound and/or the crosslinker will suitably be in a gaseous state in the plasma. The plasma may simply comprise a vapour of the monomer compound and the crosslinker. Such a vapour may be formed in-situ, with the compounds being introduced into the chamber in liquid form. The monomer may also be combined with a carrier gas, in particular, an inert gas such as helium or argon.

In preferred embodiments, the monomer and/or the crosslinker may be delivered into the chamber by way of an aerosol device such as a nebuliser or the like, as described for example in WO2003/097245 and WO03/101621, the content of which is incorporated herein by reference. In such an arrangement a carrier gas may not be required, which advantageously assists in achieving high flow rates.

In one embodiment the monomer compound and/or the crosslinking reagent are in gaseous form and are fed into the plasma at a rate of from 10-5000 mg/minute depending on chamber volume, whilst the pulsed voltage is applied.

Optionally the plasma is created with an average power of from 0.001 to 40W/litre.

The crosslinker may be miscible with the monomer and so introduced together or separately into the plasma chamber. Or the crosslinker may be immiscible with the monomer and introduced separately into the plasma chamber. In this context, the term "miscible" means that the crosslinker is soluble in the monomer, and when mixed they form a solution of uniform composition. The term "immiscible" is used to mean that the crosslinker is only partly soluble or insoluble in the monomer, and so either forms an emulsion or separates out into two layers.

The crosslinking reagent is preferably present in an amount of from 10 to 60 (v/v) %, optionally 20 to 40 (v/v) %, optionally 25 to 30 (v/v)%, optionally 30 to 50 (v/v)% of the total volume of monomer compound and crosslinking reagent, depending on the specific crosslinker. The skilled person would understand that the amount will vary to some extent depending on whether the coating is required to

be liquid repellent or provide a barrier to mass and electron transport. The skilled person would understand that the (v/v) percentages are those which give a stable crosslinked polymer coating and the highest water contact angle.

Electronic or electrical device or component thereof

Although the invention is of benefit in the context of a wide variety of substrates, the substrate may, in all aspects of the invention, advantageously be an electronic substrate.

In some embodiments of the invention, the electronic substrate may comprise an electronic or electrical device, i.e. any piece of electrical or electronic equipment. Non-limiting examples of electrical and electronic devices include communications devices such as mobile phones, smartphones and pagers, radios, and sound and audio systems such as loudspeakers, microphones, ringers or buzzers, hearing aids, personal audio equipment such as personal CD, tape cassette or MP3 players, televisions, DVD players including portable DVD players, video recorders, digi and other set-top boxes such as Sky, computers and related components such as laptop, notebook, tablet, phablet or palmtop computers, personal digital assistants (PDAs), keyboards, or instrumentation, games consoles in particular hand-held playstations and the like, data storage devices, outdoor lighting systems or radio antenna and other forms of communication equipment.

In preferred embodiments of the invention, the substrate may comprise or consist of an electronic component, e.g. a printed circuit board (PCB), a printed circuit board array (PCBA), a transistor, resistor, or semi-conductor chip. The electronic component may thus be an internal component of an electronic device, e.g. a mobile phone. The coatings of the invention are particularly valuable in preventing electrochemical migration in such components.

In all aspects of the invention, the precise conditions under which the protective polymeric coating is formed in an effective manner will vary depending upon factors such as, without limitation, the nature of the monomer compound, the crosslinking reagent, the substrate, as well as the desired properties of the coating. These conditions can be determined using routine methods or, preferably, using the techniques and preferred features of the invention described herein, which work in particular synergy with the invention.

The present invention will now be further described with reference to the following non-limiting examples and the accompanying illustrative drawings, of which:

Figure 1 illustrates shows the effect of increasing the power to monomer flow ratio for CW plasmas in a 3 litre chamber in a prior art process.

Figure 2 shows the same effect for pulsed plasma conditions.

Figure 3 shows a tapping mode image over $10 \times 10 \mu\text{m}^2$ field of view of a specimen example (thickness $d=1230 \text{ nm}$) prepared according to Example 1 (left hand side) and a contour line plot showing the data used for calculation of RMS roughness (right hand side). The ΔZ value indicated on the plot has been taken over an area of the graph that represents the majority of the coating. Peaks that lie above the ΔZ range indicate large particles and troughs that fall below the ΔZ range show voids or pinholes in the coating. The width of the peaks also gives an indication of the particle size.

Figure 4 illustrates the effect of adding the divinyl adipate crosslinker to perfluorooctyl acrylate on water contact angle from a process on a 125 Litre chamber.

Figure 5 shows the effect of adding crosslinkers in accordance with the invention on the electrical resistance per nm of the coatings.

Figure 6 shows a tapping mode image of a 90 nm thick coating, prepared as described in Example 3, over $2 \times 2 \mu\text{m}^2$ field of view (top left), a representative contour line indicating height variation (z-axis) of the coating (top right) and a phase image indicating full substrate coverage (bottom left); RMS roughness of the coating is 1.65 nm and $\Delta z/d = 0.05$

Figure 7 shows a FTIR/ATR spectrum of a coating formed according to Example 5.

Example 1

Example of plasma polymerising PFAC6 with no crosslinker in a 3 litre chamber

PFAC6 was polymerised in a 3 litre glass chamber using a continuous plasma process. In the experimental matrix, the continuous plasma was produced using RF power levels of 50, 100 and 200 Watts. The process pressure was run at 30, 60 and 90 mTorr and the monomer flow rate run at 50, 100 and 200 microlitres of liquid per minute. The duration of the process was either 10 or 40 minutes. The coated substrate was silicon wafer and whether the coating was tacky or not was determined by wiping the substrate with a finger and determining by eye if the coating had smeared. The results in Figure 1 indicate that for lower CW powers, the coating could easily be smeared (i.e. tacky), but by increasing the power to flow ratio, the coating became resistant to smearing (i.e. non-tacky) and this is expected to be due to additional crosslinking caused by more monomer fragmentation. The water contact angle was also seen to decline with increasing power to flow ratio, and this too is indicative of more power increasing the level of monomer fragmentation.

The same 3 litre chamber was used to also look at the effect of power to flow ratios with pulsed plasma polymerisation. In these experiments, the peak power was either 250 or 500 W. The RF power feed was pulsed with an on time of 35 microseconds and off times of 5.5 and 1 millisecond. Process pressure was either 20, 35, 60 or 80 mTorr. The monomer flow was either 50 or 200 microlitres of liquid per minute. Figure 2 shows that low power to flow conditions can generate coatings that can be smeared (tacky) and only by increasing the power could the smear be reduced. This too had a concomitant decline in water contact angle.

The results from Figures 1 and 2 show that plasma processing PFAC6 on its own could only lead to smear –free coatings when the powers were high enough to decrease the other desirable property of the water contact angle. This illustrates the need to add a crosslinker to prevent the coating smearing and maintain the water contact angle.

Example 2

Examples of Perfluoro Alkyl Acrylate co-polymerisation with Crosslinker to show improvements in electrical resistance

Perfluorooctyl acrylate monomer was mixed with a single crosslinker from the following list:

VINYL ESTERS: Divinyl adipate (DVA)

VINYL ETHERS: 1,4-Butanediol divinyl ether (BDVE); 1,4 Cyclohexanedimethanol divinyl ether (CDDE)

DI- or TRI- VINYLS: 1,7-Octadiene (1,7-OD); 1,2,4-Trivinylcyclohexane (TVCH);

DI VINYL with alkyl fluoro group: 1,6-divinylperfluorohexane (DVPFH)

DI VINYL with silicon group: 1,3-Divinyl tetramethyldisiloxane (DVTMDS)

DI VINYL with cyclic ring and carboxylate groups: Diallyl 1,4-cyclohexanedicarboxylate (DCHC)

DI ACRYLATE: 1H1H,6H,6H-perfluorohexanediol diacrylate (PFHDA)

The crosslinker was mixed with the PFAC6 as a percentage by volume for the following percentages: 20, 40, 60, 80, and 100%.

Plasma initiated polymerisation reactions were carried out in a 3 litre glass plasma chamber. The substrates were test circuit boards. The PFAC6/crosslinker mixture was introduced at a rate of 0.04 – 0.06 ul/min and the process pressure was 40 mTorr. The process plasma consisted of a 1 minute continuous plasma (CW) step of 50W followed by 10 - 20 minute pulsed plasma (PW) step of 50W and RF power being delivered by a pulse sequence with a duty cycle of 6.9%. The coated circuit boards were immersed in tap water and an 8V potential was applied for 13 minutes. The final current readings, together with coating thickness measurements, were used to calculate the electrical resistance per nm of coating. Figure 5 below shows the effect of adding a range of crosslinkers on the electrical resistance per nm of the barrier coatings. Figure 5 clearly shows the improvement in resistance that can be gained by adding the crosslinker to the PFAC6 monomer. Furthermore, all of the crosslinkers tested gave non-smear/non-tacky coatings with concentration levels of 20% and above, with the exceptions of DVTMDS, DCHD, PFHDA and DVPFH which were non-smear at levels of 40% and above.

Example 3**Example of crosslinker concentration on water repellent coating.**

Water repellent coatings were prepared in a 125 litre volume chamber using PFAC6 with different levels of divinyl adipate (DVA) and using helium as a carrier gas. The deposition process consisted of a 3 minute CW step with 300W power and a pulsed step with 150W power and an RF pulse duty cycle of 0.018%. Silicon wafer was used as the test substrates and the contact angle of the coated wafer was determined by applying a 3ul drop of deionised wafer onto the coated wafer and measuring the contact angle using a VCA Optima (AST products) with image analysis software. The variation of contact angle with (v/v)% DVA crosslinker is shown in Figure 4. It was also noted that for the 10 (v/v) % DVA concentration, the water drop left a mark on the coated wafer where it's circumference had been. This indicated that a higher level of crosslinker was required to give a more stable coating. This observation, in combination with the results in figure 4, suggest an optimum range of 20 – 40 (v/v) % DVA crosslinker, though this is likely to vary, typically within the range of from 10-60 (v/v) % for different monomers and crosslinkers and different chamber sizes.

Pulsed plasma polymerisation of perfluoro alkyl acrylate in the presence of divinyl adipate (DVA) was carried out in a 125 litre chamber. Figure 4 shows the effect of adding different liquid volume percentages of DVA crosslinker to perfluorooctyl acrylate on water contact angle on a silicon wafer substrate.

Example 4**Example of AFM measurement of monomer/crosslinker co-polymer coating**

A barrier-style coating was prepared in a 22 litre volume chamber using PFAC6 with 10% DVA. The deposition process consisted of a 1 minute CW step with a CW power to flow ratio of 3.9 (W/microliter/min), and a pulsed step with a PW power/monomer flow ratio of 0.28 (W/microlitres/min). Figure 6 shows the representative topographical and phase contrast images were obtained from all the samples. High spatial resolution images show mainly the structures of areas between the raised features. The RMS roughness of the coating is 1.65 nm and the $\Delta z/d = 0.05$. As the $\Delta z/d$ value is < 0.15, then this indicates that the physical layer is substantially pinhole free.

Example 5**Example of FTIR/ATR measurement of monomer/crosslinker co-polymer coating**

Two barrier-style coatings were prepared in a 22 litre volume chamber using PFAC6 only and PFAC6 with 10% DVA as described in example 4. The deposition process consisted of a 1 minute CW step with a CW power to flow ratio of 3.9 (W/microliter/min), and a pulsed step with a PW power/monomer flow ratio of 0.28 (W/microlitres/min). Figure 7 shows the representative FTIR/ATR spectrum obtained from both samples.

The FTIR/ATR intensity ratios of peaks attributed to stretching mode of CF_3 and C=O groups, $\text{CF}_3/\text{C=O}$, of the coating is indicative of sufficient cross linking in the coating to form a physical barrier. CF_3 refers to the terminal groups in the side chain of PFAC6.

Formation of the barrier coatings is believed to be caused by a mixture of cross linking and controlled fragmentation of the monomers during polymerisation. Cross linking is believed to be predominantly via the $\text{CF}_2\text{-CF}_3$ chain, whilst fragmentation is thought to be predominantly through loss of the C=O group during polymerisation and to a lesser extent shortening of the CF_2 chain. Cross linking effects the abundance of $-\text{CF}_3$ groups in the coating and controlled fragmentation controls the amount of C=O groups in the coating. The ratio of these two functional groups is an indication that sufficient cross-linking and fragmentation has taken place and can be measured by the ratio of the intensities of the corresponding FTIR/ATR peaks.

It has been shown that coatings with reduced $\text{CF}_3 / \text{C=O}$ ratios give higher resistance values in the electrical test, as described in Example 2, showing an improved coating performance on increased cross linking (for CF_3) and fragmentation (for C=O). If the FTIR/ATR intensity ratio of the peaks attributed to $-\text{CF}_3$ stretching and C=O stretching, $\text{CF}_3/\text{C=O}$, is less than $0.6e^{-0.1n}$ (where $n=6$ for PFAC6) the resistance of the coating at the electrical test is expected to be higher than 8MΩ.

The peak intensity ratio $\text{CF}_3/\text{C=O}$ the coating thickness and the final current readings during the electrical test are shown in table 2:

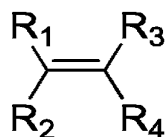
| | Thickness (nm) | Resistance water at 8V (Ohms) | in $\text{CF}_3/\text{C=O}$ ATR peak intensity ratio |
|------------------------------------|-------------------|-------------------------------------|---|
| PFAC6 | 1480 | 2.1×10^7 | 0.23 |
| PFAC6 and 10%DVA (in volume) | 1450 | 9.2×10^9 | 0.11 |

Table 2

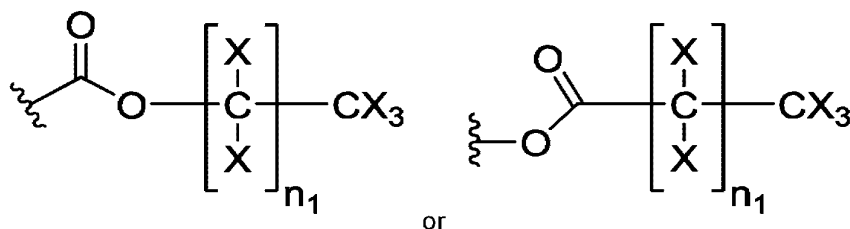
Claims:

1. An electronic or electrical device or component thereof comprising a protective cross-linked polymeric coating on a surface of the electronic or electrical device or component thereof; wherein the protective cross-linked polymeric coating is obtainable by exposing the electronic or electrical device or component thereof to a plasma comprising a monomer compound and a crosslinking reagent for a period of time sufficient to allow formation of the protective cross-linked polymeric coating on a surface thereof,

wherein the monomer compound has the following formula:

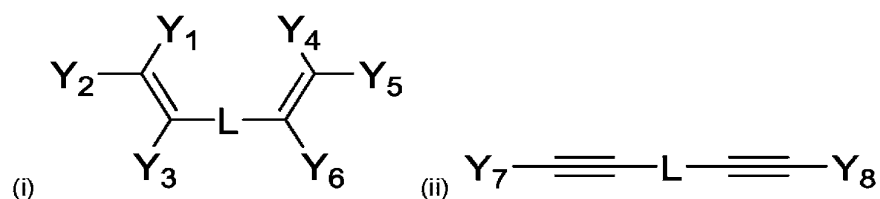


where R_1 , R_2 and R_4 are each independently selected from hydrogen, optionally substituted branched or straight chain C_1 - C_6 alkyl or halo alkyl or aryl optionally substituted by halo, and R_3 is selected from:



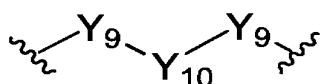
where each X is independently selected from hydrogen, a halogen, optionally substituted branched or straight chain C_1 - C_6 alkyl, halo alkyl or aryl optionally substituted by halo; and n_1 is an integer from 1 to 27; and wherein the crosslinking reagent comprises two or more unsaturated bonds attached by means of one or more linker moieties and has a boiling point of less than 500 °C at standard pressure.

2. An electronic or electrical device or component thereof according to claim 1, wherein the protective cross-linked polymeric coating is a physical barrier to mass and electron transport.
3. An electronic or electrical device or component thereof according to claim 1 or claim 2, wherein the protective cross-linked polymeric coating forms a liquid repellent surface defined by a static water contact angle (WCA) of at least 90°.
4. An electronic or electrical device or component thereof according to any preceding claim, wherein the crosslinking reagent has one of the following structures:



where Y_1 , Y_2 , Y_3 , Y_4 , Y_5 , Y_6 , Y_7 and Y_8 are each independently selected from hydrogen, optionally substituted cyclic, branched or straight chain C_1 - C_8 alkyl or aryl; and L is a linker moiety.

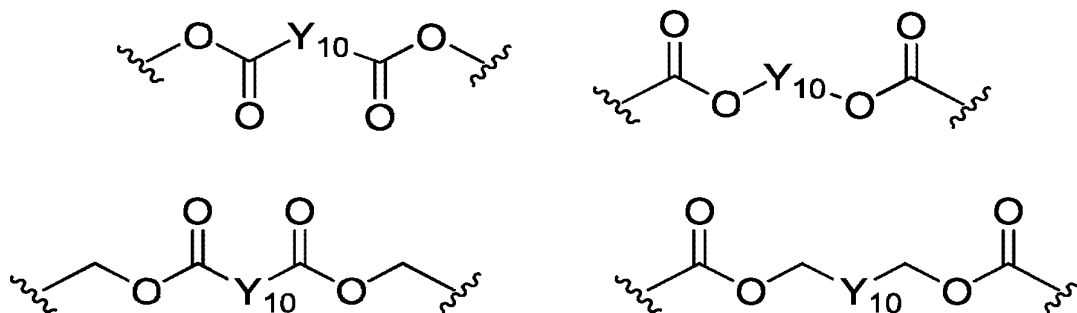
5. An electronic or electrical device or component thereof according to claim 4, wherein for compound (i) L has the following formula:



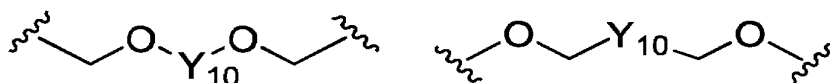
each Y_9 is independently selected from, a bond, -O-, -O-C(O)-, -C(O)-O-, - Y_{11} -O-C(O)-, -C(O)-O- Y_{11} -, -O Y_{11} -, and - Y_{11} O-, where Y_{11} is an optionally substituted cyclic, branched or straight chain C_1 - C_8 alkylene; and

Y_{10} is selected from optionally substituted cyclic, branched or straight chain C_1 - C_8 alkylene and a siloxane group.

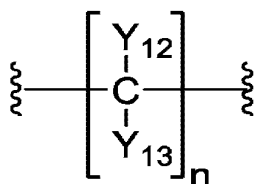
6. An electronic or electrical device or component thereof according to claim 5, wherein for compound (i) L has one of the following structures:



7. An electronic or electrical device or component thereof according to claim 5, wherein for compound (i) L has one of the following structures:

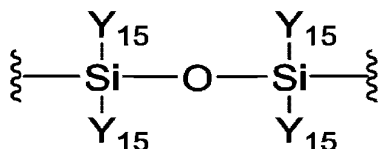


8. An electronic or electrical device or component thereof according to any of claims 5 to 7, wherein Y_{10} has the following formula:



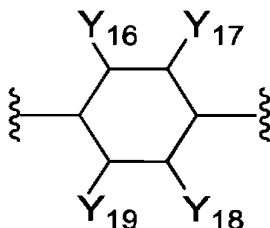
wherein each Y_{12} and Y_{13} is independently selected from H, halo, optionally substituted cyclic, branched or straight chain alkyl, or $-OY_{14}$, where Y_{14} is selected from optionally substituted branched or straight chain C_1 - C_8 alkyl or alkenyl, and n is an integer from 1 to 10.

9. An electronic or electrical device or component thereof according to claim 8, wherein each Y_{12} is H and each Y_{13} is H.
10. An electronic or electrical device or component thereof according to claim 8, wherein each Y_{12} is fluoro and each Y_{13} is fluoro.
11. An electronic or electrical device or component thereof according to any of claims 8 to 10, wherein n is from 4 to 6.
12. An electronic or electrical device or component thereof according to any of claims 5 to 7, wherein Y_{10} has the following formula:



Wherein each Y_{15} is independently selected from optionally substituted branched or straight chain C_1 - C_8 alkyl.

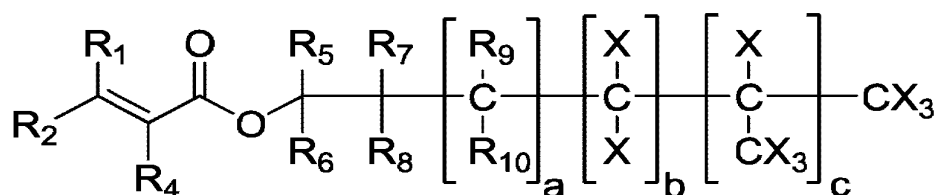
13. An electronic or electrical device or component thereof according to claim 12, wherein each Y_{15} is methyl, and each Y_9 is a bond.
14. An electronic or electrical device or component thereof according to any of claims 5 to 7, wherein Y_{10} has the following formula:



wherein Y_{16} to Y_{19} are each independently selected from H and optionally substituted branched or straight chain C_1 - C_8 alkyl or alkenyl.

15. An electronic or electrical device or component thereof according to claim 14, wherein Y_{18} is H or vinylene, and Y_{16} , Y_{17} and Y_{19} are each H.
16. An electronic or electrical device or component thereof according to any preceding claim wherein the crosslinking reagent is selected from Divinyl Adipate (DVA), 1,4 Butanediol divinyl ether (BDVE), 1,4 Cyclohexanedimethanol divinyl ether (CDDE), 1,7 -Octadiene (17OD), 1,2,4 -Trivinylcyclohexane (TVCH), 1,3-Divinyltetramethyldisiloxane (DVTMDS), Diallyl 1,4-cyclohexanedicarboxylate (DCHD), 1,6-Divinylperfluorohexane (DVPFH), 1H,1H,6H,6H-Perfluorohexanediol diacrylate (PFHDA) and glyoxal bis (diallyl acetal) (GBDA).
17. An electronic or electrical device or component thereof according to claim 4, wherein for compound (ii), L is selected from a branched or straight chain C_1 - C_8 alkylene or an ether group.
18. An electronic or electrical device or component thereof according to any preceding claim wherein the monomer is a compound of formula I(a):

I(a)

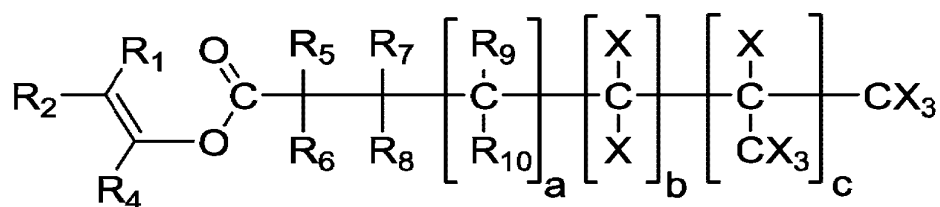


wherein each of R_1 , R_2 , R_4 , and R_5 to R_{10} is independently selected from hydrogen or an optionally substituted C_1 - C_8 branched or straight chain alkyl group; each X is independently selected from hydrogen or halogen; a is from 0-10; b is from 2 to 14; and c is 0 or 1;

; or

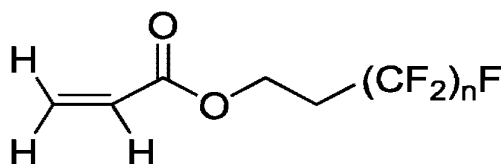
wherein the monomer is a compound of formula I(b):

I(b)



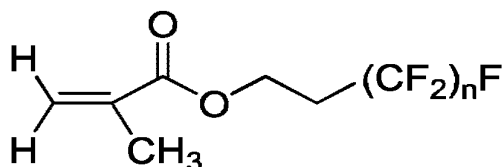
wherein each of R_1 , R_2 , R_4 , and R_5 to R_{10} is independently selected from hydrogen or an optionally substituted C_1 - C_6 branched or straight chain alkyl group; each X is independently selected from hydrogen or halogen; a is from 0-10; b is from 2 to 14; and c is 0 or 1.

19. An electronic or electrical device or component thereof according to claim 18, wherein the halogen is fluorine.
20. An electronic or electrical device or component thereof according to claim 18 or claim 19, wherein each of R_1 , R_2 , R_4 , and R_5 to R_{10} is independently selected from hydrogen, methyl, ethyl, n-propyl, isopropyl, n-butyl, isobutyl, tert-butyl, sec-butyl, n-pentyl, neopentyl, n-hexyl, isohexyl, and 3-methylpentyl.
21. An electronic or electrical device or component thereof according to claim 20, wherein each of R_1 , R_2 , R_4 , and R_5 to R_{10} is independently selected from hydrogen or methyl.
22. An electronic or electrical device or component thereof according to any of claims 18 to 20, wherein a and c are each independently 0 or 1; and b is from 3 to 7.
23. An electronic or electrical device or component thereof according to any preceding claim wherein each X is H.
24. An electronic or electrical device or component thereof according to any of claims 1 to 22, wherein each X is F.
25. An electronic or electrical device or component thereof according to any of claims 18 to 24, wherein R_1 and R_2 are both hydrogen.
26. An electronic or electrical device or component thereof according to any of claims 18 to 25, wherein R_4 is hydrogen or methyl.
27. An electronic or electrical device or component thereof according to any of claims 18 to 26, wherein R_9 is hydrogen and R_{10} is branched or straight chain C_1 - C_6 alkyl group.
28. An electronic or electrical device or component thereof according to claim 27, wherein R_{10} is methyl.
29. An electronic or electrical device or component thereof according to any of claims 18 to 28, wherein each of R_5 to R_8 is hydrogen.
30. An electronic or electrical device or component thereof according to any of claims 18 to 29 wherein each of each of R_1 , R_2 , R_4 , and R_5 to R_{10} is hydrogen, each X is H, a=0 and c=0.
31. An electronic or electrical device or component thereof according to any of claims 24 to 30, wherein the compound of formula I(a) has the following formula:



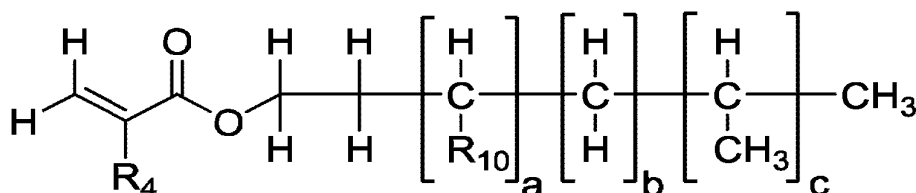
where n is from 2 to 10.

32. An electronic or electrical device or component thereof according to any of claims 24 to 30, wherein the compound of formula I(a) has the following formula:



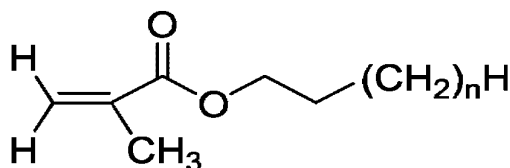
where n is from 2 to 10.

33. An electronic or electrical device or component thereof according to claim 31, wherein the compound of formula I(a) is selected from 1H,1H,2H,2H-perfluorohexyl acrylate (PFAC4), 1H,1H,2H,2H-perfluorooctyl acrylate (PFAC6), 1H,1H,2H,2H-perfluorodecyl acrylate (PFAC8) and 1H,1H,2H,2H-perfluorododecyl acrylate (PFAC10).
34. An electronic or electrical device or component thereof according to claim 32, wherein the compound of formula I(a) is selected from 1H,1H,2H,2H-perfluorohexyl methacrylate (PFMAC4), 1H,1H,2H,2H-perfluorooctyl methacrylate (PFMAC6) and 1H,1H,2H,2H-perfluorodecyl methacrylate (PFMAC8).
35. An electronic or electrical device or component thereof according to any of claims 18 to 23, wherein the compound of formula I(a) has the following formula:



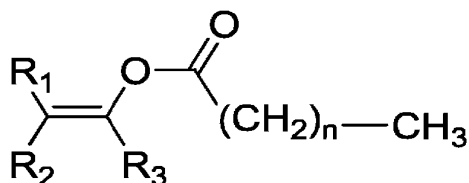
wherein a and c are each independently 0 or 1, b = 3-7 and n is 4 to 10, where n = a + b + c + 1.

36. An electronic or electrical device or component thereof according to any of claims 18 to 23, wherein the compound of formula I(a) has the following formula:



where n is 2 to 12.

37. An electronic or electrical device or component thereof according to claim 35 or claim 36, wherein the compound of formula I(a) is selected from ethyl hexyl acrylate, hexyl acrylate, decyl acrylate, lauryl dodecyl acrylate and iso decyl acrylate.
38. An electronic or electrical device or component thereof according to any of claims 18 to 23, wherein the monomer is a compound of formula I(b) and has the following formula:

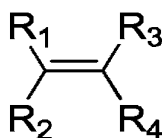


where n is from 4 to 14, optionally where R₁, R₂ and R₃ are each H.

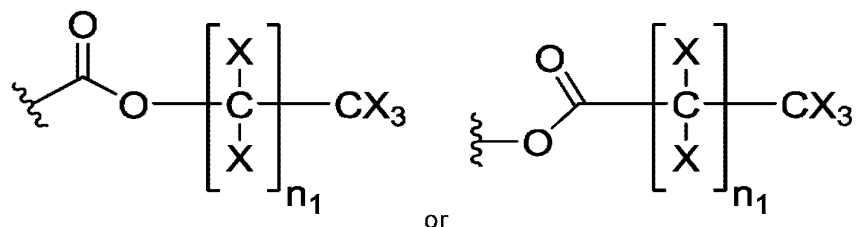
39. An electronic or electrical device or component thereof according to any of the preceding claims, wherein the electronic or electrical device or component thereof is selected from mobile phones, smartphones, pagers, radios, sound and audio systems such as loudspeakers, microphones, ringers and/or buzzers, hearing aids, personal audio equipment such as personal CD, tape cassette or MP3 players, televisions, DVD players including portable DVD players, video recorders, digi and other set-top boxes, computers and related components such as laptop, notebook, tablet, phablet or palmtop computers, personal digital assistants (PDAs), keyboards, or instrumentation, games consoles, data storage devices, outdoor lighting systems, radio antennae and other forms of communication equipment, and printed circuit boards.
40. A method for treating an electronic or electrical device or component thereof comprising:

exposing the electronic or electrical device or component thereof to a plasma comprising a monomer compound and a crosslinking reagent for a period of time sufficient to allow a protective cross-linked polymeric coating to form on a surface of the electronic or electrical device or component thereof;

wherein the monomer compound has the following formula:

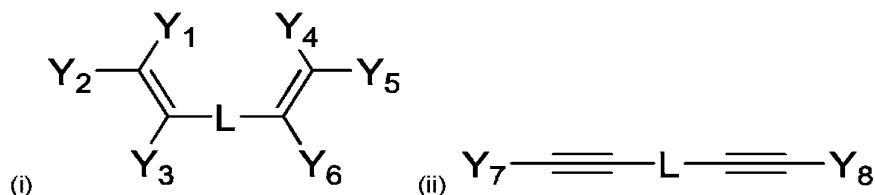


where R_1 , R_2 and R_4 are each independently selected from hydrogen, optionally substituted branched or straight chain C_1 - C_6 alkyl or halo alkyl or aryl optionally substituted by halo, and R_3 is selected from:



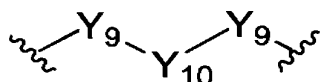
where each X is independently selected from hydrogen, optionally substituted branched or straight chain C_1 - C_6 alkyl, halo alkyl or aryl optionally substituted by halo; and n_1 is an integer from 1 to 27; and wherein the crosslinking reagent comprises two or more unsaturated bonds attached by means of one or more linker moieties and has a boiling point at standard pressure of less than 500°C .

41. A method according to claim 40, wherein the crosslinking reagent has one of the following structures:



where Y_1 , Y_2 , Y_3 , Y_4 , Y_5 , Y_6 , Y_7 and Y_8 are each independently selected from hydrogen, optionally substituted cyclic, branched or straight chain C_1 - C_6 alkyl or aryl; and L is a linker moiety.

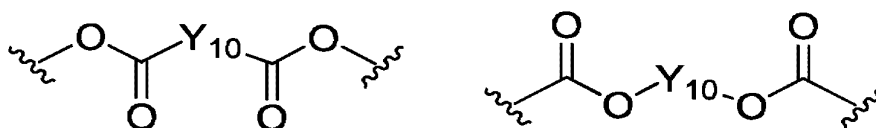
42. A method according to claim 41, wherein for compound (i) L has the following formula:

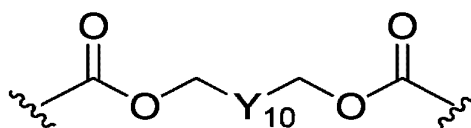
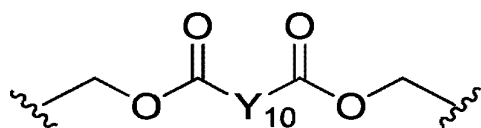


each Y_9 is independently selected from, a bond, $-\text{O}-$, $-\text{O}-\text{C}(\text{O})-$, $-\text{C}(\text{O})-\text{O}-$, $-\text{Y}_{11}-\text{O}-\text{C}(\text{O})-$, $-\text{C}(\text{O})-\text{O}-\text{Y}_{11}-$, $-\text{OY}_{11}-$, and $-\text{Y}_{11}\text{O}-$, where Y_{11} is an optionally substituted cyclic, branched or straight chain C_1 - C_8 alkylene; and

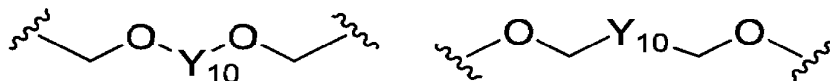
Y_{10} is selected from optionally substituted cyclic, branched or straight chain C_1 - C_8 alkylene and a siloxane group.

43. A method according to claim 42, wherein for compound (i) L has one of the following structures:

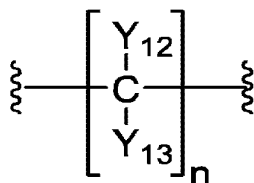




44. A method according to claim 42, wherein for compound (i) L has one of the following structures:

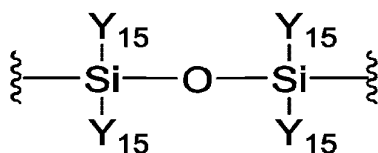


45. An method according to any of claims 42 to 44, wherein Y₁₀ has the following formula:



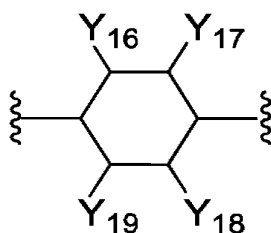
wherein each Y₁₂ and Y₁₃ is independently selected from H, halo, optionally substituted cyclic, branched or straight chain alkyl, or -OY₁₄, where Y₁₄ is selected from optionally substituted branched or straight chain C₁-C₈ alkyl or alkenyl, and n is an integer from 1 to 10.

46. A method according to claim 45, wherein each Y₁₂ is H and each Y₁₃ is H.
 47. A method according to claim 45, wherein each Y₁₂ is fluoro and each Y₁₃ is fluoro.
 48. A method according to any of claims 45 to 47, wherein n is from 4 to 6.
 49. A method according to any of claims 42 to 44, wherein Y₁₀ has the following formula:



Wherein each Y₁₅ is independently selected from optionally substituted branched or straight chain C₁-C₈ alkyl.

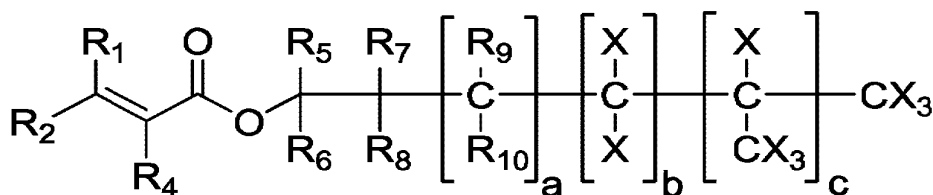
50. A method according to claim 49, wherein each Y₁₅ is methyl, and each Y₉ is a bond.
 51. A method according to any of claims 42 to 44, wherein Y₁₀ has the following formula:



wherein Y_{16} to Y_{19} are each independently selected from H and optionally substituted branched or straight chain C_1 - C_8 alkyl or alkenyl.

52. A method according to claim 51, wherein Y_{18} is vinylene or H, and Y_{16} , Y_{17} and Y_{19} are each H.
53. A method according to any of claims 40 to 52, wherein the crosslinking reagent is selected from Divinyl Adipate (DVA), 1,4 Butanediol divinyl ether (BDVE), 1,4 Cyclohexanedimethanol divinyl ether (CDDE), 1,7 -Octadiene (17OD), 1,2,4 -Trivinylcyclohexane (TVCH), 1,3-Divinyltetramethyldisiloxane (DVTMDS), Diallyl 1,4-cyclohexanedicarboxylate (DCHD), 1,6-Divinylperfluorohexane (DVPFH), 1H,1H,6H,6H-Perfluorohexanediol diacrylate (PFHDA) and glyoxal bis (diallyl acetal) (GBDA).
54. A method according to claim 41, wherein for compound (ii), L is selected from a branched or straight chain C_1 - C_8 alkylene or an ether group.
55. A method according to any of claims 40 to 54 wherein the monomer is a compound of formula I(a):

I(a)

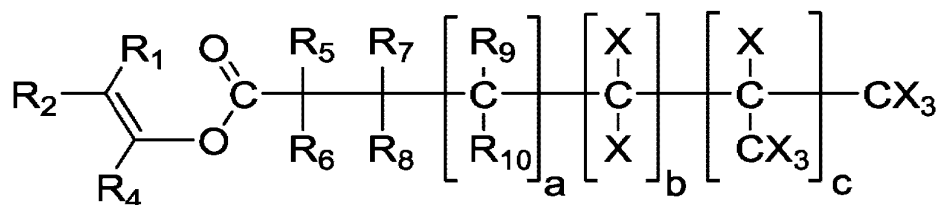


wherein each of R_1 , R_2 , R_4 , and R_5 to R_{10} is independently selected from hydrogen or an optionally substituted C_1 - C_8 branched or straight chain alkyl group; each X is independently selected from hydrogen or halogen; a is from 0-10; b is from 2 to 14; and c is 0 or 1;

; or

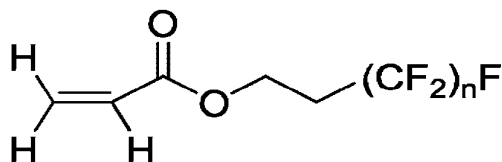
wherein the monomer is a compound of formula I(b):

I(b)



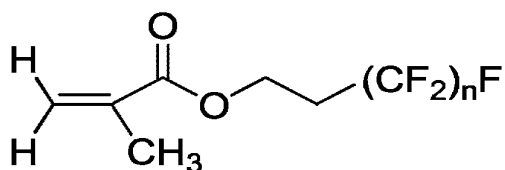
wherein each of R_1 , R_2 , R_4 , and R_5 to R_{10} is independently selected from hydrogen or an optionally substituted C_1 - C_6 branched or straight chain alkyl group; each X is independently selected from hydrogen or halogen; a is from 0-10; b is from 2 to 14; and c is 0 or 1.

56. A method according to claim 55, wherein the halogen is fluorine.
57. A method according to claim 55 or claim 56, wherein each of R_1 , R_2 , R_4 , and R_5 to R_{10} is independently selected from hydrogen, methyl, ethyl, n-propyl, isopropyl, n-butyl, isobutyl, tert-butyl, sec-butyl, n-pentyl, neopentyl, n-hexyl, isohexyl, and 3-methylpentyl.
58. A method according to claim 57, wherein each of R_1 , R_2 , R_4 , and R_5 to R_{10} is independently selected from hydrogen or methyl.
59. A method according to any of claims 55 to 58, wherein a and c are each independently 0 or 1; and b is from 3 to 7.
60. A method according to any preceding claim wherein each X is H.
61. A method according to any of claims 41 to 59, wherein each X is F.
62. A method according to any of claims 41 to 61, wherein R_1 and R_2 are both hydrogen.
63. A method according to any of claims 41 to 62, wherein R_4 is hydrogen or methyl.
64. A method according to any of claims 41 to 63, wherein R_9 is hydrogen and R_{10} is branched or straight chain C_1 - C_6 alkyl group.
65. A method according to claim 64, wherein R_{10} is methyl.
66. A method to any of claims 41 to 65, wherein each of R_5 to R_8 is hydrogen.
67. A method according to any of claims 41 to 66 wherein each of each of R_1 , R_2 , R_4 , and R_5 to R_{10} is hydrogen, each X is H, $a=0$ and $c=0$.
68. A method according to any of claims 61 to 67, wherein the compound of formula I(a) has the following formula:



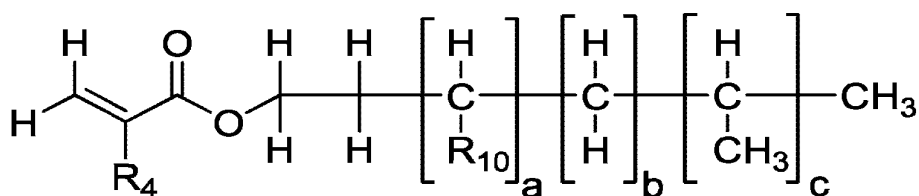
where n is from 2 to 10.

69. A method according to any of claims 61 to 67, wherein the compound of formula I(a) has the following formula:



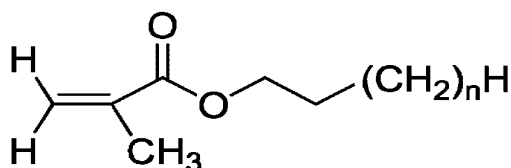
where n is from 2 to 10.

70. A method according to claim 68, wherein the compound of formula I(a) is selected from 1H,1H,2H,2H-perfluorohexyl acrylate (PFAC4), 1H,1H,2H,2H-perfluorooctyl acrylate (PFAC6), 1H,1H,2H,2H-perfluorodecyl acrylate (PFAC8) and 1H,1H,2H,2H-perfluorododecyl acrylate (PFAC10).
71. A method according to claim 69, wherein the compound of formula I(a) is selected from 1H,1H,2H,2H-perfluorohexyl methacrylate (PFMAC4), 1H,1H,2H,2H-perfluorooctyl methacrylate (PFMAC6) and 1H,1H,2H,2H-perfluorodecyl methacrylate (PFMAC8).
72. A method according to any of claims 41 to 61, wherein the compound of formula I(a) has the following formula:



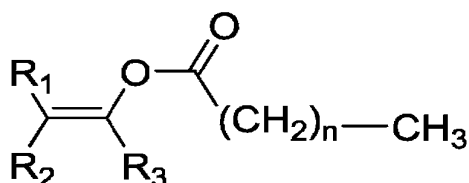
wherein a and c are each independently 0 or 1, b = 3-7 and n is 4 to 10, where n = a + b + c + 1.

73. A method according to any of claims 41 to 61, wherein the compound of formula I(a) has the following formula:



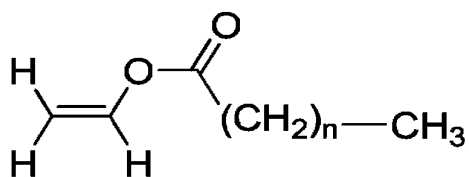
where n is 2 to 12.

74. A method according to claim 72 or claim 73, wherein the compound of formula I(a) is selected from ethyl hexyl acrylate, hexyl acrylate, decyl acrylate, lauryl dodecyl acrylate and iso decyl acrylate.
75. A method according to any of claims 41 to 60, wherein the monomer is a compound of formula I(b) and has the following formula:



where n is from 4 to 14.

76. A method according to claim 75 wherein the monomer is a compound of formula I(b) and has the following formula:



where n is from 4 to 14.

77. A method according to any of claims 40 to 76 wherein the electronic or electrical device or component thereof is placed in a plasma deposition chamber, a glow discharge is ignited within said chamber, and a voltage applied as a pulsed field.
78. A method according to claim 77 wherein applied voltage is at a power of from 40 to 500W.
79. A method according to claim 77 or claim 78 wherein the voltage is pulsed in a sequence in which the ratio of the time on : time off is in the range of from 1:500 to 1:1500.
80. A method according to any of claims 77 to 79 wherein the voltage is pulsed in a sequence where power is on for 20-50μs, and off for from 1000μs to 30000μs.
81. A method according to any of claims 77 to 80 wherein the voltage is applied as a pulsed field for a period of from 30 seconds to 90 minutes.
82. A method according to claim 81 wherein the voltage is applied as a pulsed field for from 5 to 60 minutes.
83. A method according to any one of claims 77 to 82, wherein in a preliminary step, a continuous power plasma is applied to the electronic or electrical device or component thereof.

1 / 6

○ Tack Free
× Tacky

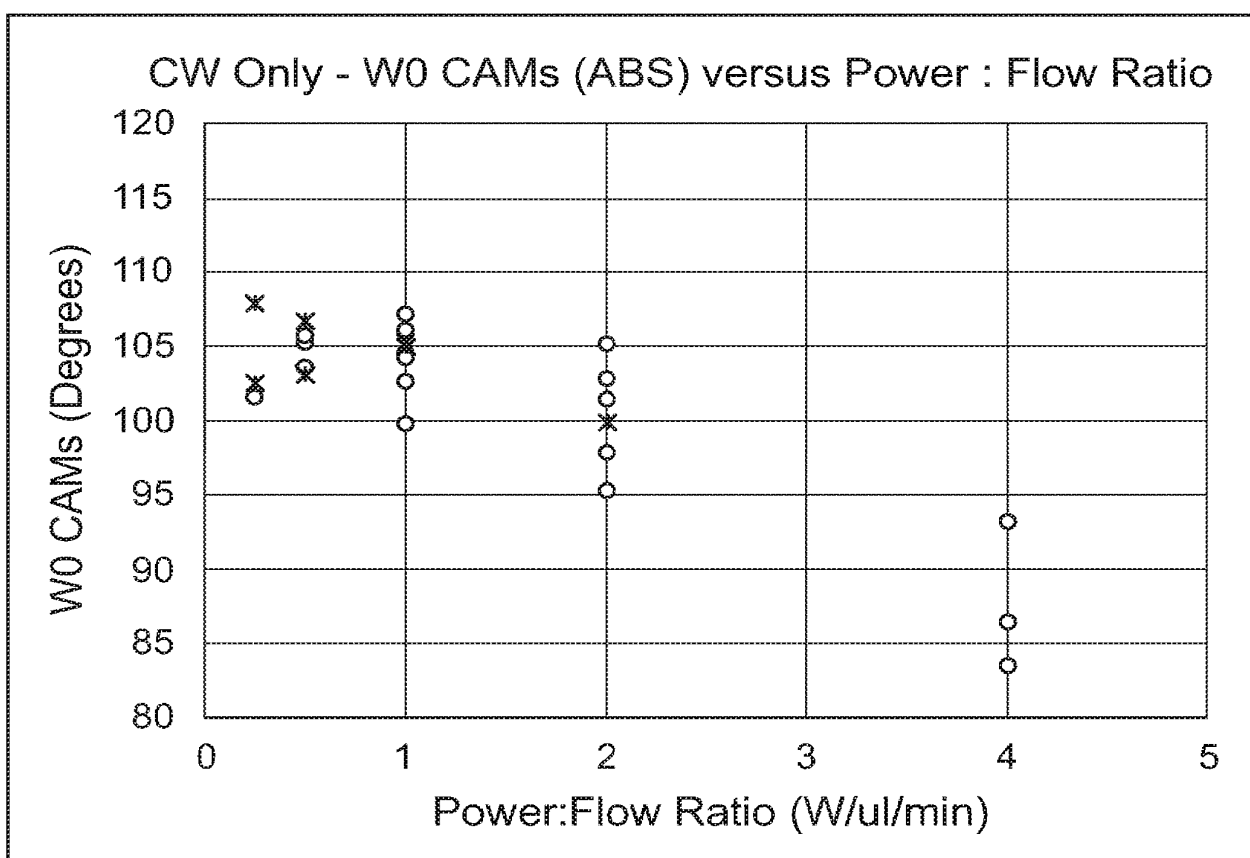


Figure 1

2 / 6

○ Tack Free
× Tacky

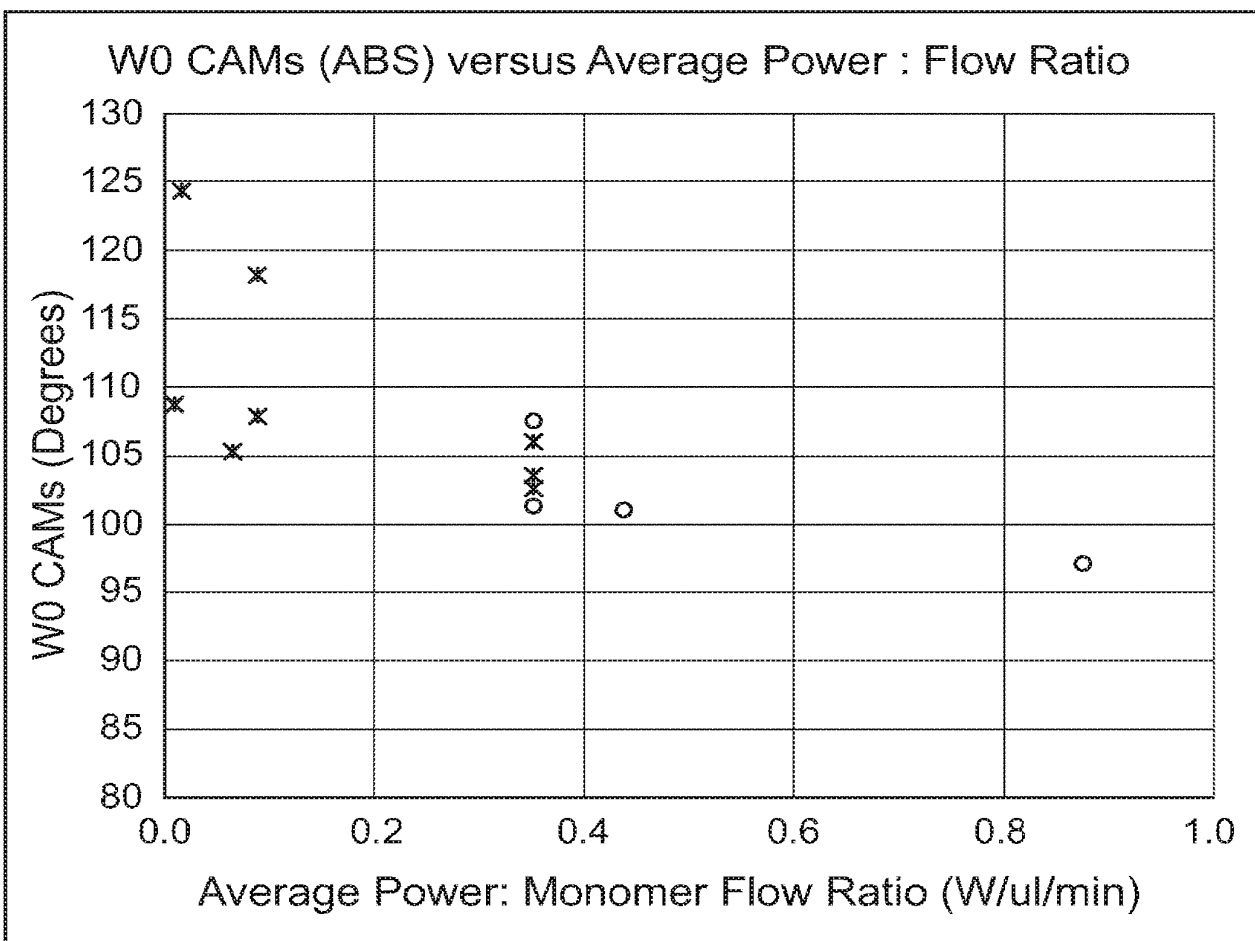


Figure 2

3 / 6

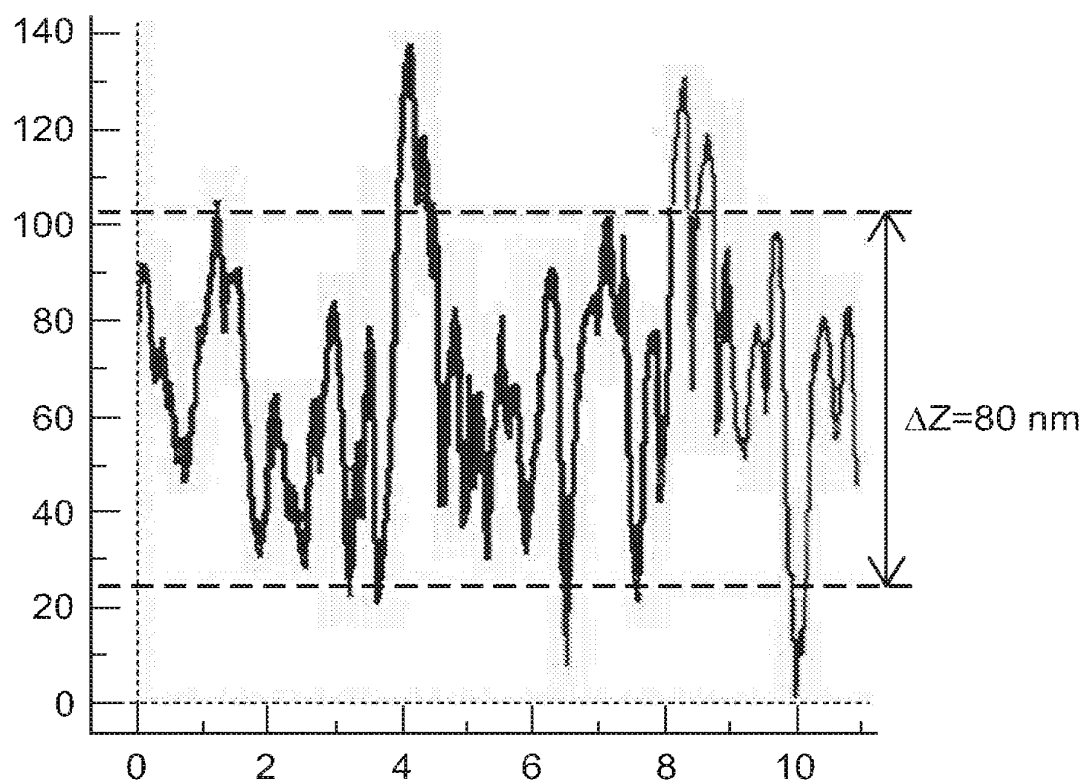
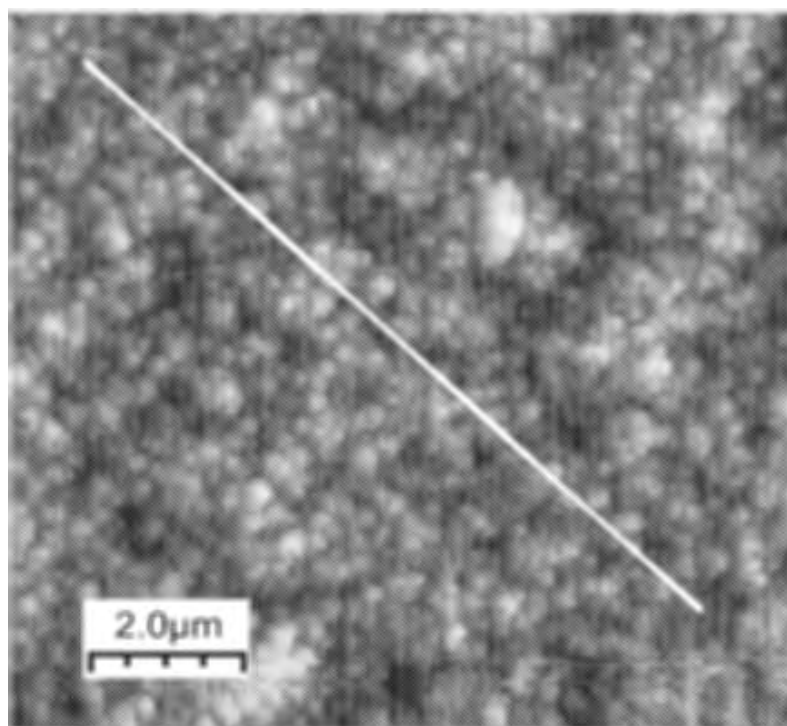


Figure 3

4 / 6

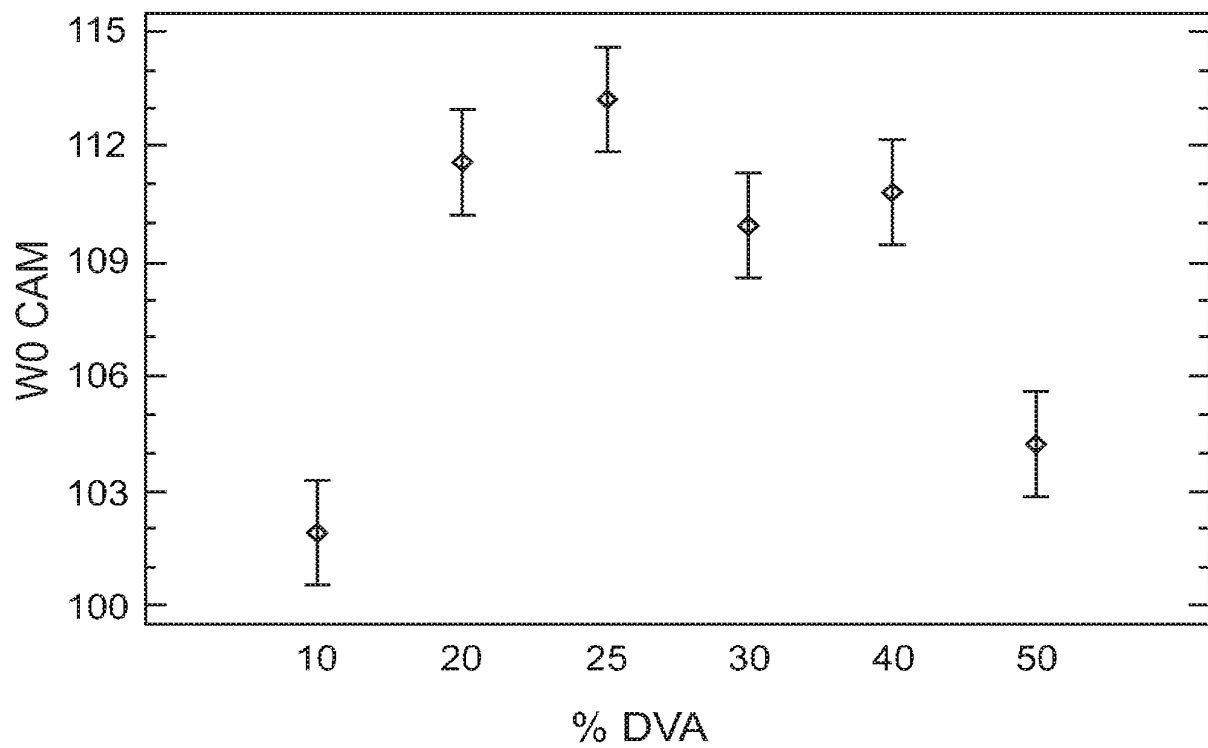


Figure 4

5 / 6

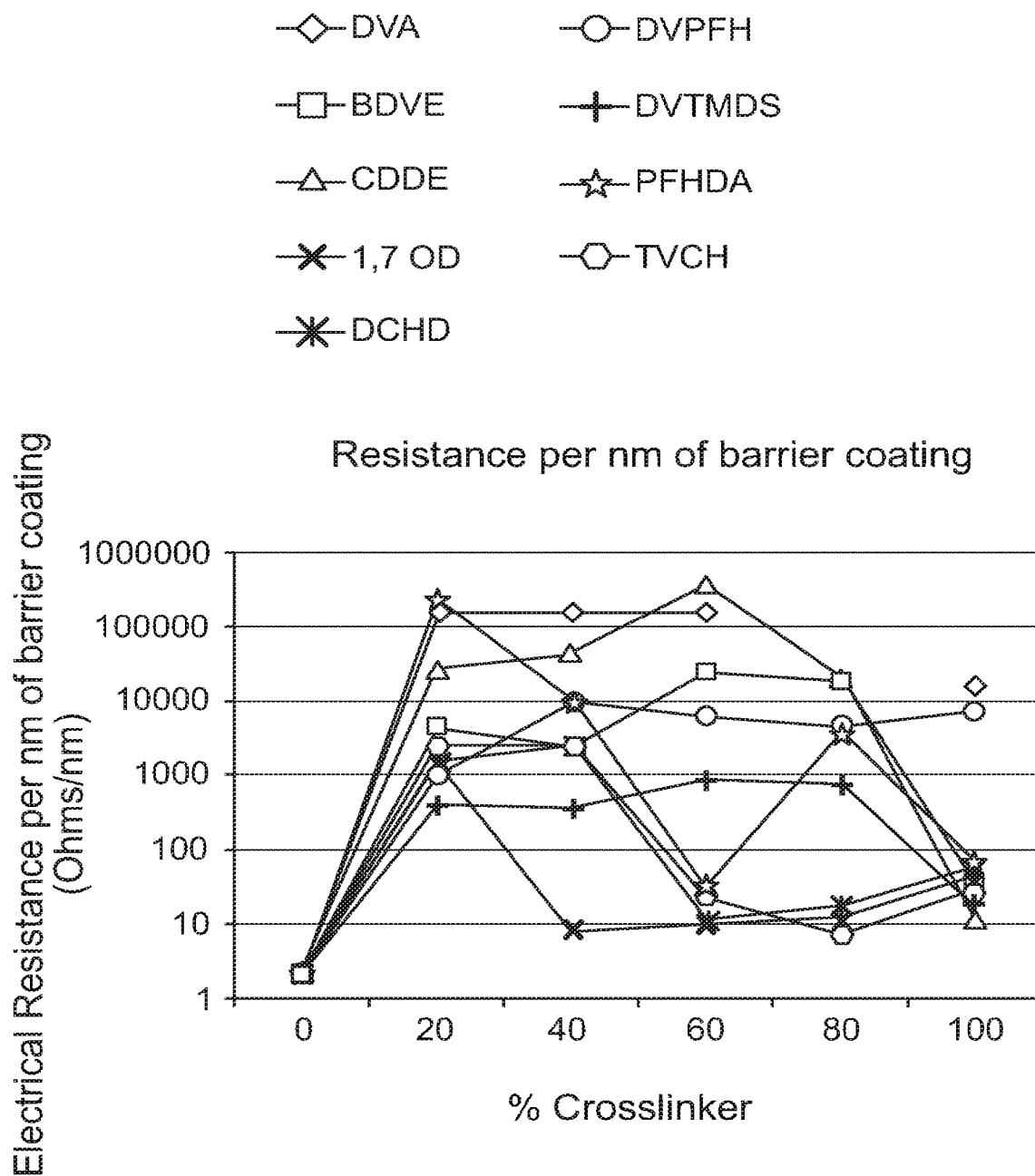


Figure 5

6 / 6

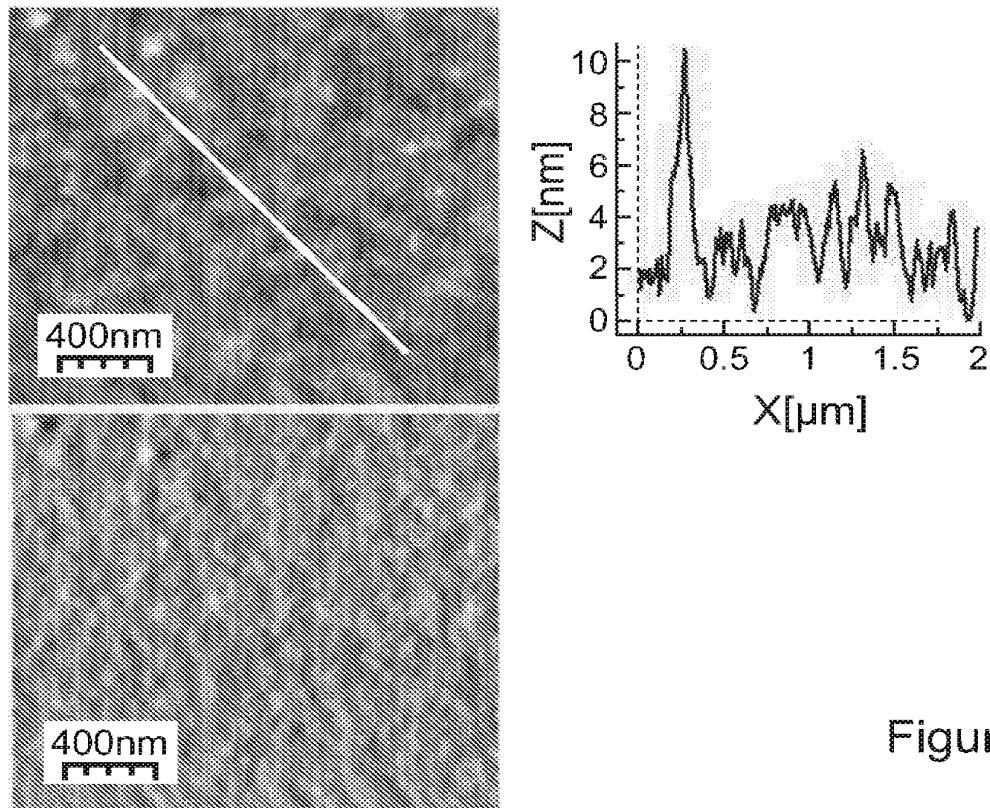
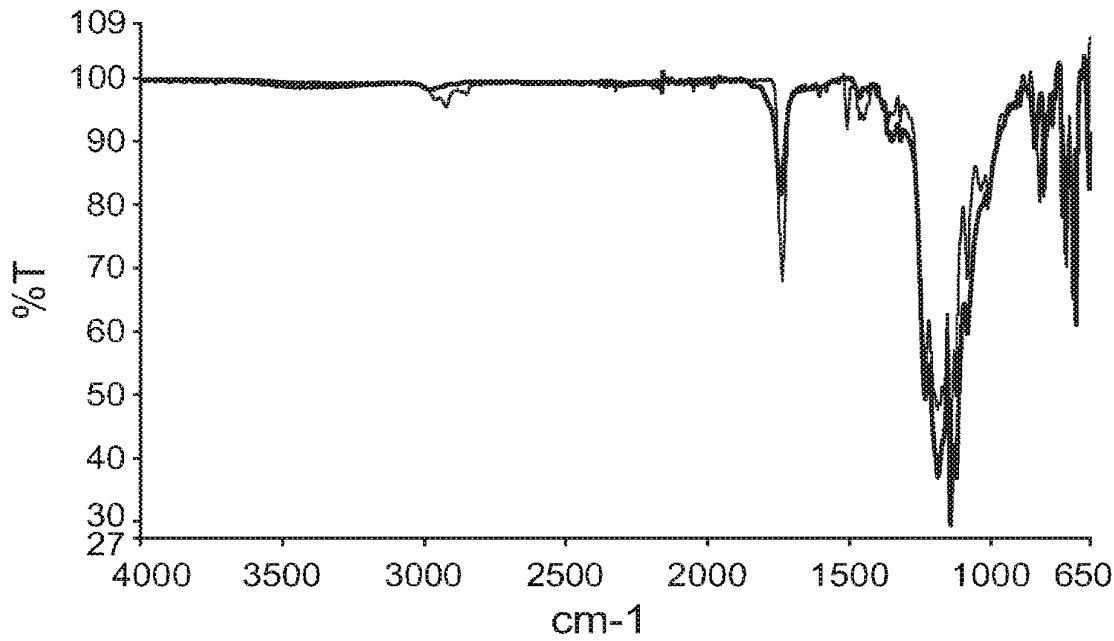


Figure 6



| Name | Description |
|------------------|--|
| — PFAC6 | Sample 350 By Administrator Date Thursday, February 13 2014 |
| — PFAC6-DVA(10%) | Sample 389 By PEService Date Tuesday, July 14 2015 |

INTERNATIONAL SEARCH REPORT

International application No

PCT/GB2016/051688

A. CLASSIFICATION OF SUBJECT MATTER

INV. C09D5/00
ADD.

According to International Patent Classification (IPC) or to both national classification and IPC

B. FIELDS SEARCHED

Minimum documentation searched (classification system followed by classification symbols)

C09D

Documentation searched other than minimum documentation to the extent that such documents are included in the fields searched

Electronic data base consulted during the international search (name of data base and, where practicable, search terms used)

EPO-Internal, WPI Data

C. DOCUMENTS CONSIDERED TO BE RELEVANT

| Category* | Citation of document, with indication, where appropriate, of the relevant passages | Relevant to claim No. |
|-----------|---|-----------------------|
| Y | WO 2007/083122 A1 (P2I LTD [GB]; COULSON STEPHEN [GB]) 26 July 2007 (2007-07-26) claims 10, 11, 27; example 1 ----- | 1-91 |
| Y | EP 0 834 352 A1 (CIBA GEIGY AG [CH]) 8 April 1998 (1998-04-08) claim 8; page 5 line 59 - page 6 lines 1-2; example B-14 ----- | 1-91 |
| A | WO 2014/155099 A1 (SEMBLANT LTD [GB]) 2 October 2014 (2014-10-02) claims 1, 5 together with examples 1, 2 and 3 ----- | 1-91 |



Further documents are listed in the continuation of Box C.



See patent family annex.

* Special categories of cited documents :

"A" document defining the general state of the art which is not considered to be of particular relevance

"E" earlier application or patent but published on or after the international filing date

"L" document which may throw doubts on priority claim(s) or which is cited to establish the publication date of another citation or other special reason (as specified)

"O" document referring to an oral disclosure, use, exhibition or other means

"P" document published prior to the international filing date but later than the priority date claimed

"T" later document published after the international filing date or priority date and not in conflict with the application but cited to understand the principle or theory underlying the invention

"X" document of particular relevance; the claimed invention cannot be considered novel or cannot be considered to involve an inventive step when the document is taken alone

"Y" document of particular relevance; the claimed invention cannot be considered to involve an inventive step when the document is combined with one or more other such documents, such combination being obvious to a person skilled in the art

"&" document member of the same patent family

Date of the actual completion of the international search

28 October 2016

Date of mailing of the international search report

08/11/2016

Name and mailing address of the ISA/

European Patent Office, P.B. 5818 Patentlaan 2
NL - 2280 HV Rijswijk
Tel. (+31-70) 340-2040,
Fax: (+31-70) 340-3016

Authorized officer

Kositza, Matthias

INTERNATIONAL SEARCH REPORT

Information on patent family members

International application No

PCT/GB2016/051688

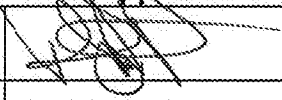
| Patent document cited in search report | Publication date | Patent family member(s) | Publication date |
|---|---------------------|----------------------------|---------------------|
| WO 2007083122 A1 | 26-07-2007 | AU 2007206779 A1 | 26-07-2007 |
| | | CA 2637625 A1 | 26-07-2007 |
| | | CN 101370877 A | 18-02-2009 |
| | | CN 103396688 A | 20-11-2013 |
| | | EP 1976942 A1 | 08-10-2008 |
| | | GB 2434369 A | 25-07-2007 |
| | | IL 192905 A | 27-06-2013 |
| | | JP 2009523914 A | 25-06-2009 |
| | | KR 20080097201 A | 04-11-2008 |
| | | KR 20140063887 A | 27-05-2014 |
| | | NZ 570462 A | 26-11-2010 |
| | | TW I400014 B | 21-06-2013 |
| | | US 2010189914 A1 | 29-07-2010 |
| | | WO 2007083122 A1 | 26-07-2007 |
| | | ZA 200806846 B | 25-11-2009 |
| ----- | | | |
| EP 0834352 A1 | 08-04-1998 | AR 004877 A1 | 10-03-1999 |
| | | EP 0834352 A1 | 08-04-1998 |
| | | US 6169127 B1 | 02-01-2001 |
| ----- | | | |
| WO 2014155099 A1 | 02-10-2014 | CN 105075408 A | 18-11-2015 |
| | | EP 2979524 A1 | 03-02-2016 |
| | | JP 2016521296 A | 21-07-2016 |
| | | KR 20150135420 A | 02-12-2015 |
| | | TW 201446890 A | 16-12-2014 |
| | | WO 2014155099 A1 | 02-10-2014 |
| ----- | | | |

TRANSMITTAL FOR POWER OF ATTORNEY TO ONE OR MORE REGISTERED PRACTITIONERS

NOTE: This form is to be submitted with the Power of Attorney by Applicant form (PTO/AIA/82B) to identify the application to which the Power of Attorney is directed, in accordance with 37 CFR 1.5, unless the application number and filing date are identified in the Power of Attorney by Applicant form. If neither form PTO/AIA/82A nor form PTO/AIA/82B identifies the application to which the Power of Attorney is directed, the Power of Attorney will not be recognized in the application.

| | |
|------------------------|-------------------------|
| Application Number | Not Yet Assigned |
| Filing Date | Herewith |
| First Named Inventor | Stephen Richard COULSON |
| Title | COATINGS |
| Art Unit | Not Yet Assigned |
| Examiner Name | Not Yet Assigned |
| Attorney Docket Number | GBL0007/US |

SIGNATURE of Applicant or Patent Practitioner

| | | | |
|--|---|---------------------|------------------|
| Signature |  | Date (Optional) | December 8, 2017 |
| Name | Paul J. Parins | Registration Number | 54,358 |
| Title (if Applicant is a juristic entity) | | | |
| Applicant Name (if Applicant is a juristic entity) | | | |

NOTE: This form must be signed in accordance with 37 CFR 1.33. See 37 CFR 1.4(d) for signature requirements and certifications. If more than one applicant, use multiple forms.



*Total of 1 forms are submitted.

This collection of information is required by 37 CFR 1.131, 1.32, and 1.33. The information is required to obtain or retain a benefit by the public which is to file (and by the USPTO to process) an application. Confidentiality is governed by 35 U.S.C. 122 and 37 CFR 1.11 and 1.14. This collection is estimated to take 3 minutes to complete, including gathering, preparing, and submitting the completed application form to the USPTO. Time will vary depending upon the individual case. Any comments on the amount of time you require to complete this form and/or suggestions for reducing this burden, should be sent to the Chief Information Officer, U.S. Patent and Trademark Office, U.S. Department of Commerce, P.O. Box 1450, Alexandria, VA 22313-1450. DO NOT SEND FEES OR COMPLETED FORMS TO THIS ADDRESS. **SEND TO: Commissioner for Patents, P.O. Box 1450, Alexandria, VA 22313-1450.**

If you need assistance in completing the form, call 1-800-PTO-9199 and select option 2.

POWER OF ATTORNEY BY APPLICANT

I hereby revoke all previous powers of attorney given in the application identified in either the attached transmittal letter or the boxes below.

| Application Number | Filing Date |
|--------------------|-------------|
| | |

(Note: The boxes above may be left blank if information is provided on form PTO/AIA/82A.)

☒ I hereby appoint the Patent Practitioner(s) associated with the following Customer Number as my/our attorney(s) or agent(s), and to transact all business in the United States Patent and Trademark Office connected therewith for the application referenced in the attached transmittal letter (form PTO/AIA/82A) or identified above:

33072

OR

☐ I hereby appoint Practitioner(s) named in the attached list (form PTO/AIA/82C) as my/our attorney(s) or agent(s), and to transact all business in the United States Patent and Trademark Office connected therewith for the patent application referenced in the attached transmittal letter (form PTO/AIA/82A) or identified above. (Note: Complete form PTO/AIA/82C.)

Please recognize or change the correspondence address for the application identified in the attached transmittal letter or the boxes above to:

☒ The address associated with the above-mentioned Customer Number

OR

☐ The address associated with Customer Number:

OR

| | | | | | |
|-------------------------|--|-------|--|-----|--|
| Firm or Individual Name | | | | | |
| Address | | | | | |
| City | | State | | Zip | |
| Country | | | | | |
| Telephone | | Email | | | |

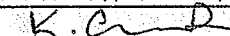
I am the Applicant (if the Applicant is a juristic entity, list the Applicant name in the box):

P2i Ltd

- ☐ Inventor or Joint Inventor (title not required below)
- ☐ Legal Representative of a Deceased or Legally Incapacitated Inventor (title not required below)
- ☒ Assignee or Person to Whom the Inventor is Under an Obligation to Assign (provide signer's title if applicant is a juristic entity)
- ☐ Person Who Otherwise Shows Sufficient Proprietary Interest (e.g., a petition under 37 CFR 1.46(b)(2) was granted in the application or is concurrently being filed with this document) (provide signer's title if applicant is a juristic entity)

SIGNATURE of Applicant for Patent

The undersigned (whose title is supplied below) is authorized to act on behalf of the applicant (e.g., where the applicant is a juristic entity).

| | | | |
|-----------|---|-----------------|-----------------|
| Signature |  | Date (Optional) | 26 October 2016 |
| Name | Dr. Kenneth Armour | | |
| Title | In-House Counsel, P2i LTD | | |

NOTE: Signature - This form must be signed by the applicant in accordance with 37 CFR 1.33. See 37 CFR 1.4 for signature requirements and certifications. If more than one applicant, use multiple forms.

☒ Total of 1 forms are submitted.

This collection of information is required by 37 CFR 1.131, 1.32, and 1.33. The information is required to obtain or retain a benefit by the public which is to file (and by the USPTO to process) an application. Confidentiality is governed by 35 U.S.C. 122 and 37 CFR 1.11 and 1.14. This collection is estimated to take 3 minutes to complete, including gathering, preparing, and submitting the completed application form to the USPTO. Time will vary depending upon the individual case. Any comments on the amount of time you require to complete this form and/or suggestions for reducing this burden, should be sent to the Chief Information Officer, U.S. Patent and Trademark Office, U.S. Department of Commerce, P.O. Box 1450, Alexandria, VA 22313-1450. DO NOT SEND FEES OR COMPLETED FORMS TO THIS ADDRESS. SEND TO: Commissioner for Patents, P.O. Box 1450, Alexandria, VA 22313-1450.

If you need assistance in completing the form, call 1-800-PTO-9199 and select option 2.

PATENT COOPERATION TREATY

PCT

INTERNATIONAL SEARCH REPORT

(PCT Article 18 and Rules 43 and 44)

| | | |
|--|--|--|
| Applicant's or agent's file reference P2494PC00 | FOR FURTHER ACTION <small>see Form PCT/ISA/220 as well as, where applicable, item 5 below.</small> | |
| International application No. PCT/GB2016/051688 | International filing date (<i>day/month/year</i>) 8 June 2016 (08-06-2016) | (Earliest) Priority Date (<i>day/month/year</i>) 9 June 2015 (09-06-2015) |
| Applicant P2I LTD | | |

This international search report has been prepared by this International Searching Authority and is transmitted to the applicant according to Article 18. A copy is being transmitted to the International Bureau.

This international search report consists of a total of 3 sheets.

☒ It is also accompanied by a copy of each prior art document cited in this report.

1. Basis of the report

a. With regard to the **language**, the international search was carried out on the basis of:

- ☒ the international application in the language in which it was filed
☐ a translation of the international application into _____, which is the language of a translation furnished for the purposes of international search (Rules 12.3(a) and 23.1(b))

b. ☐ This international search report has been established taking into account the **rectification of an obvious mistake** authorized by or notified to this Authority under Rule 91 (Rule 43.6**bis**(a)).

c. ☐ With regard to any **nucleotide and/or amino acid sequence** disclosed in the international application, see Box No. I.

2. ☐ **Certain claims were found unsearchable** (See Box No. II)

3. ☐ **Unity of invention is lacking** (see Box No III)

4. With regard to the **title**,

- ☒ the text is approved as submitted by the applicant
☐ the text has been established by this Authority to read as follows:

5. With regard to the **abstract**,

- ☒ the text is approved as submitted by the applicant
☐ the text has been established, according to Rule 38.2, by this Authority as it appears in Box No. IV. The applicant may, within one month from the date of mailing of this international search report, submit comments to this Authority

6. With regard to the **drawings**,

- a. the figure of the **drawings** to be published with the abstract is Figure No. _____
☐ as suggested by the applicant
☐ as selected by this Authority, because the applicant failed to suggest a figure
☐ as selected by this Authority, because this figure better characterizes the invention
- b. ☒ none of the figures is to be published with the abstract

INTERNATIONAL SEARCH REPORT

International application No

PCT/GB2016/051688

A. CLASSIFICATION OF SUBJECT MATTER

INV. C09D5/00
ADD.

According to International Patent Classification (IPC) or to both national classification and IPC

B. FIELDS SEARCHED

Minimum documentation searched (classification system followed by classification symbols)

C09D

Documentation searched other than minimum documentation to the extent that such documents are included in the fields searched

Electronic data base consulted during the international search (name of data base and, where practicable, search terms used)

EPO-Internal, WPI Data

C. DOCUMENTS CONSIDERED TO BE RELEVANT

| Category* | Citation of document, with indication, where appropriate, of the relevant passages | Relevant to claim No. |
|-----------|---|-----------------------|
| Y | WO 2007/083122 A1 (P2I LTD [GB]; COULSON STEPHEN [GB]) 26 July 2007 (2007-07-26) claims 10, 11, 27; example 1 ----- | 1-91 |
| Y | EP 0 834 352 A1 (CIBA GEIGY AG [CH]) 8 April 1998 (1998-04-08) claim 8; page 5 line 59 - page 6 lines 1-2; example B-14 ----- | 1-91 |
| A | WO 2014/155099 A1 (SEMBLANT LTD [GB]) 2 October 2014 (2014-10-02) claims 1, 5 together with examples 1, 2 and 3 ----- | 1-91 |



Further documents are listed in the continuation of Box C.



See patent family annex.

* Special categories of cited documents :

"A" document defining the general state of the art which is not considered to be of particular relevance

"E" earlier application or patent but published on or after the international filing date

"L" document which may throw doubts on priority claim(s) or which is cited to establish the publication date of another citation or other special reason (as specified)

"O" document referring to an oral disclosure, use, exhibition or other means

"P" document published prior to the international filing date but later than the priority date claimed

"T" later document published after the international filing date or priority date and not in conflict with the application but cited to understand the principle or theory underlying the invention

"X" document of particular relevance; the claimed invention cannot be considered novel or cannot be considered to involve an inventive step when the document is taken alone

"Y" document of particular relevance; the claimed invention cannot be considered to involve an inventive step when the document is combined with one or more other such documents, such combination being obvious to a person skilled in the art

"&" document member of the same patent family

Date of the actual completion of the international search

28 October 2016

Date of mailing of the international search report

08/11/2016

Name and mailing address of the ISA/

European Patent Office, P.B. 5818 Patentlaan 2
NL - 2280 HV Rijswijk
Tel. (+31-70) 340-2040,
Fax: (+31-70) 340-3016

Authorized officer

Kositza, Matthias

INTERNATIONAL SEARCH REPORT

Information on patent family members

International application No

PCT/GB2016/051688

| Patent document cited in search report | Publication date | Patent family member(s) | Publication date |
|---|---------------------|----------------------------|---------------------|
| WO 2007083122 A1 | 26-07-2007 | AU 2007206779 A1 | 26-07-2007 |
| | | CA 2637625 A1 | 26-07-2007 |
| | | CN 101370877 A | 18-02-2009 |
| | | CN 103396688 A | 20-11-2013 |
| | | EP 1976942 A1 | 08-10-2008 |
| | | GB 2434369 A | 25-07-2007 |
| | | IL 192905 A | 27-06-2013 |
| | | JP 2009523914 A | 25-06-2009 |
| | | KR 20080097201 A | 04-11-2008 |
| | | KR 20140063887 A | 27-05-2014 |
| | | NZ 570462 A | 26-11-2010 |
| | | TW I400014 B | 21-06-2013 |
| | | US 2010189914 A1 | 29-07-2010 |
| | | WO 2007083122 A1 | 26-07-2007 |
| | | ZA 200806846 B | 25-11-2009 |
| ----- | | | |
| EP 0834352 A1 | 08-04-1998 | AR 004877 A1 | 10-03-1999 |
| | | EP 0834352 A1 | 08-04-1998 |
| | | US 6169127 B1 | 02-01-2001 |
| ----- | | | |
| WO 2014155099 A1 | 02-10-2014 | CN 105075408 A | 18-11-2015 |
| | | EP 2979524 A1 | 03-02-2016 |
| | | JP 2016521296 A | 21-07-2016 |
| | | KR 20150135420 A | 02-12-2015 |
| | | TW 201446890 A | 16-12-2014 |
| | | WO 2014155099 A1 | 02-10-2014 |
| ----- | | | |

Information on Search Strategy - Pilot phase (see OJ 2015, A86)

The type of information contained in this sheet may change during the pilot for improving the usefulness of this new service.

Application Number

PCT/GB2016/051688

TITLE: COATINGS

APPLICANT: P2I LTD

IPC CLASSIFICATION: C09D5/00

EXAMINER: Kositza, Matthias

CONSULTED DATABASES: EPODOC, WPI, NPL

CLASSIFICATION SYMBOLS DEFINING EXTENT OF THE SEARCH:

IPC:

CPC: C09D5/++

FI/F-TERMS:

KEYWORDS OR OTHER ELEMENTS FEATURING THE INVENTION:

PATENT COOPERATION TREATY

From the
INTERNATIONAL SEARCHING AUTHORITY

PCT

WRITTEN OPINION OF THE INTERNATIONAL SEARCHING AUTHORITY (PCT Rule 43bis.1)

To:

see form PCT/ISA/220

Date of mailing
(day/month/year) see form PCT/ISA/210 (second sheet)

Applicant's or agent's file reference
see form PCT/ISA/220

FOR FURTHER ACTION
See paragraph 2 below

International application No.
PCT/GB2016/051688

International filing date (day/month/year)
08.06.2016

Priority date (day/month/year)
09.06.2015

International Patent Classification (IPC) or both national classification and IPC
INV. C09D5/00

Applicant
P2I LTD

1. This opinion contains indications relating to the following items:

- ☒ Box No. I Basis of the opinion
- ☐ Box No. II Priority
- ☐ Box No. III Non-establishment of opinion with regard to novelty, inventive step and industrial applicability
- ☐ Box No. IV Lack of unity of invention
- ☒ Box No. V Reasoned statement under Rule 43bis.1(a)(i) with regard to novelty, inventive step and industrial applicability; citations and explanations supporting such statement
- ☐ Box No. VI Certain documents cited
- ☐ Box No. VII Certain defects in the international application
- ☐ Box No. VIII Certain observations on the international application

2. FURTHER ACTION

If a demand for international preliminary examination is made, this opinion will usually be considered to be a written opinion of the International Preliminary Examining Authority ("IPEA") except that this does not apply where the applicant chooses an Authority other than this one to be the IPEA and the chosen IPEA has notified the International Bureau under Rule 66.1bis(b) that written opinions of this International Searching Authority will not be so considered.

If this opinion is, as provided above, considered to be a written opinion of the IPEA, the applicant is invited to submit to the IPEA a written reply together, where appropriate, with amendments, before the expiration of 3 months from the date of mailing of Form PCT/ISA/220 or before the expiration of 22 months from the priority date, whichever expires later.

For further options, see Form PCT/ISA/220.

Name and mailing address of the ISA:



European Patent Office
D-80298 Munich
Tel. +49 89 2399 - 0
Fax: +49 89 2399 - 4465

Date of completion of
this opinion

see form
PCT/ISA/210

Authorized Officer

Kositza, Matthias

Telephone No. +49 89 2399-0



Box No. I Basis of the opinion

1. With regard to the **language**, this opinion has been established on the basis of:
 - ☒ the international application in the language in which it was filed.
 - ☐ a translation of the international application into , which is the language of a translation furnished for the purposes of international search (Rules 12.3(a) and 23.1 (b)).
2. ☐ This opinion has been established taking into account the **rectification of an obvious mistake** authorized by or notified to this Authority under Rule 91 (Rule 43*bis*.1(a))
3. ☐ With regard to any **nucleotide and/or amino acid sequence** disclosed in the international application, this opinion has been established on the basis of a sequence listing:
 - a. ☐ forming part of the international application as filed:
 - ☐ in the form of an Annex C/ST.25 text file.
 - ☐ on paper or in the form of an image file.
 - b. ☐ furnished together with the international application under PCT Rule 13*ter*.1(a) for the purposes of international search only in the form of an Annex C/ST.25 text file.
 - c. ☐ furnished subsequent to the international filing date for the purposes of international search only:
 - ☐ in the form of an Annex C/ST.25 text file (Rule 13*ter*.1(a)).
 - ☐ on paper or in the form of an image file (Rule 13*ter*.1(b) and Administrative Instructions, Section 713).
4. ☐ In addition, in the case that more than one version or copy of a sequence listing has been filed or furnished, the required statements that the information in the subsequent or additional copies is identical to that forming part of the application as filed or does not go beyond the application as filed, as appropriate, were furnished.
5. Additional comments:

**WRITTEN OPINION OF THE
INTERNATIONAL SEARCHING AUTHORITY**

International application No.
PCT/GB2016/051688

Box No. V Reasoned statement under Rule 43bis.1(a)(i) with regard to novelty, inventive step or industrial applicability; citations and explanations supporting such statement

1. Statement

| | | |
|-------------------------------|-------------|-------------|
| Novelty (N) | Yes: Claims | <u>1-91</u> |
| | No: Claims | |
| Inventive step (IS) | Yes: Claims | |
| | No: Claims | <u>1-91</u> |
| Industrial applicability (IA) | Yes: Claims | <u>1-91</u> |
| | No: Claims | |

2. Citations and explanations

see separate sheet

item V

1. Documents

D1 WO 2007/083122 A1

D2 EP 0 834 352 A1

D3 WO 2014/155099 A1

2. Independent claims

Claim 1 relates to an electronic or electrical device or component thereof having a protective cross-linked polymeric coating made by plasma polymerization of a combination of components (1) + (2):

- (1) unsaturated monomer having the formula detailed in the claim
- (2) (poly)unsaturated cross-linking agent.

Claim 40 relates to a method for providing an electronic or electrical device or component thereof having a protective cross-linked polymeric coating made by plasma polymerization of a combination of components (1) + (2) according to claim 1.

3. Novelty

Claims 1 and 40, and the claims dependent thereon, are considered novel.

4. Inventive step

Claims 1 and 40 do not involve an inventive step. Starting from document D1 (see claims 10, 11, 27; example 1) describing the plasma polymerization of component (1) for making protective coatings for electronic / electrical components the skilled person would easily recognize by combining document D2 (see claim 8; p. 5 l. 59 - p. 6, l. 1-2, example B-14) that adding an additional cross-linking component (2) allows to vary / control the degree of cross-linking of the coating. See the application page 2, lines 1-10. At present it cannot be seen that the addition of the cross-linking component (2) is connected with any surprising beneficial technical effect which could substantiate the presence of an inventive step. The dependent claims represent trivial modifications of claims 1 and 40 which are obvious to the skilled person familiar with the present technical field.

5. Industrial applicability

The claimed subject-matter is industrially applicable.

Electronic Patent Application Fee Transmittal

| | | | | |
|---|-------------------------------|-----------------|---------------|-----------------------------|
| Application Number: | | | | |
| Filing Date: | | | | |
| Title of Invention: | COATINGS | | | |
| First Named Inventor/Applicant Name: | Stephen Richard COULSON | | | |
| Filer: | Paul John Parins/Sawyer Imrie | | | |
| Attorney Docket Number: | GBL0007/US | | | |
| Filed as Large Entity | | | | |
| Filing Fees for U.S. National Stage under 35 USC 371 | | | | |
| Description | Fee Code | Quantity | Amount | Sub-Total in USD(\$) |
| Basic Filing: | | | | |
| NATIONAL STAGE FEE | 1631 | 1 | 280 | 280 |
| NATL STAGE SEARCH FEE - REPORT PROVIDED | 1642 | 1 | 480 | 480 |
| NATIONAL STAGE EXAM - ALL OTHER CASES | 1633 | 1 | 720 | 720 |
| Pages: | | | | |
| Claims: | | | | |
| Miscellaneous-Filing: | | | | |
| OATH/DECL > 30 MOS FROM 371 COMMENCEMENT | 1617 | 1 | 140 | 140 |
| Petition: | | | | |

| Description | Fee Code | Quantity | Amount | Sub-Total in USD(\$) |
|-----------------------------------|----------|----------|--------|----------------------|
| Patent-Appeals-and-Interference: | | | | |
| Post-Allowance-and-Post-Issuance: | | | | |
| Extension-of-Time: | | | | |
| Miscellaneous: | | | | |
| Total in USD (\$) | | | | 1620 |

Electronic Acknowledgement Receipt

| | |
|---|--------------------------------------|
| EFS ID: | 31178417 |
| Application Number: | 15735130 |
| International Application Number: | PCT/GB2016/051688 |
| Confirmation Number: | 7101 |
| Title of Invention: | COATINGS |
| First Named Inventor/Applicant Name: | Stephen Richard COULSON |
| Customer Number: | 33072 |
| Filer: | Paul John Parins/Sawyer Imrie |
| Filer Authorized By: | Paul John Parins |
| Attorney Docket Number: | GBL0007/US |
| Receipt Date: | 08-DEC-2017 |
| Filing Date: | |
| Time Stamp: | 19:01:32 |
| Application Type: | U.S. National Stage under 35 USC 371 |

Payment information:

| | |
|--|-----------------------|
| Submitted with Payment | yes |
| Payment Type | CARD |
| Payment was successfully received in RAM | \$1620 |
| RAM confirmation Number | 121117INTEFSW19015300 |
| Deposit Account | |
| Authorized User | |

The Director of the USPTO is hereby authorized to charge indicated fees and credit any overpayment as follows:

| | | | | | |
|---|--|-------------------------------|--|------------------|------------------|
| | | | | | |
| File Listing: | | | | | |
| Document Number | Document Description | File Name | File Size(Bytes)/ Message Digest | Multi Part /.zip | Pages (if appl.) |
| 1 | Transmittal of New Application | GBL0007US_371_Transmittal.pdf | 2757957 | no | 3 |
| | | | 7997e6c01eb01f494443d52e5af6ff14aea46cc4 | | |
| Warnings: | | | | | |
| Information: | | | | | |
| 2 | Application Data Sheet | GBL0007US_ADS.pdf | 4711371 | no | 8 |
| | | | a71b5dee05f78c5930ebe218717a81f9aab1238c | | |
| Warnings: | | | | | |
| Information: | | | | | |
| This is not an USPTO supplied ADS fillable form | | | | | |
| 3 | Information Disclosure Statement (IDS) Form (SB08) | GBL0007US_IDS.pdf | 1212053 | no | 4 |
| | | | 425baf35e33440977faf12283507d2f1bee0d65 | | |
| Warnings: | | | | | |
| Information: | | | | | |
| This is not an USPTO supplied IDS fillable form | | | | | |
| 4 | Documents submitted with 371 Applications | GBL0007US_PCT_Publication.pdf | 1871945 | no | 44 |
| | | | 161e1fe6ab54042e3e24a799d4de1d0a60af17f9 | | |
| Warnings: | | | | | |
| Information: | | | | | |
| 5 | Power of Attorney | GBL0007US_POA.pdf | 1103599 | no | 2 |
| | | | cad992be926ca3b3f2722ee99b5b6cb003a6a943 | | |
| Warnings: | | | | | |
| Information: | | | | | |
| 6 | Foreign Reference | EP0834352A1.pdf | 1526775 | no | 22 |
| | | | b97ca728d51a3a6c53022fda81cfd0f67da6c6d9 | | |
| Ex 1028, Covered, p 74 | | | | | |

| | | | | | |
|----------------------------|-----------------------|-------------------------------|--|----|----|
| Warnings: | | | | | |
| Information: | | | | | |
| 7 | Foreign Reference | GBL0002US_PCT_Publication.pdf | 1278890 | no | 33 |
| | | | 22a5c3738381a2ef0e835e442b170c7ad5bafa53 | | |
| Warnings: | | | | | |
| Information: | | | | | |
| 8 | Foreign Reference | GBL0005US_PCT_Publication.pdf | 3484345 | no | 92 |
| | | | 69eba55ebd18d46a8fba59b48e329a6a039698ea | | |
| Warnings: | | | | | |
| Information: | | | | | |
| 9 | Foreign Reference | GBL0006US_PCT_Publication.pdf | 1826295 | no | 35 |
| | | | d9ba60957218b228167b15711b4d878414ea725b | | |
| Warnings: | | | | | |
| Information: | | | | | |
| 10 | Non Patent Literature | NPL_ISRWO.pdf | 249320 | no | 9 |
| | | | 900d3143e1f29562af8f384181cce8ff7047afbe | | |
| Warnings: | | | | | |
| Information: | | | | | |
| 11 | Foreign Reference | WO1998058117.pdf | 984480 | no | 33 |
| | | | 528f1689796e2cb1923f459e39436fdb5775cd98 | | |
| Warnings: | | | | | |
| Information: | | | | | |
| 12 | Foreign Reference | WO2003097245.pdf | 1001835 | no | 29 |
| | | | fef5e661f02a1201f650b30901128ee137f30d96 | | |
| Warnings: | | | | | |
| Information: | | | | | |
| 13 | Foreign Reference | WO2003101621.pdf | 808052 | no | 19 |
| | | | 2e5a81f330b397c713ca98edae718fa313075222 | | |
| Warnings: | | | | | |
| Information: | | | | | |
| Ex. 1028 - Favored - p. 75 | | | | | |

| | | | | | |
|---|----------------------|------------------|--|----|----|
| 14 | Foreign Reference | WO2007083122.pdf | 892913 4f215476b9dd0e30101f2024b1aeae47e2895573 | no | 24 |
| Warnings: | | | | | |
| Information: | | | | | |
| 15 | Foreign Reference | WO2014155099.pdf | 1122008 60ce32c3ec3352fa4aea72aaba866551eea9fd0 | no | 28 |
| Warnings: | | | | | |
| Information: | | | | | |
| 16 | Fee Worksheet (SB06) | fee-info.pdf | 37014 7df29816925b054d8b5559a10f8c728ecce5bf78 | no | 2 |
| Warnings: | | | | | |
| Information: | | | | | |
| Total Files Size (in bytes): | | | 24868852 | | |
| <p>This Acknowledgement Receipt evidences receipt on the noted date by the USPTO of the indicated documents, characterized by the applicant, and including page counts, where applicable. It serves as evidence of receipt similar to a Post Card, as described in MPEP 503.</p> <p><u>New Applications Under 35 U.S.C. 111</u> If a new application is being filed and the application includes the necessary components for a filing date (see 37 CFR 1.53(b)-(d) and MPEP 506), a Filing Receipt (37 CFR 1.54) will be issued in due course and the date shown on this Acknowledgement Receipt will establish the filing date of the application.</p> <p><u>National Stage of an International Application under 35 U.S.C. 371</u> If a timely submission to enter the national stage of an international application is compliant with the conditions of 35 U.S.C. 371 and other applicable requirements a Form PCT/DO/EO/903 indicating acceptance of the application as a national stage submission under 35 U.S.C. 371 will be issued in addition to the Filing Receipt, in due course.</p> <p><u>New International Application Filed with the USPTO as a Receiving Office</u> If a new international application is being filed and the international application includes the necessary components for an international filing date (see PCT Article 11 and MPEP 1810), a Notification of the International Application Number and of the International Filing Date (Form PCT/RO/105) will be issued in due course, subject to prescriptions concerning national security, and the date shown on this Acknowledgement Receipt will establish the international filing date of the application.</p> | | | | | |

PCT REQUEST

Print Out (Original in Electronic Form)

| | | |
|--------------|--|--|
| 0 | For receiving Office use only | |
| 0-1 | International Application No. | PCT/GB2016/051688 |
| 0-2 | International Filing Date | 08 June 2016 (08.06.2016) |
| 0-3 | Name of receiving Office and "PCT International Application" | RO/GB |
| 0-4 | Form PCT/RO/101 PCT Request | |
| 0-4-1 | Prepared Using | PCT Online Filing Version 3.5.000.244e MT/FOP 20141031/0.20.5.20 |
| 0-5 | Petition The undersigned requests that the present international application be processed according to the Patent Cooperation Treaty | |
| 0-6 | Receiving Office (specified by the applicant) | UK Intellectual Property Office (RO/GB) |
| 0-7 | Applicant's or agent's file reference | P2494PC00 |
| I | Title of Invention | COATINGS |
| II | Applicant | |
| II-1 | This person is | Applicant only |
| II-2 | Applicant for | All designated States |
| II-4 | Name | P2i Ltd |
| II-5 | Address | 9-12 North Central 127 Olympic Avenue Milton Park Abingdon Oxfordshire OX14 4SA United Kingdom |
| II-6 | State of nationality | GB |
| II-7 | State of residence | GB |
| III-1 | Applicant and/or inventor | |
| III-1-1 | This person is | Inventor only |
| III-1-3 | Inventor for | All designated States |
| III-1-4 | Name (LAST, First) | COULSON, Stephen Richard |
| III-1-5 | Address | c/o P2i Ltd 9-12 North Central, 127 Olympic Avenue, Milton Park Abingdon Oxfordshire OX14 4SA United Kingdom |

PCT REQUEST

Print Out (Original in Electronic Form)

| | | |
|--------------|----------------------------------|--|
| III-2 | Applicant and/or inventor | |
| III-2-1 | This person is | Inventor only |
| III-2-3 | Inventor for | All designated States |
| III-2-4 | Name (LAST, First) | EVANS, Delwyn |
| III-2-5 | Address | c/o P2i Ltd 9-12 North Central, 127 Olympic Avenue, Milton Park Abingdon Oxfordshire OX14 4SA United Kingdom |
| III-3 | Applicant and/or inventor | |
| III-3-1 | This person is | Inventor only |
| III-3-3 | Inventor for | All designated States |
| III-3-4 | Name (LAST, First) | SIOKOU, Angeliki |
| III-3-5 | Address | c/o P2i Ltd 9-12 North Central, 127 Olympic Avenue, Milton Park Abingdon Oxfordshire OX14 4SA United Kingdom |
| III-4 | Applicant and/or inventor | |
| III-4-1 | This person is | Inventor only |
| III-4-3 | Inventor for | All designated States |
| III-4-4 | Name (LAST, First) | TELFORD, Clive |
| III-4-5 | Address | c/o P2i Ltd 9-12 North Central, 127 Olympic Avenue, Milton Park Abingdon Oxfordshire OX14 4SA United Kingdom |

PCT REQUEST

Print Out (Original in Electronic Form)

| | | |
|--------------|--|---|
| IV-1 | Agent or common representative; or address for correspondence The person identified below is hereby/ has been appointed to act on behalf of the applicant(s) before the competent International Authorities as: | Agent |
| IV-1-1 | Name (LAST, First) | BLAKE, Michael |
| IV-1-2 | Address | Greaves Brewster LLP Copa House Station Road Cheddar Somerset BS27 3AH United Kingdom |
| IV-1-3 | Telephone No. | + 44 (0)1934 745880 |
| IV-1-4 | Facsimile No. | + 44 (0)1934 745881 |
| V | DESIGNATIONS | |
| V-1 | The filing of this request constitutes under Rule 4.9(a), the designation of all Contracting States bound by the PCT on the international filing date, for the grant of every kind of protection available and, where applicable, for the grant of both regional and national patents. | |
| VI-1 | Priority claim of earlier regional application | |
| VI-1-1 | Filing date | 09 June 2015 (09.06.2015) |
| VI-1-2 | Number | 15386017.6 |
| VI-1-3 | Regional Office | EP |
| VI-2 | Incorporation by reference : where an element of the international application referred to in Article 11(1)(iii)(d) or (e) or a part of the description, claims or drawings referred to in Rule 20.5(a) is not otherwise contained in this international application but is completely contained in an earlier application whose priority is claimed on the date on which one or more elements referred to in Article 11(1)(iii) were first received by the receiving Office, that element or part is, subject to confirmation under Rule 20.6, incorporated by reference in this international application for the purposes of Rule 20.6. | |
| VII-1 | International Searching Authority Chosen | European Patent Office (EPO) (ISA/EP) |

PCT REQUEST

Print Out (Original in Electronic Form)

| | | | |
|-------------|--|-----------------------------------|------------------------------------|
| VIII | Declarations | Number of declarations | |
| VIII-1 | Declaration as to the identity of the inventor | — | |
| VIII-2 | Declaration as to the applicant's entitlement, as at the international filing date, to apply for and be granted a patent | — | |
| VIII-3 | Declaration as to the applicant's entitlement, as at the international filing date, to claim the priority of the earlier application | — | |
| VIII-4 | Declaration of inventorship (only for the purposes of the designation of the United States of America) | — | |
| VIII-5 | Declaration as to non-prejudicial disclosures or exceptions to lack of novelty | — | |
| IX | Check list | Number of sheets | Electronic file(s) attached |
| IX-1 | Request (including declaration sheets) | 5 | ✓ |
| IX-2 | Description | 21 | ✓ |
| IX-3 | Claims | 14 | ✓ |
| IX-4 | Abstract | 1 | ✓ |
| IX-5 | Drawings | 6 | ✓ |
| IX-7 | TOTAL | 47 | |
| | Accompanying Items | Paper document(s) attached | Electronic file(s) attached |
| IX-8 | Fee calculation sheet | — | ✓ |
| IX-19 | Other | Pre-conversion archive | ✓ |
| IX-19 | Other | List of additional agents | ✓ |
| IX-20 | Figure of the drawings which should accompany the abstract | | |
| IX-21 | Language of filing of the international application | English | |
| X-1 | Signature of applicant, agent or common representative | (PKCS7 Digital Signature) | |
| X-1-1 | Name (LAST, First) | Blake, Michael | |
| X-1-3 | Capacity (if such capacity is not obvious from reading the request) | (Representative) | |

PCT REQUEST

Print Out (Original in Electronic Form)

FOR RECEIVING OFFICE USE ONLY

| | | |
|--------|---|---------------------------|
| 10-1 | Date of actual receipt of the purported international application | 08 June 2016 (08.06.2016) |
| 10-2 | Drawings: | |
| 10-2-1 | Received | |
| 10-2-2 | Not received | |
| 10-3 | Corrected date of actual receipt due to later but timely received papers or drawings completing the purported international application | |
| 10-4 | Date of timely receipt of the required corrections under PCT Article 11(2) | |
| 10-5 | International Searching Authority | ISA/EP |
| 10-6 | Transmittal of search copy delayed until search fee is paid | |

FOR INTERNATIONAL BUREAU USE ONLY

| | | |
|------|--|--|
| 11-1 | Date of receipt of the record copy by the International Bureau | |
|------|--|--|

Continuation of Box IV-1

Additional Agents

Our reference: P2494PC00-QR8AQ

TURNER, Rhiannon Rosalind
WALLIS, Naomi Rachel
BUMKE, Jakob Wenzel
LELAND, Emma Clare
GLANSDORP, Freija Gwendolyn
BLAKE, Michael Edward
SPINNER, David Richard
DUTTON, Erica

of Greaves Brewster LLP
Copa House
Station Road
Cheddar
Somerset
BS27 3AH
United Kingdom.

(51) International Patent Classification:
C09D 5/00 (2006.01)(21) International Application Number:
PCT/GB2016/051688(22) International Filing Date:
8 June 2016 (08.06.2016)

(25) Filing Language: English

(26) Publication Language: English

(30) Priority Data:
15386017.6 9 June 2015 (09.06.2015) EP(71) Applicant: **P2i LTD** [GB/GB]; 9-12 North Central, 127 Olympic Avenue, Milton Park, Abingdon Oxfordshire OX14 4SA (GB).(72) Inventors: **COULSON, Stephen Richard**; c/o P2i Ltd, 9-12 North Central, 127 Olympic Avenue, Milton Park, Abingdon Oxfordshire OX14 4SA (GB). **EVANS, Delwyn**; c/o P2i Ltd, 9-12 North Central, 127 Olympic Avenue, Milton Park, Abingdon Oxfordshire OX14 4SA (GB). **SIKOU, Angeliki**; c/o P2i Ltd, 9-12 North Central, 127 Olympic Avenue, Milton Park, Abingdon Oxfordshire OX14 4SA (GB). **TELFORD, Clive**; c/o P2i Ltd, 9-12 North Central, 127 Olympic Avenue, Milton Park, Abingdon Oxfordshire OX14 4SA (GB).(74) Agent: **BLAKE, Michael**; Greaves Brewster LLP, Copahouse, Station Road, Cheddar Somerset BS27 3AH (GB).

(81) Designated States (unless otherwise indicated, for every kind of national protection available): AE, AG, AL, AM, AO, AT, AU, AZ, BA, BB, BG, BH, BN, BR, BW, BY, BZ, CA, CH, CL, CN, CO, CR, CU, CZ, DE, DK, DM, DO, DZ, EC, EE, EG, ES, FI, GB, GD, GE, GH, GM, GT, HN, HR, HU, ID, IL, IN, IR, IS, JP, KE, KG, KN, KP, KR, KZ, LA, LC, LK, LR, LS, LU, LY, MA, MD, ME, MG, MK, MN, MW, MX, MY, MZ, NA, NG, NI, NO, NZ, OM, PA, PE, PG, PH, PL, PT, QA, RO, RS, RU, RW, SA, SC, SD, SE, SG, SK, SL, SM, ST, SV, SY, TH, TJ, TM, TN, TR, TT, TZ, UA, UG, US, UZ, VC, VN, ZA, ZM, ZW.

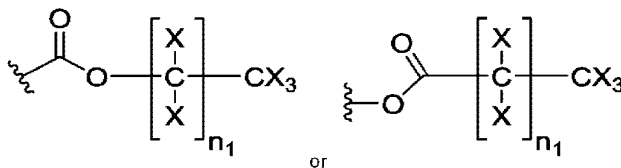
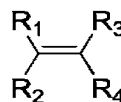
(84) Designated States (unless otherwise indicated, for every kind of regional protection available): ARIPO (BW, GH, GM, KE, LR, LS, MW, MZ, NA, RW, SD, SL, ST, SZ, TZ, UG, ZM, ZW), Eurasian (AM, AZ, BY, KG, KZ, RU, TJ, TM), European (AL, AT, BE, BG, CH, CY, CZ, DE, DK, EE, ES, FI, FR, GB, GR, HR, HU, IE, IS, IT, LT, LU, LV, MC, MK, MT, NL, NO, PL, PT, RO, RS, SE, SI, SK, SM, TR), OAPI (BF, BJ, CF, CG, CI, CM, GA, GN, GQ, GW, KM, ML, MR, NE, SN, TD, TG).

Published:

— with international search report (Art. 21(3))

[Continued on next page]

(54) Title: COATINGS



(57) Abstract: The present invention provides an electronic or electrical device or component thereof comprising a cross-linked polymeric coating on a surface of the electronic or electrical device or component thereof; wherein the cross-linked polymeric coating is obtainable by exposing the electronic or electrical device or component thereof to a plasma comprising a monomer compound and a crosslinking reagent for a period of time sufficient to allow formation of the cross-linked polymeric coating on a surface thereof, wherein the monomer compound has the following formula: where R_1 , R_2 and R_4 are each independently selected from hydrogen, optionally substituted branched or straight chain C_1 - C_6 alkyl or halo alkyl or aryl optionally substituted by halo, and R_3 is selected from: or where each X is independently selected from hydrogen, a halogen, optionally substituted branched or straight chain C_1 - C_6 alkyl, halo alkyl or aryl optionally substituted by halo; and n_1 is an integer from 2 to 27; and wherein the crosslinking reagent comprises two or more unsaturated bonds attached by means of one or more linker moieties and has a boiling point at standard pressure of less than 500°C.



-
- *before the expiration of the time limit for amending the claims and to be republished in the event of receipt of amendments (Rule 48.2(h))*

Coatings

Field of the invention

This invention relates to protective coatings. Specifically, the present invention relates to protective coatings for electronic or electrical devices and components thereof, and methods of forming such coatings. The coatings can protect by being hydrophobic and so resist the ingress of water-based liquid into electronic devices, or they can protect by forming a barrier coating and so provide electrical resistance between the electrical parts of the phone and water based liquid.

Background of the invention

Monounsaturated monomers are used to make barrier coatings using plasma polymerisation processes (see co-pending application).

The perfluoroalkyl chain monomers are also used for generating hydrophobic surfaces from pulsed plasma deposition processes (see WO 9858117 A1).

The power of the plasma initiated polymerisation affects the nature of the polymer produced. The higher average energy inputs of continuous wave plasmas lead to more fragmentation of the monomer, and so the polymer loses structural properties of the monomer. In the case of 1H,1H,2H,2H- perfluorodecyl acrylate (PFAC8), there's less retention of the perfluoroalkyl chain and the contact angle of the surface coating is compromised. Higher plasma energies also lead to more crosslinking. For the lower average energy inputs of pulsed plasmas, there's better retention of monomer structure and less crosslinking. The greater retention of the perfluoro chain under low energy, pulsed plasma conditions leads to best levels of contact angles for the surface coating.

When the perfluoroalkyl chains have eight or more fluorinated carbons (long chain), the polymer made from the monomer has a crystalline structure. When the perfluoroalkyl chains have less than eight fluorinated carbons, the resulting polymer is amorphous and so can be unstable in the presence of water (see "Molecular Aggregation Structure and Surface Properties of Poly(fluoroalkyl acrylate) Thin Films, *Macromolecules*, 2005, vol 38, p5699-5705)

When long chain perfluoroalkyl polymers are produced by high average power (continuous wave or CW) or low average power (pulsed wave or PW) plasmas, then because of the crystalline structure of the long chains, the polymers are non-stick to the touch and stable in the presence of water.

However, the feel and water stability of shorter chain polymer coatings is affected by the plasma power levels used. For example, when PFAC6 (1H,1H,2H,2H-perfluorooctyl acrylate) is polymerised in low power plasma conditions, the resulting polymer coating can have several disadvantages. For example the coating can cause water drops to spread out a little (slump), be marked by the presence of a water drop on its surface, have a tacky feel, or can be easily smeared (for example on substrates of silicon wafer and ABS plastic).

By increasing the power of the plasma used for polymerisation, the polymer becomes more crosslinked and becomes more resistant to smearing. However, increasing the power has the concomitant effect of decreasing the water contact angle through more monomer fragmentation (as described above). Figure 1 shows the effect of increasing the power to monomer flow ratio for CW plasmas in a 125 litre chamber: at a ratio of 4 W/ μ l/min, the water contact angle is ~85-95 degrees and the coating is tack-free. However, as the ratio drops, the contact angle increases and the occurrence of tacky/smudgy coatings increases too. Figure 2 shows the same effect for pulsed plasma conditions. These results show that the process window for producing tack and smudge-free coatings have a limited plasma processing range and the final coating has a compromised water contact angle.

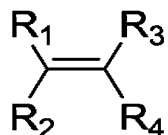
Accordingly, it is an aim to solve one or more of the above-mentioned problems with the prior art coatings.

Statement of Invention

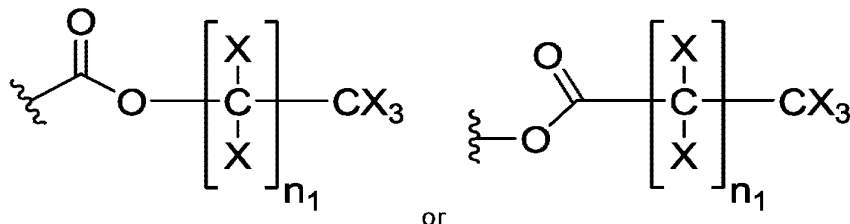
An aspect of the present invention provides an electronic or electrical device or component thereof comprising a protective cross-linked polymeric coating on a surface of the electronic or electrical device or component thereof;

wherein the protective cross-linked polymeric coating is obtainable by exposing the electronic or electrical device or component thereof to a plasma comprising a monomer compound and a crosslinking reagent for a period of time sufficient to allow formation of the protective cross-linked polymeric coating on a surface thereof,

wherein the monomer compound has the following formula:



where R_1 , R_2 and R_4 are each independently selected from hydrogen, optionally substituted branched or straight chain C_1 - C_6 alkyl or halo alkyl or aryl optionally substituted by halo, and R_3 is selected from:



where each X is independently selected from hydrogen, a halogen, optionally substituted branched or straight chain C₁-C₆ alkyl, halo alkyl or aryl optionally substituted by halo; and n₁ is an integer from 1 to 27; and wherein the crosslinking reagent comprises two or more unsaturated bonds attached by means of one or more linker moieties and has a boiling point of less than 500 °C at standard pressure.

High levels of polymer crosslinking (formerly only achievable with high average power continuous wave plasmas) can be achieved by adding a crosslinking molecule to the monomer to produce a cross-linked co-polymer. This has the advantage of increasing the plasma processing range so stable coatings can now be produced in low average energy pulse plasma conditions.

The high degree of retention of the hydrophobic monomer structures from the low energy pulsed plasma gives coatings of the co-polymer a good hydrophobic coating (as evidenced by water contact angles) and a coating that is not sticky or smeary to the touch. Thus, these properties make it suitable for the coating of electronic devices to prevent the ingress of water-based liquids from accidental damage. Such coatings typically have a thickness between 1 and 100 nm, preferably 1 and 50 nm.

The coating may be described as a liquid repellent layer, typically a water repellent layer. The coating may repel water, an aqueous liquid such as rainwater, or oil.

Alternatively, or in addition to providing a liquid repellent layer, the coating may be a physical barrier layer.

The coating may be a physical barrier i.e. it provides a physical barrier to mass and electron transport.

The physical barrier layer restricts diffusion of water, oxygen and ions. When the coating is a physical barrier it typically has a thickness of greater than 50 nm.

The coating is a protective layer i.e. it prevents damage by contact with water or other liquids. The coating may provide protective functionality by forming a liquid repellent layer and/or a physical barrier layer.

The coating is preferably substantially pin-hole free to enable it to provide a physical barrier. Preferably $\Delta Z/d < 0.15$, where ΔZ is the average height variation on an AFM line scan in nm (as shown in Figure 3) and d is coating thickness in nm.

The value of $\Delta Z/d$ tells us to what extent defects/voids on the surface of the coating extend into the coating, i.e. the percentage value of the depth of defect over total coating thickness. For example, $\Delta Z/d = 0.15$ means that the voids on the surface only extend down to 15% of the coating thickness. A coating with a $\Delta Z/d < 0.15$ is defined herein as being substantially pinhole free. If voids are bigger than this, the desired functionality is unlikely to be achieved.

The coating is preferably conformal, which means that it takes the 3D shape of the electronic or electrical device or component thereof and covers substantially an entire surface of the device. This has the advantage of ensuring that the coating has sufficient thickness to give optimal functionality over an entire surface of the device or component. The meaning of the term “covers substantially an entire surface” will depend to some extent on the type of surface to be covered. For example, for some components, it may be necessary for there to be complete coverage of the surface in order for the component to function after submersion in water. However, for other components or housings, small gaps in coverage may be tolerated.

The coating may have a thickness of 50 to 10,000nm, optionally 50 to 8000nm, 100 to 5000nm, preferably 250nm – 5000nm, most preferably 250nm – 2000nm.

The coating may be electrically insulating and be sufficiently compliant that electrical connectors can be joined to the electronic or electrical device or component thereof and an electrical connection made between the electrical connectors and electronic or electrical device or component thereof without the requirement to first remove the coating. In this case, the force exerted by the electrical connector on the coating is sufficient to alter the structure of or even break through the coating local to the electrical connector, allowing the electrical connection to be made. Electrical connectors can typically be joined to the electronic or electrical device or component in this way for coating thicknesses of under 5000nm, and for high performance coatings below 2000nm.

The electronic or electrical device or component thereof can typically withstand immersion in up to 1m of water for over 30 minutes without failure or corrosion whilst power applied to the electronic or electrical device or component. The effectiveness of the coating can be determined by measuring its electrical resistance at a fixed voltage when submerged in water for a set time period; for example, applying a voltage of 8V to the coating of a device which is submerged in water for 13 minutes. Coatings having a resistance value of 1×10^7 Ohms or higher in this test are effective barrier coatings and the coated electronic or electrical device or component thereof will pass successfully an IPX7 test. The IPX7 test is the Ingress Protection Marking which classifies and rates the degree of protection provided against water. In the IPX7 test for phones, the device is immersed in water under defined conditions of pressure and time (up to 1 m of submersion) for a duration of 30 minutes. The device has to be powered on during testing and functional after 24 hrs.

In one embodiment, the coating is electrically insulating and has a thickness of less than 1 micron and wherein a force of 5- 20g applied to the coating using a round probe with 1 mm diameter allows an electrical connection to be made with the electronic or electrical device or component thereof in the local area where the force has been applied.

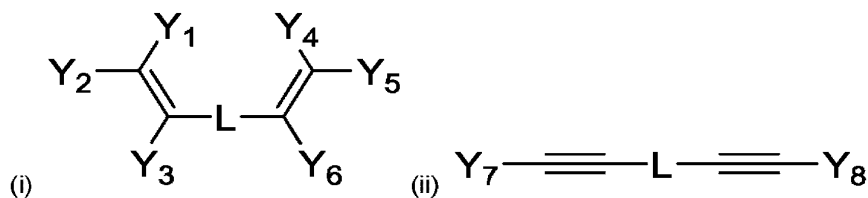
In another embodiment, the coating is electrically insulating and has a thickness of 1-2.5 microns and wherein a force of 20-100g applied to the coating using a round probe with 1 mm diameter allows an electrical connection to be made in the local area of the coating where the force has been applied.

The coating may have a higher density than that of the corresponding monomer from which it is formed. For example, the increase in density may be at least 0.1 g/cm^3 . The increase in density is explained by the highly crosslinked coating. The high density of the coating improves the barrier properties of the coating.

The coating may form a surface defined by a static water contact angle (WCA) of at least 70° . A coating with a WCA of at least 90° is a liquid repellent, typically a water repellent layer. For fluorinated polymers, the coating may have a static water contact angle of at least 100° . The contact angle of a liquid on a solid substrate gives an indication of the surface energy which in turn illustrates the substrate's liquid repellence. Contact angles were measured on a VCA Optima contact angle analyser, using $3 \mu\text{l}$ droplets of deionised water at room temperature.

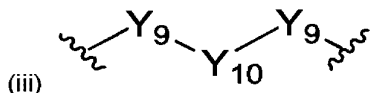
Crosslinker

A basic requirement for the cross-linking reagent is the presence two or more unsaturated bonds, for example, -C=C- or alkyne groups. The unsaturated bonds are attached by one or more linker moieties. The linker moiety is not especially limited provided it joins together the two or more unsaturated bonds. The cross-linking reagent must have a boiling point of less than 500°C , preferably from -10 to 300°C , optionally 180 to 270°C , most preferably 205 to 260°C at standard pressure. It is a preference, but not a requirement, that the cross-linking reagent is not unduly hazardous for use in plasma processing, that is to say it can be used in a production environment where low levels of vapour do not present any significant health and safety issues (e.g. strongly oxidising, explosive, toxic, or has an offensive odour (e.g. stench reagent)). The crosslinking reagent preferably has one of the following structures:



where Y_1 , Y_2 , Y_3 , Y_4 , Y_5 , Y_6 , Y_7 and Y_8 are each independently selected from hydrogen, optionally substituted cyclic, branched or straight chain $\text{C}_1\text{-C}_6$ alkyl or aryl; and L is a linker moiety.

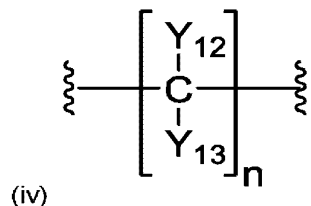
Most preferably L has the following formula:



where each Y_9 is independently selected from, a bond, -O- , -O-C(O)- , -C(O)-O- , $\text{-Y}_{11}\text{-O-C(O)-}$, $\text{-C(O)-O-Y}_{11}\text{-}$, $\text{-OY}_{11}\text{-}$, and $\text{-Y}_{11}\text{O-}$, where Y_{11} is an optionally substituted cyclic, branched or straight chain $\text{C}_1\text{-C}_8$ alkylene; and

Y₁₀ is selected from optionally substituted cyclic, branched or straight chain C₁-C₈ alkylene and a siloxane containing group.

In one most preferred embodiment Y₁₀ has the following formula:



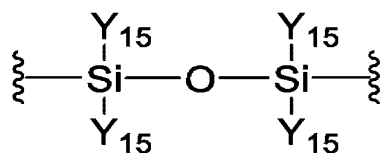
wherein each Y₁₂ and Y₁₃ is independently selected from H, halo, optionally substituted cyclic, branched or straight chain C₁-C₈ alkyl, or -OY₁₄, where Y₁₄ is selected from optionally substituted branched or straight chain C₁-C₈ alkyl or alkenyl, and n is an integer from 1 to 10.

Optionally, each Y₁₂ is H and each Y₁₃ is H, i.e. Y₁₀ is a linear alkylene chain. For this embodiment Y₉ is preferably a vinyl ester or vinyl ether group.

Optionally, each Y₁₂ is fluoro and each Y₁₃ is fluoro, i.e. Y₁₀ is a linear perfluoroalkylene chain.

Typically, n is from 4 to 6.

In another embodiment Y₁₀ has the following formula:

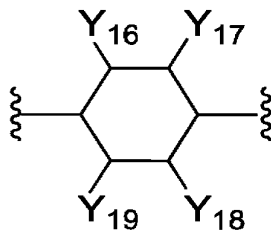


(v)

wherein each Y₁₅ is independently selected from optionally substituted branched or straight chain C₁-C₆ alkyl.

In one embodiment each Y₁₅ is methyl, and each Y₉ is a bond.

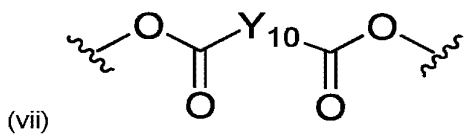
In a further embodiment Y₁₀ has the following formula:



(vi)

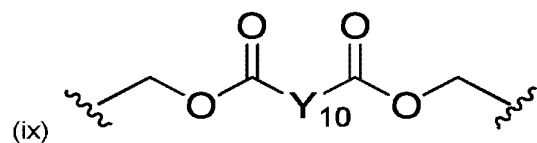
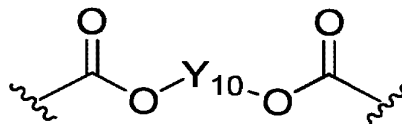
wherein Y_{16} to Y_{19} are each independently selected from H and optionally substituted branched or straight chain C_1 - C_8 alkyl or alkenyl. A preferred alkenyl group is vinyl. Optionally Y_{18} is H or vinyl, and Y_{16} , Y_{17} and Y_{19} are each H. In one embodiment each of Y_{16} to Y_{19} is H. In another embodiment Y_{18} is vinyl, and Y_{16} , Y_{17} and Y_{19} are each H.

In preferable embodiments for compound (i), L has one of the following structures:

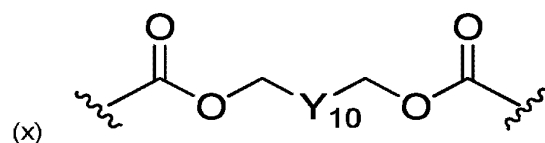


(vii)

(viii)

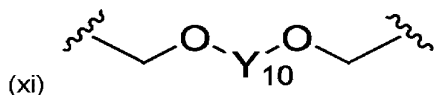


(ix)

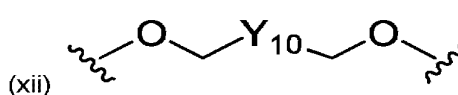


(x)

In another embodiment for compound (i) L has one of the following structures:



(xi)



(xii)

For L according structure (vii), Y_{10} is preferably an alkylene chain or a cycloalkylene, such as those shown in structures iv) and vi) above. The alkylene chain may be a straight chain alkylene chain. When Y_{10} is a cycloalkylene, cyclohexylene is preferred, most preferably 1,4 cyclohexylene.

For L according to structure (viii), Y₁₀ is preferably structure (iv), i.e. an alkylene or fluoroalkylene chain.

For L according to structure (ix), Y₁₀ is preferably cycloalkylene, such as the cyclohexylene according to structure (vi).

For L according to structure (x), Y₁₀ is preferably structure (iv) wherein each Y₁₂ and Y₁₃ is F, i.e. a perfluoroalkylene chain.

For L according to structure (xi) or structure (xii) Y₁₀ is preferably alkylene or cycloalkylene. Optionally the alkylene or cycloalkylene may be substituted with one or more vinyl groups or alkenyl ether groups, preferably one or more vinyl ether groups.

When each Y₉ is a bond, each Y₁₀ may be any of structures (iv), (v) and (vi). Preferably Y₁₀ is a straight chain alkylene such that the crosslinking reagent is a diene, such as a heptadiene, octadiene, or nonadiene, most preferably 1,7-octadiene.

When each Y₉ is O, each Y₁₀ is preferably a branched or straight chain C₁-C₆ alkylene, preferably a straight chain alkylene, most preferably a C₄ straight chain alkylene, i.e the crosslinking reagent is 1,4-butanediol divinyl ether

It will be understood that each Y₉ group can be combined with any other Y₉ group and Y₁₀ group to form the crosslinking reagent.

The skilled person will be aware of possible substituents for each of the cyclic, branched or straight chain C₁-C₈ alkylene groups mentioned above. The alkylene groups may be substituted at one or more positions by a suitable chemical group. Halo substituents are preferred, with fluoro substituents most preferred. Each C₁-C₈ alkylene group may be a C₁-C₃, C₂-C₆, or C₆-C₈ alkylene group.

Particularly preferred embodiments of the crosslinker have alkyl chains for Y₁₀ and vinyl ester or vinyl ether groups on either side.

A particularly preferred cross-linking reagent is divinyl adipate. (a divinyl ester)

Another preferred cross-linking reagent is 1,4-butanediol divinyl ether (a divinyl ether)

For the most preferred embodiments the crosslinking reagent is selected from Divinyl Adipate (DVA), 1,4 Butanediol divinyl ether (BDVE), 1,4 Cyclohexanedimethanol divinyl ether (CDDE), 1,7 -Octadiene (17OD), 1,2,4 -Trivinylcyclohexane (TVCH), 1,3-Divinyltetramethyldisiloxane (DVTMDS), Diallyl 1,4-cyclohexanedicarboxylate (DCHD), 1,6-Divinylperfluorohexane (DVPFH), 1H,1H,6H,6H-Perfluorohexanediol diacrylate (PFHDA) and glyoxal bis (diallyl acetal) (GBDA).

For the alkyne crosslinking reagents according to compound (ii), L is preferably selected from a branched or straight chain C₁-C₈ alkylene or an ether group. L may be a C₃, C₄, C₅, or C₆ alkylene,

preferably a straight chain alkylene. Chemical structures for the particularly preferred crosslinking reagents are set out below in Table 1:

| | |
|---|--|
| Divinyl Adipate (DVA) | |
| 1,4 Butanediol divinyl ether (BDVE) | |
| 1,4 Cyclohexanedimethanol divinyl ether (CDDE) | |
| 1,7 -Octadiene (17OD) | |
| 1,2,4 -Trivinylcyclohexane (TVCH) | |
| 1,3- Divinyltetramethyldisiloxane (DVTMDS) | |
| Diallyl 1,4- cyclohexanedicarboxylate (DCHD) | |
| 1,6-Divinylperfluorohexane (DVPFH) | |
| 1H,1H,6H,6H- Perfluorohexanediol diacrylate (PFHDA) | |
| GBDA | |

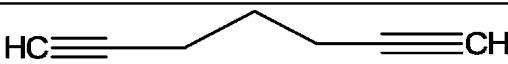
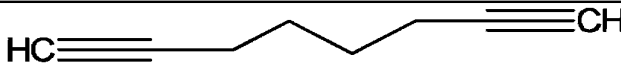
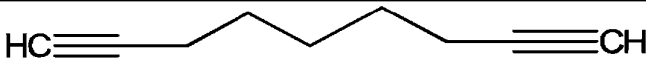
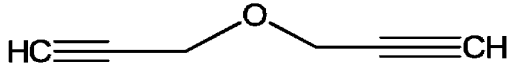
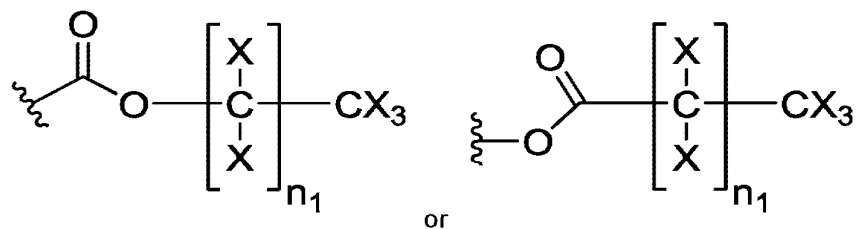
| | |
|-----------------|--|
| 1,6-heptadiyne |  |
| 1,7-heptadiyne |  |
| 1,8-heptadiyne |  |
| Propargyl ether |  |

Table 1*Monomer Compound*

The monomer compound has the following formula:

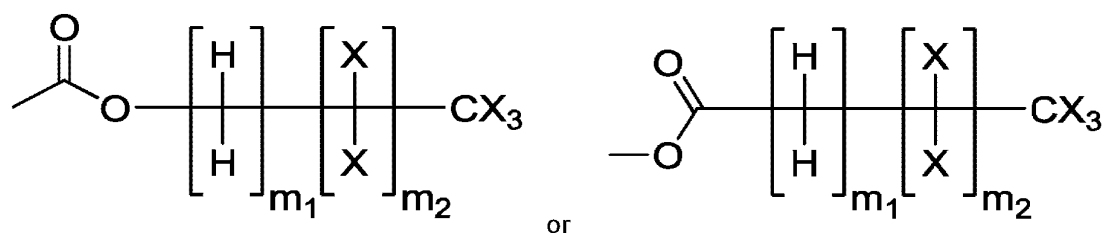


where R_1 , R_2 and R_4 are each independently selected from hydrogen, optionally substituted branched or straight chain C_1 - C_6 alkyl or halo alkyl or aryl optionally substituted by halo, and R_3 is selected from:



where each X is independently selected from hydrogen, a halogen, optionally substituted branched or straight chain C_1 - C_6 alkyl, halo alkyl or aryl optionally substituted by halo; and n_1 is an integer from 1 to 27. Preferably, n_1 is from 1 to 12. Optionally n_1 is from 4 to 12, optionally 6 to 8.

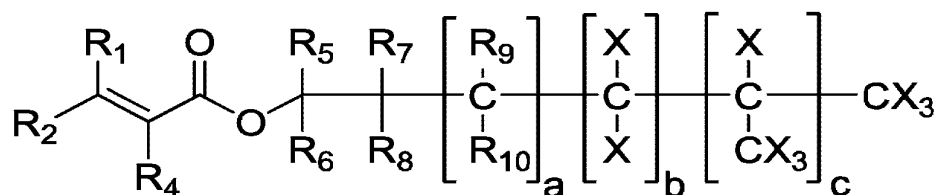
In a preferred embodiment R_3 is selected from:



where m_1 is an integer from 0 to 13 and each X is independently selected from hydrogen, a halogen, optionally substituted branched or straight chain C_1 - C_8 alkyl, halo alkyl or aryl optionally substituted by halo; and m_2 is an integer from 2 to 14;

In a particularly preferred embodiment the monomer is a compound of formula I(a):

I(a)

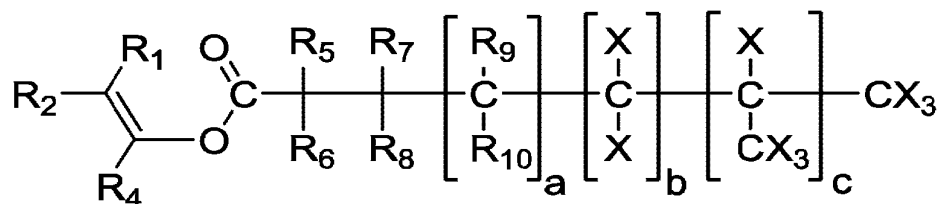


wherein each of R_1 , R_2 , R_4 , and R_5 to R_{10} is independently selected from hydrogen or an optionally substituted C_1 - C_8 branched or straight chain alkyl group; each X is independently selected from hydrogen or halogen; a is from 0-10; b is from 2 to 14; and c is 0 or 1;

; or

the monomer is a compound of formula I(b):

I(b)



wherein each of R_1 , R_2 , R_4 , and R_5 to R_{10} is independently selected from hydrogen or an optionally substituted C_1 - C_8 branched or straight chain alkyl group; each X is independently selected from hydrogen or halogen; a is from 0-10; b is from 2 to 14; and c is 0 or 1.

The halogen may be chlorine or bromine, but is preferably fluorine for compliance with RoHS regulations (Restriction of Hazardous Substances).

a is from 0 to 10, preferably from 0 to 6, optionally 2 to 4, most preferably 0 or 1. b is from 2 to 14, optionally from 2 to 10, preferably 3 to 7.

Each of R₁, R₂, R₄, and R₅ to R₁₀ is independently selected from hydrogen or a C₁-C₆ branched or straight chain alkyl group. The alkyl group may be substituted or unsubstituted, saturated or unsaturated. When the alkyl group is substituted, the location or type of the substituent is not especially limited provided the resultant polymer provides an appropriate liquid repellent and/or barrier layer. The skilled person would be aware of suitable substituents. If the alkyl group is substituted, a preferred substituent is halo, i.e. any of R₁, R₂, R₄, and R₅ to R₁₀ may be haloalkyl, preferably fluoroalkyl. Other possible substituents may be hydroxyl or amine groups. If the alkyl group is unsaturated it may comprise one or more alkene or alkyne groups.

Each of R₁, R₂, R₄, and R₅ to R₁₀ may be independently selected from hydrogen, methyl, ethyl, n-propyl, isopropyl, n-butyl, isobutyl, tert-butyl, sec-butyl, n-pentyl, neopentyl, n-hexyl, isohexyl, and 3-methylpentyl.

In a preferred embodiment each of R₁, R₂, R₄, and R₅ to R₁₀ is independently selected from hydrogen or methyl.

In a preferred embodiment, a and c are each independently 0 or 1; and b is from 3 to 7.

In one preferred embodiment each X is H. In an alternative preferred embodiment each X is F.

Optionally R₁ and R₂ are both hydrogen.

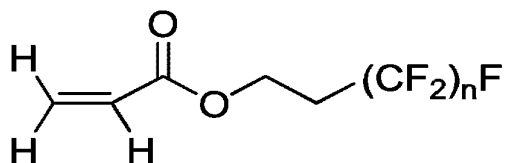
Optionally R₄ is hydrogen or methyl. Preferably R₁ and R₂ are both hydrogen and R₄ is hydrogen or methyl.

Optionally R₉ is hydrogen and R₁₀ is branched or straight chain C₁-C₆ alkyl group. In a preferred embodiment R₁₀ is methyl.

In one embodiment each of R₅ to R₈ is hydrogen.

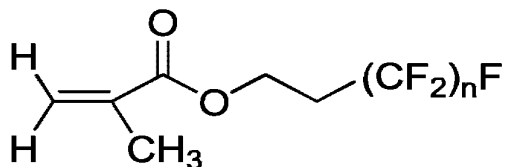
In one embodiment each of R₁, R₂, R₄, and R₅ to R₁₀ is hydrogen, each X is H, a=0 and c=0.

In a particularly preferred embodiment the monomer compound has the following formula:



where n is from 2 to 10.

In another preferred embodiment the monomer compound has the following formula:

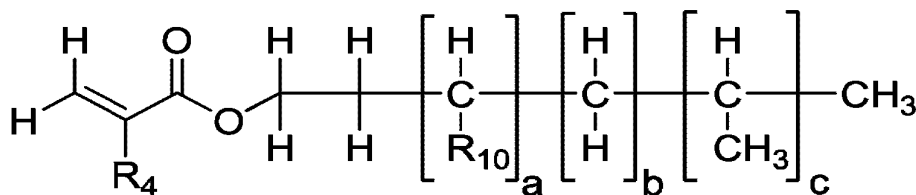


where n is from 2 to 10.

The monomer compound may be selected from 1H,1H,2H,2H-perfluorohexyl acrylate (PFAC4), 1H,1H,2H,2H-perfluorooctyl acrylate (PFAC6), 1H,1H,2H,2H-perfluorodecyl acrylate (PFAC8) and 1H,1H,2H,2H-perfluorododecyl acrylate (PFAC10).

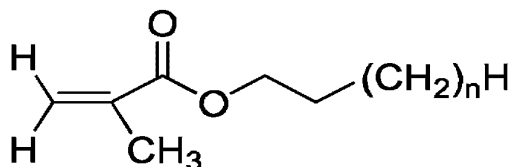
The monomer compound may be selected from 1H,1H,2H,2H-perfluorohexyl methacrylate (PFMAC4), 1H,1H,2H,2H-perfluorooctyl methacrylate (PFMAC6) and 1H,1H,2H,2H-perfluorodecyl methacrylate (PFMAC8).

The monomer compound of formula I(a) may have the following formula:



wherein a and c are each independently 0 or 1, b = 3-7 and n is 4 to 10, where n = a + b + c + 1.

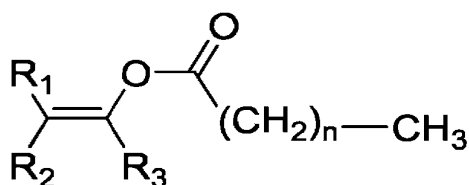
The monomer compound of formula I(a) may have the following formula:



where n is 2 to 12.

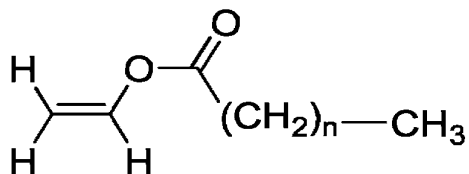
The monomer compound may be selected from ethyl hexyl acrylate, hexyl acrylate, decyl acrylate, lauryl dodecyl acrylate and iso decyl acrylate.

The monomer may have the following formula:



where n is from 4 to 14.

The monomer may have the following formula:

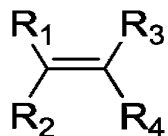


where n is from 4 to 14.

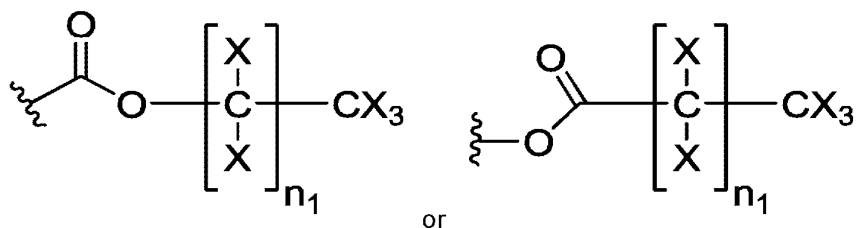
In an additional aspect, the present invention provides a method for treating an electronic device or component thereof comprising:

exposing the electronic or electrical device or component thereof to a plasma comprising a monomer compound and a crosslinking reagent for a period of time sufficient to allow a protective cross-linked polymeric coating to form on a surface of the electronic or electrical device or component thereof;

wherein the monomer compound has the following formula:



where R_1 , R_2 and R_4 are each independently selected from hydrogen, optionally substituted branched or straight chain C_1 - C_8 alkyl or halo alkyl or aryl optionally substituted by halo, and R_3 is selected from:



where each X is independently selected from hydrogen, optionally substituted branched or straight chain C_1 - C_8 alkyl, halo alkyl or aryl optionally substituted by halo; and n_1 is an integer from 1 to 27; and wherein the crosslinking reagent comprises two or more unsaturated bonds and has a boiling point of less than 500 °C at standard pressure.

The monomer compound and the crosslinking reagent utilised in the method are described in further detail above.

Typically the electronic or electrical device or component thereof is placed in a plasma deposition chamber, a glow discharge is ignited within said chamber, and a voltage applied as a pulsed field. .

Preferably the applied voltage is at a power of from 30 to 800W. Optionally the voltage is pulsed in a sequence in which the ratio of the time on/time off is in the range of from 0.001 to 1, optionally 0.002 to 0.5. For example, time on may be 10-500 μ s, preferably 35-45 μ s, or 30-40 μ s, such as about 36 μ s and time off may be from 0.1 to 30 ms, preferably 0.1 to 20 ms, optionally from 5 to 15 ms, for example 6ms. Time on may be 35 μ s, 40 μ s, 45 μ s. Time off may be 0.1, 1, 2, 3, 6, 8, 10, 15, 20, 25 or 30 ms.

The term pulsed may mean that the plasma cycles between a state of no (or substantially no) plasma emission (off-state) and a state where a particular amount of plasma is emitted (on-state).

Alternatively, pulsed may mean that there is continuous emission of plasma but that the amount of plasma cycles between an upper limit (on-state) and lower limit (off-state).

In a further embodiment, the invention resides in a method of forming a coating on an electronic or electrical device or component thereof as defined above, the method comprising: exposing said substrate in a chamber to a plasma comprising a monomer compound, preferably a continuous plasma, for a sufficient period of time to allow a protective polymeric coating to form on the substrate,

wherein during exposure of the substrate the continuous plasma has a power density of at least 2W/litre, preferably 20W/litre.

Optionally the voltage is applied as a pulsed field for a period of from 30 seconds to 90 minutes. Optionally the voltage is applied as a pulsed field for from 5 to 60 minutes. Optionally in a preliminary step, a continuous power plasma is applied to the electronic or electrical device or component thereof. The preliminary step may be conducted in the presence of an inert gas.

Before the crosslinker and/or the monomer compound enter the deposition chamber each may be in the form of a gas, liquid or a solid (for example a powder) at room temperature. However, it is the preferred that the crosslinking reagent and the monomer compound are both liquid at room temperature, and most preferably that the monomer and crosslinker liquids are miscible.

The monomer compound and/or the crosslinker will suitably be in a gaseous state in the plasma. The plasma may simply comprise a vapour of the monomer compound and the crosslinker. Such a vapour may be formed in-situ, with the compounds being introduced into the chamber in liquid form. The monomer may also be combined with a carrier gas, in particular, an inert gas such as helium or argon.

In preferred embodiments, the monomer and/or the crosslinker may be delivered into the chamber by way of an aerosol device such as a nebuliser or the like, as described for example in WO2003/097245 and WO03/101621, the content of which is incorporated herein by reference. In such an arrangement a carrier gas may not be required, which advantageously assists in achieving high flow rates.

In one embodiment the monomer compound and/or the crosslinking reagent are in gaseous form and are fed into the plasma at a rate of from 10-5000 mg/minute depending on chamber volume, whilst the pulsed voltage is applied.

Optionally the plasma is created with an average power of from 0.001 to 40W/litre.

The crosslinker may be miscible with the monomer and so introduced together or separately into the plasma chamber. Or the crosslinker may be immiscible with the monomer and introduced separately into the plasma chamber. In this context, the term "miscible" means that the crosslinker is soluble in the monomer, and when mixed they form a solution of uniform composition. The term "immiscible" is used to mean that the crosslinker is only partly soluble or insoluble in the monomer, and so either forms an emulsion or separates out into two layers.

The crosslinking reagent is preferably present in an amount of from 10 to 60 (v/v) %, optionally 20 to 40 (v/v) %, optionally 25 to 30 (v/v)%, optionally 30 to 50 (v/v)% of the total volume of monomer compound and crosslinking reagent, depending on the specific crosslinker. The skilled person would understand that the amount will vary to some extent depending on whether the coating is required to

be liquid repellent or provide a barrier to mass and electron transport. The skilled person would understand that the (v/v) percentages are those which give a stable crosslinked polymer coating and the highest water contact angle.

Electronic or electrical device or component thereof

Although the invention is of benefit in the context of a wide variety of substrates, the substrate may, in all aspects of the invention, advantageously be an electronic substrate.

In some embodiments of the invention, the electronic substrate may comprise an electronic or electrical device, i.e. any piece of electrical or electronic equipment. Non-limiting examples of electrical and electronic devices include communications devices such as mobile phones, smartphones and pagers, radios, and sound and audio systems such as loudspeakers, microphones, ringers or buzzers, hearing aids, personal audio equipment such as personal CD, tape cassette or MP3 players, televisions, DVD players including portable DVD players, video recorders, digi and other set-top boxes such as Sky, computers and related components such as laptop, notebook, tablet, phablet or palmtop computers, personal digital assistants (PDAs), keyboards, or instrumentation, games consoles in particular hand-held playstations and the like, data storage devices, outdoor lighting systems or radio antenna and other forms of communication equipment.

In preferred embodiments of the invention, the substrate may comprise or consist of an electronic component, e.g. a printed circuit board (PCB), a printed circuit board array (PCBA), a transistor, resistor, or semi-conductor chip. The electronic component may thus be an internal component of an electronic device, e.g. a mobile phone. The coatings of the invention are particularly valuable in preventing electrochemical migration in such components.

In all aspects of the invention, the precise conditions under which the protective polymeric coating is formed in an effective manner will vary depending upon factors such as, without limitation, the nature of the monomer compound, the crosslinking reagent, the substrate, as well as the desired properties of the coating. These conditions can be determined using routine methods or, preferably, using the techniques and preferred features of the invention described herein, which work in particular synergy with the invention.

The present invention will now be further described with reference to the following non-limiting examples and the accompanying illustrative drawings, of which:

Figure 1 illustrates shows the effect of increasing the power to monomer flow ratio for CW plasmas in a 3 litre chamber in a prior art process.

Figure 2 shows the same effect for pulsed plasma conditions.

Figure 3 shows a tapping mode image over $10 \times 10 \mu\text{m}^2$ field of view of a specimen example (thickness $d=1230 \text{ nm}$) prepared according to Example 1 (left hand side) and a contour line plot showing the data used for calculation of RMS roughness (right hand side). The ΔZ value indicated on the plot has been taken over an area of the graph that represents the majority of the coating. Peaks that lie above the ΔZ range indicate large particles and troughs that fall below the ΔZ range show voids or pinholes in the coating. The width of the peaks also gives an indication of the particle size.

Figure 4 illustrates the effect of adding the divinyl adipate crosslinker to perfluorooctyl acrylate on water contact angle from a process on a 125 Litre chamber.

Figure 5 shows the effect of adding crosslinkers in accordance with the invention on the electrical resistance per nm of the coatings.

Figure 6 shows a tapping mode image of a 90 nm thick coating, prepared as described in Example 3, over $2 \times 2 \mu\text{m}^2$ field of view (top left), a representative contour line indicating height variation (z-axis) of the coating (top right) and a phase image indicating full substrate coverage (bottom left); RMS roughness of the coating is 1.65 nm and $\Delta z/d = 0.05$

Figure 7 shows a FTIR/ATR spectrum of a coating formed according to Example 5.

Example 1

Example of plasma polymerising PFAC6 with no crosslinker in a 3 litre chamber

PFAC6 was polymerised in a 3 litre glass chamber using a continuous plasma process. In the experimental matrix, the continuous plasma was produced using RF power levels of 50, 100 and 200 Watts. The process pressure was run at 30, 60 and 90 mTorr and the monomer flow rate run at 50, 100 and 200 microlitres of liquid per minute. The duration of the process was either 10 or 40 minutes. The coated substrate was silicon wafer and whether the coating was tacky or not was determined by wiping the substrate with a finger and determining by eye if the coating had smeared. The results in Figure 1 indicate that for lower CW powers, the coating could easily be smeared (i.e. tacky), but by increasing the power to flow ratio, the coating became resistant to smearing (i.e. non-tacky) and this is expected to be due to additional crosslinking caused by more monomer fragmentation. The water contact angle was also seen to decline with increasing power to flow ratio, and this too is indicative of more power increasing the level of monomer fragmentation.

The same 3 litre chamber was used to also look at the effect of power to flow ratios with pulsed plasma polymerisation. In these experiments, the peak power was either 250 or 500 W. The RF power feed was pulsed with an on time of 35 microseconds and off times of 5.5 and 1 millisecond. Process pressure was either 20, 35, 60 or 80 mTorr. The monomer flow was either 50 or 200 microlitres of liquid per minute. Figure 2 shows that low power to flow conditions can generate coatings that can be smeared (tacky) and only by increasing the power could the smear be reduced. This too had a concomitant decline in water contact angle.

The results from Figures 1 and 2 show that plasma processing PFAC6 on its own could only lead to smear –free coatings when the powers were high enough to decrease the other desirable property of the water contact angle. This illustrates the need to add a crosslinker to prevent the coating smearing and maintain the water contact angle.

Example 2

Examples of Perfluoro Alkyl Acrylate co-polymerisation with Crosslinker to show improvements in electrical resistance

Perfluorooctyl acrylate monomer was mixed with a single crosslinker from the following list:

VINYL ESTERS: Divinyl adipate (DVA)

VINYL ETHERS: 1,4-Butanediol divinyl ether (BDVE); 1,4 Cyclohexanedimethanol divinyl ether (CDDE)

DI- or TRI- VINYLS: 1,7-Octadiene (1,7-OD); 1,2,4-Trivinylcyclohexane (TVCH);

DI VINYL with alkyl fluoro group: 1,6-divinylperfluorohexane (DVPFH)

DI VINYL with silicon group: 1,3-Divinyl tetramethyldisiloxane (DVTMDS)

DI VINYL with cyclic ring and carboxylate groups: Diallyl 1,4-cyclohexanedicarboxylate (DCHC)

DI ACRYLATE: 1H1H,6H,6H-perfluorohexanediol diacrylate (PFHDA)

The crosslinker was mixed with the PFAC6 as a percentage by volume for the following percentages: 20, 40, 60, 80, and 100%.

Plasma initiated polymerisation reactions were carried out in a 3 litre glass plasma chamber. The substrates were test circuit boards. The PFAC6/crosslinker mixture was introduced at a rate of 0.04 – 0.06 ul/min and the process pressure was 40 mTorr. The process plasma consisted of a 1 minute continuous plasma (CW) step of 50W followed by 10 - 20 minute pulsed plasma (PW) step of 50W and RF power being delivered by a pulse sequence with a duty cycle of 6.9%. The coated circuit boards were immersed in tap water and an 8V potential was applied for 13 minutes. The final current readings, together with coating thickness measurements, were used to calculate the electrical resistance per nm of coating. Figure 5 below shows the effect of adding a range of crosslinkers on the electrical resistance per nm of the barrier coatings. Figure 5 clearly shows the improvement in resistance that can be gained by adding the crosslinker to the PFAC6 monomer. Furthermore, all of the crosslinkers tested gave non-smear/non-tacky coatings with concentration levels of 20% and above, with the exceptions of DVTMDS, DCHD, PFHDA and DVPFH which were non-smear at levels of 40% and above.

Example 3**Example of crosslinker concentration on water repellent coating.**

Water repellent coatings were prepared in a 125 litre volume chamber using PFAC6 with different levels of divinyl adipate (DVA) and using helium as a carrier gas. The deposition process consisted of a 3 minute CW step with 300W power and a pulsed step with 150W power and an RF pulse duty cycle of 0.018%. Silicon wafer was used as the test substrates and the contact angle of the coated wafer was determined by applying a 3ul drop of deionised wafer onto the coated wafer and measuring the contact angle using a VCA Optima (AST products) with image analysis software. The variation of contact angle with (v/v)% DVA crosslinker is shown in Figure 4. It was also noted that for the 10 (v/v) % DVA concentration, the water drop left a mark on the coated wafer where it's circumference had been. This indicated that a higher level of crosslinker was required to give a more stable coating. This observation, in combination with the results in figure 4, suggest an optimum range of 20 – 40 (v/v) % DVA crosslinker, though this is likely to vary, typically within the range of from 10-60 (v/v) % for different monomers and crosslinkers and different chamber sizes.

Pulsed plasma polymerisation of perfluoro alkyl acrylate in the presence of divinyl adipate (DVA) was carried out in a 125 litre chamber. Figure 4 shows the effect of adding different liquid volume percentages of DVA crosslinker to perfluorooctyl acrylate on water contact angle on a silicon wafer substrate.

Example 4**Example of AFM measurement of monomer/crosslinker co-polymer coating**

A barrier-style coating was prepared in a 22 litre volume chamber using PFAC6 with 10% DVA. The deposition process consisted of a 1 minute CW step with a CW power to flow ratio of 3.9 (W/microliter/min), and a pulsed step with a PW power/monomer flow ratio of 0.28 (W/microlitres/min). Figure 6 shows the representative topographical and phase contrast images were obtained from all the samples. High spatial resolution images show mainly the structures of areas between the raised features. The RMS roughness of the coating is 1.65 nm and the $\Delta z/d = 0.05$. As the $\Delta z/d$ value is < 0.15, then this indicates that the physical layer is substantially pinhole free.

Example 5**Example of FTIR/ATR measurement of monomer/crosslinker co-polymer coating**

Two barrier-style coatings were prepared in a 22 litre volume chamber using PFAC6 only and PFAC6 with 10% DVA as described in example 4. The deposition process consisted of a 1 minute CW step with a CW power to flow ratio of 3.9 (W/microliter/min), and a pulsed step with a PW power/monomer flow ratio of 0.28 (W/microlitres/min). Figure 7 shows the representative FTIR/ATR spectrum obtained from both samples.

The FTIR/ATR intensity ratios of peaks attributed to stretching mode of CF_3 and C=O groups, $\text{CF}_3/\text{C=O}$, of the coating is indicative of sufficient cross linking in the coating to form a physical barrier. CF_3 refers to the terminal groups in the side chain of PFAC6.

Formation of the barrier coatings is believed to be caused by a mixture of cross linking and controlled fragmentation of the monomers during polymerisation. Cross linking is believed to be predominantly via the $\text{CF}_2\text{-CF}_3$ chain, whilst fragmentation is thought to be predominantly through loss of the C=O group during polymerisation and to a lesser extent shortening of the CF_2 chain. Cross linking effects the abundance of $-\text{CF}_3$ groups in the coating and controlled fragmentation controls the amount of C=O groups in the coating. The ratio of these two functional groups is an indication that sufficient cross-linking and fragmentation has taken place and can be measured by the ratio of the intensities of the corresponding FTIR/ATR peaks.

It has been shown that coatings with reduced $\text{CF}_3 / \text{C=O}$ ratios give higher resistance values in the electrical test, as described in Example 2, showing an improved coating performance on increased cross linking (for CF_3) and fragmentation (for C=O). If the FTIR/ATR intensity ratio of the peaks attributed to $-\text{CF}_3$ stretching and C=O stretching, $\text{CF}_3/\text{C=O}$, is less than $0.6e^{-0.1n}$ (where $n=6$ for PFAC6) the resistance of the coating at the electrical test is expected to be higher than 8MOhms.

The peak intensity ratio $\text{CF}_3/\text{C=O}$ the coating thickness and the final current readings during the electrical test are shown in table 2:

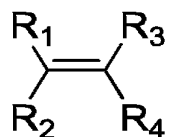
| | Thickness (nm) | Resistance water at 8V (Ohms) | in $\text{CF}_3/\text{C=O}$ ATR peak intensity ratio |
|------------------------------------|-------------------|-------------------------------------|---|
| PFAC6 | 1480 | 2.1×10^7 | 0.23 |
| PFAC6 and 10%DVA (in volume) | 1450 | 9.2×10^9 | 0.11 |

Table 2

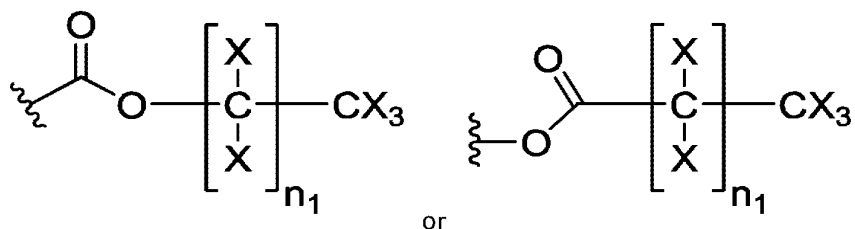
Claims:

1. An electronic or electrical device or component thereof comprising a protective cross-linked polymeric coating on a surface of the electronic or electrical device or component thereof; wherein the protective cross-linked polymeric coating is obtainable by exposing the electronic or electrical device or component thereof to a plasma comprising a monomer compound and a crosslinking reagent for a period of time sufficient to allow formation of the protective cross-linked polymeric coating on a surface thereof,

wherein the monomer compound has the following formula:

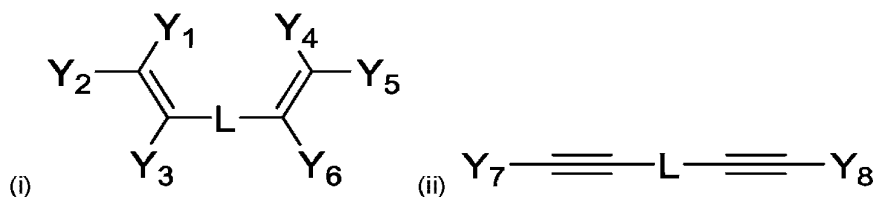


where R_1 , R_2 and R_4 are each independently selected from hydrogen, optionally substituted branched or straight chain C_1 - C_6 alkyl or halo alkyl or aryl optionally substituted by halo, and R_3 is selected from:



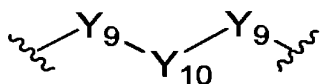
where each X is independently selected from hydrogen, a halogen, optionally substituted branched or straight chain C_1 - C_6 alkyl, halo alkyl or aryl optionally substituted by halo; and n_1 is an integer from 1 to 27; and wherein the crosslinking reagent comprises two or more unsaturated bonds attached by means of one or more linker moieties and has a boiling point of less than 500 °C at standard pressure.

2. An electronic or electrical device or component thereof according to claim 1, wherein the protective cross-linked polymeric coating is a physical barrier to mass and electron transport.
3. An electronic or electrical device or component thereof according to claim 1 or claim 2, wherein the protective cross-linked polymeric coating forms a liquid repellent surface defined by a static water contact angle (WCA) of at least 90°.
4. An electronic or electrical device or component thereof according to any preceding claim, wherein the crosslinking reagent has one of the following structures:



where Y_1 , Y_2 , Y_3 , Y_4 , Y_5 , Y_6 , Y_7 and Y_8 are each independently selected from hydrogen, optionally substituted cyclic, branched or straight chain C_1 - C_8 alkyl or aryl; and L is a linker moiety.

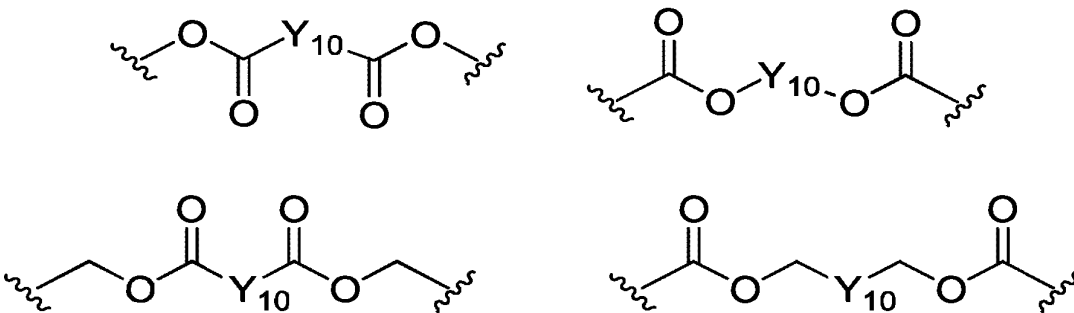
5. An electronic or electrical device or component thereof according to claim 4, wherein for compound (i) L has the following formula:



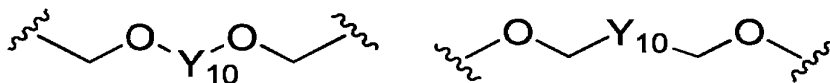
each Y_9 is independently selected from, a bond, -O-, -O-C(O)-, -C(O)-O-, - Y_{11} -O-C(O)-, -C(O)-O- Y_{11} -, -O Y_{11} -, and - Y_{11} O-, where Y_{11} is an optionally substituted cyclic, branched or straight chain C_1 - C_8 alkylene; and

Y_{10} is selected from optionally substituted cyclic, branched or straight chain C_1 - C_8 alkylene and a siloxane group.

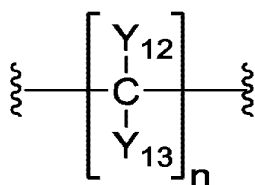
6. An electronic or electrical device or component thereof according to claim 5, wherein for compound (i) L has one of the following structures:



7. An electronic or electrical device or component thereof according to claim 5, wherein for compound (i) L has one of the following structures:

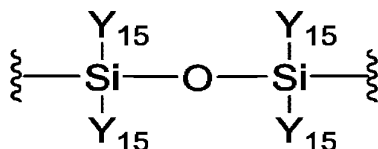


8. An electronic or electrical device or component thereof according to any of claims 5 to 7, wherein Y_{10} has the following formula:



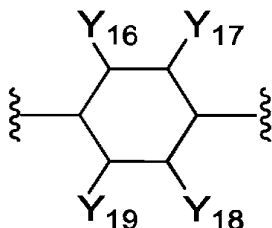
wherein each Y_{12} and Y_{13} is independently selected from H, halo, optionally substituted cyclic, branched or straight chain alkyl, or $-\text{OY}_{14}$, where Y_{14} is selected from optionally substituted branched or straight chain $\text{C}_1\text{-C}_8$ alkyl or alkenyl, and n is an integer from 1 to 10.

9. An electronic or electrical device or component thereof according to claim 8, wherein each Y_{12} is H and each Y_{13} is H.
10. An electronic or electrical device or component thereof according to claim 8, wherein each Y_{12} is fluoro and each Y_{13} is fluoro.
11. An electronic or electrical device or component thereof according to any of claims 8 to 10, wherein n is from 4 to 6.
12. An electronic or electrical device or component thereof according to any of claims 5 to 7, wherein Y_{10} has the following formula:



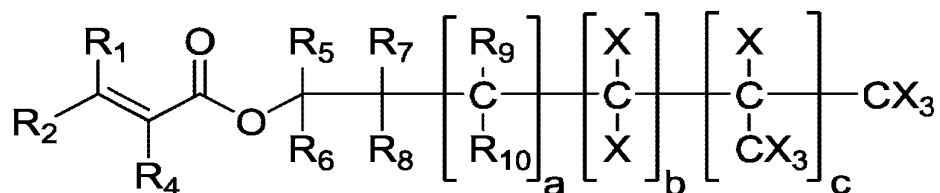
Wherein each Y_{15} is independently selected from optionally substituted branched or straight chain $\text{C}_1\text{-C}_8$ alkyl.

13. An electronic or electrical device or component thereof according to claim 12, wherein each Y_{15} is methyl, and each Y_9 is a bond.
14. An electronic or electrical device or component thereof according to any of claims 5 to 7, wherein Y_{10} has the following formula:



wherein Y_{16} to Y_{19} are each independently selected from H and optionally substituted branched or straight chain $\text{C}_1\text{-C}_8$ alkyl or alkenyl.

15. An electronic or electrical device or component thereof according to claim 14, wherein Y₁₈ is H or vinylene, and Y₁₆, Y₁₇ and Y₁₉ are each H.
16. An electronic or electrical device or component thereof according to any preceding claim wherein the crosslinking reagent is selected from Divinyl Adipate (DVA), 1,4 Butanediol divinyl ether (BDVE), 1,4 Cyclohexanedimethanol divinyl ether (CDDE), 1,7 -Octadiene (17OD), 1,2,4 -Trivinylcyclohexane (TVCH), 1,3-Divinyltetramethyldisiloxane (DVTMDS), Diallyl 1,4-cyclohexanedicarboxylate (DCHD), 1,6-Divinylperfluorohexane (DVPFH), 1H,1H,6H,6H-Perfluorohexanediol diacrylate (PFHDA) and glyoxal bis (diallyl acetal) (GBDA).
17. An electronic or electrical device or component thereof according to claim 4, wherein for compound (ii), L is selected from a branched or straight chain C₁-C₈ alkylene or an ether group.
18. An electronic or electrical device or component thereof according to any preceding claim wherein the monomer is a compound of formula I(a):

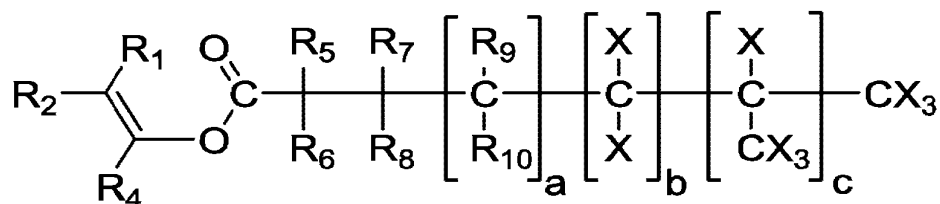
$$l(a)$$


wherein each of R₁, R₂, R₄, and R₅ to R₁₀ is independently selected from hydrogen or an optionally substituted C₁-C₆ branched or straight chain alkyl group; each X is independently selected from hydrogen or halogen; a is from 0-10; b is from 2 to 14; and c is 0 or 1;

; or

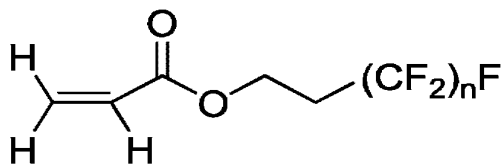
wherein the monomer is a compound of formula I(b):

1(b)



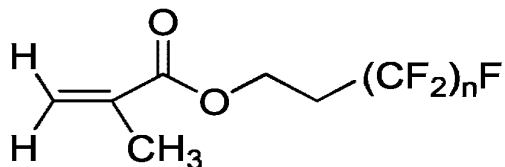
wherein each of R_1 , R_2 , R_4 , and R_5 to R_{10} is independently selected from hydrogen or an optionally substituted C_1 - C_6 branched or straight chain alkyl group; each X is independently selected from hydrogen or halogen; a is from 0-10; b is from 2 to 14; and c is 0 or 1.

19. An electronic or electrical device or component thereof according to claim 18, wherein the halogen is fluorine.
20. An electronic or electrical device or component thereof according to claim 18 or claim 19, wherein each of R_1 , R_2 , R_4 , and R_5 to R_{10} is independently selected from hydrogen, methyl, ethyl, n-propyl, isopropyl, n-butyl, isobutyl, tert-butyl, sec-butyl, n-pentyl, neopentyl, n-hexyl, isohexyl, and 3-methylpentyl.
21. An electronic or electrical device or component thereof according to claim 20, wherein each of R_1 , R_2 , R_4 , and R_5 to R_{10} is independently selected from hydrogen or methyl.
22. An electronic or electrical device or component thereof according to any of claims 18 to 20, wherein a and c are each independently 0 or 1; and b is from 3 to 7.
23. An electronic or electrical device or component thereof according to any preceding claim wherein each X is H.
24. An electronic or electrical device or component thereof according to any of claims 1 to 22, wherein each X is F.
25. An electronic or electrical device or component thereof according to any of claims 18 to 24, wherein R_1 and R_2 are both hydrogen.
26. An electronic or electrical device or component thereof according to any of claims 18 to 25, wherein R_4 is hydrogen or methyl.
27. An electronic or electrical device or component thereof according to any of claims 18 to 26, wherein R_9 is hydrogen and R_{10} is branched or straight chain C_1 - C_6 alkyl group.
28. An electronic or electrical device or component thereof according to claim 27, wherein R_{10} is methyl.
29. An electronic or electrical device or component thereof according to any of claims 18 to 28, wherein each of R_5 to R_8 is hydrogen.
30. An electronic or electrical device or component thereof according to any of claims 18 to 29 wherein each of each of R_1 , R_2 , R_4 , and R_5 to R_{10} is hydrogen, each X is H, a=0 and c=0.
31. An electronic or electrical device or component thereof according to any of claims 24 to 30, wherein the compound of formula I(a) has the following formula:



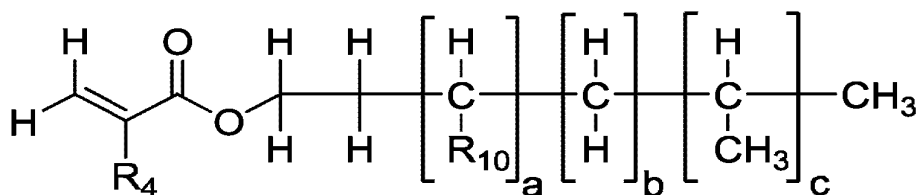
where n is from 2 to 10.

32. An electronic or electrical device or component thereof according to any of claims 24 to 30, wherein the compound of formula I(a) has the following formula:



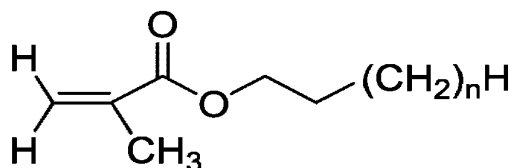
where n is from 2 to 10.

33. An electronic or electrical device or component thereof according to claim 31, wherein the compound of formula I(a) is selected from 1H,1H,2H,2H-perfluorohexyl acrylate (PFAC4), 1H,1H,2H,2H-perfluorooctyl acrylate (PFAC6), 1H,1H,2H,2H-perfluorodecyl acrylate (PFAC8) and 1H,1H,2H,2H-perfluorododecyl acrylate (PFAC10).
34. An electronic or electrical device or component thereof according to claim 32, wherein the compound of formula I(a) is selected from 1H,1H,2H,2H-perfluorohexyl methacrylate (PFMAC4), 1H,1H,2H,2H-perfluorooctyl methacrylate (PFMAC6) and 1H,1H,2H,2H-perfluorodecyl methacrylate (PFMAC8).
35. An electronic or electrical device or component thereof according to any of claims 18 to 23, wherein the compound of formula I(a) has the following formula:



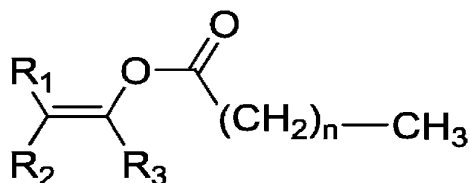
wherein a and c are each independently 0 or 1, b = 3-7 and n is 4 to 10, where $n = a + b + c + 1$.

36. An electronic or electrical device or component thereof according to any of claims 18 to 23, wherein the compound of formula I(a) has the following formula:



where n is 2 to 12.

37. An electronic or electrical device or component thereof according to claim 35 or claim 36, wherein the compound of formula I(a) is selected from ethyl hexyl acrylate, hexyl acrylate, decyl acrylate, lauryl dodecyl acrylate and iso decyl acrylate.
38. An electronic or electrical device or component thereof according to any of claims 18 to 23, wherein the monomer is a compound of formula I(b) and has the following formula:

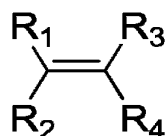


where n is from 4 to 14, optionally where R₁, R₂ and R₃ are each H.

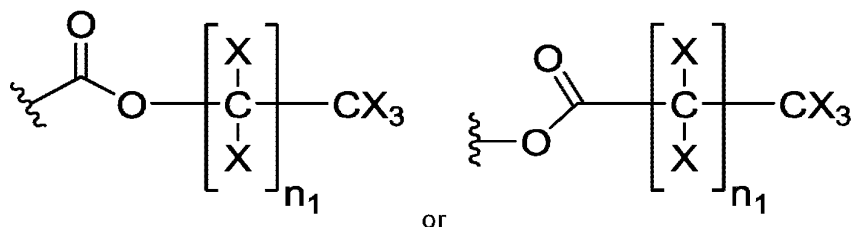
39. An electronic or electrical device or component thereof according to any of the preceding claims, wherein the electronic or electrical device or component thereof is selected from mobile phones, smartphones, pagers, radios, sound and audio systems such as loudspeakers, microphones, ringers and/or buzzers, hearing aids, personal audio equipment such as personal CD, tape cassette or MP3 players, televisions, DVD players including portable DVD players, video recorders, digi and other set-top boxes, computers and related components such as laptop, notebook, tablet, phablet or palmtop computers, personal digital assistants (PDAs), keyboards, or instrumentation, games consoles, data storage devices, outdoor lighting systems, radio antennae and other forms of communication equipment, and printed circuit boards.
40. A method for treating an electronic or electrical device or component thereof comprising:

exposing the electronic or electrical device or component thereof to a plasma comprising a monomer compound and a crosslinking reagent for a period of time sufficient to allow a protective cross-linked polymeric coating to form on a surface of the electronic or electrical device or component thereof;

wherein the monomer compound has the following formula:

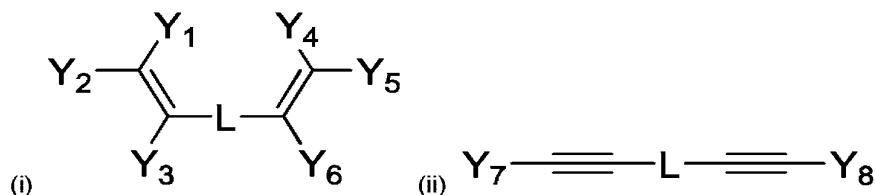


where R_1 , R_2 and R_4 are each independently selected from hydrogen, optionally substituted branched or straight chain C_1 - C_6 alkyl or halo alkyl or aryl optionally substituted by halo, and R_3 is selected from:



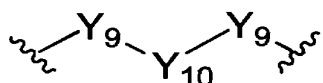
where each X is independently selected from hydrogen, optionally substituted branched or straight chain C_1 - C_6 alkyl, halo alkyl or aryl optionally substituted by halo; and n_1 is an integer from 1 to 27; and wherein the crosslinking reagent comprises two or more unsaturated bonds attached by means of one or more linker moieties and has a boiling point at standard pressure of less than 500°C .

41. A method according to claim 40, wherein the crosslinking reagent has one of the following structures:



where Y_1 , Y_2 , Y_3 , Y_4 , Y_5 , Y_6 , Y_7 and Y_8 are each independently selected from hydrogen, optionally substituted cyclic, branched or straight chain C_1 - C_6 alkyl or aryl; and L is a linker moiety.

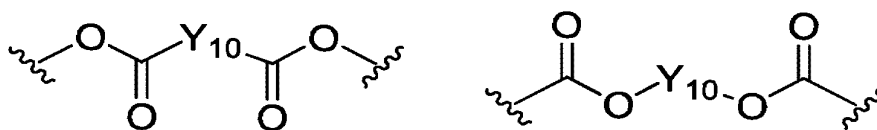
42. A method according to claim 41, wherein for compound (i) L has the following formula:

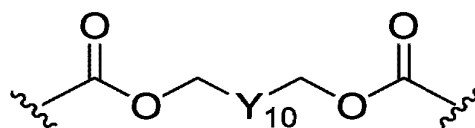
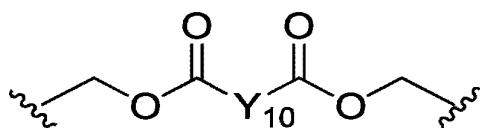


each Y_9 is independently selected from, a bond, $-\text{O}-$, $-\text{O}-\text{C}(\text{O})-$, $-\text{C}(\text{O})-\text{O}-$, $-\text{Y}_{11}-\text{O}-\text{C}(\text{O})-$, $-\text{C}(\text{O})-\text{O}-\text{Y}_{11}-$, $-\text{OY}_{11}-$, and $-\text{Y}_{11}\text{O}-$, where Y_{11} is an optionally substituted cyclic, branched or straight chain C_1 - C_8 alkylene; and

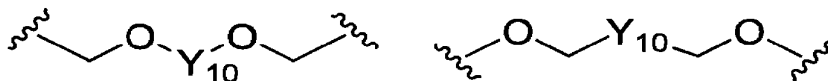
Y_{10} is selected from optionally substituted cyclic, branched or straight chain C_1 - C_8 alkylene and a siloxane group.

43. A method according to claim 42, wherein for compound (i) L has one of the following structures:

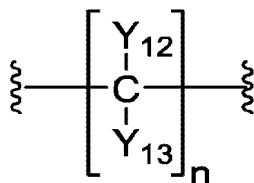




44. A method according to claim 42, wherein for compound (i) L has one of the following structures:

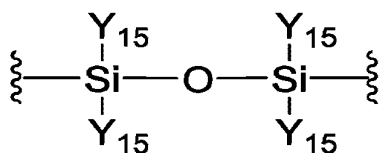


45. An method according to any of claims 42 to 44, wherein Y₁₀ has the following formula:



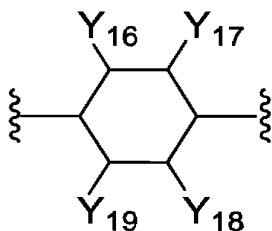
wherein each Y₁₂ and Y₁₃ is independently selected from H, halo, optionally substituted cyclic, branched or straight chain alkyl, or -OY₁₄, where Y₁₄ is selected from optionally substituted branched or straight chain C₁-C₈ alkyl or alkenyl, and n is an integer from 1 to 10.

46. A method according to claim 45, wherein each Y₁₂ is H and each Y₁₃ is H.
 47. A method according to claim 45, wherein each Y₁₂ is fluoro and each Y₁₃ is fluoro.
 48. A method according to any of claims 45 to 47, wherein n is from 4 to 6.
 49. A method according to any of claims 42 to 44, wherein Y₁₀ has the following formula:



Wherein each Y₁₅ is independently selected from optionally substituted branched or straight chain C₁-C₈ alkyl.

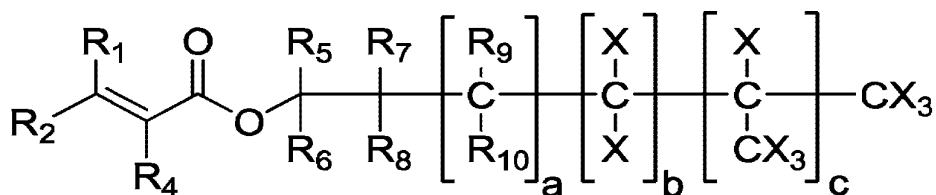
50. A method according to claim 49, wherein each Y₁₅ is methyl, and each Y₉ is a bond.
 51. A method according to any of claims 42 to 44, wherein Y₁₀ has the following formula:



wherein Y_{16} to Y_{19} are each independently selected from H and optionally substituted branched or straight chain C_1 - C_8 alkyl or alkenyl.

52. A method according to claim 51, wherein Y_{18} is vinylene or H, and Y_{16} , Y_{17} and Y_{19} are each H.
53. A method according to any of claims 40 to 52, wherein the crosslinking reagent is selected from Divinyl Adipate (DVA), 1,4 Butanediol divinyl ether (BDVE), 1,4 Cyclohexanedimethanol divinyl ether (CDDE), 1,7 -Octadiene (17OD), 1,2,4 -Trivinylcyclohexane (TVCH), 1,3-Divinyltetramethyldisiloxane (DVTMDS), Diallyl 1,4-cyclohexanedicarboxylate (DCHD), 1,6-Divinylperfluorohexane (DVPFH), 1H,1H,6H,6H-Perfluorohexanediol diacrylate (PFHDA) and glyoxal bis (diallyl acetal) (GBDA).
54. A method according to claim 41, wherein for compound (ii), L is selected from a branched or straight chain C_1 - C_8 alkylene or an ether group.
55. A method according to any of claims 40 to 54 wherein the monomer is a compound of formula I(a):

I(a)

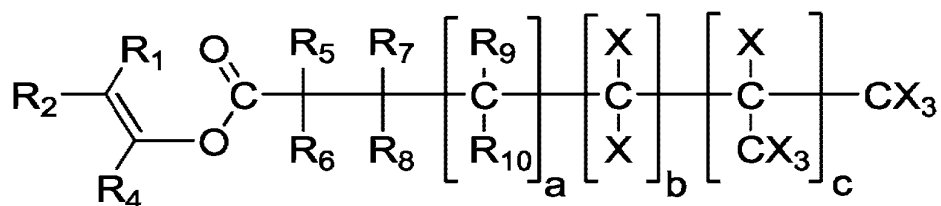


wherein each of R_1 , R_2 , R_4 , and R_5 to R_{10} is independently selected from hydrogen or an optionally substituted C_1 - C_8 branched or straight chain alkyl group; each X is independently selected from hydrogen or halogen; a is from 0-10; b is from 2 to 14; and c is 0 or 1;

; or

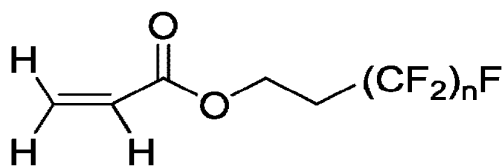
wherein the monomer is a compound of formula I(b):

I(b)



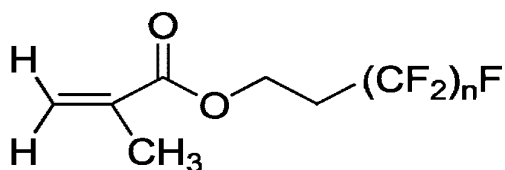
wherein each of R_1 , R_2 , R_4 , and R_5 to R_{10} is independently selected from hydrogen or an optionally substituted C_1 - C_6 branched or straight chain alkyl group; each X is independently selected from hydrogen or halogen; a is from 0-10; b is from 2 to 14; and c is 0 or 1.

56. A method according to claim 55, wherein the halogen is fluorine.
57. A method according to claim 55 or claim 56, wherein each of R_1 , R_2 , R_4 , and R_5 to R_{10} is independently selected from hydrogen, methyl, ethyl, n-propyl, isopropyl, n-butyl, isobutyl, tert-butyl, sec-butyl, n-pentyl, neopentyl, n-hexyl, isohexyl, and 3-methylpentyl.
58. A method according to claim 57, wherein each of R_1 , R_2 , R_4 , and R_5 to R_{10} is independently selected from hydrogen or methyl.
59. A method according to any of claims 55 to 58, wherein a and c are each independently 0 or 1; and b is from 3 to 7.
60. A method according to any preceding claim wherein each X is H.
61. A method according to any of claims 41 to 59, wherein each X is F.
62. A method according to any of claims 41 to 61, wherein R_1 and R_2 are both hydrogen.
63. A method according to any of claims 41 to 62, wherein R_4 is hydrogen or methyl.
64. A method according to any of claims 41 to 63, wherein R_9 is hydrogen and R_{10} is branched or straight chain C_1 - C_6 alkyl group.
65. A method according to claim 64, wherein R_{10} is methyl.
66. A method to any of claims 41 to 65, wherein each of R_5 to R_8 is hydrogen.
67. A method according to any of claims 41 to 66 wherein each of each of R_1 , R_2 , R_4 , and R_5 to R_{10} is hydrogen, each X is H, $a=0$ and $c=0$.
68. A method according to any of claims 61 to 67, wherein the compound of formula I(a) has the following formula:



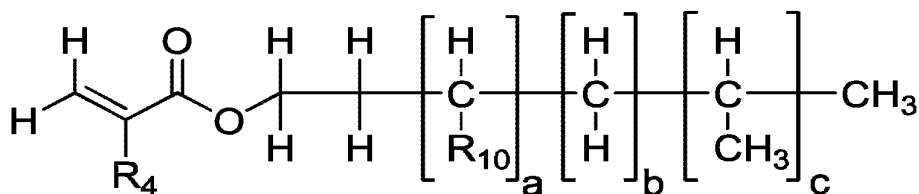
where n is from 2 to 10.

69. A method according to any of claims 61 to 67, wherein the compound of formula I(a) has the following formula:



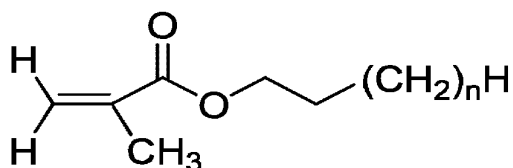
where n is from 2 to 10.

70. A method according to claim 68, wherein the compound of formula I(a) is selected from 1H,1H,2H,2H-perfluorohexyl acrylate (PFAC4), 1H,1H,2H,2H-perfluorooctyl acrylate (PFAC6), 1H,1H,2H,2H-perfluorodecyl acrylate (PFAC8) and 1H,1H,2H,2H-perfluorododecyl acrylate (PFAC10).
71. A method according to claim 69, wherein the compound of formula I(a) is selected from 1H,1H,2H,2H-perfluorohexyl methacrylate (PFMAC4), 1H,1H,2H,2H-perfluorooctyl methacrylate (PFMAC6) and 1H,1H,2H,2H-perfluorodecyl methacrylate (PFMAC8).
72. A method according to any of claims 41 to 61, wherein the compound of formula I(a) has the following formula:



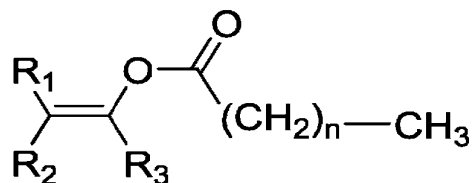
wherein a and c are each independently 0 or 1, b = 3-7 and n is 4 to 10, where n = a + b + c + 1.

73. A method according to any of claims 41 to 61, wherein the compound of formula I(a) has the following formula:



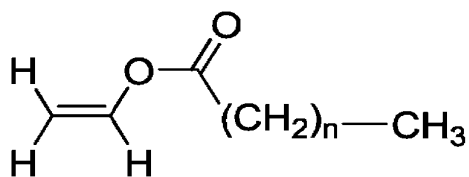
where n is 2 to 12.

74. A method according to claim 72 or claim 73, wherein the compound of formula I(a) is selected from ethyl hexyl acrylate, hexyl acrylate, decyl acrylate, lauryl dodecyl acrylate and iso decyl acrylate.
75. A method according to any of claims 41 to 60, wherein the monomer is a compound of formula I(b) and has the following formula:



where n is from 4 to 14.

76. A method according to claim 75 wherein the monomer is a compound of formula I(b) and has the following formula:



where n is from 4 to 14.

77. A method according to any of claims 40 to 76 wherein the electronic or electrical device or component thereof is placed in a plasma deposition chamber, a glow discharge is ignited within said chamber, and a voltage applied as a pulsed field.
78. A method according to claim 77 wherein applied voltage is at a power of from 40 to 500W.
79. A method according to claim 77 or claim 78 wherein the voltage is pulsed in a sequence in which the ratio of the time on : time off is in the range of from 1:500 to 1:1500.
80. A method according to any of claims 77 to 79 wherein the voltage is pulsed in a sequence where power is on for 20-50μs, and off for from 1000μs to 30000μs.
81. A method according to any of claims 77 to 80 wherein the voltage is applied as a pulsed field for a period of from 30 seconds to 90 minutes.
82. A method according to claim 81 wherein the voltage is applied as a pulsed field for from 5 to 60 minutes.
83. A method according to any one of claims 77 to 82, wherein in a preliminary step, a continuous power plasma is applied to the electronic or electrical device or component thereof.

1 / 6

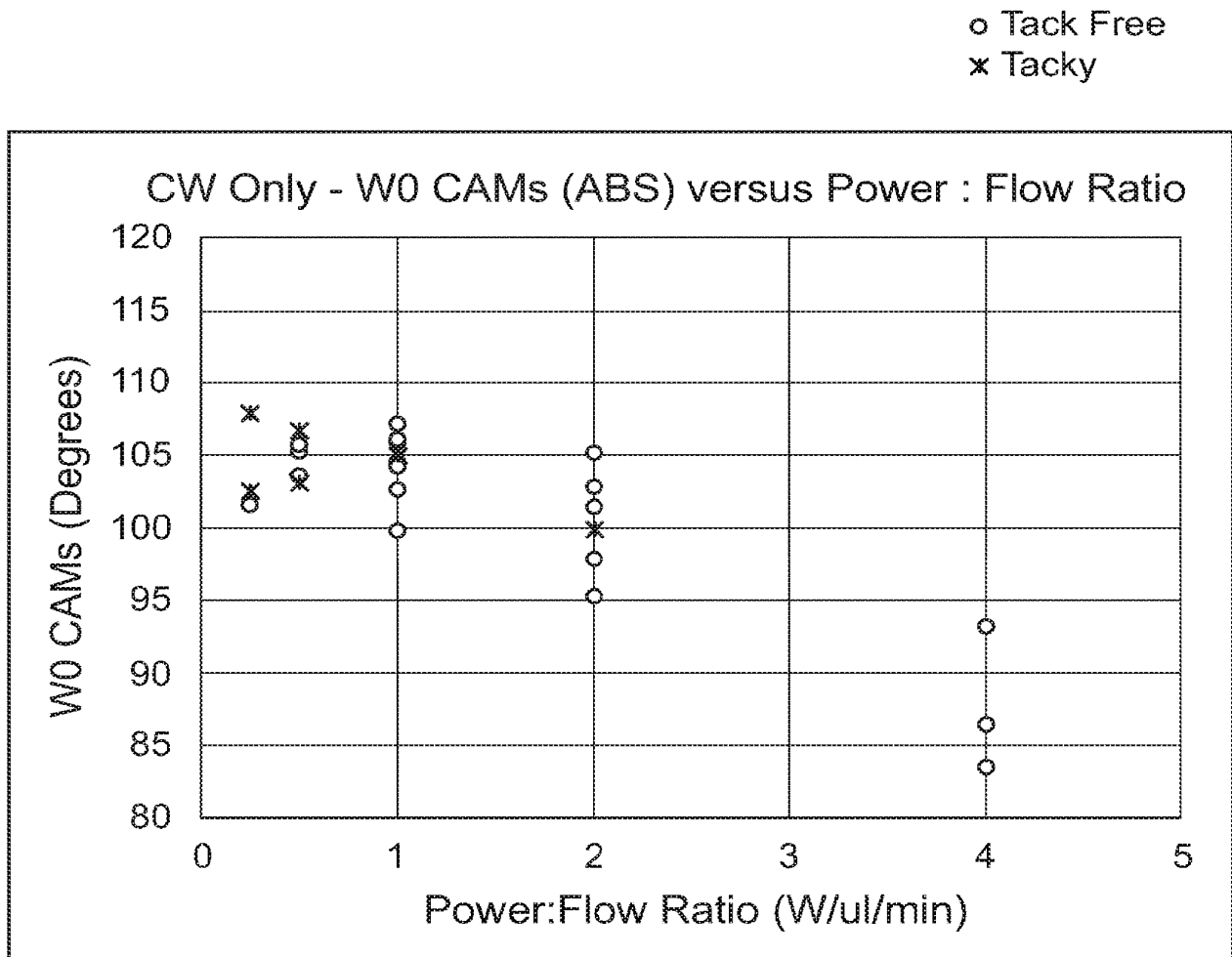


Figure 1

2 / 6

○ Tack Free
× Tacky

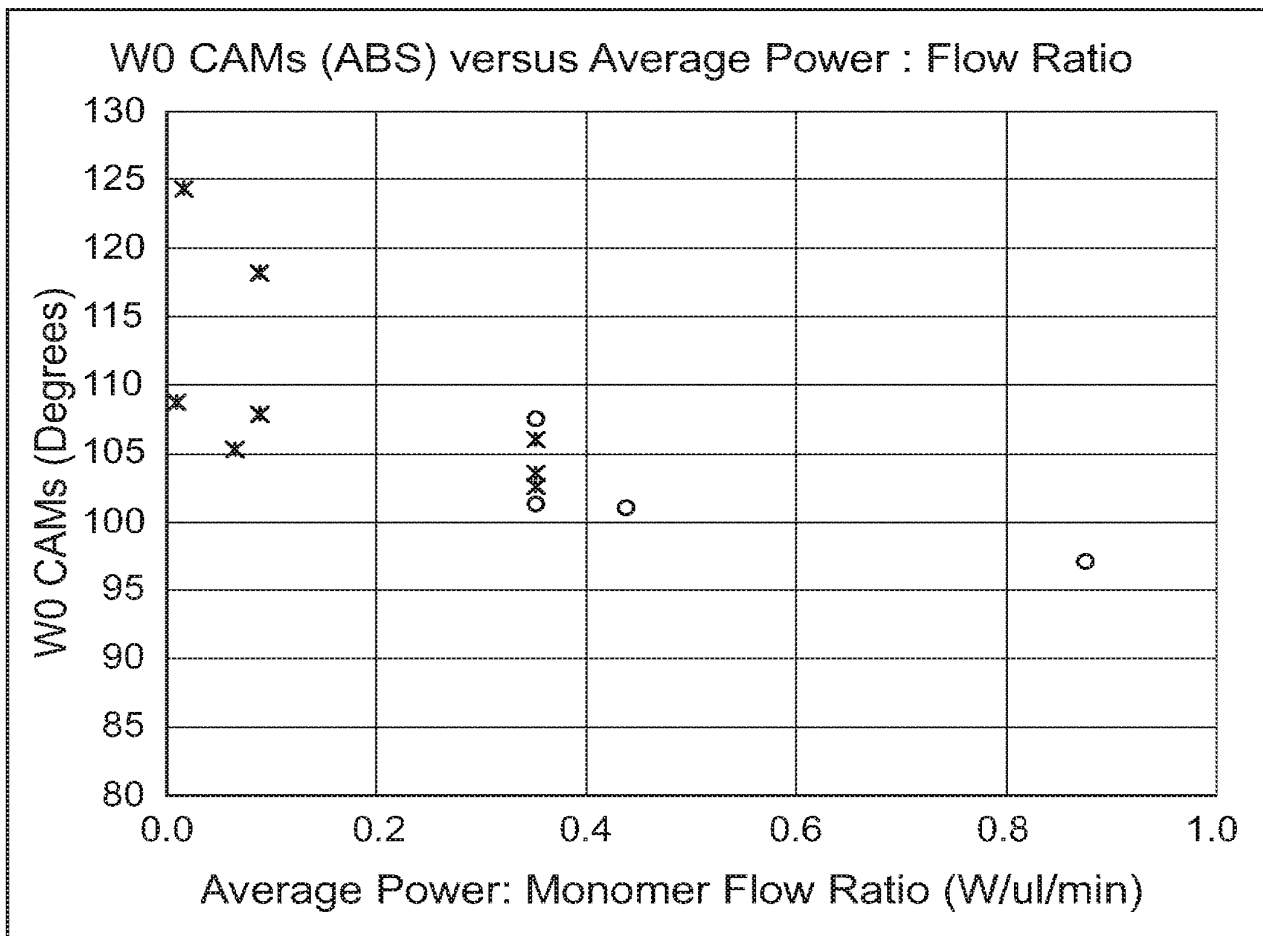


Figure 2

3 / 6

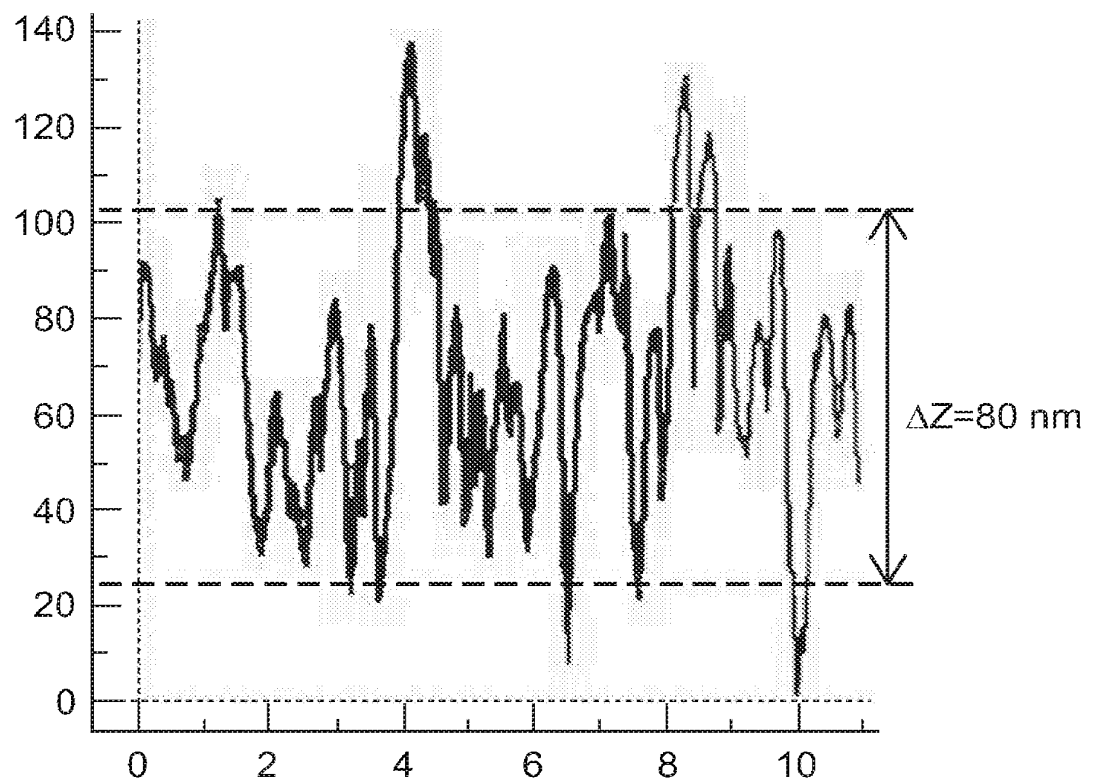
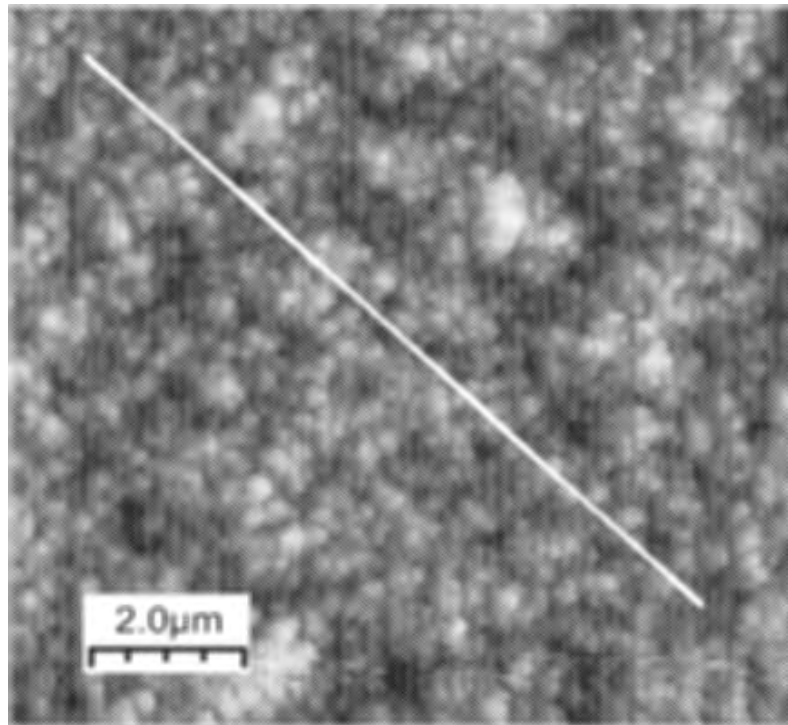


Figure 3

4 / 6

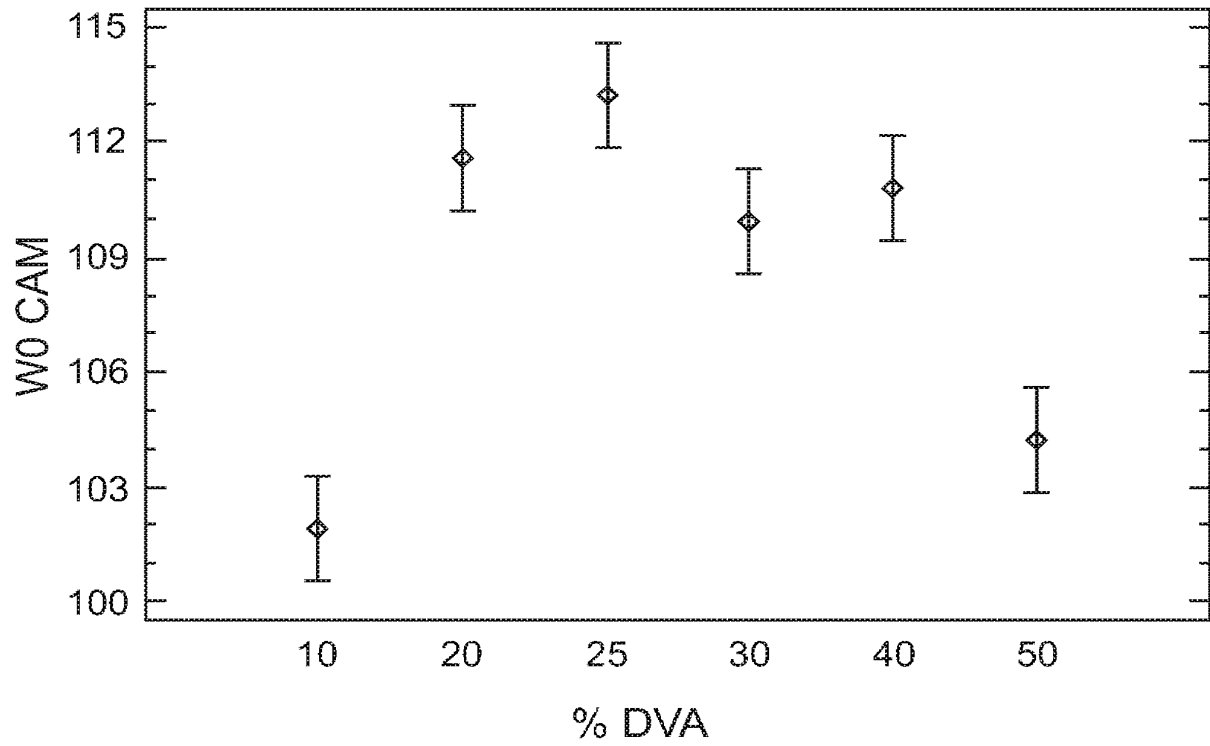


Figure 4

5 / 6

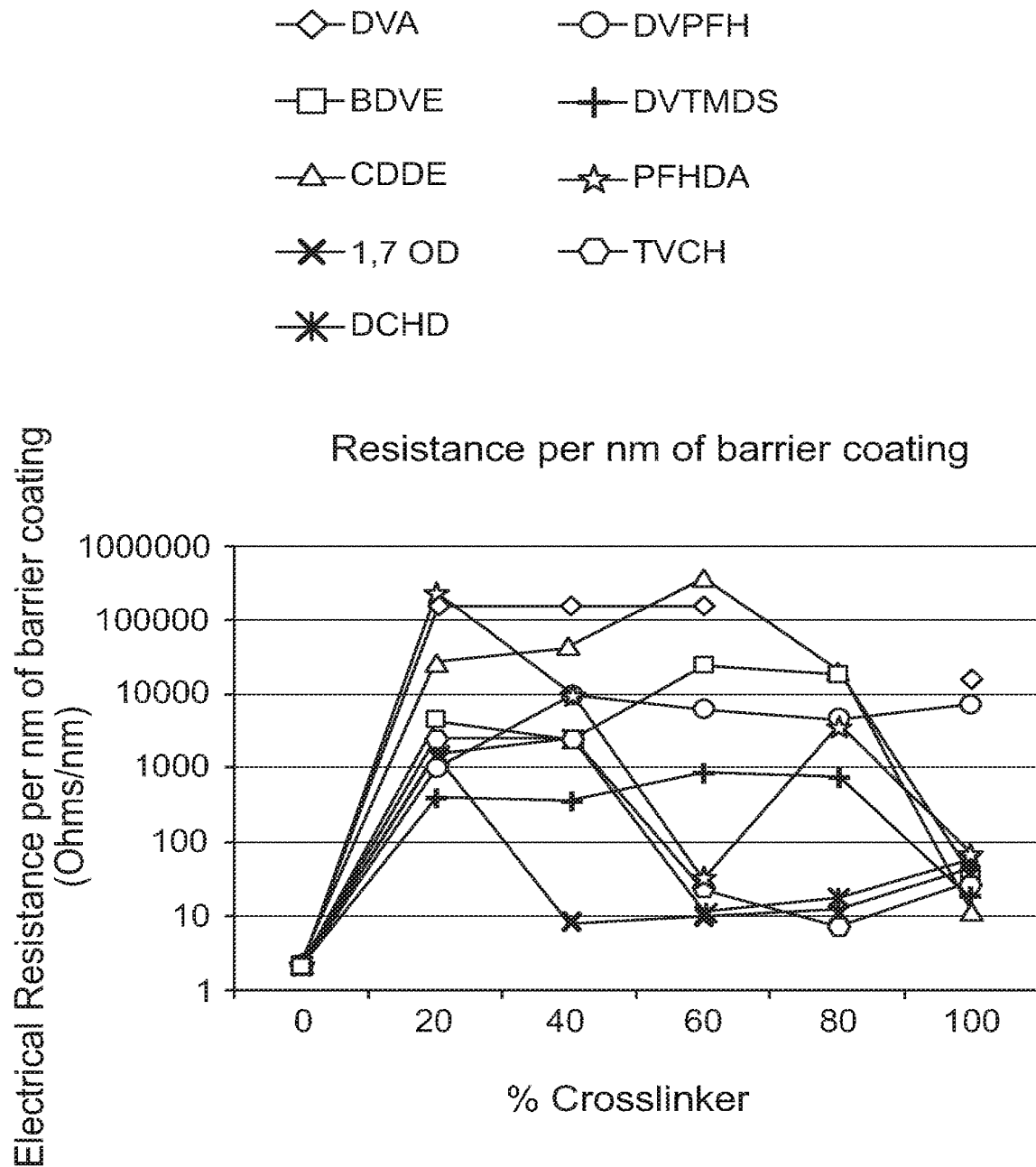


Figure 5

6 / 6

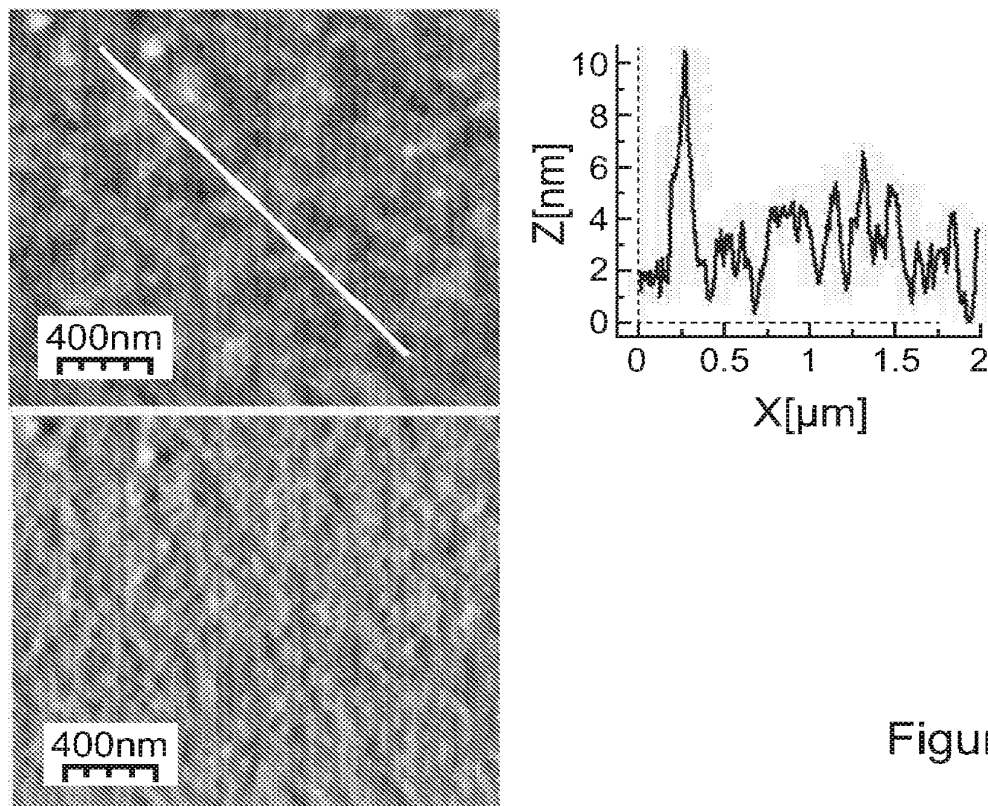
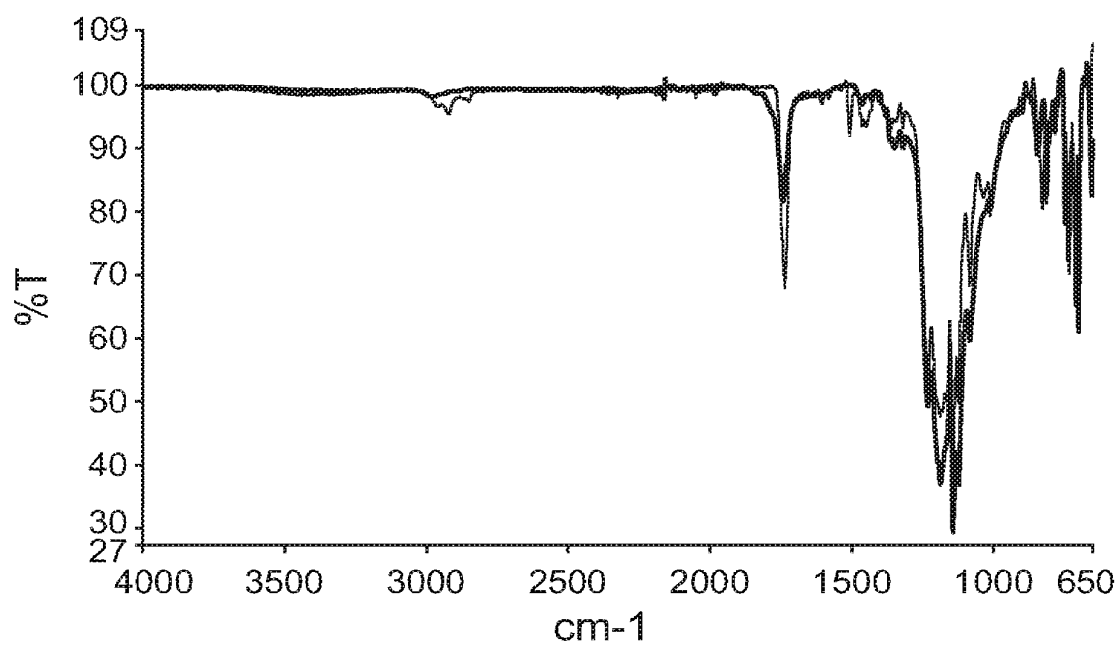


Figure 6



| Name | Description |
|------------------|--|
| — PFAC6 | Sample 350 By Administrator Date Thursday, February 13 2014 |
| — PFAC6-DVA(10%) | Sample 389 By PEService Date Tuesday, July 14 2015 |

Information on Search Strategy - Pilot phase (see OJ 2015, A86)

The type of information contained in this sheet may change during the pilot for improving the usefulness of this new service.

Application Number

PCT/GB2016/051688

TITLE: COATINGS

APPLICANT: P2I LTD

IPC CLASSIFICATION: C09D5/00

EXAMINER: Kositza, Matthias

CONSULTED DATABASES: EPODOC, WPI, NPL

CLASSIFICATION SYMBOLS DEFINING EXTENT OF THE SEARCH:

IPC:

CPC: C09D5/++

FI/F-TERMS:

KEYWORDS OR OTHER ELEMENTS FEATURING THE INVENTION:



Bescheinigung

Die angehefteten
Unterlagen stimmen mit der
als ursprünglich eingereicht
geltenden Fassung der auf
dem nächsten Blatt
bezeichneten europäischen
Patentanmeldung überein.

Certificate

The attached documents are
exact copies of the text in
which the European patent
application described on the
following page is deemed to
have been filed.

Attestation

Les documents joints à la
présente attestation sont
conformes au texte,
considéré comme
initialement déposé, de la
demande de brevet
européen qui est spécifiée à
la page suivante.

Patentanmeldung Nr.

Patent application No.

Demande de brevet n°

15386017.6 / EP15386017

The organization code and number of your priority application, to be used for filing abroad under the Paris Convention, is EP15386017.

Der Präsident des Europäischen Patentamts;
Im Auftrag

For the President of the European Patent Office

Le Président de l'Office européen des brevets
p.o.



V. Joseph

Anmeldung Nr:
Application no.: 15386017.6
Demande no :

Anmeldetag:
Date of filing: 09.06.15
Date de dépôt :

Anmelder / Applicant(s) / Demandeur(s):

p2i Limited
Global Headquarters
9-12 North Central
127 Olympic Avenue
Milton Park
Abingdon
Oxfordshire OX14 4SA/GB

Bezeichnung der Erfindung / Title of the invention / Titre de l'invention:

(Falls die Bezeichnung der Erfindung nicht angegeben ist, oder falls die Anmeldung in einer Nicht-Amtssprache des EPA eingereicht wurde, siehe Beschreibung bezüglich ursprünglicher Bezeichnung.

If no title is shown, or if the application has been filed in a non-EPO language, please refer to the description for the original title.

Si aucun titre n'est indiqué, ou si la demande a été déposée dans une langue autre qu'une langue officielle de l'OEB, se référer à la description pour le titre original.)

COATINGS

In Anspruch genommene Priorität(en) / Priority(Priorities) claimed / Priorité(s) revendiquée(s)
Staat/Tag/Aktenzeichen / State/Date/File no. / Pays/Date/Numéro de dépôt:

Am Anmeldetag benannte Vertragsstaaten / Contracting States designated at date of filing / Etats contractants désignées lors du dépôt:

AL AT BE BG CH CY CZ DE DK EE ES FI FR GB GR HR HU IE IS IT LI LT LU LV MC MK MT NL NO PL
PT RO RS SE SI SK SM TR

Coatings

Field of the invention

This invention relates to protective coatings. Specifically, the present invention relates to protective coatings for electronic or electrical devices and components thereof, and methods of forming such coatings. The coatings can protect by being hydrophobic and so resist the ingress of water-based liquid into electronic devices, or they can protect by forming a barrier coating and so provide electrical resistance between the electrical parts of the phone and water based liquid.

Background of the invention

Monounsaturated monomers are used to make barrier coatings using plasma polymerisation processes (see co-pending application).

The perfluoroalkyl chain monomers are also used for generating hydrophobic surfaces from pulsed plasma deposition processes (see WO 9858117 A1).

The power of the plasma initiated polymerisation affects the nature of the polymer produced. The higher average energy inputs of continuous wave plasmas lead to more fragmentation of the monomer, and so the polymer loses structural properties of the monomer. In the case of 1H,1H,2H,2H-perfluorodecyl acrylate (PFAC8), there's less retention of the perfluoroalkyl chain and the contact angle of the surface coating is compromised. Higher plasma energies also lead to more crosslinking. For the lower average energy inputs of pulsed plasmas, there's better retention of monomer structure and less crosslinking. The greater retention of the perfluoro chain under low energy, pulsed plasma conditions leads to best levels of contact angles for the surface coating.

When the perfluoroalkyl chains have eight or more fluorinated carbons (long chain), the polymer made from the monomer has a crystalline structure. When the perfluoroalkyl chains have less than eight fluorinated carbons, the resulting polymer is amorphous and so can be unstable in the presence of water (see "Molecular Aggregation Structure and Surface Properties of Poly(fluoroalkyl acrylate) Thin Films, Macromolecules, 2005, vol 38, p5699-5705)

When long chain perfluoroalkyl polymers are produced by high average power (continuous wave or CW) or low average power (pulsed wave or PW) plasmas, then because of the crystalline structure of the long chains, the polymers are non-stick to the touch and stable in the presence of water.

However, the feel and water stability of shorter chain polymer coatings is affected by the plasma power levels used. For example, when PFAC6 (1H,1H,2H,2H-perfluorooctyl acrylate) is polymerised in low power plasma conditions, the resulting polymer coating can have several disadvantages. For example the coating can cause water drops to spread out a little (slump), be marked by the presence of a water drop on its surface, have a tacky feel, or can be easily smeared (for example on substrates of silicon wafer and ABS plastic).

By increasing the power of the plasma used for polymerisation, the polymer becomes more crosslinked and becomes more resistant to smearing. However, increasing the power has the concomitant effect of decreasing the water contact angle through more monomer fragmentation (as described above). Figure 1 shows the effect of increasing the power to monomer flow ratio for CW plasmas in a 125 litre chamber: at a ratio of 4 W/ μ l/min, the water contact angle is ~85-95 degrees and the coating is tack-free. However, as the ratio drops, the contact angle increases and the occurrence of tacky/smudgy coatings increases too. Figure 2 shows the same effect for pulsed plasma conditions. These results show that the process window for producing tack and smudge-free coatings have a limited plasma processing range and the final coating has a compromised water contact angle.

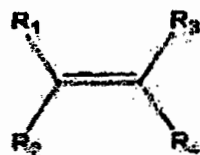
Accordingly, it is an aim to solve one or more of the above-mentioned problems with the prior art coatings.

Statement of Invention

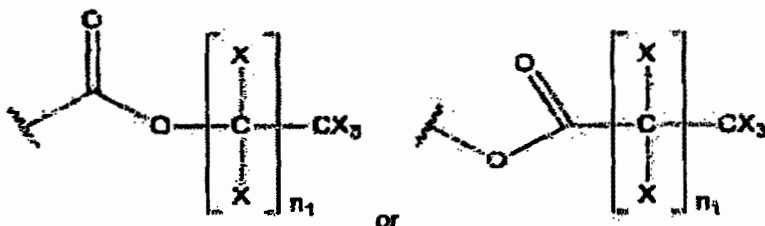
An aspect of the present invention provides an electronic or electrical device or component thereof comprising a protective cross-linked polymeric coating on a surface of the electronic or electrical device or component thereof;

wherein the protective cross-linked polymeric coating is obtainable by exposing the electronic or electrical device or component thereof to a plasma comprising a monomer compound and a crosslinking reagent for a period of time sufficient to allow formation of the protective cross-linked polymeric coating on a surface thereof,

wherein the monomer compound has the following formula:



where R_1 , R_2 and R_4 are each independently selected from hydrogen, optionally substituted branched or straight chain C_1 - C_8 alkyl or halo alkyl or aryl optionally substituted by halo, and R_3 is selected from:



where each X is independently selected from hydrogen, a halogen, optionally substituted branched or straight chain C₁-C₈ alkyl, halo alkyl or aryl optionally substituted by halo; and n₁ is an integer from 1 to 27; and wherein the crosslinking reagent comprises two or more unsaturated bonds attached by means of one or more linker moieties and has a boiling point of less than 500 °C at standard pressure.

High levels of polymer crosslinking (formerly only achievable with high average power continuous wave plasmas) can be achieved by adding a crosslinking molecule to the monomer to produce a cross-linked co-polymer. This has the advantage of increasing the plasma processing range so stable coatings can now be produced in low average energy pulse plasma conditions.

10 The high degree of retention of the hydrophobic monomer structures from the low energy pulsed plasma gives coatings of the co-polymer a good hydrophobic coating (as evidenced by water contact angles) and a coating that is not sticky or smeary to the touch. Thus, these properties make it suitable for the coating of electronic devices to prevent the ingress of water-based liquids from accidental damage. Such coatings typically have a thickness between 1 and 100 nm, preferably 1 and 50 nm.

15 The coating may be described as a liquid repellant layer, typically a water repellant layer. The coating may repel water, an aqueous liquid such as rainwater, or oil.

Alternatively, or in addition to providing a liquid repellent layer, the coating may be a physical barrier layer.

The coating may be a physical barrier i.e. it provides a physical barrier to mass and electron transport.

20 The physical barrier layer restricts diffusion of water, oxygen and ions. When the coating is a physical barrier it typically has a thickness of greater than 50 nm.

The coating is a protective layer i.e. it prevents damage by contact with water or other liquids. The coating may provide its protective functionality by forming a liquid repellent layer and/or a physical barrier layer.

25 The coating is preferably substantially pin-hole free to enable it to provide a physical barrier. Preferably $\Delta Z/d < 0.15$, where ΔZ is the average height variation on an AFM line scan in nm (as shown in Figure 3) and d is coating thickness in nm.

The value of $\Delta Z/d$ tells us to what extent defects/voids on the surface of the coating extend into the coating, i.e. the percentage value of the depth of defect over total coating thickness. For example,
30 $\Delta Z/d = 0.15$ means that the voids on the surface only extend down to 15% of the coating thickness. A coating with a $\Delta Z/d < 0.15$ is defined herein as being substantially pinhole free. If voids are bigger than this, the desired functionality is unlikely to be achieved.

The coating is preferably conformal, which means that it takes the 3D shape of the electronic or electrical device or component thereof and covers substantially an entire surface of the device. This has the advantage of ensuring that the coating has sufficient thickness to give optimal functionality over an entire surface of the device or component. The meaning of the term "covers substantially an entire surface" will depend to some extent on the type of surface to be covered. For example, for some components, it may be necessary for there to be complete coverage of the surface in order for the component to function after submersion in water. However, for other components or housings, small gaps in coverage may be tolerated.

The coating may have a thickness of 50 to 10,000nm, optionally 50 to 8000nm, 100 to 5000nm, preferably 250nm – 5000nm, most preferably 250nm – 2000nm.

The coating may be electrically insulating and be sufficiently compliant that electrical connectors can be joined to the electronic or electrical device or component thereof and an electrical connection made between the electrical connectors and electronic or electrical device or component thereof without the requirement to first remove the coating. In this case, the force exerted by the electrical connector on the coating is sufficient to alter the structure of or even break through the coating local to the electrical connector, allowing the electrical connection to be made. Electrical connectors can typically be joined to the electronic or electrical device or component in this way for coating thicknesses of under 5000nm, and for high performance coatings below 2000nm.

The electronic or electrical device or component thereof can typically withstand immersion in up to 1m of water for over 30 minutes without failure or corrosion whilst power applied to the electronic or electrical device or component. The effectiveness of the coating can be determined by measuring its electrical resistance at a fixed voltage when submerged in water for a set time period; for example, applying a voltage of 8V to the coating of a device which is submerged in water for 13 minutes. Coatings having a resistance value of 1×10^7 Ohms or higher in this test are effective barrier coatings and the coated electronic or electrical device or component thereof will pass successfully an IPX7 test. The IPX7 test is the Ingress Protection Marking which classifies and rates the degree of protection provided against water. In the IPX7 test for phones, the device is immersed in water under defined conditions of pressure and time (up to 1 m of submersion) for a duration of 30 minutes. The device has to be powered on during testing and functional after 24 hrs.

In one embodiment, the coating is electrically insulating and has a thickness of less than 1 micron and wherein a force of 5- 20g applied to the coating using a round probe with 1 mm diameter allows an electrical connection to be made with the electronic or electrical device or component thereof in the local area where the force has been applied.

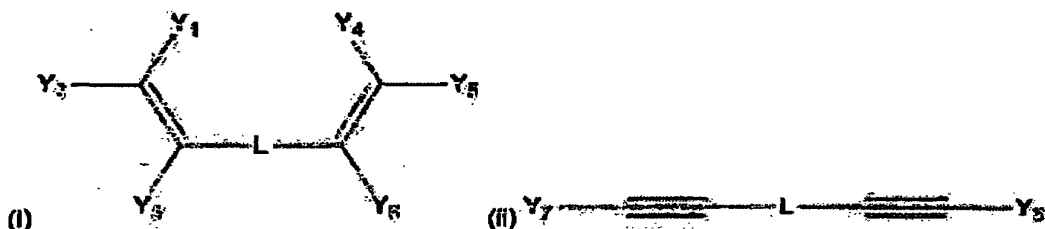
In another embodiment, the coating is electrically insulating and has a thickness of 1-2.5 microns and wherein a force of 20-100g applied to the coating using a round probe with 1 mm diameter allows an electrical connection to be made in the local area of the coating where the force has been applied.

The coating may have a higher density than that of the corresponding monomer from which it is formed. For example, the increase in density may be at least 0.1 g/cm^3 . The increase in density is explained by the highly crosslinked coating. The high density of the coating improves the barrier properties of the coating.

- 5 The coating may form a surface defined by a static water contact angle (WCA) of at least 70° . A coating with a WCA of at least 90° is a liquid repellent, typically a water repellent layer. For fluorinated polymers, the coating may have a static water contact angle of at least 100° . The contact angle of a liquid on a solid substrate gives an indication of the surface energy which in turn illustrates the substrate's liquid repellence. Contact angles were measured on a VCA Optima contact angle analyser, using $3 \mu\text{l}$ droplets of deionised water at room temperature.

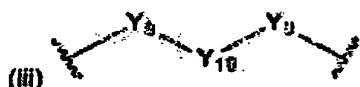
Crosslinker

- A basic requirement for the cross-linking reagent is the presence two or more unsaturated bonds, for example, -C=C- or alkyne groups. The unsaturated bonds are attached by one or more linker moieties. The linker moiety is not especially limited provided it joins together the two or more unsaturated bonds. The cross-linking reagent must have a boiling point of less than 500°C , preferably from -10 to 300°C , optionally 180 to 270°C , most preferably 205 to 260°C at standard pressure. It is a preference, but not a requirement, that the cross-linking reagent is not unduly hazardous for use in plasma processing, that is to say it can be used in a production environment where low levels of vapour do not present any significant health and safety issues (e.g. strongly oxidising, explosive, toxic, or has an offensive odour (e.g. stench reagent)). The crosslinking reagent preferably has one of the following structures:



- where Y_1 , Y_2 , Y_3 , Y_4 , Y_5 , Y_6 , Y_7 and Y_8 are each independently selected from hydrogen, optionally substituted cyclic, branched or straight chain $\text{C}_1\text{--C}_8$ alkyl or aryl; and L is a linker moiety.

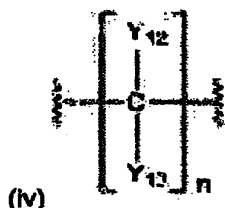
Most preferably L has the following formula:



- where each Y_8 is independently selected from, a bond, -O- , -O-C(O)- , -C(O)-O- , $\text{-Y}_{11}\text{-O-C(O)-}$, $\text{-C(O)-O-Y}_{11}\text{-}$, $\text{-OY}_{11}\text{-}$, and $\text{-Y}_{11}\text{-O-}$, where Y_{11} is an optionally substituted cyclic, branched or straight chain $\text{C}_1\text{--C}_8$ alkylene; and

Y_{10} is selected from optionally substituted cyclic, branched or straight chain C_1-C_8 alkylene and a siloxane containing group.

In one most preferred embodiment Y_{10} has the following formula:



5

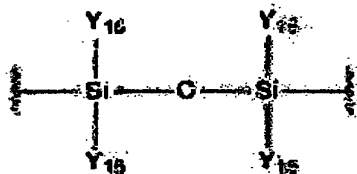
wherein each Y_{12} and Y_{13} is independently selected from H, halo, optionally substituted cyclic, branched or straight chain C_1-C_8 alkyl, or $-OY_{14}$, where Y_{14} is selected from optionally substituted branched or straight chain C_1-C_8 alkyl or alkenyl, and n is an integer from 1 to 10.

Optionally, each Y_{12} is H and each Y_{13} is H, i.e. Y_{10} is a linear alkylene chain. For this embodiment Y_9 is preferably a vinyl ester or vinyl ether group.

Optionally, each Y_{12} is fluoro and each Y_{13} is fluoro, i.e. Y_{10} is a linear perfluoroalkylene chain.

Typically, n is from 4 to 6.

In another embodiment Y_{10} has the following formula:

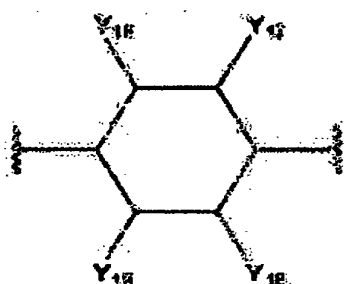


15 (v)

wherein each Y_{16} is independently selected from optionally substituted branched or straight chain C_1-C_8 alkyl.

In one embodiment each Y_{16} is methyl, and each Y_9 is a bond.

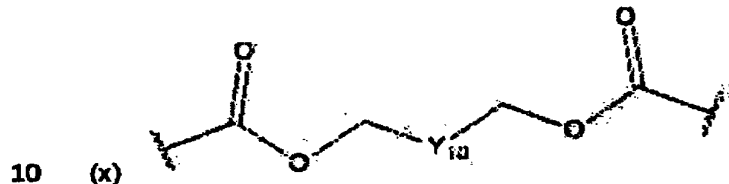
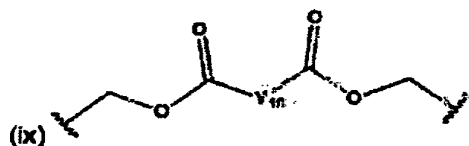
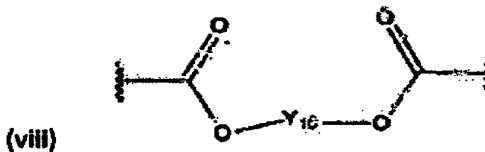
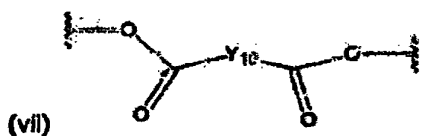
In a further embodiment Y_{10} has the following formula:



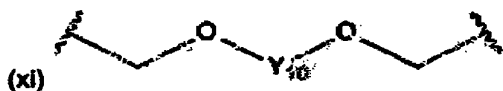
(vi)

wherein Y_{16} to Y_{19} are each independently selected from H and optionally substituted branched or straight chain C_1 - C_8 alkyl or alkenyl. A preferred alkenyl group is vinyl. Optionally Y_{18} is H or vinyl, and Y_{16} , Y_{17} and Y_{19} are each H. In one embodiment each of Y_{16} to Y_{19} is H. In another embodiment Y_{18} is vinyl, and Y_{16} , Y_{17} and Y_{19} are each H.

In preferable embodiments for compound (I), L has one of the following structures:



In another embodiment for compound (I) L has one of the following structures:



For L according structure (vii), Y_{10} is preferably an alkylene chain or a cycloalkylene, such as those shown in structures iv) and vi) above. The alkylene chain may be a straight chain alkylene chain.

When Y_{10} is a cycloalkylene, cyclohexylene is preferred, most preferably 1,4 cyclohexylene.

For L according to structure (viii), Y_{10} is preferably structure (iv), i.e. an alkylene or fluoroalkylene chain.

For L according to structure (ix), Y_{10} is preferably cycloalkylene, such as the cyclohexylene according to structure (vi).

- 5 For L according to structure (x), Y_{10} is preferably structure (iv) wherein each Y_{12} and Y_{13} is F, i.e. a perfluoroalkylene chain.

For L according to structure (xi) or structure (xii) Y_{10} is preferably alkylene or cycloalkylene. Optionally the alkylene or cycloalkylene may be substituted with one or more vinyl groups or alkenyl ether groups, preferably one or more vinyl ether groups.

- 10 When each Y_9 is a bond, each Y_{10} may be any of structures (iv), (v) and (vi). Preferably Y_{10} is a straight chain alkylene such that the crosslinking reagent is a diene, such as a heptadiene, octadiene, or nonadiene, most preferably 1,7-octadiene.

- When each Y_9 is O, each Y_{10} is preferably a branched or straight chain C_1 - C_8 alkylene, preferably a straight chain alkylene, most preferably a C_4 straight chain alkylene, i.e. the crosslinking reagent is 1,4-butanediol divinyl ether
- 15

It will be understood that each Y_9 group can be combined with any other Y_9 group and Y_{10} group to form the crosslinking reagent.

- The skilled person will be aware of possible substituents for each of the cyclic, branched or straight chain C_1 - C_8 alkylene groups mentioned above. The alkylene groups may be substituted at one or more positions by a suitable chemical group. Halo substituents are preferred, with fluoro substituents most preferred. Each C_1 - C_8 alkylene group may be a C_1 - C_3 , C_2 - C_6 , or C_6 - C_8 alkylene group.
- 20

Particularly preferred embodiments of the crosslinker have alkyl chains for Y_{10} and vinyl ester or vinyl ether groups on either side.

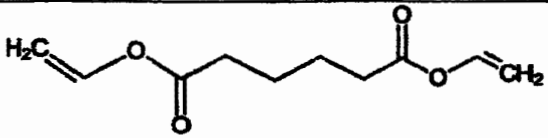
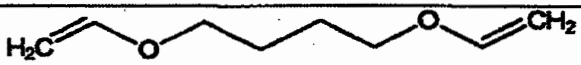
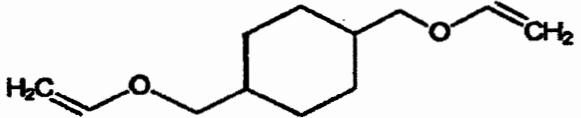

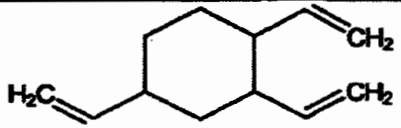
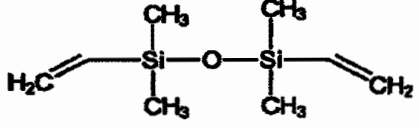
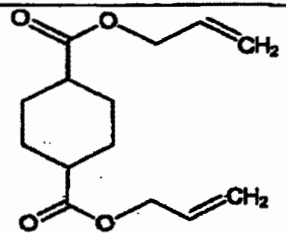
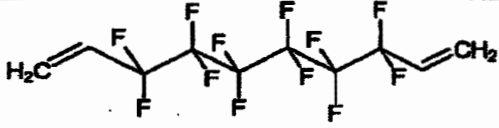
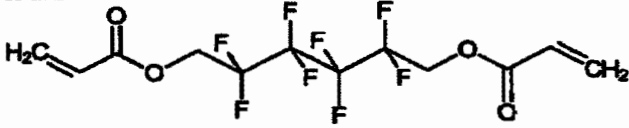
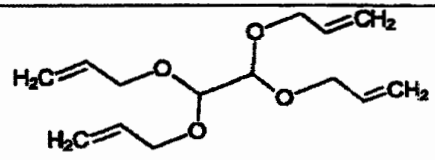
- A particularly preferred cross-linking reagent is divinyl adipate. (a divinyl ester)
- 25

Another preferred cross-linking reagent is 1,4-butanediol divinyl ether (a divinyl ether)

- For the most preferred embodiments the crosslinking reagent is selected from Divinyl Adipate (DVA), 1,4 Butanediol divinyl ether (BDVE), 1,4 Cyclohexanedimethanol divinyl ether (CDDE), 1,7 -Octadiene (17OD), 1,2,4 -Trivinylcyclohexane (TVCH), 1,3-Divinyltetramethyldisiloxane (DVTMDS), Diallyl 1,4-cyclohexanedicarboxylate (DCHD), 1,6-Divinylperfluorohexane (DVPFH), 1H,1H,6H,6H-Perfluorohexanediol diacrylate (PFHDA) and glyoxal bis (diallyl acetal) (GBDA).
- 30

For the alkyne crosslinking reagents according to compound (ii), L is preferably selected from a branched or straight chain C_1 - C_8 alkylene or an ether group. L may be a C_3 , C_4 , C_5 , or C_6 alkylene,

preferably a straight chain alkylene. Chemical structures for the particularly preferred crosslinking reagents are set out below in Table 1:

| | |
|---|--|
| Divinyl Adipate (DVA) |  |
| 1,4 Butanediol divinyl ether (BDVE) |  |
| 1,4 Cyclohexanedimethanol divinyl ether (CDDE) |  |
| 1,7-Octadiene (17OD) |  |
| 1,2,4-Trivinylcyclohexane (TVCH) |  |
| 1,3-Divinyltetramethyldisiloxane (DVTMDS) |  |
| Diallyl 1,4-cyclohexanedicarboxylate (DCHD) |  |
| 1,6-Divinylperfluorohexane (DVPFH) |  |
| 1H,1H,6H,6H-Perfluorohexanediol diacrylate (PFHDA) |  |
| GBDA |  |


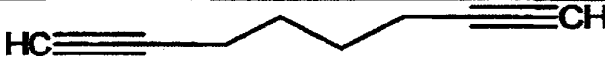
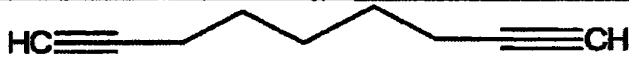
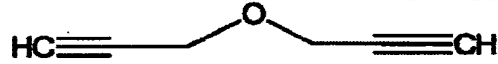
| | |
|-----------------|--|
| 1,6-heptadiyne |  |
| 1,7-heptadiyne |  |
| 1,8-heptadiyne |  |
| Propargyl ether |  |

Table 1

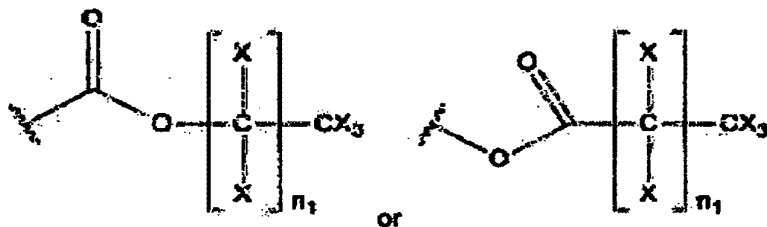
Monomer Compound

The monomer compound has the following formula:



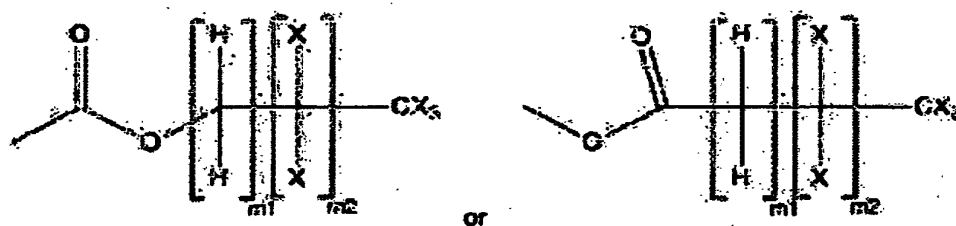
5

where R_1 , R_2 and R_4 are each independently selected from hydrogen, optionally substituted branched or straight chain C_1 - C_8 alkyl or halo alkyl or aryl optionally substituted by halo, and R_3 is selected from:



where each X is independently selected from hydrogen, a halogen, optionally substituted branched or straight chain C_1 - C_8 alkyl, halo alkyl or aryl optionally substituted by halo; and n_1 is an integer from 1 to 27. Preferably, n_1 is from 1 to 12. Optionally n_1 is from 4 to 12, optionally 6 to 8.

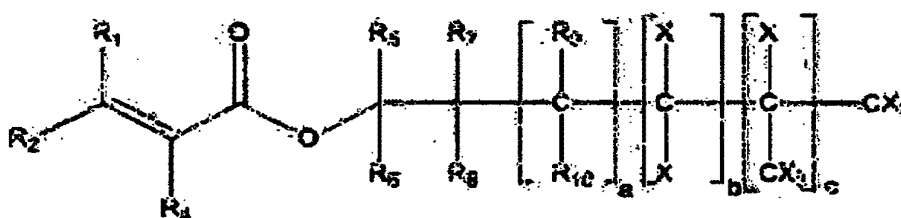
In a preferred embodiment R_3 is selected from:



where m_1 is an integer from 0 to 13 and each X is independently selected from hydrogen, a halogen, optionally substituted branched or straight chain C_1 - C_6 alkyl, halo alkyl or aryl optionally substituted by halo; and m_2 is an integer from 2 to 14;

In a particularly preferred embodiment the monomer is a compound of formula I(a):

I(a)

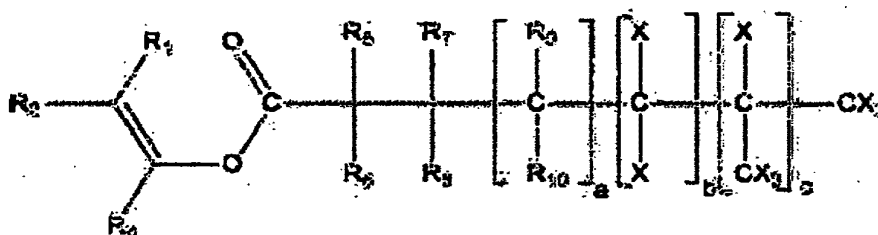


wherein each of R_1 , R_2 , R_4 , and R_5 to R_{10} is independently selected from hydrogen or an optionally substituted C_1 - C_6 branched or straight chain alkyl group; each X is independently selected from hydrogen or halogen; a is from 0-10; b is from 2 to 14; and c is 0 or 1;

or

the monomer is a compound of formula I(b):

I(b)



wherein each of R_1 , R_2 , R_4 , and R_5 to R_{10} is independently selected from hydrogen or an optionally substituted C_1 - C_6 branched or straight chain alkyl group; each X is independently selected from hydrogen or halogen; a is from 0-10; b is from 2 to 14; and c is 0 or 1.

The halogen may be chlorine or bromine, but is preferably fluorine for compliance with RoHS regulations (Restriction of Hazardous Substances).

a is from 0 to 10, preferably from 0 to 6, optionally 2 to 4, most preferably 0 or 1. b is from 2 to 14, optionally from 2 to 10, preferably 3 to 7.

- 5 Each of R_1 , R_2 , R_4 , and R_5 to R_{10} is independently selected from hydrogen or a C_1 - C_6 branched or straight chain alkyl group. The alkyl group may be substituted or unsubstituted, saturated or unsaturated. When the alkyl group is substituted, the location or type of the substituent is not especially limited provided the resultant polymer provides an appropriate liquid repellent and/or barrier layer. The skilled person would be aware of suitable substituents. If the alkyl group is substituted, a
- 10 preferred substituent is halo, i.e. any of R_1 , R_2 , R_4 , and R_5 to R_{10} may be haloalkyl, preferably fluoro alkyl. Other possible substituents may be hydroxyl or amine groups. If the alkyl group is unsaturated it may comprise one or more alkene or alkyne groups.

- 15 Each of R_1 , R_2 , R_4 , and R_5 to R_{10} may be independently selected from hydrogen, methyl, ethyl, n-propyl, isopropyl, n-butyl, isobutyl, tert-butyl, sec-butyl, n-pentyl, neopentyl, n-hexyl, isohexyl, and 3-methylpentyl.

In a preferred embodiment each of R_1 , R_2 , R_4 , and R_5 to R_{10} is independently selected from hydrogen or methyl.

- 20 In a preferred embodiment, a and c are each independently 0 or 1; and b is from 3 to 7.

In one preferred embodiment each X is H. In an alternative preferred embodiment each X is F.

Optionally R_1 and R_2 are both hydrogen.

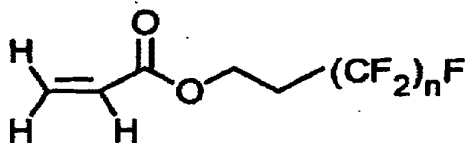
Optionally R_4 is hydrogen or methyl. Preferably R_1 and R_2 are both hydrogen and R_4 is hydrogen or methyl.

- 25 Optionally R_9 is hydrogen and R_{10} is branched or straight chain C_1 - C_6 alkyl group. In a preferred embodiment R_{10} is methyl.

In one embodiment each of R_3 to R_8 is hydrogen.

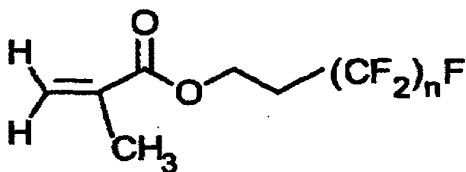
- 30 In one embodiment each of R_1 , R_2 , R_4 , and R_5 to R_{10} is hydrogen, each X is H, a=0 and c=0.

In a particularly preferred embodiment the monomer compound has the following formula:



where n is from 2 to 10.

In another preferred embodiment the monomer compound has the following formula:

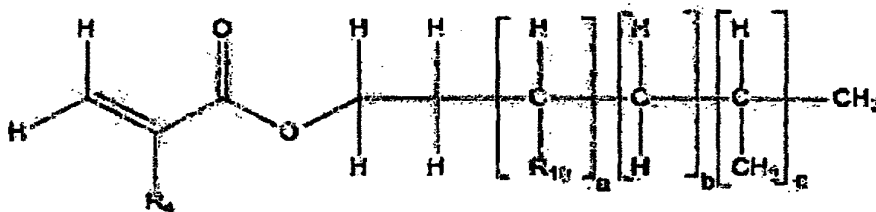


5 where n is from 2 to 10.

The monomer compound may be selected from 1H,1H,2H,2H-perfluorohexyl acrylate (PFAC4), 1H,1H,2H,2H-perfluorooctyl acrylate (PFAC6), 1H,1H,2H,2H-perfluorodecyl acrylate (PFAC8) and 1H,1H,2H,2H-perfluorododecyl acrylate (PFAC10).

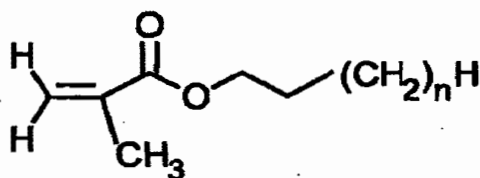
10 The monomer compound may be selected from 1H,1H,2H,2H-perfluorohexyl methacrylate (PFMAC4), 1H,1H,2H,2H-perfluorooctyl methacrylate (PFMAC6) and 1H,1H,2H,2H-perfluorodecyl methacrylate (PFMAC8).

The monomer compound of formula I(a) may have the following formula:



wherein a and c are each independently 0 or 1, $b = 3-7$ and n is 4 to 10, where $n = a + b + c + 1$.

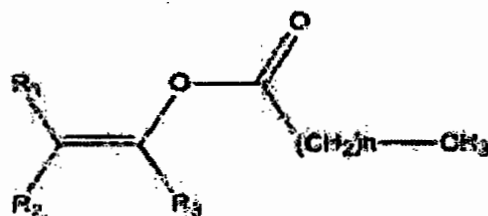
15 The monomer compound of formula I(a) may have the following formula:



where n is 2 to 12.

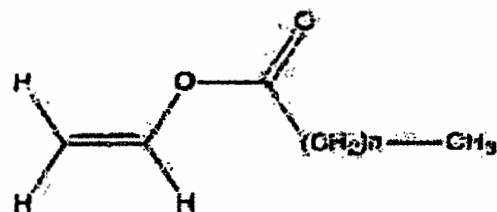
The monomer compound may be selected from ethyl hexyl acrylate, hexyl acrylate, decyl acrylate, lauryl dodecyl acrylate and iso decyl acrylate.

- 5 The monomer may have the following formula:



where n is from 4 to 14.

The monomer may have the following formula:



10

where n is from 4 to 14.

In an additional aspect, the present invention provides a method for treating an electronic device or component thereof comprising:

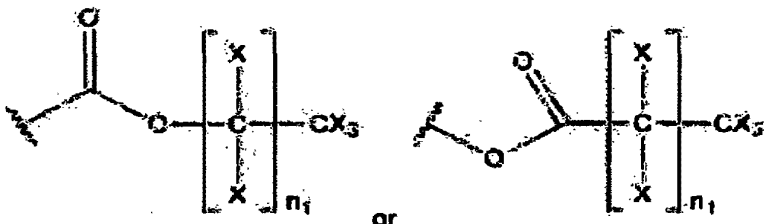
exposing the electronic or electrical device or component thereof to a plasma comprising a monomer compound and a crosslinking reagent for a period of time sufficient to allow a protective cross-linked polymeric coating to form on a surface of the electronic or electrical device or component thereof;

15

wherein the monomer compound has the following formula:



where R_1 , R_2 and R_4 are each independently selected from hydrogen, optionally substituted branched or straight chain C_1 - C_8 alkyl or halo alkyl or aryl optionally substituted by halo, and R_3 is selected from:



- 5 where each X is independently selected from hydrogen, optionally substituted branched or straight chain C_1 - C_8 alkyl, halo alkyl or aryl optionally substituted by halo; and n_1 is an integer from 1 to 27; and wherein the crosslinking reagent comprises two or more unsaturated bonds and has a boiling point of less than 500 °C at standard pressure.

10 The monomer compound and the crosslinking reagent utilised in the method are described in further detail above.

Typically the electronic or electrical device or component thereof is placed in a plasma deposition chamber, a glow discharge is ignited within said chamber, and a voltage applied as a pulsed field. .

- 15 Preferably the applied voltage is at a power of from 30 to 800W. Optionally the voltage is pulsed in a sequence in which the ratio of the time on/time off is in the range of from 0.001 to 1, optionally 0.002 to 0.5. For example, time on may be 10-500 μ s, preferably 35-45 μ s, or 30-40 μ s, such as about 36 μ s and time off may be from 0.1 to 30 ms, preferably 0.1 to 20 ms, optionally from 5 to 15 ms, for example 6ms. Time on may be 35 μ s, 40 μ s, 45 μ s. Time off may be 0.1, 1, 2, 3, 6, 8, 10, 15, 20, 25 or 30 ms.

- 20 The term pulsed may mean that the plasma cycles between a state of no (or substantially no) plasma emission (off-state) and a state where a particular amount of plasma is emitted (on-state).

Alternatively, pulsed may mean that there is continuous emission of plasma but that the amount of plasma cycles between an upper limit (on-state) and lower limit (off-state).

- 25 In a further embodiment, the invention resides in a method of forming a coating on an electronic or electrical device or component thereof as defined above, the method comprising: exposing said substrate in a chamber to a plasma comprising a monomer compound, preferably a continuous plasma, for a sufficient period of time to allow a protective polymeric coating to form on the substrate,

wherein during exposure of the substrate the continuous plasma has a power density of at least 2W/litre, preferably 20W/litre.

Optionally the voltage is applied as a pulsed field for a period of from 30 seconds to 90 minutes. Optionally the voltage is applied as a pulsed field for from 5 to 60 minutes. Optionally in a preliminary step, a continuous power plasma is applied to the electronic or electrical device or component thereof. The preliminary step may be conducted in the presence of an inert gas.

Before the crosslinker and/or the monomer compound enter the deposition chamber each may be in the form of a gas, liquid or a solid (for example a powder) at room temperature. However, it is the preferred that the crosslinking reagent and the monomer compound are both liquid at room temperature, and most preferably that the monomer and crosslinker liquids are miscible.

The monomer compound and/or the crosslinker will suitably be in a gaseous state in the plasma. The plasma may simply comprise a vapour of the monomer compound and the crosslinker. Such a vapour may be formed in-situ, with the compounds being introduced into the chamber in liquid form. The monomer may also be combined with a carrier gas, in particular, an inert gas such as helium or argon.

In preferred embodiments, the monomer and/or the crosslinker may be delivered into the chamber by way of an aerosol device such as a nebuliser or the like, as described for example in WO2003/097245 and WO03/101621, the content of which is incorporated herein by reference. In such an arrangement a carrier gas may not be required, which advantageously assists in achieving high flow rates.

In one embodiment the monomer compound and/or the crosslinking reagent are in gaseous form and are fed into the plasma at a rate of from 10-5000 mg/minute depending on chamber volume, whilst the pulsed voltage is applied.

Optionally the plasma is created with an average power of from 0.001 to 40W/litre.

The crosslinker may be miscible with the monomer and so introduced together or separately into the plasma chamber. Or the crosslinker may be immiscible with the monomer and introduced separately into the plasma chamber. In this context, the term "miscible" means that the crosslinker is soluble in the monomer, and when mixed they form a solution of uniform composition. The term "immiscible" is used to mean that the crosslinker is only partly soluble or insoluble in the monomer, and so either forms an emulsion or separates out into two layers.

The crosslinking reagent is preferably present in an amount of from 10 to 60 (v/v) %, optionally 20 to 40 (v/v) %, optionally 25 to 30 (v/v) %, optionally 30 to 50 (v/v)% of the total volume of monomer compound and crosslinking reagent, depending on the specific crosslinker. The skilled person would understand that the amount will vary to some extent depending on whether the coating is required to

be liquid repellent or provide a barrier to mass and electron transport. The skilled person would understand that the (v/v) percentages are those which give a stable crosslinked polymer coating and the highest water contact angle.

Electronic or electrical device or component thereof

5

Although the invention is of benefit in the context of a wide variety of substrates, the substrate may, in all aspects of the invention, advantageously be an electronic substrate.

10

In some embodiments of the invention, the electronic substrate may comprise an electronic or electrical device, i.e. any piece of electrical or electronic equipment. Non-limiting examples of electrical and electronic devices include communications devices such as mobile phones, smartphones and pagers, radios, and sound and audio systems such as loudspeakers, microphones, ringers or buzzers, hearing aids, personal audio equipment such as personal CD, tape cassette or MP3 players, televisions, DVD players including portable DVD players, video recorders, digi and other set-top boxes such as Sky, computers and related components such as laptop, notebook, tablet, phablet or palmtop computers, personal digital assistants (PDAs), keyboards, or instrumentation, games consoles in particular hand-held playstations and the like, data storage devices, outdoor lighting systems or radio antenna and other forms of communication equipment.

15

20

In preferred embodiments of the invention, the substrate may comprise or consist of an electronic component, e.g. a printed circuit board (PCB), a printed circuit board array (PCBA), a transistor, resistor, or semi-conductor chip. The electronic component may thus be an internal component of an electronic device, e.g. a mobile phone. The coatings of the invention are particularly valuable in preventing electrochemical migration in such components.

25

In all aspects of the invention, the precise conditions under which the protective polymeric coating is formed in an effective manner will vary depending upon factors such as, without limitation, the nature of the monomer compound, the crosslinking reagent, the substrate, as well as the desired properties of the coating. These conditions can be determined using routine methods or, preferably, using the techniques and preferred features of the invention described herein, which work in particular synergy with the invention.

30

The present invention will now be further described with reference to the following non-limiting examples and the accompanying illustrative drawings, of which:

Figure 1 illustrates shows the effect of increasing the power to monomer flow ratio for CW plasmas in a 3 litre chamber in a prior art process.

Figure 2 shows the same effect for pulsed plasma conditions.

Figure 3 shows a tapping mode image over $10 \times 10 \mu\text{m}^2$ field of view of a specimen example (thickness $d=1230 \text{ nm}$) prepared according to Example 1 (left hand side) and a contour line plot showing the data used for calculation of RMS roughness (right hand side). The ΔZ value indicated on the plot has been taken over an area of the graph that represents the majority of the coating. Peaks that lie above the ΔZ range indicate large particles and troughs that fall below the ΔZ range show voids or pinholes in the coating. The width of the peaks also gives an indication of the particle size.

Figure 4 illustrates the effect of adding the divinyl adipate crosslinker to perfluorooctyl acrylate on water contact angle from a process on a 125 Litre chamber.

Figure 5 shows the effect of adding crosslinkers in accordance with the invention on the electrical resistance per nm of the coatings.

Figure 6 shows a tapping mode image of a 90 nm thick coating, prepared as described in Example 3, over $2 \times 2 \mu\text{m}^2$ field of view (top left), a representative contour line indicating height variation (z-axis) of the coating (top right) and a phase image indicating full substrate coverage (bottom left); RMS roughness of the coating is 1.65 nm and $\Delta z/d = 0.05$

Example 1

Example of plasma polymerising PFAC6 with no crosslinker in a 3 litre chamber

PFAC6 was polymerised in a 3 litre glass chamber using a continuous plasma process. In the experimental matrix, the continuous plasma was produced using RF power levels of 50, 100 and 200 Watts. The process pressure was run at 30, 60 and 90 mTorr and the monomer flow rate run at 50, 100 and 200 microlitres of liquid per minute. The duration of the process was either 10 or 40 minutes. The coated substrate was silicon wafer and whether the coating was tacky or not was determined by wiping the substrate with a finger and determining by eye if the coating had smeared. The results in Figure 1 indicate that for lower CW powers, the coating could easily be smeared (i.e. tacky), but by increasing the power to flow ratio, the coating became resistant to smearing (i.e. non-tacky) and this is expected to be due to additional crosslinking caused by more monomer fragmentation. The water contact angle was also seen to decline with increasing power to flow ratio, and this too is indicative of more power increasing the level of monomer fragmentation.

The same 3 litre chamber was used to also look at the effect of power to flow ratios with pulsed plasma polymerisation. In these experiments, the peak power was either 250 or 500 W. The RF power feed was pulsed with an on time of 35 microseconds and off times of 5.5 and 1 millisecond. Process pressure was either 20, 35, 60 or 80 mTorr. The monomer flow was either 50 or 200 microlitres of liquid per minute. Figure 2 shows that low power to flow conditions can generate coatings that can be smeared (tacky) and only by increasing the power could the smear be reduced. This too had a concomitant decline in water contact angle.

The results from Figures 1 and 2 show that plasma processing PFAC6 on its own could only lead to smear-free coatings when the powers were high enough to decrease the other desirable property of the water contact angle. This illustrates the need to add a crosslinker to prevent the coating smearing and maintain the water contact angle.

5 Example 2

Examples of Perfluoro Alkyl Acrylate co-polymerisation with Crosslinker to show Improvements in electrical resistance

Perfluorooctyl acrylate monomer was mixed with a single crosslinker from the following list:

VINYL ESTERS: Divinyl adipate (DVA)

10 VINYL ETHERS: 1,4-Butanediol divinyl ether (BDVE); 1,4 Cyclohexanedimethanol divinyl ether (CDDE)

DI- or TRI- VINYLS: 1,7-Octadiene (1,7-OD); 1,2,4-Trivinylcyclohexane (TVCH);

DI VINYL with alkyl fluoro group: 1,6-divinylperfluorohexane (DVPFH)

DI VINYL with silicon group: 1,3-Divinyl tetramethyldisiloxane (DVTMDS)

15 DI VINYL with cyclic ring and carboxylate groups: Diallyl 1,4-cyclohexanedicarboxylate (DCHC)

DI ACRYLATE: 1H1H,6H,6H-perfluorohexanediol diacrylate (PFHDA)

The crosslinker was mixed with the PFAC6 as a percentage by volume for the following percentages: 20, 40, 60, 80, and 100%.

20 Plasma initiated polymerisation reactions were carried out in a 3 litre glass plasma chamber. The substrates were test circuit boards. The PFAC6/crosslinker mixture was introduced at a rate of 0.04 – 0.06 ul/min and the process pressure was 40 mTorr. The process plasma consisted of a 1 minute continuous plasma (CW) step of 50W followed by 10 - 20 minute pulsed plasma (PW) step of 50W and RF power being delivered by a pulse sequence with a duty cycle of 6.9%. The coated circuit boards were immersed in tap water and an 8V potential was applied for 13 minutes. The final current

25 readings, together with coating thickness measurements, were used to calculate the electrical resistance per nm of coating. Figure 5 below shows the effect of adding a range of crosslinkers on the electrical resistance per nm of the barrier coatings. Figure 5 clearly shows the improvement in resistance that can be gained by adding the crosslinker to the PFAC6 monomer. Furthermore, all of the crosslinkers tested gave non-smear/non-tacky coatings with concentration levels of 20% and

30 above, with the exceptions of DVTMDS, DCHD, PFHDA and DVPFH which were non-smear at levels of 40% and above.

Example 3

Example of crosslinker concentration on water repellent coating.

Water repellent coatings were prepared in a 125 litre volume chamber using PFAC6 with different levels of divinyl adipate (DVA) and using helium as a carrier gas. The deposition process consisted of a 3 minute CW step with 300W power and a pulsed step with 150W power and an RF pulse duty cycle of 0.018%. Silicon wafer was used as the test substrates and the contact angle of the coated wafer was determined by applying a 3ul drop of deionised water onto the coated wafer and measuring the contact angle using a VCA Optima (AST products) with image analysis software. The variation of contact angle with (v/v)% DVA crosslinker is shown in Figure 4. It was also noted that for the 10 (v/v) % DVA concentration, the water drop left a mark on the coated wafer where it's circumference had been. This indicated that a higher level of crosslinker was required to give a more stable coating. This observation, in combination with the results in figure 4, suggest an optimum range of 20 – 40 (v/v) % DVA crosslinker, though this is likely to vary, typically within the range of from 10-60 (v/v) % for different monomers and crosslinkers and different chamber sizes.

Pulsed plasma polymerisation of perfluoro alkyl acrylate in the presence of divinyl adipate (DVA) was carried out in a 125 litre chamber. Figure 4 shows the effect of adding different liquid volume percentages of DVA crosslinker to perfluorooctyl acrylate on water contact angle on a silicon wafer substrate.

Example 4

Example of AFM measurement of monomer/crosslinker co-polymer coating

A barrier-style coating was prepared in a 22 litre volume chamber using PFAC6 with 10% DVA. The deposition process consisted of a 1 minute CW step with a CW power to flow ratio of 3.9 (W/microliter/min), and a pulsed step with a PW power/monomer flow ratio of 0.28 (W/microlitres/min). Figure 6 shows the representative topographical and phase contrast images were obtained from all the samples. High spatial resolution images show mainly the structures of areas between the raised features. The RMS roughness of the coating is 1.65 nm and the $\Delta z/d = 0.05$. As the $\Delta z/d$ value is < 0.15, then this indicates that the physical layer is substantially pinhole free.

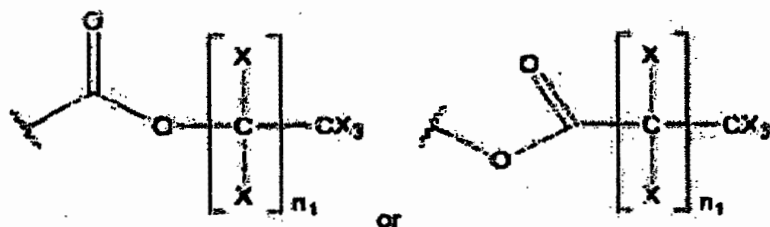
Claims:

1. An electronic or electrical device or component thereof comprising a protective cross-linked polymeric coating on a surface of the electronic or electrical device or component thereof;
 wherein the protective cross-linked polymeric coating is obtainable by exposing the electronic or electrical device or component thereof to a plasma comprising a monomer compound and a crosslinking reagent for a period of time sufficient to allow formation of the protective cross-linked polymeric coating on a surface thereof;

wherein the monomer compound has the following formula:

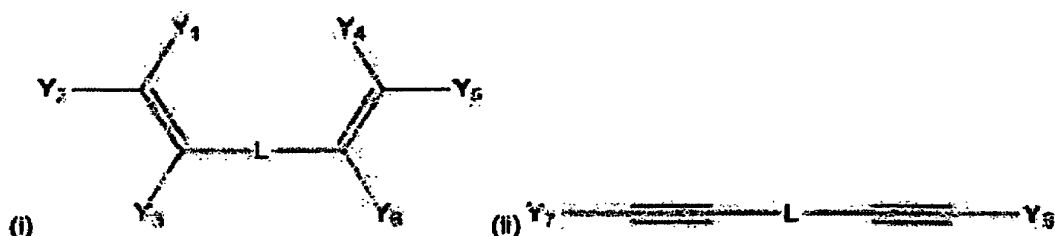


- 10 where R_1 , R_2 and R_3 are each independently selected from hydrogen, optionally substituted branched or straight chain C_1 - C_8 alkyl or halo alkyl or aryl optionally substituted by halo, and R_3 is selected from:



- 15 where each X is independently selected from hydrogen, a halogen, optionally substituted branched or straight chain C_1 - C_8 alkyl, halo alkyl or aryl optionally substituted by halo; and n_1 is an integer from 1 to 27; and wherein the crosslinking reagent comprises two or more unsaturated bonds attached by means of one or more linker moieties and has a boiling point of less than 500 °C at standard pressure.

2. An electronic or electrical device or component thereof according to claim 1, wherein the protective cross-linked polymeric coating is a physical barrier to mass and electron transport.
- 20 3. An electronic or electrical device or component thereof according to claim 1 or claim 2, wherein the protective cross-linked polymeric coating forms a liquid repellent surface defined by a static water contact angle (WCA) of at least 90°.
4. An electronic or electrical device or component thereof according to any preceding claim, wherein the crosslinking reagent has one of the following structures:



where Y_1 , Y_2 , Y_3 , Y_4 , Y_5 , Y_6 , Y_7 and Y_8 are each independently selected from hydrogen, optionally substituted cyclic, branched or straight chain C_1 - C_8 alkyl or aryl; and L is a linker moiety.

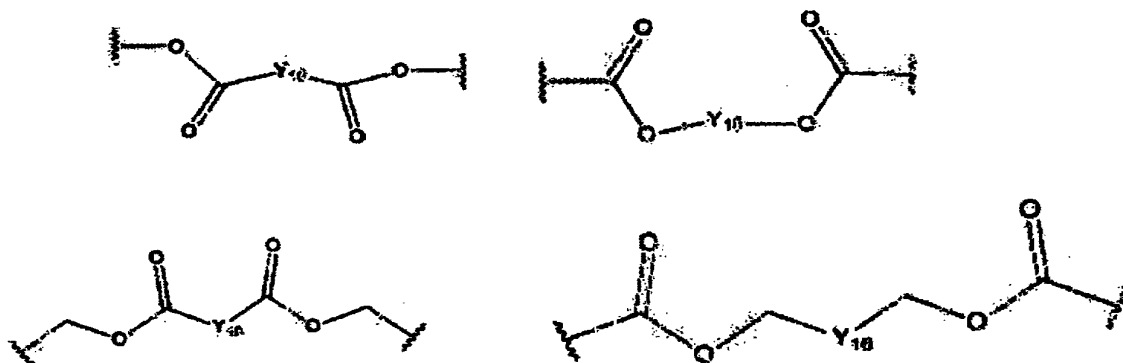
5. An electronic or electrical device or component thereof according to claim 4, wherein for compound (i) L has the following formula:



each Y_9 is independently selected from, a bond, $-O-$, $-O-C(O)-$, $-C(O)-O-$, $-Y_{11}-O-C(O)-$, $-C(O)-O-Y_{11}-$, $-OY_{11}-$, and $-Y_{11}O-$, where Y_{11} is an optionally substituted cyclic, branched or straight chain C_1 - C_8 alkylene; and

Y_{10} is selected from optionally substituted cyclic, branched or straight chain C_1 - C_8 alkylene and a siloxane group.

6. An electronic or electrical device or component thereof according to claim 5, wherein for compound (i) L has one of the following structures:



7. An electronic or electrical device or component thereof according to claim 5, wherein for compound (i) L has one of the following structures:



8. An electronic or electrical device or component thereof according to any of claims 5 to 7, wherein Y_{10} has the following formula:



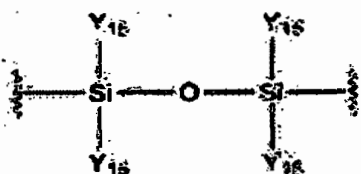
wherein each Y_{12} and Y_{13} is independently selected from H, halo, optionally substituted cyclic, branched or straight chain alkyl, or $-OY_{14}$, where Y_{14} is selected from optionally substituted branched or straight chain C_1-C_8 alkyl or alkenyl, and n is an integer from 1 to 10.

9. An electronic or electrical device or component thereof according to claim 8, wherein each Y_{12} is H and each Y_{13} is H.

10. An electronic or electrical device or component thereof according to claim 8, wherein each Y_{12} is fluoro and each Y_{13} is fluoro.

11. An electronic or electrical device or component thereof according to any of claims 8 to 10, wherein n is from 4 to 6.

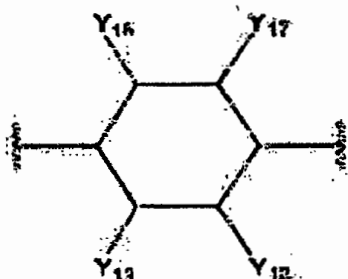
12. An electronic or electrical device or component thereof according to any of claims 5 to 7, wherein Y_{10} has the following formula:



Wherein each Y_{15} is independently selected from optionally substituted branched or straight chain C_1-C_8 alkyl.

13. An electronic or electrical device or component thereof according to claim 12, wherein each Y_{16} is methyl, and each Y_9 is a bond.

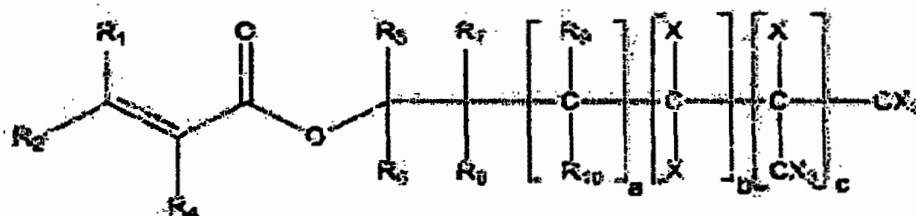
14. An electronic or electrical device or component thereof according to any of claims 5 to 7, wherein Y_{10} has the following formula:



wherein Y_{16} to Y_{19} are each independently selected from H and optionally substituted branched or straight chain C_1-C_8 alkyl or alkenyl.

15. An electronic or electrical device or component thereof according to claim 14, wherein Y_{18} is H or vinylene, and Y_{16} , Y_{17} and Y_{19} are each H.
16. An electronic or electrical device or component thereof according to any preceding claim wherein the crosslinking reagent is selected from Divinyl Adipate (DVA), 1,4 Butanediol divinyl ether (BDVE), 1,4 Cyclohexanedimethanol divinyl ether (CDDE), 1,7 -Octadiene (17OD), 1,2,4 -Trivinylcyclohexane (TVCH), 1,3-Divinyltetramethyldisiloxane (DVTMDS), Diallyl 1,4-cyclohexanedicarboxylate (DCHD), 1,6-Divinylperfluorohexane (DVPFH), 1H,1H,6H,6H-Perfluorohexanediol diacrylate (PFHDA) and glyoxal bis (diallyl acetal) (GBDA).
17. An electronic or electrical device or component thereof according to claim 4, wherein for compound (ii), L is selected from a branched or straight chain C_1 - C_8 alkylene or an ether group.
18. An electronic or electrical device or component thereof according to any preceding claim wherein the monomer is a compound of formula I(a):

I(a)

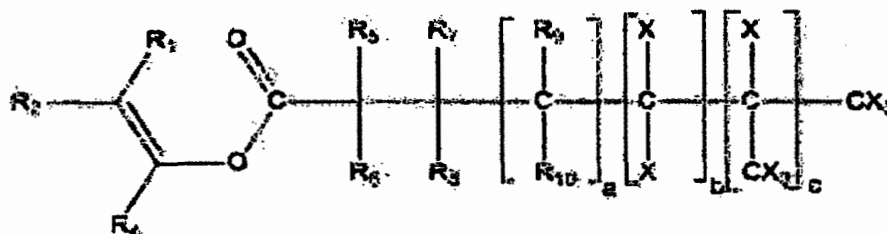


wherein each of R_1 , R_2 , R_4 , and R_5 to R_{10} is independently selected from hydrogen or an optionally substituted C_1 - C_8 branched or straight chain alkyl group; each X is independently selected from hydrogen or halogen; a is from 0-10; b is from 2 to 14; and c is 0 or 1;

; or

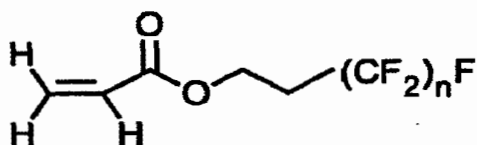
- wherein the monomer is a compound of formula I(b):

I(b)



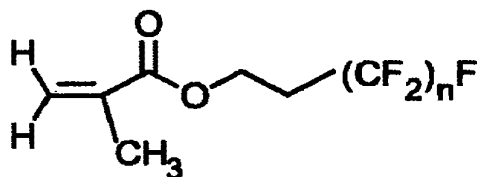
wherein each of R_1 , R_2 , R_4 , and R_5 to R_{10} is independently selected from hydrogen or an optionally substituted C_1 - C_6 branched or straight chain alkyl group; each X is independently selected from hydrogen or halogen; a is from 0-10; b is from 2 to 14; and c is 0 or 1.

19. An electronic or electrical device or component thereof according to claim 18, wherein the
5 halogen is fluorine.
20. An electronic or electrical device or component thereof according to claim 18 or claim 19,
wherein each of R_1 , R_2 , R_4 , and R_5 to R_{10} is independently selected from hydrogen, methyl,
ethyl, n-propyl, isopropyl, n-butyl, isobutyl, tert-butyl, sec-butyl, n-pentyl, neopentyl, n-hexyl,
isohexyl, and 3-methylpentyl.
- 10 21. An electronic or electrical device or component thereof according to claim 20, wherein each of
 R_1 , R_2 , R_4 , and R_5 to R_{10} is independently selected from hydrogen or methyl.
22. An electronic or electrical device or component thereof according to any of claims 18 to 20,
wherein a and c are each independently 0 or 1; and b is from 3 to 7.
23. An electronic or electrical device or component thereof according to any preceding claim
15 wherein each X is H.
24. An electronic or electrical device or component thereof according to any of claims 1 to 22,
wherein each X is F.
25. An electronic or electrical device or component thereof according to any of claims 18 to 24,
wherein R_1 and R_2 are both hydrogen.
- 20 26. An electronic or electrical device or component thereof according to any of claims 18 to 25,
wherein R_4 is hydrogen or methyl.
27. An electronic or electrical device or component thereof according to any of claims 18 to 26,
wherein R_6 is hydrogen and R_{10} is branched or straight chain C_1 - C_6 alkyl group.
28. An electronic or electrical device or component thereof according to claim 27, wherein R_{10} is
25 methyl.
29. An electronic or electrical device or component thereof according to any of claims 18 to 28,
wherein each of R_5 to R_9 is hydrogen.
30. An electronic or electrical device or component thereof according to any of claims 18 to 29
wherein each of each of R_1 , R_2 , R_4 , and R_5 to R_{10} is hydrogen, each X is H, a=0 and c=0.
- 30 31. An electronic or electrical device or component thereof according to any of claims 24 to 30,
wherein the compound of formula I(a) has the following formula:



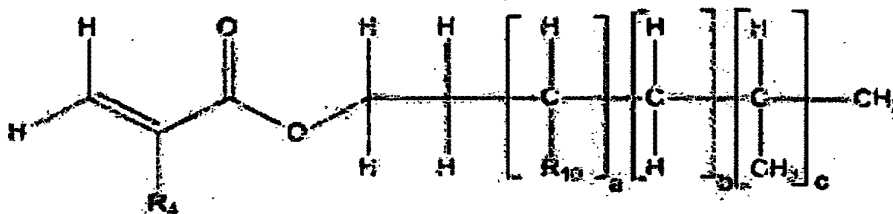
where n is from 2 to 10.

32. An electronic or electrical device or component thereof according to any of claims 24 to 30, wherein the compound of formula I(a) has the following formula:



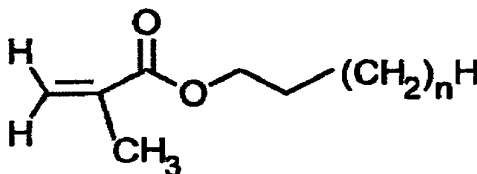
where n is from 2 to 10.

33. An electronic or electrical device or component thereof according to claim 31, wherein the compound of formula I(a) is selected from 1H,1H,2H,2H-perfluorohexyl acrylate (PFAC4), 1H,1H,2H,2H-perfluorooctyl acrylate (PFAC6), 1H,1H,2H,2H-perfluorodecyl acrylate (PFAC8) and 1H,1H,2H,2H-perfluorododecyl acrylate (PFAC10).
34. An electronic or electrical device or component thereof according to claim 32, wherein the compound of formula I(a) is selected from 1H,1H,2H,2H-perfluorohexyl methacrylate (PFMAC4), 1H,1H,2H,2H-perfluorooctyl methacrylate (PFMAC6) and 1H,1H,2H,2H-perfluorodecyl methacrylate (PFMAC8).
35. An electronic or electrical device or component thereof according to any of claims 18 to 23, wherein the compound of formula I(a) has the following formula:



wherein a and c are each independently 0 or 1, b=3-7 and n is 4 to 10, where n=a+b+c+1.

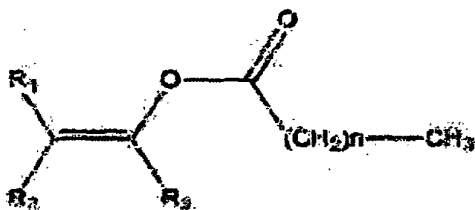
36. An electronic or electrical device or component thereof according to any of claims 18 to 23, wherein the compound of formula I(a) has the following formula:



where n is 2 to 12.

37. An electronic or electrical device or component thereof according to claim 35 or claim 36, wherein the compound of formula I(a) is selected from ethyl hexyl acrylate, hexyl acrylate, decyl acrylate, lauryl dodecyl acrylate and iso decyl acrylate.

5 38. An electronic or electrical device or component thereof according to any of claims 18 to 23, wherein the monomer is a compound of formula I(b) and has the following formula:



where n is from 4 to 14, optionally where R₁, R₂ and R₃ are each H.

10 39. An electronic or electrical device or component thereof according to any of the preceding claims, wherein the electronic or electrical device or component thereof is selected from mobile phones, smartphones, pagers, radios, sound and audio systems such as loudspeakers, microphones, ringers and/or buzzers, hearing aids, personal audio equipment such as personal CD, tape cassette or MP3 players, televisions, DVD players including portable DVD players, video recorders, digi and other set-top boxes, computers and related components such as
15 laptop, notebook, tablet, phablet or palmtop computers, personal digital assistants (PDAs), keyboards, or instrumentation, games consoles, data storage devices, outdoor lighting systems, radio antennae and other forms of communication equipment, and printed circuit boards.

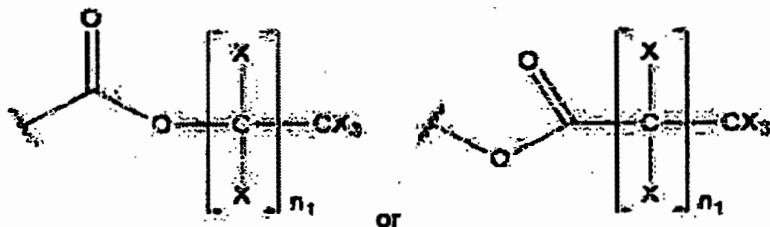
40. A method for treating an electronic or electrical device or component thereof comprising:

20 exposing the electronic or electrical device or component thereof to a plasma comprising a monomer compound and a crosslinking reagent for a period of time sufficient to allow a protective cross-linked polymeric coating to form on a surface of the electronic or electrical device or component thereof;

wherein the monomer compound has the following formula:

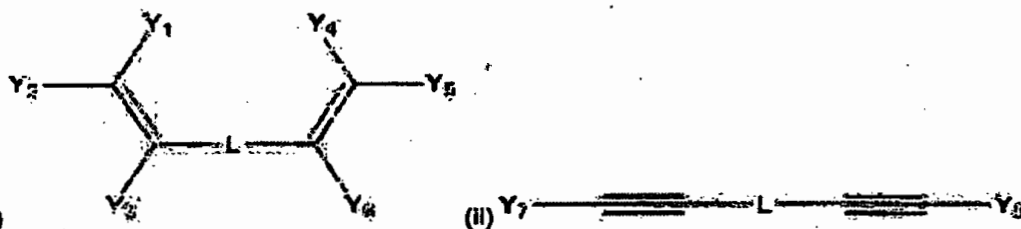


where R_1 , R_2 and R_4 are each independently selected from hydrogen, optionally substituted branched or straight chain C_1 - C_6 alkyl or halo alkyl or aryl optionally substituted by halo, and R_3 is selected from:



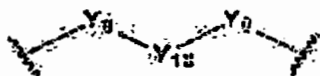
where each X is independently selected from hydrogen, optionally substituted branched or straight chain C_1 - C_6 alkyl, halo alkyl or aryl optionally substituted by halo; and n_1 is an integer from 1 to 27; and wherein the crosslinking reagent comprises two or more unsaturated bonds attached by means of one or more linker moieties and has a boiling point at standard pressure of less than 500°C .

41. A method according to claim 40, wherein the crosslinking reagent has one of the following structures:



where Y_1 , Y_2 , Y_3 , Y_4 , Y_5 , Y_6 , Y_7 and Y_8 are each independently selected from hydrogen, optionally substituted cyclic, branched or straight chain C_1 - C_6 alkyl or aryl; and L is a linker moiety.

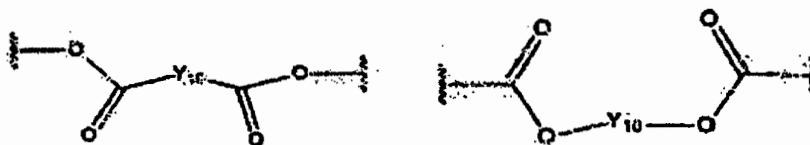
42. A method according to claim 41, wherein for compound (i) L has the following formula:



each Y_9 is independently selected from, a bond, $-\text{O}-$, $-\text{O}-\text{C}(\text{O})-$, $-\text{C}(\text{O})-\text{O}-$, $-\text{Y}_{11}-\text{O}-\text{C}(\text{O})-$, $-\text{C}(\text{O})-\text{O}-\text{Y}_{11}-$, $-\text{OY}_{11}-$, and $-\text{Y}_{11}\text{O}-$, where Y_{11} is an optionally substituted cyclic, branched or straight chain C_1 - C_6 alkylene; and

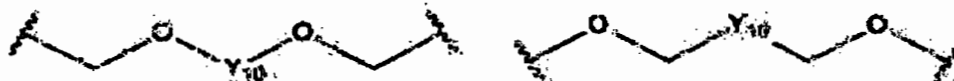
Y_{10} is selected from optionally substituted cyclic, branched or straight chain C_1 - C_6 alkylene and a siloxane group.

43. A method according to claim 42, wherein for compound (i) L has one of the following structures:

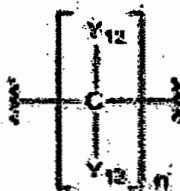




44. A method according to claim 42, wherein for compound (i) L has one of the following structures:

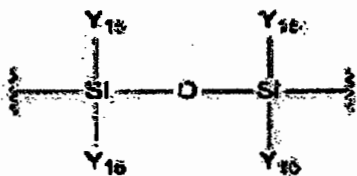


- 5 45. An method according to any of claims 42 to 44, wherein Y_{10} has the following formula:



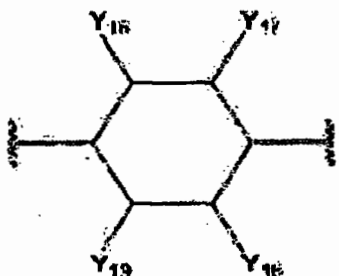
- 10 wherein each Y_{12} and Y_{13} is independently selected from H, halo, optionally substituted cyclic, branched or straight chain alkyl, or $-OY_{14}$, where Y_{14} is selected from optionally substituted branched or straight chain C_1 - C_8 alkyl or alkenyl, and n is an integer from 1 to 10.

46. A method according to claim 45, wherein each Y_{12} is H and each Y_{13} is H.
 47. A method according to claim 45, wherein each Y_{12} is fluoro and each Y_{13} is fluoro.
 48. A method according to any of claims 45 to 47, wherein n is from 4 to 6.
 15 49. A method according to any of claims 42 to 44, wherein Y_{10} has the following formula:



Wherein each Y_{15} is independently selected from optionally substituted branched or straight chain C_1 - C_8 alkyl.

50. A method according to claim 49, wherein each Y_{15} is methyl, and each Y_9 is a bond.
 20 51. A method according to any of claims 42 to 44, wherein Y_{10} has the following formula:



wherein Y_{16} to Y_{19} are each independently selected from H and optionally substituted branched or straight chain C_1 - C_8 alkyl or alkenyl.

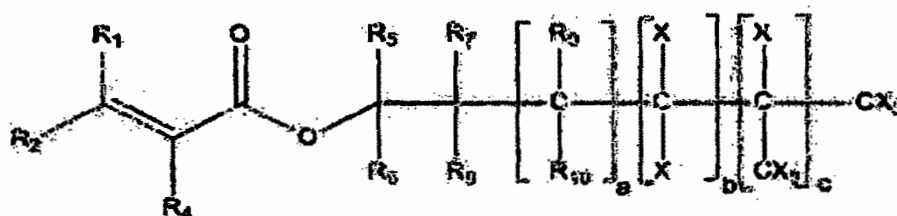
52. An method according to claim 51, wherein Y_{16} is vinylene or H, and Y_{16} , Y_{17} and Y_{19} are each H.

53. A method according to any of claims 40 to 52, wherein the crosslinking reagent is selected from Divinyl Adipate (DVA), 1,4 Butanediol divinyl ether (BDVE), 1,4 Cyclohexanedimethanol divinyl ether (CDDE), 1,7 -Octadiene (17OD), 1,2,4 -Trivinylcyclohexane (TVCH), 1,3-Divinyltetramethyldisiloxane (DVTMDS), Diallyl 1,4-cyclohexanedicarboxylate (DCHD), 1,6-Divinylperfluorohexane (DVPFH), 1H,1H,6H,6H-Perfluorohexanediol diacrylate (PFHDA) and glyoxal bis (diallyl acetal) (GBDA).

54. A method according to claim 41, wherein for compound (ii), L is selected from a branched or straight chain C_1 - C_8 alkylene or an ether group.

55. A method according to any of claims 40 to 54 wherein the monomer is a compound of formula I(a):

I(a)

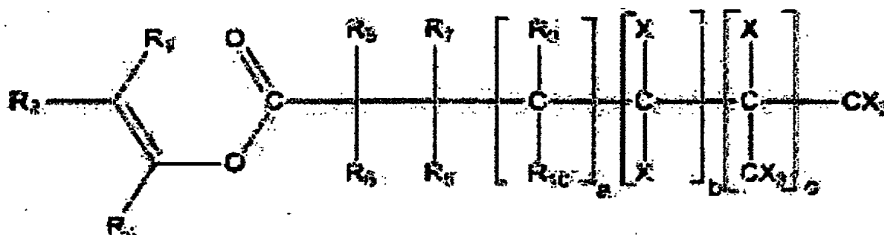


wherein each of R_1 , R_2 , R_4 , and R_5 to R_{10} is independently selected from hydrogen or an optionally substituted C_1 - C_8 branched or straight chain alkyl group; each X is independently selected from hydrogen or halogen; a is from 0-10; b is from 2 to 14; and c is 0 or 1;

; or

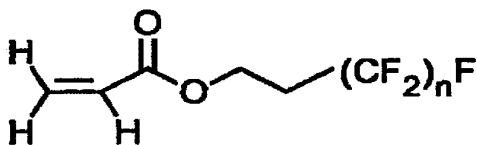
wherein the monomer is a compound of formula I(b):

I(b)



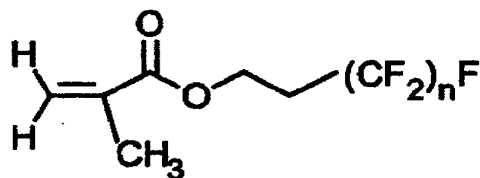
wherein each of R_1 , R_2 , R_4 , and R_5 to R_{10} is independently selected from hydrogen or an optionally substituted C_1 - C_8 branched or straight chain alkyl group; each X is independently selected from hydrogen or halogen; a is from 0-10; b is from 2 to 14; and c is 0 or 1.

56. A method according to claim 55, wherein the halogen is fluorine.
57. A method according to claim 55 or claim 56, wherein each of R_1 , R_2 , R_4 , and R_5 to R_{10} is independently selected from hydrogen, methyl, ethyl, *n*-propyl, isopropyl, *n*-butyl, isobutyl, *tert*-butyl, *sec*-butyl, *n*-pentyl, neopentyl, *n*-hexyl, isohexyl, and 3-methylpentyl.
58. A method according to claim 57, wherein each of R_1 , R_2 , R_4 , and R_5 to R_{10} is independently selected from hydrogen or methyl.
59. A method according to any of claims 55 to 58, wherein a and c are each independently 0 or 1; and b is from 3 to 7.
60. A method according to any preceding claim wherein each X is H.
61. A method according to any of claims 41 to 59, wherein each X is F.
62. A method according to any of claims 41 to 61, wherein R_1 and R_2 are both hydrogen.
63. A method according to any of claims 41 to 62, wherein R_4 is hydrogen or methyl.
64. A method according to any of claims 41 to 63, wherein R_9 is hydrogen and R_{10} is branched or straight chain C_1 - C_8 alkyl group.
65. A method according to claim 64, wherein R_{10} is methyl.
66. A method to any of claims 41 to 65, wherein each of R_5 to R_8 is hydrogen.
67. A method according to any of claims 41 to 66 wherein each of each of R_1 , R_2 , R_4 , and R_5 to R_{10} is hydrogen, each X is H, $a=0$ and $c=0$.
68. A method according to any of claims 61 to 67, wherein the compound of formula I(a) has the following formula:



where n is from 2 to 10.

69. A method according to any of claims 61 to 67, wherein the compound of formula I(a) has the following formula:

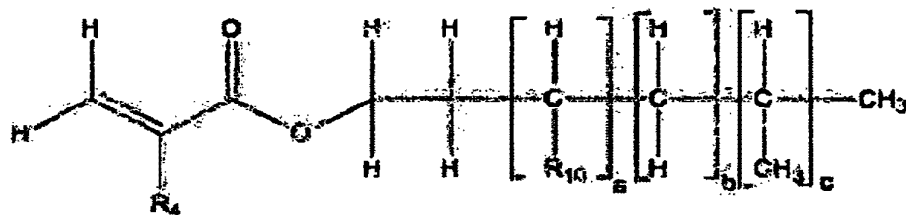


where n is from 2 to 10.

70. A method according to claim 68, wherein the compound of formula I(a) is selected from 1H,1H,2H,2H-perfluorohexyl acrylate (PFAC4), 1H,1H,2H,2H-perfluorooctyl acrylate (PFAC6),
 5 1H,1H,2H,2H-perfluorodecyl acrylate (PFAC8) and 1H,1H,2H,2H-perfluorododecyl acrylate (PFAC10).

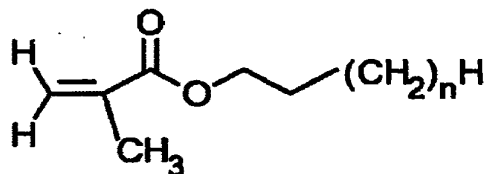
71. A method according to claim 69, wherein the compound of formula I(a) is selected from 1H,1H,2H,2H-perfluorohexyl methacrylate (PFMAC4), 1H,1H,2H,2H-perfluorooctyl methacrylate (PFMAC6) and 1H,1H,2H,2H-perfluorodecyl methacrylate (PFMAC8).
 10

72. A method according to any of claims 41 to 61, wherein the compound of formula I(a) has the following formula:



15 wherein a and c are each independently 0 or 1, b=3-7 and n is 4 to 10, where n=a+b+c+1.

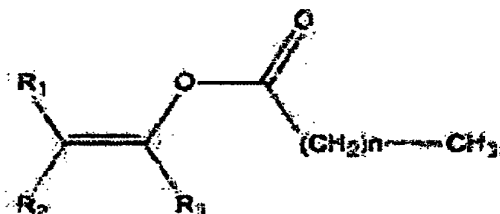
73. A method according to any of claims 41 to 61, wherein the compound of formula I(a) has the following formula:



where n is 2 to 12.

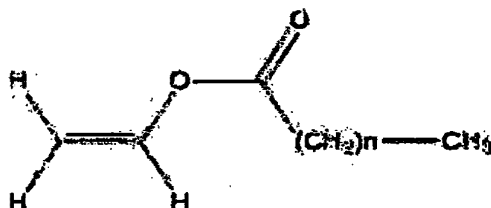
74. A method according to claim 72 or claim 73, wherein the compound of formula I(a) is selected from ethyl hexyl acrylate, hexyl acrylate, decyl acrylate, lauryl dodecyl acrylate and iso decyl acrylate.

75. A method according to any of claims 41 to 60, wherein the monomer is a compound of formula I(b) and has the following formula:



where n is from 4 to 14.

76. A method according to claim 75 wherein the monomer is a compound of formula I(b) and has the following formula:



where n is from 4 to 14.

77. A method according to any of claims 40 to 76 wherein the electronic or electrical device or component thereof is placed in a plasma deposition chamber, a glow discharge is ignited within said chamber, and a voltage applied as a pulsed field.

78. A method according to claim 77 wherein applied voltage is at a power of from 40 to 500W.

79. A method according to claim 77 or claim 78 wherein the voltage is pulsed in a sequence in which the ratio of the time on : time off is in the range of from 1:500 to 1:1500.

80. A method according to any of claims 77 to 79 wherein the voltage is pulsed in a sequence where power is on for 20-50 μ s, and off for from 1000 μ s to 30000 μ s.

81. A method according to any of claims 77 to 80 wherein the voltage is applied as a pulsed field for a period of from 30 seconds to 90 minutes.

82. A method according to claim 81 wherein the voltage is applied as a pulsed field for from 5 to 60 minutes.

83. A method according to any one of claims 77 to 82, wherein in a preliminary step, a continuous power plasma is applied to the electronic or electrical device or component thereof.

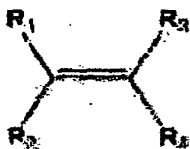
84. A method according to claim 83, wherein the preliminary step is conducted in the presence of an inert gas.
85. A method according to any one of claims 77 to 84, wherein the monomer compound and/or the crosslinking reagent in gaseous form is fed into the plasma at a rate of from 80-300 mg/minute, whilst the pulsed voltage is applied.
86. A method according to any one of claims 77 to 85, wherein the plasma is created with a voltage at an average power of from 0.001 to 500w/m³.
87. A method according to any of claims 40 to 86, wherein the monomer compound is miscible with the crosslinking reagent.
88. A method according to any of claims 40 to 86, wherein the monomer compound is immiscible with the crosslinking reagent.
89. A method according to claim 87, wherein the step of exposing the electronic or electrical device or component thereof to a plasma occurs in a plasma chamber; and the monomer compound and crosslinking reagent are mixed prior to entering the chamber.
90. A method according to claim 87 or claim 88 wherein the step of exposing the electronic or electrical device or component thereof to a plasma occurs in a plasma chamber; and the monomer compound and crosslinking reagent are introduced separately into the chamber.
91. A method according to any of claims 40 to 90, wherein the crosslinking reagent is present in an amount of from 10 to 80 (v/v) % of the total volume of monomer compound and crosslinking reagent.

Coatings

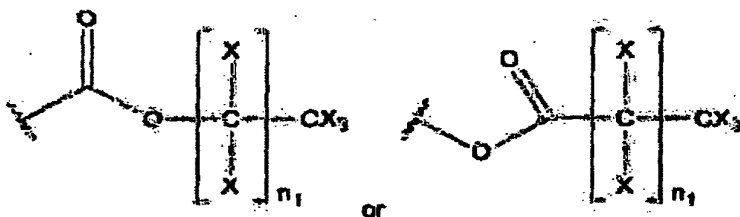
Abstract

The present invention provides an electronic or electrical device or component thereof comprising a cross-linked polymeric coating on a surface of the electronic or electrical device or component thereof; wherein the cross-linked polymeric coating is obtainable by exposing the electronic or electrical device or component thereof to a plasma comprising a monomer compound and a crosslinking reagent for a period of time sufficient to allow formation of the cross-linked polymeric coating on a surface thereof,

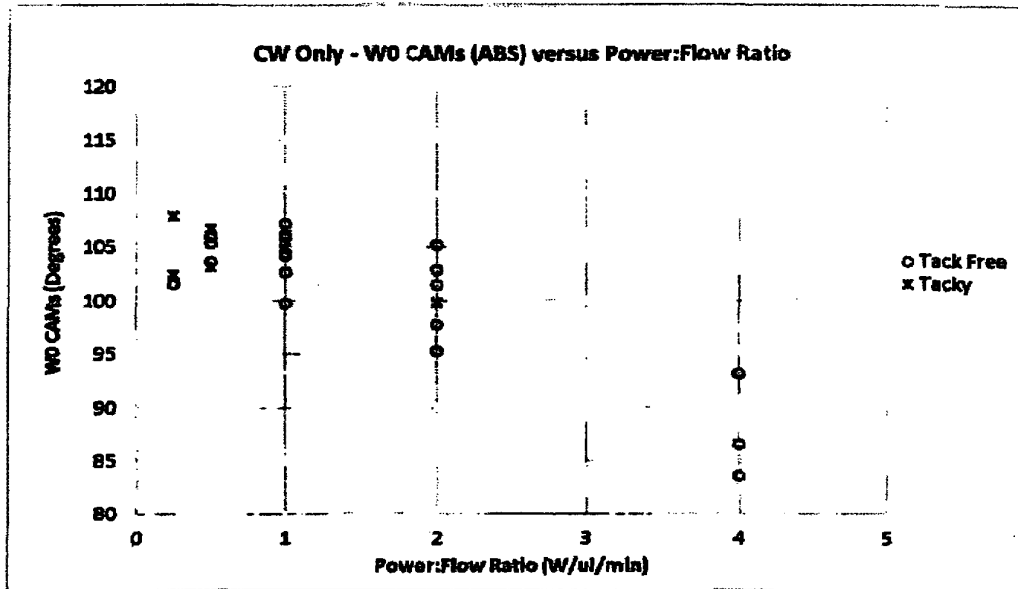
wherein the monomer compound has the following formula:



where R_1 , R_2 and R_4 are each independently selected from hydrogen, optionally substituted branched or straight chain C_1 - C_8 alkyl or halo alkyl or aryl optionally substituted by halo, and R_3 is selected from:



where each X is independently selected from hydrogen, a halogen, optionally substituted branched or straight chain C_1 - C_8 alkyl, halo alkyl or aryl optionally substituted by halo; and n_1 is an integer from 1 to 27; and wherein the crosslinking reagent comprises two or more unsaturated bonds attached by means of one or more linker moieties and has a boiling point at standard pressure of less than 500°C .

**Figure 1**

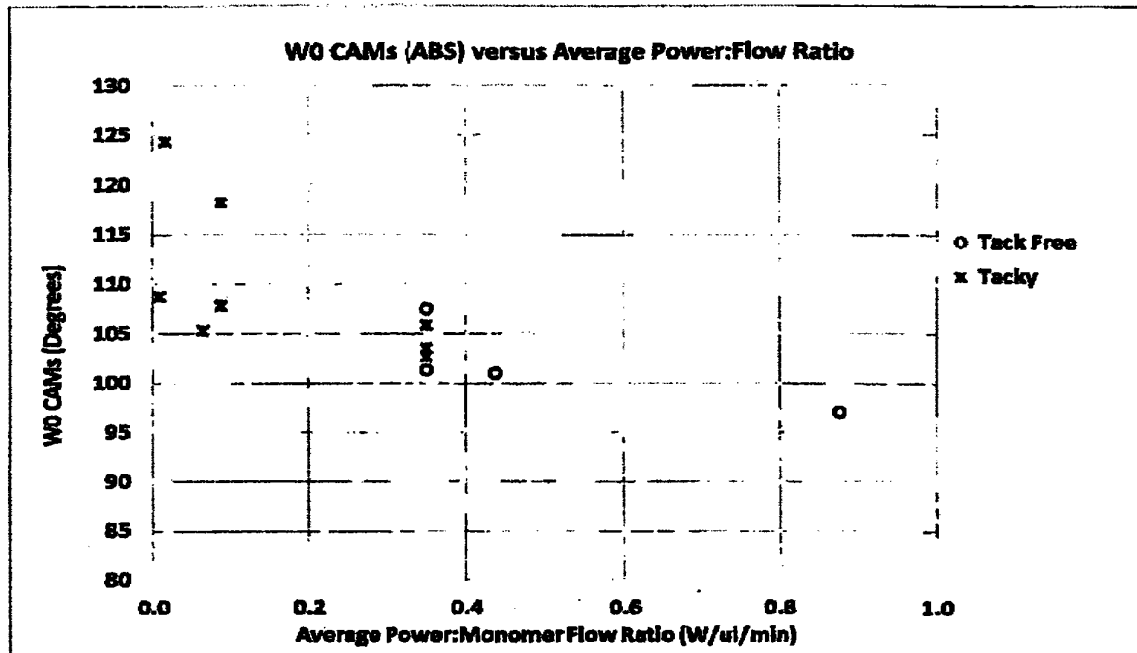


Figure 2

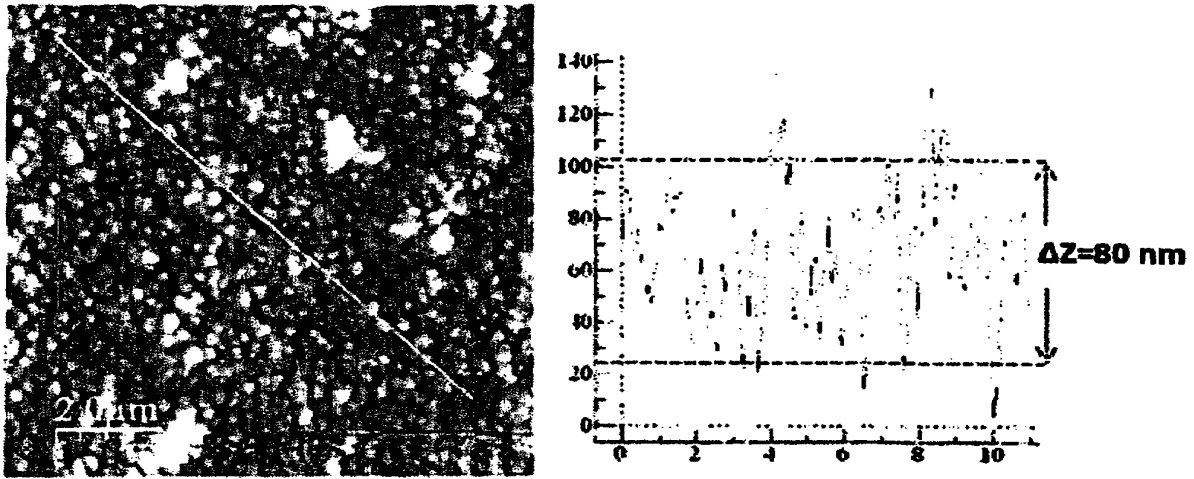


Figure 3

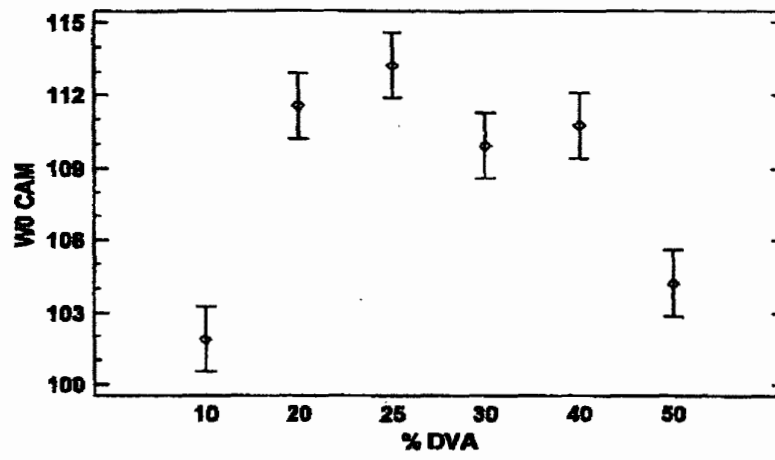


Figure 4

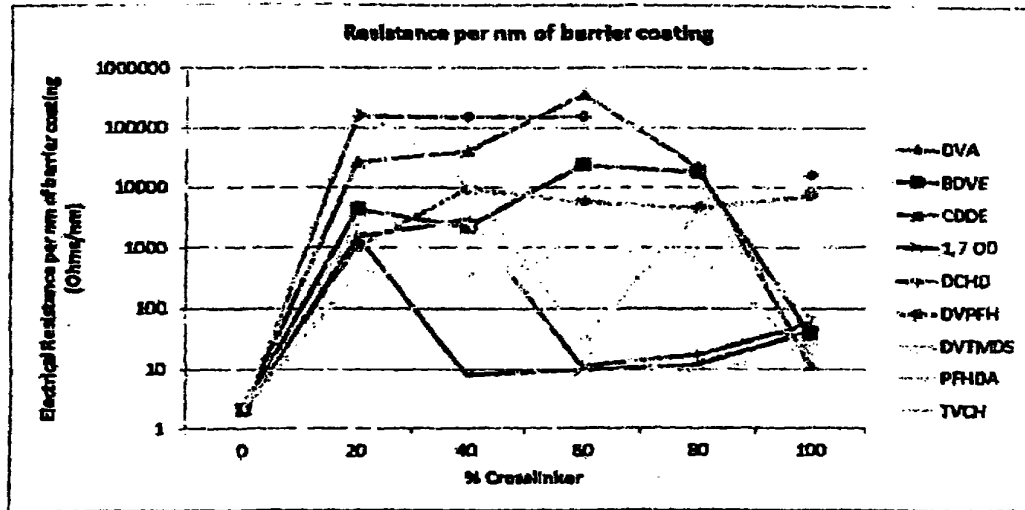
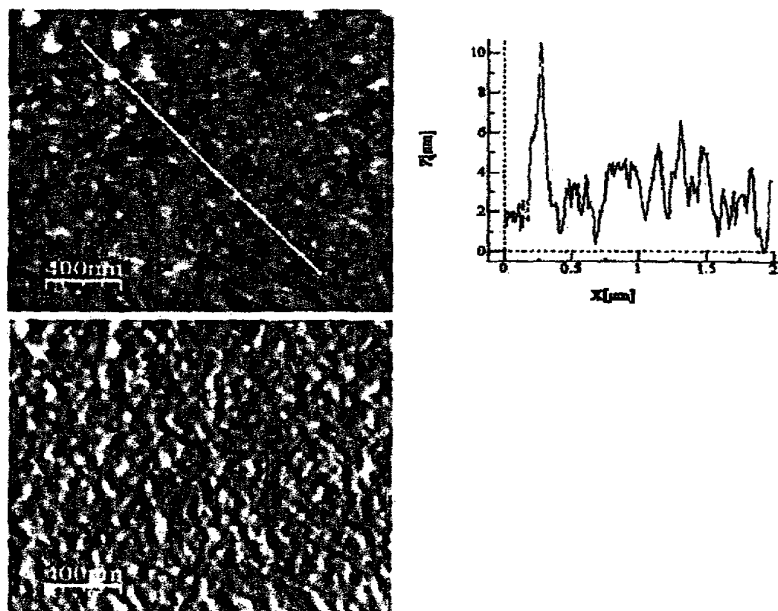


Figure 5

Figure 6



DOCUMENT MADE AVAILABLE UNDER THE PATENT COOPERATION TREATY (PCT)

| | |
|--|---|
| International application number: | PCT/GB2016/051688 |
| International filing date: | 08 June 2016 (08.06.2016) |
| Document type: | Certified copy of priority document |
| Document details: | Country/Office: EP |
| | Number: 15386017.6 |
| | Filing date: 09 June 2015 (09.06.2015) |
| Date of receipt at the International Bureau: | 29 July 2016 (29.07.2016) |

Remark: Priority document submitted or transmitted to the International Bureau in compliance with Rule 17.1(a),(b) or (b-bis)



UNITED STATES PATENT AND TRADEMARK OFFICE

UNITED STATES DEPARTMENT OF COMMERCE
United States Patent and Trademark Office
Address: COMMISSIONER FOR PATENTS
P.O. Box 1450
Alexandria, Virginia 22313-1450
www.uspto.gov

| APPLICATION NUMBER | FILING or 371(c) DATE | GRP ART UNIT | FIL FEE REC'D | ATTY. DOCKET NO | TOT CLAIMS | IND CLAIMS |
|-----------------------|--------------------------|-----------------|---------------|-----------------|------------|------------|
| 15/735,130 | 12/08/2017 | | 1620 | GBL0007/US | 83 | 2 |

CONFIRMATION NO. 7101

FILING RECEIPT



0000000098077872

33072
KAGAN BINDER, PLLC
SUITE 200, MAPLE ISLAND BUILDING
221 MAIN STREET NORTH
STILLWATER, MN 55082

Date Mailed: 03/16/2018

Receipt is acknowledged of this non-provisional patent application. The application will be taken up for examination in due course. Applicant will be notified as to the results of the examination. Any correspondence concerning the application must include the following identification information: the U.S. APPLICATION NUMBER, FILING DATE, NAME OF APPLICANT, and TITLE OF INVENTION. Fees transmitted by check or draft are subject to collection. Please verify the accuracy of the data presented on this receipt. **If an error is noted on this Filing Receipt, please submit a written request for a Filing Receipt Correction. Please provide a copy of this Filing Receipt with the changes noted thereon. If you received a "Notice to File Missing Parts" for this application, please submit any corrections to this Filing Receipt with your reply to the Notice. When the USPTO processes the reply to the Notice, the USPTO will generate another Filing Receipt incorporating the requested corrections**

Inventor(s)

Stephen Richard COULSON, Abingdon, UNITED KINGDOM;
Delwyn EVANS, Abingdon, UNITED KINGDOM;
Angeliki SIOKOU, Abingdon, UNITED KINGDOM;
Clive TELFORD, Abingdon, UNITED KINGDOM;

Applicant(s)

P2i Ltd, Abingdon, UNITED KINGDOM

Power of Attorney: The patent practitioners associated with Customer Number 33072

Domestic Priority data as claimed by applicant

This application is a 371 of PCT/GB2016/051688 06/08/2016

Foreign Applications (You may be eligible to benefit from the **Patent Prosecution Highway** program at the USPTO. Please see <http://www.uspto.gov> for more information.)
EUROPEAN PATENT OFFICE (EPO) 15386017.6 06/09/2015

Permission to Access Application via Priority Document Exchange: Yes

Permission to Access Search Results: Yes

Applicant may provide or rescind an authorization for access using Form PTO/SB/39 or Form PTO/SB/69 as appropriate.

If Required, Foreign Filing License Granted: 03/14/2018

The country code and number of your priority application, to be used for filing abroad under the Paris Convention, is **US 15/735,130**

Projected Publication Date: 06/21/2018

Non-Publication Request: No

Early Publication Request: No

Title

COATINGS

Preliminary Class

Statement under 37 CFR 1.55 or 1.78 for AIA (First Inventor to File) Transition Applications: No

PROTECTING YOUR INVENTION OUTSIDE THE UNITED STATES

Since the rights granted by a U.S. patent extend only throughout the territory of the United States and have no effect in a foreign country, an inventor who wishes patent protection in another country must apply for a patent in a specific country or in regional patent offices. Applicants may wish to consider the filing of an international application under the Patent Cooperation Treaty (PCT). An international (PCT) application generally has the same effect as a regular national patent application in each PCT-member country. The PCT process **simplifies** the filing of patent applications on the same invention in member countries, but **does not result** in a grant of "an international patent" and does not eliminate the need of applicants to file additional documents and fees in countries where patent protection is desired.

Almost every country has its own patent law, and a person desiring a patent in a particular country must make an application for patent in that country in accordance with its particular laws. Since the laws of many countries differ in various respects from the patent law of the United States, applicants are advised to seek guidance from specific foreign countries to ensure that patent rights are not lost prematurely.

Applicants also are advised that in the case of inventions made in the United States, the Director of the USPTO must issue a license before applicants can apply for a patent in a foreign country. The filing of a U.S. patent application serves as a request for a foreign filing license. The application's filing receipt contains further information and guidance as to the status of applicant's license for foreign filing.

Applicants may wish to consult the USPTO booklet, "General Information Concerning Patents" (specifically, the section entitled "Treaties and Foreign Patents") for more information on timeframes and deadlines for filing foreign patent applications. The guide is available either by contacting the USPTO Contact Center at 800-786-9199, or it can be viewed on the USPTO website at <http://www.uspto.gov/web/offices/pac/doc/general/index.html>.

For information on preventing theft of your intellectual property (patents, trademarks and copyrights), you may wish to consult the U.S. Government website, <http://www.stopfakes.gov>. Part of a Department of Commerce initiative, this website includes self-help "toolkits" giving innovators guidance on how to protect intellectual property in specific countries such as China, Korea and Mexico. For questions regarding patent enforcement issues, applicants may call the U.S. Government hotline at 1-866-999-HALT (1-866-999-4258).

LICENSE FOR FOREIGN FILING UNDER
Title 35, United States Code, Section 184
Title 37, Code of Federal Regulations, 5.11 & 5.15

GRANTED

The applicant has been granted a license under 35 U.S.C. 184, if the phrase "IF REQUIRED, FOREIGN FILING LICENSE GRANTED" followed by a date appears on this form. Such licenses are issued in all applications where the conditions for issuance of a license have been met, regardless of whether or not a license may be required as set forth in 37 CFR 5.15. The scope and limitations of this license are set forth in 37 CFR 5.15(a) unless an earlier license has been issued under 37 CFR 5.15(b). The license is subject to revocation upon written notification. The date indicated is the effective date of the license, unless an earlier license of similar scope has been granted under 37 CFR 5.13 or 5.14.

This license is to be retained by the licensee and may be used at any time on or after the effective date thereof unless it is revoked. This license is automatically transferred to any related applications(s) filed under 37 CFR 1.53(d). This license is not retroactive.

The grant of a license does not in any way lessen the responsibility of a licensee for the security of the subject matter as imposed by any Government contract or the provisions of existing laws relating to espionage and the national security or the export of technical data. Licensees should apprise themselves of current regulations especially with respect to certain countries, of other agencies, particularly the Office of Defense Trade Controls, Department of State (with respect to Arms, Munitions and Implements of War (22 CFR 121-128)); the Bureau of Industry and Security, Department of Commerce (15 CFR parts 730-774); the Office of Foreign Assets Control, Department of Treasury (31 CFR Parts 500+) and the Department of Energy.

NOT GRANTED

No license under 35 U.S.C. 184 has been granted at this time, if the phrase "IF REQUIRED, FOREIGN FILING LICENSE GRANTED" DOES NOT appear on this form. Applicant may still petition for a license under 37 CFR 5.12, if a license is desired before the expiration of 6 months from the filing date of the application. If 6 months has lapsed from the filing date of this application and the licensee has not received any indication of a secrecy order under 35 U.S.C. 181, the licensee may foreign file the application pursuant to 37 CFR 5.15(b).

SelectUSA

The United States represents the largest, most dynamic marketplace in the world and is an unparalleled location for business investment, innovation, and commercialization of new technologies. The U.S. offers tremendous resources and advantages for those who invest and manufacture goods here. Through SelectUSA, our nation works to promote and facilitate business investment. SelectUSA provides information assistance to the international investor community; serves as an ombudsman for existing and potential investors; advocates on behalf of U.S. cities, states, and regions competing for global investment; and counsels U.S. economic development organizations on investment attraction best practices. To learn more about why the United States is the best country in the world to develop technology, manufacture products, deliver services, and grow your business, visit <http://www.SelectUSA.gov> or call +1-202-482-6800.

| PATENT APPLICATION FEE DETERMINATION RECORD Substitute for Form PTO-875 | | | | | | Application or Docket Number 15/735,130 | | | |
|--|---|------------------------------------|---|--------------------|--------------------|--|----------|-------------------------------|--|
| APPLICATION AS FILED - PART I | | | | | | | | | |
| (Column 1) | | (Column 2) | | SMALL ENTITY | | OR OTHER THAN SMALL ENTITY | | | |
| FOR | NUMBER FILED | NUMBER EXTRA | RATE(\$) | FEE(\$) | | RATE(\$) | FEE(\$) | | |
| BASIC FEE (37 CFR 1.16(a), (b), or (c)) | N/A | N/A | N/A | | | N/A | 300 | | |
| SEARCH FEE (37 CFR 1.16(k), (i), or (m)) | N/A | N/A | N/A | | | N/A | 520 | | |
| EXAMINATION FEE (37 CFR 1.16(o), (p), or (q)) | N/A | N/A | N/A | | | N/A | 760 | | |
| TOTAL CLAIMS (37 CFR 1.16(j)) | 90 | minus 20 = * | 70 | | | x 100 = | 7000 | | |
| INDEPENDENT CLAIMS (37 CFR 1.16(h)) | 2 | minus 3 = * | | | | x 460 = | 0.00 | | |
| APPLICATION SIZE FEE (37 CFR 1.16(s)) | | | If the specification and drawings exceed 100 sheets of paper, the application size fee due is \$310 (\$155 for small entity) for each additional 50 sheets or fraction thereof. See 35 U.S.C. 41(a)(1)(G) and 37 CFR 1.16(s). | | | 0.00 | | | |
| MULTIPLE DEPENDENT CLAIM PRESENT (37 CFR 1.16(j)) | | | | | | | 820 | | |
| * If the difference in column 1 is less than zero, enter "0" in column 2. | | | | TOTAL | | TOTAL | 9400 | | |
| APPLICATION AS AMENDED - PART II | | | | | | | | | |
| (Column 1) | | (Column 2) | | (Column 3) | | SMALL ENTITY | | OR OTHER THAN SMALL ENTITY | |
| AMENDMENT A | CLAIMS REMAINING AFTER AMENDMENT | HIGHEST NUMBER PREVIOUSLY PAID FOR | PRESENT EXTRA | RATE(\$) | ADDITIONAL FEE(\$) | | RATE(\$) | ADDITIONAL FEE(\$) | |
| | Total (37 CFR 1.16(i)) | * | Minus | ** | = | | x | = | |
| | Independent (37 CFR 1.16(h)) | * | Minus | *** | = | | x | = | |
| | Application Size Fee (37 CFR 1.16(s)) | | | | | | | | |
| | FIRST PRESENTATION OF MULTIPLE DEPENDENT CLAIM (37 CFR 1.16(j)) | | | | | | | | |
| | | | | TOTAL ADD'L FEE | | TOTAL ADD'L FEE | | | |
| AMENDMENT B | CLAIMS REMAINING AFTER AMENDMENT | HIGHEST NUMBER PREVIOUSLY PAID FOR | PRESENT EXTRA | RATE(\$) | ADDITIONAL FEE(\$) | | RATE(\$) | ADDITIONAL FEE(\$) | |
| | Total (37 CFR 1.16(i)) | * | Minus | ** | = | | x | = | |
| | Independent (37 CFR 1.16(h)) | * | Minus | *** | = | | x | = | |
| | Application Size Fee (37 CFR 1.16(s)) | | | | | | | | |
| | FIRST PRESENTATION OF MULTIPLE DEPENDENT CLAIM (37 CFR 1.16(j)) | | | | | | | | |
| | | | | TOTAL ADD'L FEE | | TOTAL ADD'L FEE | | | |
| * If the entry in column 1 is less than the entry in column 2, write "0" in column 3. ** If the "Highest Number Previously Paid For" IN THIS SPACE is less than 20, enter "20". *** If the "Highest Number Previously Paid For" IN THIS SPACE is less than 3, enter "3". The "Highest Number Previously Paid For" (Total or Independent) is the highest found in the appropriate box in column 1. | | | | | | | | | |



UNITED STATES PATENT AND TRADEMARK OFFICE

UNITED STATES DEPARTMENT OF COMMERCE
United States Patent and Trademark Office
Address: COMMISSIONER FOR PATENTS
P.O. Box 1450
Alexandria, Virginia 22313-1450
www.uspto.gov

| | | |
|-----------------------------|-------------------------|------------------|
| U.S. APPLICATION NUMBER NO. | FIRST NAMED INVENTOR | ATTY. DOCKET NO. |
| 15/735,130 | Stephen Richard COULSON | GBL0007/US |

33072
KAGAN BINDER, PLLC
SUITE 200, MAPLE ISLAND BUILDING
221 MAIN STREET NORTH
STILLWATER, MN 55082

| | |
|-------------------------------|---------------|
| INTERNATIONAL APPLICATION NO. | |
| PCT/GB2016/051688 | |
| I.A. FILING DATE | PRIORITY DATE |
| 06/08/2016 | 06/09/2015 |

CONFIRMATION NO. 7101
371 FORMALITIES LETTER



Date Mailed: 03/16/2018

Notification of Insufficiency Under 37 CFR 1.492 and/or 1.495 (DO/EO/US)

The following items have been submitted by the applicant or the International Bureau to the United States Patent and Trademark Office as a Designated / Elected Office (37 CFR 1.495).

- Priority Document
- Copy of the International Application filed on 12/08/2017
- Copy of the International Search Report filed on 12/08/2017
- Information Disclosure Statements filed on 12/08/2017
- Request for Immediate Examination filed on 12/08/2017
- U.S. Basic National Fees filed on 12/08/2017
- Authorize Access to Search Results filed on 12/08/2017
- Priority Documents filed on 12/08/2017
- Power of Attorney filed on 12/08/2017
- Authorization to Permit Access filed on 12/08/2017
- Application Data Sheet (37 CFR 1.76) filed on 12/08/2017

The following items **MUST** be furnished within the period set forth below:

- Additional claim fees of **\$7820** as a undiscounted entity, including any required multiple dependent claim fee, are required. Applicant must submit the additional claim fees or cancel the additional claims for which fees are due.

SUMMARY OF FEES DUE:

Total additional fees required for this application is **\$7820** for an Undiscounted Entity:

Applicant is notified that the above-identified application contains the deficiencies noted below. No period for reply is set forth in this notice for correction of these deficiencies. However, if a deficiency relates to the inventor's oath or declaration, the applicant must file an oath or declaration in compliance with 37 CFR 1.63, or a substitute statement in compliance with 37 CFR 1.64, executed by or with respect to each actual inventor no later than the expiration of the time period set in the "Notice of Allowability" to avoid abandonment. See 37 CFR 1.495(c).

- Properly executed inventor's oath or declaration for the following inventor(s) has not been submitted: **Stephen Richard COULSON, Delwyn EVANS, Angeliki SIOKOU, and Clive TELFORD**

Total additional claim fee(s) for this application is **\$7820**

- **\$7000** for **70** total claims over 20.
- **\$820** for multiple dependent claim surcharge.

ALL OF THE ITEMS SET FORTH ABOVE MUST BE SUBMITTED WITHIN TWO (2) MONTHS FROM THE DATE OF THIS NOTICE. FAILURE TO PROPERLY RESPOND WILL RESULT IN ABANDONMENT.

The time period set above may be extended by filing a petition and fee for extension of time under the provisions of 37 CFR 1.136(a).

Applicant is reminded that any communications to the United States Patent and Trademark Office must be mailed to the address given in the heading and include the U.S. application no. shown above (37 CFR 1.5)

Registered users of EFS-Web may alternatively submit their reply to this notice via EFS-Web.

<https://sportal.uspto.gov/authenticate/AuthenticateUserLocalEPF.html>

For more information about EFS-Web please call the USPTO Electronic Business Center at **1-866-217-9197** or visit our website at <http://www.uspto.gov/ebc>.

If you are not using EFS-Web to submit your reply, you must include a copy of this notice.

MAMYE I WAGSTAFF

Telephone: (571) 272-1057

| MULTIPLE DEPENDENT CLAIM FEE CALCULATION SHEET Substitute for Form PTO-1360 (For use with Form PTO/SB/06) | | | | | | | Application Number 15735130 | | Filing Date | | | | |
|--|----------|--------|-----------------------|--------|------------------------|--------|---|-------|-------------|-------|--------|-------|--------|
| | | | | | | | Applicant(s) Stephen COULSON | | | | | | |
| | | | | | | | * May be used for additional claims or amendments | | | | | | |
| CLAIMS | AS FILED | | AFTER FIRST AMENDMENT | | AFTER SECOND AMENDMENT | | | * | | * | | * | |
| | Indep | Depend | Indep | Depend | Indep | Depend | | Indep | Depend | Indep | Depend | Indep | Depend |
| 1 | 1 | | | | | | | | (1) | | | | |
| 2 | | 1 | | | | | | | (1) | | | | |
| 3 | | 2 | | | | | | | (1) | | | | |
| 4 | | (1) | | | | | | | (1) | | | | |
| 5 | | (1) | | | | | | | (1) | | | | |
| 6 | | (1) | | | | | | | (1) | | | | |
| 7 | | (1) | | | | | | | (1) | | | | |
| 8 | | (1) | | | | | | | (1) | | | | |
| 9 | | (1) | | | | | | | (1) | | | | |
| 10 | | (1) | | | | | | | (1) | | | | |
| 11 | | (1) | | | | | | | (1) | | | | |
| 12 | | (1) | | | | | | | (1) | | | | |
| 13 | | (1) | | | | | | | (1) | | | | |
| 14 | | (1) | | | | | | | (1) | | | | |
| 15 | | (1) | | | | | | | (1) | | | | |
| 16 | | (1) | | | | | | | (1) | | | | |
| 17 | | (1) | | | | | | | (1) | | | | |
| 18 | | (1) | | | | | | | (1) | | | | |
| 19 | | (1) | | | | | | | (1) | | | | |
| 20 | | (1) | | | | | | | (1) | | | | |
| 21 | | (1) | | | | | | | (1) | | | | |
| 22 | | (1) | | | | | | | (1) | | | | |
| 23 | | (1) | | | | | | | (1) | | | | |
| 24 | | (1) | | | | | | | (1) | | | | |
| 25 | | (1) | | | | | | | (1) | | | | |
| 26 | | (1) | | | | | | | (1) | | | | |
| 27 | | (1) | | | | | | | (1) | | | | |
| 28 | | (1) | | | | | | | (1) | | | | |
| 29 | | (1) | | | | | | | (1) | | | | |
| 30 | | (1) | | | | | | | (1) | | | | |
| 31 | | (1) | | | | | | | (1) | | | | |
| 32 | | (1) | | | | | | | (1) | | | | |
| 33 | | (1) | | | | | | | (1) | | | | |
| 34 | | (1) | | | | | | | (1) | | | | |
| 35 | | (1) | | | | | | | (1) | | | | |
| 36 | | (1) | | | | | | | (1) | | | | |
| 37 | | (1) | | | | | | | (1) | | | | |
| 38 | | (1) | | | | | | | (1) | | | | |
| 39 | | (1) | | | | | | | (1) | | | | |
| 40 | 1 | | | | | | | | | | | | |
| 41 | | 1 | | | | | | | | | | | |
| 42 | | 1 | | | | | | | | | | | |
| 43 | | 1 | | | | | | | | | | | |
| 44 | | 1 | | | | | | | | | | | |
| 45 | | 3 | | | | | | | | | | | |
| 46 | | 3 | | | | | | | | | | | |
| 47 | | 3 | | | | | | | | | | | |
| 48 | | (1) | | | | | | | | | | | |
| 49 | | (1) | | | | | | | | | | | |
| 50 | | (1) | | | | | | | | | | | |
| Total Indep | 2 | | 0 | | 0 | | | | | | | | |
| Total Depend | 88 | ↙ | 0 | ↙ | 0 | ↙ | | | | | | | |
| Total Claims | 90 | | 0 | | 0 | | | | | | | | |

| | | | | | | |
|-----|--|-------|--|--|--|--|
| 51 | | (1) | | | | |
| 52 | | (1) | | | | |
| 53 | | (1) | | | | |
| 54 | | (1) | | | | |
| 55 | | (1) | | | | |
| 56 | | (1) | | | | |
| 57 | | (1) | | | | |
| 58 | | (1) | | | | |
| 59 | | (1) | | | | |
| 60 | | (1) | | | | |
| 61 | | (1) | | | | |
| 62 | | (1) | | | | |
| 63 | | (1) | | | | |
| 64 | | (1) | | | | |
| 65 | | (1) | | | | |
| 66 | | (1) | | | | |
| 67 | | (1) | | | | |
| 68 | | (1) | | | | |
| 69 | | (1) | | | | |
| 70 | | (1) | | | | |
| 71 | | (1) | | | | |
| 72 | | (1) | | | | |
| 73 | | (1) | | | | |
| 74 | | (1) | | | | |
| 75 | | (1) | | | | |
| 76 | | (1) | | | | |
| 77 | | (1) | | | | |
| 78 | | (1) | | | | |
| 79 | | (1) | | | | |
| 80 | | (1) | | | | |
| 81 | | (1) | | | | |
| 82 | | (1) | | | | |
| 83 | | (1) | | | | |
| 84 | | | | | | |
| 85 | | | | | | |
| 86 | | | | | | |
| 87 | | | | | | |
| 88 | | | | | | |
| 89 | | | | | | |
| 90 | | | | | | |
| 91 | | | | | | |
| 92 | | | | | | |
| 93 | | | | | | |
| 94 | | | | | | |
| 95 | | | | | | |
| 96 | | | | | | |
| 97 | | | | | | |
| 98 | | | | | | |
| 99 | | | | | | |
| 100 | | | | | | |

PATENT

In The United States Patent And Trademark Office

| | |
|---|---------------------------------|
| Inventor: Stephen Richard COULSON | Examiner: To Be Assigned |
| Serial No. 15/735,130 | Group Art Unit: 1762 |
| International Filing Date: June 8, 2016 | Confirmation No. 7101 |
| International App. No. PCT/GB2016/051688 | Docket No. GBL0007/US |
| 371(c): December 8, 2017 | |
| For: COATINGS | |

Commissioner for Patents
P.O. Box 1450
Alexandria, Virginia 22313-1450

I CERTIFY THAT ON 06/15/2018, THIS CORRESPONDENCE IS
BEING ELECTRONICALLY TRANSMITTED TO THE UNITED STATES
PATENT AND TRADEMARK OFFICE VIA THE OFFICE'S EFS-WEB

/Sonja A. Talley/
SONJA A. TALLEY

Preliminary Amendment

Dear Sir or Madam:

This is a Preliminary Amendment in the above-captioned application. Kindly enter this Amendment prior to calculating the filing fee for this application.

No fee is believed to be required in connection with the filing of this Preliminary Amendment.

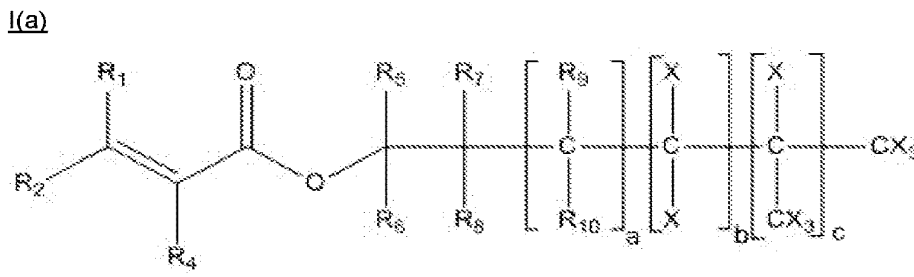
Claim Amendments

Please amend claims 1-3, 5-16, and 31-34; and cancel claims 4, 17-30, and 35-91 as follows:

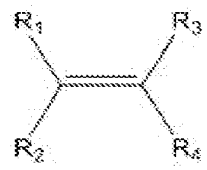
1. (Currently Amended) An electronic or electrical device or electronic or electrical component thereof comprising a protective cross-linked polymeric coating on a surface of ~~the electronic or electrical~~ said device or component ~~thereof~~;

wherein the protective cross-linked polymeric coating is obtainable by exposing ~~the electronic or electrical~~ said device or component ~~thereof~~ to a plasma comprising a monomer compound and a crosslinking reagent for a period of time sufficient to allow formation of the protective cross-linked polymeric coating on a surface thereof,

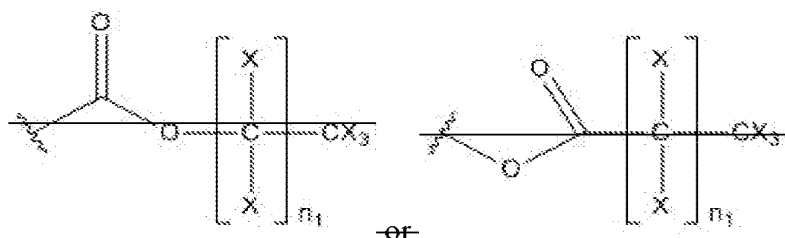
wherein the monomer compound is a compound of formula I(a):



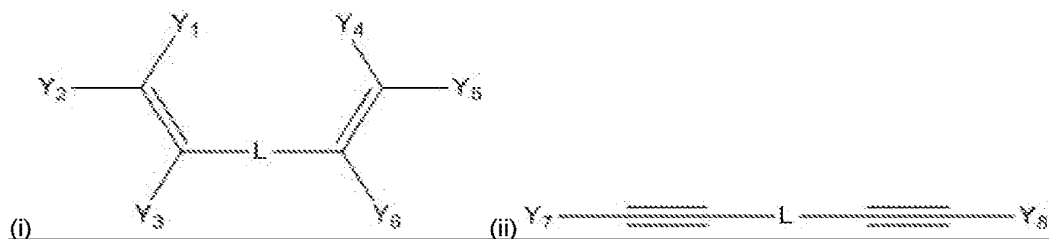
wherein each of R₁, R₂, and R₅ to R₈ is hydrogen; each of R₄, R₉ and R₁₀ is independently hydrogen or methyl; each X is independently hydrogen or halogen; a is from 0-10; b is from 3 to 7; and c is 0 or 1 ~~has the following formula:~~



~~where R₁, R₂ and R₄ are each independently selected from hydrogen, optionally substituted branched or straight chain C₁-C₆ alkyl or halo alkyl or aryl optionally substituted by halo, and R₃ is selected from:~~



~~where each X is independently selected from hydrogen, a halogen, optionally substituted branched or straight chain C₁-C₆ alkyl, halo alkyl or aryl optionally substituted by halo; and n₁ is an integer from 1 to 27; and wherein the crosslinking reagent comprises two or more unsaturated bonds attached by means of one or more linker moieties and has a boiling point of less than 500 °C at standard pressure, the crosslinking reagent having one of the following structures:~~



where Y₁, Y₂, Y₃, Y₄, Y₅, Y₆, Y₇ and Y₈ are each hydrogen; and L is a linker moiety.

2.(Currently Amended) ~~An electronic or electrical~~ The device or component thereof according to claim 1, wherein the protective cross-linked polymeric coating is a physical barrier to mass and electron transport.

3.(Currently Amended) ~~An electronic or electrical~~ The device or component thereof according to claim 1 ~~or claim 2~~, wherein the protective cross-linked polymeric coating forms a liquid repellent surface defined by a static water contact angle (WCA) of at least 90°.

4. (Cancelled)

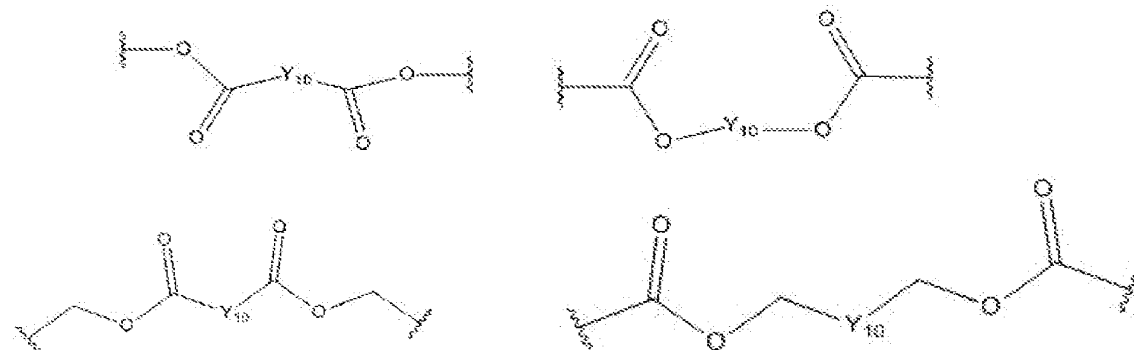
5. (Currently Amended) ~~An electronic or electrical~~ The device or component thereof according to ~~claim 4~~ claim 1, wherein for compound (i) L has the following formula:



each Y₉ is independently selected from, a bond, -O-, -O-C(O)-, -C(O)-O-, -Y₁₁-O-C(O)-, -C(O)-O-Y₁₁-, -OY₁₁-, and -Y₁₁O-, where Y₁₁ is an optionally substituted cyclic, branched or straight chain C₁-C₈ alkylene; and

Y₁₀ is selected from optionally substituted cyclic, branched or straight chain C₁-C₈ alkylene and a siloxane group.

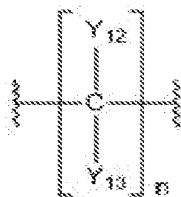
6. (Currently Amended) ~~An electronic or electrical~~ The device or component thereof according to claim 5, wherein for compound (i) L has one of the following structures:



7. (Currently Amended) ~~An electronic or electrical~~ The device or component thereof according to claim 5, wherein for compound (i) L has one of the following structures:



8. (Currently Amended) ~~An electronic or electrical~~ The device or component ~~thereof~~ according to ~~any of claims 5 to 7~~ claim 5, wherein Y_{10} has the following formula:



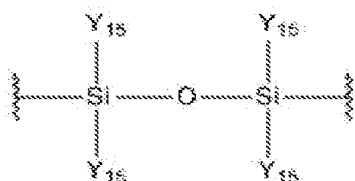
wherein each Y_{12} and Y_{13} is independently selected from H, halo, optionally substituted cyclic, branched or straight chain alkyl, or $-OY_{14}$, where Y_{14} is selected from optionally substituted branched or straight chain C_1 - C_8 alkyl or alkenyl, and n is an integer from 1 to 10.

9. (Currently Amended) ~~An electronic or electrical~~ The device or component ~~thereof~~ according to claim 8, wherein each Y_{12} is H and each Y_{13} is H.

10. (Currently Amended) ~~An electronic or electrical~~ The device or component ~~thereof~~ according to claim 8, wherein each Y_{12} is fluoro and each Y_{13} is fluoro.

11. (Currently Amended) ~~An electronic or electrical~~ The device or component ~~thereof~~ according to ~~any of claims 8 to 10~~ claim 8, wherein n is from 4 to 6.

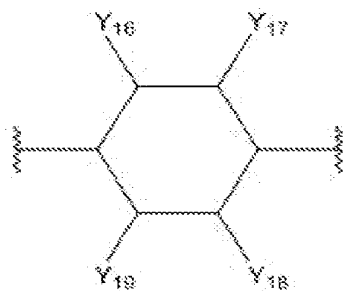
12. (Currently Amended) ~~An electronic or electrical~~ The device or component ~~thereof~~ according to ~~any of claims 5 to 7~~ claim 5, wherein Y_{10} has the following formula:



Wherein each Y_{15} is independently selected from optionally substituted branched or straight chain C_1 - C_6 alkyl.

13. (Currently Amended) ~~An electronic or electrical~~ The device or component ~~thereof~~ according to claim 12, wherein each Y₁₅ is methyl, and each Y₉ is a bond.

14. (Currently Amended) ~~An electronic or electrical~~ The device or component ~~thereof~~ according to ~~any of claims 5 to 7~~ claim 5, wherein Y₁₀ has the following formula:



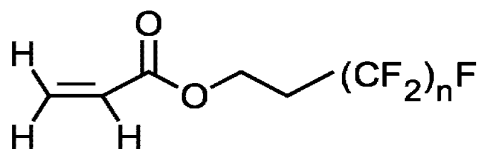
wherein Y₁₆ to Y₁₉ are each independently selected from H and optionally substituted branched or straight chain C₁-C₈ alkyl or alkenyl.

15. (Currently Amended) ~~An electronic or electrical~~ The device or component ~~thereof~~ according to claim 14, wherein Y₁₈ is H or vinylene, and Y₁₆, Y₁₇ and Y₁₉ are each H.

16. (Currently Amended) ~~An electronic or electrical~~ The device or component ~~thereof~~ according to ~~any preceding claim~~ claim 1, wherein the crosslinking reagent is selected from Divinyl Adipate (DVA), 1,4 Butanediol divinyl ether (BDVE), 1,4 Cyclohexanedimethanol divinyl ether (CDDE), 1,7 -Octadiene (17OD), 1,2,4 -Trivinylcyclohexane (TVCH), 1,3-Divinyltetramethyldisiloxane (DVTMDS), Diallyl 1,4-cyclohexanedicarboxylate (DCHD), 1,6-Divinylperfluorohexane (DVPFH), 1H,1H,6H,6H-Perfluorohexanediol diacrylate (PFHDA) and glyoxal bis (diallyl acetal) (GBDA).

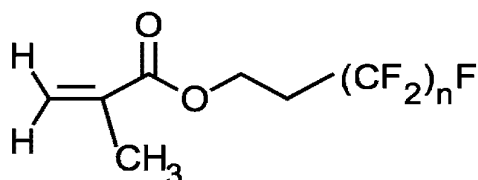
17-30. (Cancelled)

31. (Currently Amended) ~~An electronic or electrical~~ The device or component ~~thereof~~ according to ~~any of claims 24 to 30~~ claim 1, wherein the compound of formula I(a) has the following formula:



where n is from 2 to 10.

32. (Currently Amended) ~~An electronic or electrical~~ The device or component ~~thereof~~ according to ~~any of claims 24 to 30~~ claim 1, wherein the compound of formula I(a) has the following formula:



where n is from 2 to 10.

33. (Currently Amended) ~~An electronic or electrical~~ The device or component ~~thereof~~ according to claim 31, wherein the compound of formula I(a) is selected from 1H,1H,2H,2H-perfluorohexyl acrylate (PFAC4), 1H,1H,2H,2H-perfluorooctyl acrylate (PFAC6), 1H,1H,2H,2H-perfluorodecyl acrylate (PFAC8) and 1H,1H,2H,2H-perfluorododecyl acrylate (PFAC10).

34. (Currently Amended) ~~An electronic or electrical~~ The device or component ~~thereof~~ according to claim 32, wherein the compound of formula I(a) is selected from 1H,1H,2H,2H-pefluorohexyl

methacrylate (PFMAC4), 1H,1H,2H,2H-perfluorooctyl methacrylate (PFMAC6) and 1H,1H,2H,2H-perfluorodecyl methacrylate (PFMAC8).

35-91. (Cancelled)

Remarks

Applicant respectfully requests that the preliminary amendment be entered prior to examination of the above-identified patent application.

Claims 1-91 are pending in the above-identified patent application. With this Preliminary Amendment, claims 1-3, 5-16, and 31-34 are amended, and claims 4, 17-30, and 35-91 are canceled. Upon entry of the current amendment, claims 1-3, 5-16, and 31-34 are pending, which include 19 total claims and 1 independent claim.

Claims Pending Upon National Stage Entry

Applicant notes that the PCT Publication of PCT application PCT/GB2016/051688 published with 34 pages of text, which included 83 claims. The “Claims Worksheet (PTO-2022)” on PAIR identifies 83 claims as well. But, Applicant points out that the PCT Publication of PCT application PCT/GB2016/051688 erroneously left out page 35, which was originally filed with PCT application PCT/GB2016/051688 and included claims 84-91. A copy of PCT application PCT/GB2016/051688 as filed was downloaded from <http://www.wipo.int/patentscope/en/> and is submitted herewith as an attachment, which shows page 35 and claims 84-91. Applicant’s undersigned representative, Mr. Paul J. Parins, contacted the IB on June 15, 2018, and the IB concurred with respect to page 35 as explained above. The IB indicated that PCT application PCT/GB2016/051688 would be re-published in July, 2018 with page 35 and provided to the USPTO as a Designated Office.

Accordingly, the present Preliminary Amendment references claims 84-91 as canceled.

Support for Claim Amendments

Applicant submits that the claim amendments are fully supported by the application as originally filed and do not present new matter.

Support for the amendment to claim 1 can be found at, e. g., page 5, lines 23-27 and page 11, lines 6-11. Claim 1 is also amended for clarification purposes.

Claims 2-3, 5-16, and 31-34 are amended to conform to the amendment of claim 1, from which they depend.

Claims 3, 8, 11, 12, 14, 16, 31, and 32 are amended to remove multiple dependencies.

Approval of the application and allowance of the claims are earnestly solicited. In the event that a phone conference between the Examiner and the Applicant's undersigned representative would help resolve any issues in the application, the Examiner is invited to contact said attorney at (651) 275-9831.

Respectfully Submitted,

Date: June 15, 2018

By: /Paul J. Parins/

Paul J. Parins, Reg. No. 54,358

Customer Number 33072

Phone: 651-275-9831

Fax: 651-351-2954

#119892

Coatings

Field of the invention

This invention relates to protective coatings. Specifically, the present invention relates to protective coatings for electronic or electrical devices and components thereof, and methods of forming such coatings. The coatings can protect by being hydrophobic and so resist the ingress of water-based liquid into electronic devices, or they can protect by forming a barrier coating and so provide electrical resistance between the electrical parts of the phone and water based liquid.

Background of the invention

Monounsaturated monomers are used to make barrier coatings using plasma polymerisation processes (see co-pending application).

The perfluoroalkyl chain monomers are also used for generating hydrophobic surfaces from pulsed plasma deposition processes (see WO 9858117 A1).

The power of the plasma initiated polymerisation affects the nature of the polymer produced. The higher average energy inputs of continuous wave plasmas lead to more fragmentation of the monomer, and so the polymer loses structural properties of the monomer. In the case of 1H,1H,2H,2H- perfluorodecyl acrylate (PFAC8), there's less retention of the perfluoroalkyl chain and the contact angle of the surface coating is compromised. Higher plasma energies also lead to more crosslinking. For the lower average energy inputs of pulsed plasmas, there's better retention of monomer structure and less crosslinking. The greater retention of the perfluoro chain under low energy, pulsed plasma conditions leads to best levels of contact angles for the surface coating.

When the perfluoroalkyl chains have eight or more fluorinated carbons (long chain), the polymer made from the monomer has a crystalline structure. When the perfluoroalkyl chains have less than eight fluorinated carbons, the resulting polymer is amorphous and so can be unstable in the presence of water (see "Molecular Aggregation Structure and Surface Properties of Poly(fluoroalkyl acrylate) Thin Films, *Macromolecules*, 2005, vol 38, p5699-5705)

When long chain perfluoroalkyl polymers are produced by high average power (continuous wave or CW) or low average power (pulsed wave or PW) plasmas, then because of the crystalline structure of the long chains, the polymers are non-stick to the touch and stable in the presence of water.

However, the feel and water stability of shorter chain polymer coatings is affected by the plasma power levels used. For example, when PFAC6 (1H,1H,2H,2H-perfluorooctyl acrylate) is polymerised in low power plasma conditions, the resulting polymer coating can have several disadvantages. For example the coating can cause water drops to spread out a little (slump), be marked by the presence of a water drop on its surface, have a tacky feel, or can be easily smeared (for example on substrates of silicon wafer and ABS plastic).

By increasing the power of the plasma used for polymerisation, the polymer becomes more crosslinked and becomes more resistant to smearing. However, increasing the power has the concomitant effect of decreasing the water contact angle through more monomer fragmentation (as described above). Figure 1 shows the effect of increasing the power to monomer flow ratio for CW plasmas in a 125 litre chamber: at a ratio of 4 W/ μ l/min, the water contact angle is ~85-95 degrees and the coating is tack-free. However, as the ratio drops, the contact angle increases and the occurrence of tacky/smudgy coatings increases too. Figure 2 shows the same effect for pulsed plasma conditions. These results show that the process window for producing tack and smudge-free coatings have a limited plasma processing range and the final coating has a compromised water contact angle.

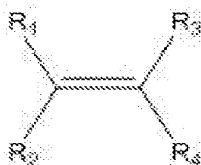
Accordingly, it is an aim to solve one or more of the above-mentioned problems with the prior art coatings.

Statement of Invention

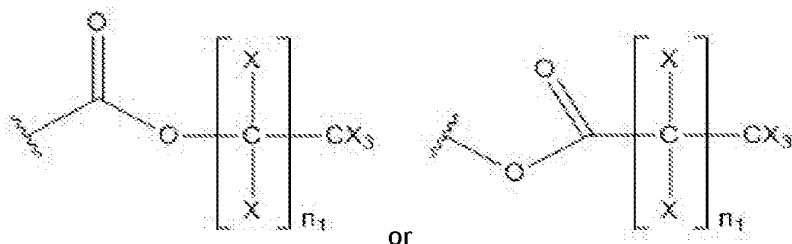
An aspect of the present invention provides an electronic or electrical device or component thereof comprising a protective cross-linked polymeric coating on a surface of the electronic or electrical device or component thereof;

wherein the protective cross-linked polymeric coating is obtainable by exposing the electronic or electrical device or component thereof to a plasma comprising a monomer compound and a crosslinking reagent for a period of time sufficient to allow formation of the protective cross-linked polymeric coating on a surface thereof,

wherein the monomer compound has the following formula:



where R_1 , R_2 and R_4 are each independently selected from hydrogen, optionally substituted branched or straight chain C_1 - C_6 alkyl or halo alkyl or aryl optionally substituted by halo, and R_3 is selected from:



where each X is independently selected from hydrogen, a halogen, optionally substituted branched or straight chain C₁-C₆ alkyl, halo alkyl or aryl optionally substituted by halo; and n₁ is an integer from 1 to 27; and wherein the crosslinking reagent comprises two or more unsaturated bonds attached by means of one or more linker moieties and has a boiling point of less than 500 °C at standard pressure.

High levels of polymer crosslinking (formerly only achievable with high average power continuous wave plasmas) can be achieved by adding a crosslinking molecule to the monomer to produce a cross-linked co-polymer. This has the advantage of increasing the plasma processing range so stable coatings can now be produced in low average energy pulse plasma conditions.

The high degree of retention of the hydrophobic monomer structures from the low energy pulsed plasma gives coatings of the co-polymer a good hydrophobic coating (as evidenced by water contact angles) and a coating that is not sticky or smeary to the touch. Thus, these properties make it suitable for the coating of electronic devices to prevent the ingress of water-based liquids from accidental damage. Such coatings typically have a thickness between 1 and 100 nm, preferably 1 and 50 nm.

The coating may be described as a liquid repellant layer, typically a water repellant layer. The coating may repel water, an aqueous liquid such as rainwater, or oil.

Alternatively, or in addition to providing a liquid repellent layer, the coating may be a physical barrier layer.

The coating may be a physical barrier i.e. it provides a physical barrier to mass and electron transport.

The physical barrier layer restricts diffusion of water, oxygen and ions. When the coating is a physical barrier it typically has a thickness of greater than 50 nm.

The coating is a protective layer i.e. it prevents damage by contact with water or other liquids. The coating may provide its protective functionality by forming a liquid repellent layer and/or a physical barrier layer.

The coating is preferably substantially pin-hole free to enable it to provide a physical barrier. Preferably $\Delta Z/d < 0.15$, where ΔZ is the average height variation on an AFM line scan in nm (as shown in Figure 3) and d is coating thickness in nm.

The value of $\Delta Z/d$ tells us to what extent defects/voids on the surface of the coating extend into the coating, i.e. the percentage value of the depth of defect over total coating thickness. For example, $\Delta Z/d = 0.15$ means that the voids on the surface only extend down to 15% of the coating thickness. A coating with a $\Delta Z/d < 0.15$ is defined herein as being substantially pinhole free. If voids are bigger than this, the desired functionality is unlikely to be achieved.

The coating is preferably conformal, which means that it takes the 3D shape of the electronic or electrical device or component thereof and covers substantially an entire surface of the device. This has the advantage of ensuring that the coating has sufficient thickness to give optimal functionality over an entire surface of the device or component. The meaning of the term “covers substantially an entire surface” will depend to some extent on the type of surface to be covered. For example, for some components, it may be necessary for there to be complete coverage of the surface in order for the component to function after submersion in water. However, for other components or housings, small gaps in coverage may be tolerated.

The coating may have a thickness of 50 to 10,000nm, optionally 50 to 8000nm, 100 to 5000nm, preferably 250nm – 5000nm, most preferably 250nm – 2000nm.

The coating may be electrically insulating and be sufficiently compliant that electrical connectors can be joined to the electronic or electrical device or component thereof and an electrical connection made between the electrical connectors and electronic or electrical device or component thereof without the requirement to first remove the coating. In this case, the force exerted by the electrical connector on the coating is sufficient to alter the structure of or even break through the coating local to the electrical connector, allowing the electrical connection to be made. Electrical connectors can typically be joined to the electronic or electrical device or component in this way for coating thicknesses of under 5000nm, and for high performance coatings below 2000nm.

The electronic or electrical device or component thereof can typically withstand immersion in up to 1m of water for over 30 minutes without failure or corrosion whilst power applied to the electronic or electrical device or component. The effectiveness of the coating can be determined by measuring its electrical resistance at a fixed voltage when submerged in water for a set time period; for example, applying a voltage of 8V to the coating of a device which is submerged in water for 13 minutes. Coatings having a resistance value of 1×10^7 Ohms or higher in this test are effective barrier coatings and the coated electronic or electrical device or component thereof will pass successfully an IPX7 test. The IPX7 test is the Ingress Protection Marking which classifies and rates the degree of protection provided against water. In the IPX7 test for phones, the device is immersed in water under defined conditions of pressure and time (up to 1 m of submersion) for a duration of 30 minutes. The device has to be powered on during testing and functional after 24 hrs.

In one embodiment, the coating is electrically insulating and has a thickness of less than 1 micron and wherein a force of 5- 20g applied to the coating using a round probe with 1 mm diameter allows an electrical connection to be made with the electronic or electrical device or component thereof in the local area where the force has been applied.

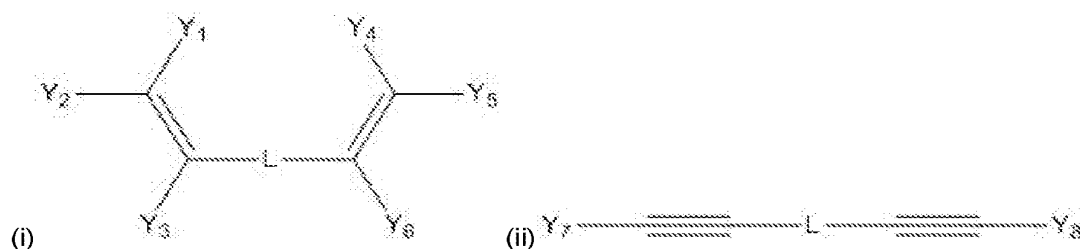
In another embodiment, the coating is electrically insulating and has a thickness of 1-2.5 microns and wherein a force of 20-100g applied to the coating using a round probe with 1 mm diameter allows an electrical connection to be made in the local area of the coating where the force has been applied.

The coating may have a higher density than that of the corresponding monomer from which it is formed. For example, the increase in density may be at least 0.1 g/cm^3 . The increase in density is explained by the highly crosslinked coating. The high density of the coating improves the barrier properties of the coating.

The coating may form a surface defined by a static water contact angle (WCA) of at least 70° . A coating with a WCA of at least 90° is a liquid repellent, typically a water repellent layer. For fluorinated polymers, the coating may have a static water contact angle of at least 100° . The contact angle of a liquid on a solid substrate gives an indication of the surface energy which in turn illustrates the substrate's liquid repellence. Contact angles were measured on a VCA Optima contact angle analyser, using $3 \mu\text{l}$ droplets of deionised water at room temperature.

Crosslinker

A basic requirement for the cross-linking reagent is the presence two or more unsaturated bonds, for example, -C=C- or alkyne groups. The unsaturated bonds are attached by one or more linker moieties. The linker moiety is not especially limited provided it joins together the two or more unsaturated bonds. The cross-linking reagent must have a boiling point of less than 500°C , preferably from -10 to 300°C , optionally 180 to 270°C , most preferably 205 to 260°C at standard pressure. It is a preference, but not a requirement, that the cross-linking reagent is not unduly hazardous for use in plasma processing, that is to say it can be used in a production environment where low levels of vapour do not present any significant health and safety issues (e.g. strongly oxidising, explosive, toxic, or has an offensive odour (e.g. stench reagent)). The crosslinking reagent preferably has one of the following structures:



where Y_1 , Y_2 , Y_3 , Y_4 , Y_5 , Y_6 , Y_7 and Y_8 are each independently selected from hydrogen, optionally substituted cyclic, branched or straight chain $\text{C}_1\text{-C}_8$ alkyl or aryl; and L is a linker moiety.

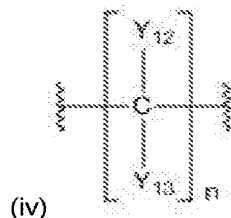
Most preferably L has the following formula:



where each Y_9 is independently selected from, a bond, -O- , -O-C(O)- , -C(O)-O- , $\text{-Y}_{11}\text{-O-C(O)-}$, $\text{-C(O)-O-Y}_{11}\text{-}$, $\text{-OY}_{11}\text{-}$, and $\text{-Y}_{11}\text{O-}$, where Y_{11} is an optionally substituted cyclic, branched or straight chain $\text{C}_1\text{-C}_8$ alkylene; and

Y_{10} is selected from optionally substituted cyclic, branched or straight chain C_1 - C_8 alkylene and a siloxane containing group.

In one most preferred embodiment Y_{10} has the following formula:



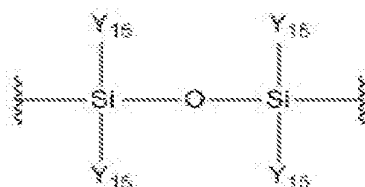
wherein each Y_{12} and Y_{13} is independently selected from H, halo, optionally substituted cyclic, branched or straight chain C_1 - C_8 alkyl, or $-OY_{14}$, where Y_{14} is selected from optionally substituted branched or straight chain C_1 - C_8 alkyl or alkenyl, and n is an integer from 1 to 10.

Optionally, each Y_{12} is H and each Y_{13} is H, i.e. Y_{10} is a linear alkylene chain. For this embodiment Y_9 is preferably a vinyl ester or vinyl ether group.

Optionally, each Y_{12} is fluoro and each Y_{13} is fluoro, i.e. Y_{10} is a linear perfluoroalkylene chain.

Typically, n is from 4 to 6.

In another embodiment Y_{10} has the following formula:

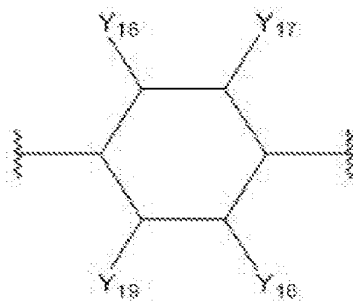


(v)

wherein each Y_{15} is independently selected from optionally substituted branched or straight chain C_1 - C_8 alkyl.

In one embodiment each Y_{15} is methyl, and each Y_9 is a bond.

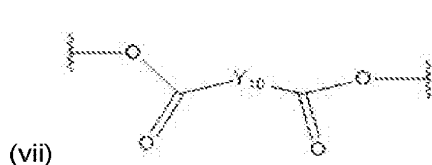
In a further embodiment Y_{10} has the following formula:



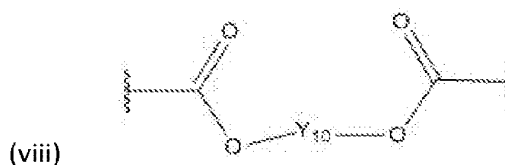
(vi)

wherein Y_{16} to Y_{19} are each independently selected from H and optionally substituted branched or straight chain C_1 - C_8 alkyl or alkenyl. A preferred alkenyl group is vinyl. Optionally Y_{18} is H or vinyl, and Y_{16} , Y_{17} and Y_{19} are each H. In one embodiment each of Y_{16} to Y_{19} is H. In another embodiment Y_{18} is vinyl, and Y_{16} , Y_{17} and Y_{19} are each H.

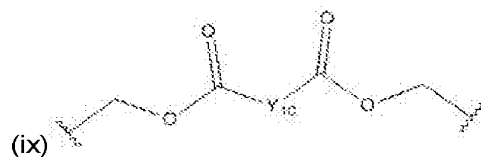
In preferable embodiments for compound (i), L has one of the following structures:



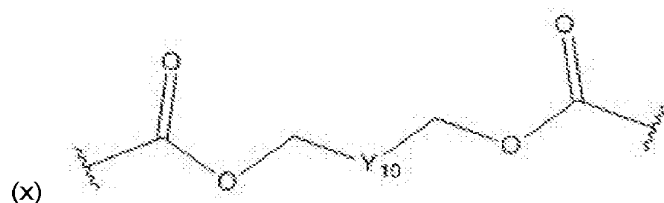
(vii)



(viii)



(ix)

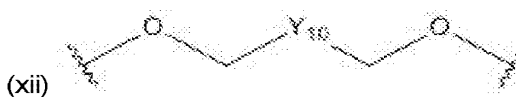


(x)

In another embodiment for compound (i) L has one of the following structures:



(xi)



(xii)

For L according structure (vii), Y_{10} is preferably an alkylene chain or a cycloalkylene, such as those shown in structures iv) and vi) above. The alkylene chain may be a straight chain alkylene chain. When Y_{10} is a cycloalkylene, cyclohexylene is preferred, most preferably 1,4 cyclohexylene.

For L according to structure (viii), Y_{10} is preferably structure (iv), i.e. an alkylene or fluoroalkylene chain.

For L according to structure (ix), Y_{10} is preferably cycloalkylene, such as the cyclohexylene according to structure (vi).

For L according to structure (x), Y_{10} is preferably structure (iv) wherein each Y_{12} and Y_{13} is F, i.e. a perfluoroalkylene chain.

For L according to structure (xi) or structure (xii) Y_{10} is preferably alkylene or cycloalkylene. Optionally the alkylene or cycloalkylene may be substituted with one or more vinyl groups or alkenyl ether groups, preferably one or more vinyl ether groups.

When each Y_9 is a bond, each Y_{10} may be any of structures (iv), (v) and (vi). Preferably Y_{10} is a straight chain alkylene such that the crosslinking reagent is a diene, such as a heptadiene, octadiene, or nonadiene, most preferably 1,7-octadiene.

When each Y_9 is O, each Y_{10} is preferably a branched or straight chain C_1 - C_6 alkylene, preferably a straight chain alkylene, most preferably a C_4 straight chain alkylene, i.e the crosslinking reagent is 1,4-butanediol divinyl ether

It will be understood that each Y_9 group can be combined with any other Y_9 group and Y_{10} group to form the crosslinking reagent.

The skilled person will be aware of possible substituents for each of the cyclic, branched or straight chain C_1 - C_8 alkylene groups mentioned above. The alkylene groups may be substituted at one or more positions by a suitable chemical group. Halo substituents are preferred, with fluoro substituents most preferred. Each C_1 - C_8 alkylene group may be a C_1 - C_3 , C_2 - C_6 , or C_6 - C_8 alkylene group.

Particularly preferred embodiments of the crosslinker have alkyl chains for Y_{10} and vinyl ester or vinyl ether groups on either side.

A particularly preferred cross-linking reagent is divinyl adipate. (a divinyl ester)

Another preferred cross-linking reagent is 1,4-butanediol divinyl ether (a divinyl ether)

For the most preferred embodiments the crosslinking reagent is selected from Divinyl Adipate (DVA), 1,4 Butanediol divinyl ether (BDVE), 1,4 Cyclohexanedimethanol divinyl ether (CDDE), 1,7 -Octadiene (17OD), 1,2,4 -Trivinylcyclohexane (TVCH), 1,3-Divinyltetramethyldisiloxane (DVTMDS), Diallyl 1,4-cyclohexanedicarboxylate (DCHD), 1,6-Divinylperfluorohexane (DVPFH), 1H,1H,6H,6H-Perfluorohexanediol diacrylate (PFHDA) and glyoxal bis (diallyl acetal) (GBDA).

For the alkyne crosslinking reagents according to compound (ii), L is preferably selected from a branched or straight chain C_1 - C_8 alkylene or an ether group. L may be a C_3 , C_4 , C_5 , or C_6 alkylene,

preferably a straight chain alkylene. Chemical structures for the particularly preferred crosslinking reagents are set out below in Table 1:

| | |
|---|--|
| Divinyl Adipate (DVA) | |
| 1,4 Butanediol divinyl ether (BDVE) | |
| 1,4 Cyclohexanedimethanol divinyl ether (CDDE) | |
| 1,7 -Octadiene (17OD) | |
| 1,2,4 -Trivinylcyclohexane (TVCH) | |
| 1,3- Divinyltetramethyldisiloxane (DVTMDS) | |
| Diallyl 1,4- cyclohexanedicarboxylate (DCHD) | |
| 1,6-Divinylperfluorohexane (DVPFH) | |
| 1H,1H,6H,6H- Perfluorohexanediol diacrylate (PFHDA) | |
| GBDA | |

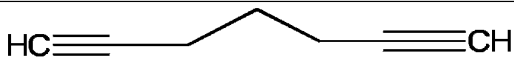
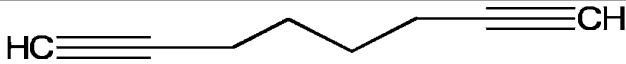
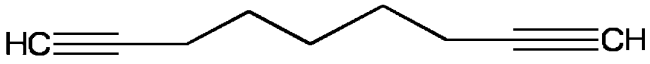
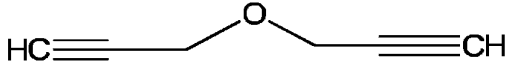
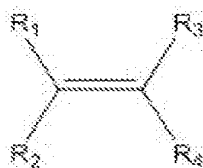
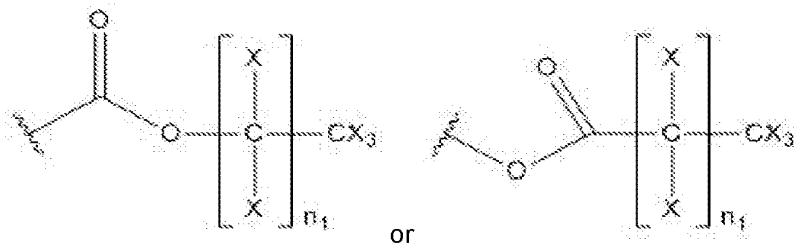
| | |
|-----------------|--|
| 1,6-heptadiyne |  |
| 1,7-heptadiyne |  |
| 1,8-heptadiyne |  |
| Propargyl ether |  |

Table 1*Monomer Compound*

The monomer compound has the following formula:

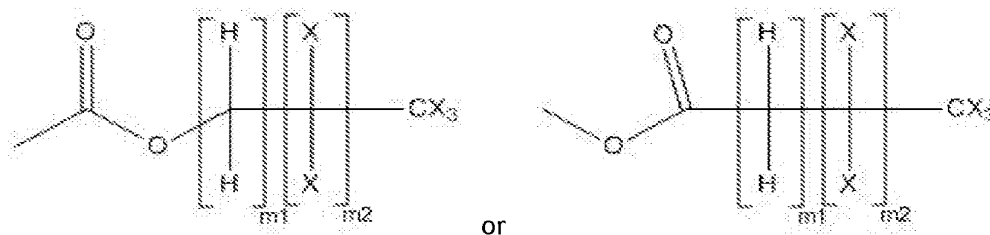


where R_1 , R_2 and R_4 are each independently selected from hydrogen, optionally substituted branched or straight chain C_1 - C_6 alkyl or halo alkyl or aryl optionally substituted by halo, and R_3 is selected from:



where each X is independently selected from hydrogen, a halogen, optionally substituted branched or straight chain C_1 - C_6 alkyl, halo alkyl or aryl optionally substituted by halo; and n_1 is an integer from 1 to 27. Preferably, n_1 is from 1 to 12. Optionally n_1 is from 4 to 12, optionally 6 to 8.

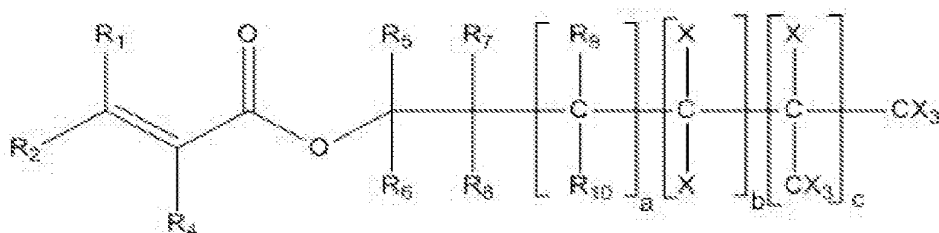
In a preferred embodiment R_3 is selected from:



where m_1 is an integer from 0 to 13 and each X is independently selected from hydrogen, a halogen, optionally substituted branched or straight chain C_1 - C_6 alkyl, halo alkyl or aryl optionally substituted by halo; and m_2 is an integer from 2 to 14;

In a particularly preferred embodiment the monomer is a compound of formula I(a):

I(a)

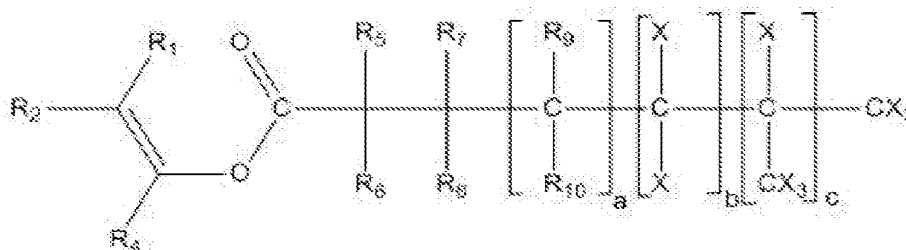


wherein each of R_1 , R_2 , R_4 , and R_5 to R_{10} is independently selected from hydrogen or an optionally substituted C_1 - C_6 branched or straight chain alkyl group; each X is independently selected from hydrogen or halogen; a is from 0-10; b is from 2 to 14; and c is 0 or 1;

; or

the monomer is a compound of formula I(b):

I(b)



wherein each of R_1 , R_2 , R_4 , and R_5 to R_{10} is independently selected from hydrogen or an optionally substituted C_1 - C_6 branched or straight chain alkyl group; each X is independently selected from hydrogen or halogen; a is from 0-10; b is from 2 to 14; and c is 0 or 1.

The halogen may be chlorine or bromine, but is preferably fluorine for compliance with RoHS regulations (Restriction of Hazardous Substances).

a is from 0 to 10, preferably from 0 to 6, optionally 2 to 4, most preferably 0 or 1. b is from 2 to 14, optionally from 2 to 10, preferably 3 to 7.

Each of R₁, R₂, R₄, and R₅ to R₁₀ is independently selected from hydrogen or a C₁-C₆ branched or straight chain alkyl group. The alkyl group may be substituted or unsubstituted, saturated or unsaturated. When the alkyl group is substituted, the location or type of the substituent is not especially limited provided the resultant polymer provides an appropriate liquid repellent and/or barrier layer. The skilled person would be aware of suitable substituents. If the alkyl group is substituted, a preferred substituent is halo, i.e. any of R₁, R₂, R₄, and R₅ to R₁₀ may be haloalkyl, preferably fluoro alkyl. Other possible substituents may be hydroxyl or amine groups. If the alkyl group is unsaturated it may comprise one or more alkene or alkyne groups.

Each of R₁, R₂, R₄, and R₅ to R₁₀ may be independently selected from hydrogen, methyl, ethyl, n-propyl, isopropyl, n-butyl, isobutyl, tert-butyl, sec-butyl, n-pentyl, neopentyl, n-hexyl, isohexyl, and 3-methylpentyl.

In a preferred embodiment each of R₁, R₂, R₄, and R₅ to R₁₀ is independently selected from hydrogen or methyl.

In a preferred embodiment, a and c are each independently 0 or 1; and b is from 3 to 7.

In one preferred embodiment each X is H. In an alternative preferred embodiment each X is F.

Optionally R₁ and R₂ are both hydrogen.

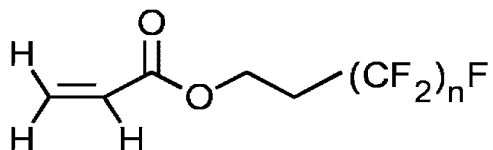
Optionally R₄ is hydrogen or methyl. Preferably R₁ and R₂ are both hydrogen and R₄ is hydrogen or methyl.

Optionally R₉ is hydrogen and R₁₀ is branched or straight chain C₁-C₆ alkyl group. In a preferred embodiment R₁₀ is methyl.

In one embodiment each of R₅ to R₈ is hydrogen.

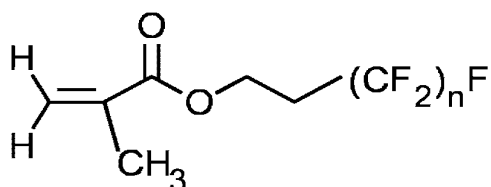
In one embodiment each of R₁, R₂, R₄, and R₅ to R₁₀ is hydrogen, each X is H, a=0 and c=0.

In a particularly preferred embodiment the monomer compound has the following formula:



where n is from 2 to 10.

In another preferred embodiment the monomer compound has the following formula:

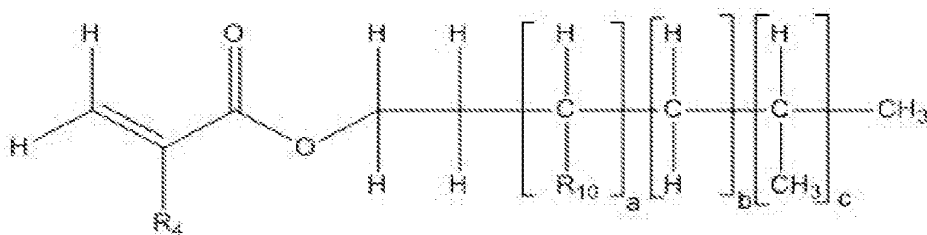


where n is from 2 to 10.

The monomer compound may be selected from 1H,1H,2H,2H-perfluorohexyl acrylate (PFAC4), 1H,1H,2H,2H-perfluorooctyl acrylate (PFAC6), 1H,1H,2H,2H-perfluorodecyl acrylate (PFAC8) and 1H,1H,2H,2H-perfluorododecyl acrylate (PFAC10).

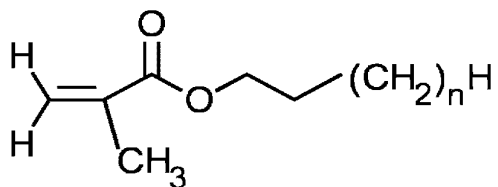
The monomer compound may be selected from 1H,1H,2H,2H-perfluorohexyl methacrylate (PFMAC4), 1H,1H,2H,2H-perfluorooctyl methacrylate (PFMAC6) and 1H,1H,2H,2H-perfluorodecyl methacrylate (PFMAC8).

The monomer compound of formula I(a) may have the following formula:



wherein a and c are each independently 0 or 1, b = 3-7 and n is 4 to 10, where n = a + b + c + 1.

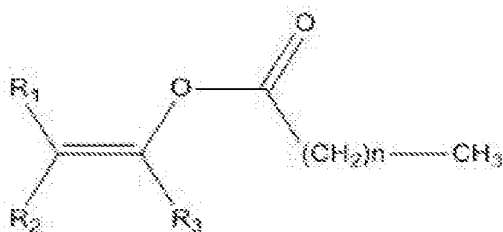
The monomer compound of formula I(a) may have the following formula:



where n is 2 to 12.

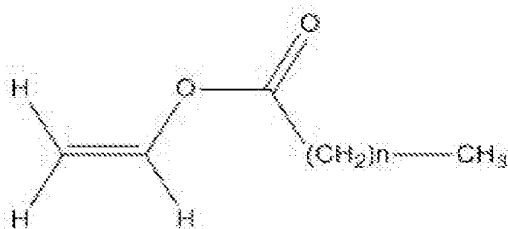
The monomer compound may be selected from ethyl hexyl acrylate, hexyl acrylate, decyl acrylate, lauryl dodecyl acrylate and iso decyl acrylate.

The monomer may have the following formula:



where n is from 4 to 14.

The monomer may have the following formula:

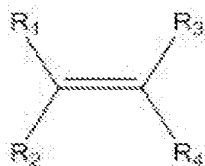


where n is from 4 to 14.

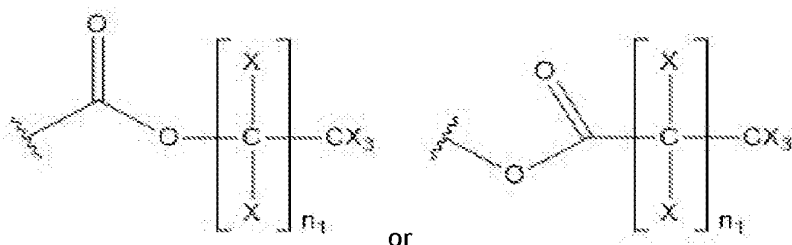
In an additional aspect, the present invention provides a method for treating an electronic device or component thereof comprising:

exposing the electronic or electrical device or component thereof to a plasma comprising a monomer compound and a crosslinking reagent for a period of time sufficient to allow a protective cross-linked polymeric coating to form on a surface of the electronic or electrical device or component thereof;

wherein the monomer compound has the following formula:



where R_1 , R_2 and R_4 are each independently selected from hydrogen, optionally substituted branched or straight chain C_1 - C_6 alkyl or halo alkyl or aryl optionally substituted by halo, and R_3 is selected from:



where each X is independently selected from hydrogen, optionally substituted branched or straight chain C_1 - C_6 alkyl, halo alkyl or aryl optionally substituted by halo; and n_1 is an integer from 1 to 27; and wherein the crosslinking reagent comprises two or more unsaturated bonds and has a boiling point of less than 500°C at standard pressure.

The monomer compound and the crosslinking reagent utilised in the method are described in further detail above.

Typically the electronic or electrical device or component thereof is placed in a plasma deposition chamber, a glow discharge is ignited within said chamber, and a voltage applied as a pulsed field. .

Preferably the applied voltage is at a power of from 30 to 800W. Optionally the voltage is pulsed in a sequence in which the ratio of the time on/time off is in the range of from 0.001 to 1, optionally 0.002 to 0.5. For example, time on may be 10-500 μs , preferably 35-45 μs , or 30-40 μs , such as about 36 μs and time off may be from 0.1 to 30 ms, preferably 0.1 to 20 ms, optionally from 5 to 15 ms, for example 6ms. Time on may be 35 μs , 40 μs , 45 μs . Time off may be 0.1, 1, 2, 3, 6, 8, 10, 15, 20, 25 or 30 ms.

The term pulsed may mean that the plasma cycles between a state of no (or substantially no) plasma emission (off-state) and a state where a particular amount of plasma is emitted (on-state).

Alternatively, pulsed may mean that there is continuous emission of plasma but that the amount of plasma cycles between an upper limit (on-state) and lower limit (off-state).

In a further embodiment, the invention resides in a method of forming a coating on an electronic or electrical device or component thereof as defined above, the method comprising: exposing said substrate in a chamber to a plasma comprising a monomer compound, preferably a continuous plasma, for a sufficient period of time to allow a protective polymeric coating to form on the substrate,

wherein during exposure of the substrate the continuous plasma has a power density of at least 2W/litre, preferably 20W/litre.

Optionally the voltage is applied as a pulsed field for a period of from 30 seconds to 90 minutes. Optionally the voltage is applied as a pulsed field for from 5 to 60 minutes. Optionally in a preliminary step, a continuous power plasma is applied to the electronic or electrical device or component thereof. The preliminary step may be conducted in the presence of an inert gas.

Before the crosslinker and/or the monomer compound enter the deposition chamber each may be in the form of a gas, liquid or a solid (for example a powder) at room temperature. However, it is the preferred that the crosslinking reagent and the monomer compound are both liquid at room temperature, and most preferably that the monomer and crosslinker liquids are miscible.

The monomer compound and/or the crosslinker will suitably be in a gaseous state in the plasma. The plasma may simply comprise a vapour of the monomer compound and the crosslinker. Such a vapour may be formed in-situ, with the compounds being introduced into the chamber in liquid form. The monomer may also be combined with a carrier gas, in particular, an inert gas such as helium or argon.

In preferred embodiments, the monomer and/or the crosslinker may be delivered into the chamber by way of an aerosol device such as a nebuliser or the like, as described for example in WO2003/097245 and WO03/101621, the content of which is incorporated herein by reference. In such an arrangement a carrier gas may not be required, which advantageously assists in achieving high flow rates.

In one embodiment the monomer compound and/or the crosslinking reagent are in gaseous form and are fed into the plasma at a rate of from 10-5000 mg/minute depending on chamber volume, whilst the pulsed voltage is applied.

Optionally the plasma is created with an average power of from 0.001 to 40W/litre.

The crosslinker may be miscible with the monomer and so introduced together or separately into the plasma chamber. Or the crosslinker may be immiscible with the monomer and introduced separately into the plasma chamber. In this context, the term "miscible" means that the crosslinker is soluble in the monomer, and when mixed they form a solution of uniform composition. The term "immiscible" is used to mean that the crosslinker is only partly soluble or insoluble in the monomer, and so either forms an emulsion or separates out into two layers.

The crosslinking reagent is preferably present in an amount of from 10 to 60 (v/v) %, optionally 20 to 40 (v/v) %, optionally 25 to 30 (v/v)%, optionally 30 to 50 (v/v)% of the total volume of monomer compound and crosslinking reagent, depending on the specific crosslinker. The skilled person would understand that the amount will vary to some extent depending on whether the coating is required to

be liquid repellent or provide a barrier to mass and electron transport. The skilled person would understand that the (v/v) percentages are those which give a stable crosslinked polymer coating and the highest water contact angle.

Electronic or electrical device or component thereof

Although the invention is of benefit in the context of a wide variety of substrates, the substrate may, in all aspects of the invention, advantageously be an electronic substrate.

In some embodiments of the invention, the electronic substrate may comprise an electronic or electrical device, i.e. any piece of electrical or electronic equipment. Non-limiting examples of electrical and electronic devices include communications devices such as mobile phones, smartphones and pagers, radios, and sound and audio systems such as loudspeakers, microphones, ringers or buzzers, hearing aids, personal audio equipment such as personal CD, tape cassette or MP3 players, televisions, DVD players including portable DVD players, video recorders, digi and other set-top boxes such as Sky, computers and related components such as laptop, notebook, tablet, phablet or palmtop computers, personal digital assistants (PDAs), keyboards, or instrumentation, games consoles in particular hand-held playstations and the like, data storage devices, outdoor lighting systems or radio antenna and other forms of communication equipment.

In preferred embodiments of the invention, the substrate may comprise or consist of an electronic component, e.g. a printed circuit board (PCB), a printed circuit board array (PCBA), a transistor, resistor, or semi-conductor chip. The electronic component may thus be an internal component of an electronic device, e.g. a mobile phone. The coatings of the invention are particularly valuable in preventing electrochemical migration in such components.

In all aspects of the invention, the precise conditions under which the protective polymeric coating is formed in an effective manner will vary depending upon factors such as, without limitation, the nature of the monomer compound, the crosslinking reagent, the substrate, as well as the desired properties of the coating. These conditions can be determined using routine methods or, preferably, using the techniques and preferred features of the invention described herein, which work in particular synergy with the invention.

The present invention will now be further described with reference to the following non-limiting examples and the accompanying illustrative drawings, of which:

Figure 1 illustrates shows the effect of increasing the power to monomer flow ratio for CW plasmas in a 3 litre chamber in a prior art process.

Figure 2 shows the same effect for pulsed plasma conditions.

Figure 3 shows a tapping mode image over $10 \times 10 \mu\text{m}^2$ field of view of a specimen example (thickness $d=1230 \text{ nm}$) prepared according to Example 1 (left hand side) and a contour line plot showing the data used for calculation of RMS roughness (right hand side). The ΔZ value indicated on the plot has been taken over an area of the graph that represents the majority of the coating. Peaks that lie above the ΔZ range indicate large particles and troughs that fall below the ΔZ range show voids or pinholes in the coating. The width of the peaks also gives an indication of the particle size.

Figure 4 illustrates the effect of adding the divinyl adipate crosslinker to perfluorooctyl acrylate on water contact angle from a process on a 125 Litre chamber.

Figure 5 shows the effect of adding crosslinkers in accordance with the invention on the electrical resistance per nm of the coatings.

Figure 6 shows a tapping mode image of a 90 nm thick coating, prepared as described in Example 3, over $2 \times 2 \mu\text{m}^2$ field of view (top left), a representative contour line indicating height variation (z-axis) of the coating (top right) and a phase image indicating full substrate coverage (bottom left); RMS roughness of the coating is 1.65 nm and $\Delta z/d = 0.05$

Figure 7 shows a FTIR/ATR spectrum of a coating formed according to Example 5.

Example 1

Example of plasma polymerising PFAC6 with no crosslinker in a 3 litre chamber

PFAC6 was polymerised in a 3 litre glass chamber using a continuous plasma process. In the experimental matrix, the continuous plasma was produced using RF power levels of 50, 100 and 200 Watts. The process pressure was run at 30, 60 and 90 mTorr and the monomer flow rate run at 50, 100 and 200 microlitres of liquid per minute. The duration of the process was either 10 or 40 minutes. The coated substrate was silicon wafer and whether the coating was tacky or not was determined by wiping the substrate with a finger and determining by eye if the coating had smeared. The results in Figure 1 indicate that for lower CW powers, the coating could easily be smeared (i.e. tacky), but by increasing the power to flow ratio, the coating became resistant to smearing (i.e. non-tacky) and this is expected to be due to additional crosslinking caused by more monomer fragmentation. The water contact angle was also seen to decline with increasing power to flow ratio, and this too is indicative of more power increasing the level of monomer fragmentation.

The same 3 litre chamber was used to also look at the effect of power to flow ratios with pulsed plasma polymerisation. In these experiments, the peak power was either 250 or 500 W. The RF power feed was pulsed with an on time of 35 microseconds and off times of 5.5 and 1 millisecond. Process pressure was either 20, 35, 60 or 80 mTorr. The monomer flow was either 50 or 200 microlitres of liquid per minute. Figure 2 shows that low power to flow conditions can generate coatings that can be smeared (tacky) and only by increasing the power could the smear be reduced. This too had a concomitant decline in water contact angle.

The results from Figures 1 and 2 show that plasma processing PFAC6 on its own could only lead to smear –free coatings when the powers were high enough to decrease the other desirable property of the water contact angle. This illustrates the need to add a crosslinker to prevent the coating smearing and maintain the water contact angle.

Example 2

Examples of Perfluoro Alkyl Acrylate co-polymerisation with Crosslinker to show improvements in electrical resistance

Perfluorooctyl acrylate monomer was mixed with a single crosslinker from the following list:

VINYL ESTERS: Divinyl adipate (DVA)

VINYL ETHERS: 1,4-Butanediol divinyl ether (BDVE); 1,4 Cyclohexanedimethanol divinyl ether (CDDE)

DI- or TRI- VINYLS: 1,7-Octadiene (1,7-OD); 1,2,4-Trivinylcyclohexane (TVCH);

DI VINYL with alkyl fluoro group: 1,6-divinylperfluorohexane (DVPFH)

DI VINYL with silicon group: 1,3-Divinyl tetramethyldisiloxane (DVTMDS)

DI VINYL with cyclic ring and carboxylate groups: Diallyl 1,4-cyclohexanedicarboxylate (DCHC)

DI ACRYLATE: 1H1H,6H,6H-perfluorohexanediol diacrylate (PFHDA)

The crosslinker was mixed with the PFAC6 as a percentage by volume for the following percentages: 20, 40, 60, 80, and 100%.

Plasma initiated polymerisation reactions were carried out in a 3 litre glass plasma chamber. The substrates were test circuit boards. The PFAC6/crosslinker mixture was introduced at a rate of 0.04 – 0.06 ul/min and the process pressure was 40 mTorr. The process plasma consisted of a 1 minute continuous plasma (CW) step of 50W followed by 10 - 20 minute pulsed plasma (PW) step of 50W and RF power being delivered by a pulse sequence with a duty cycle of 6.9%. The coated circuit boards were immersed in tap water and an 8V potential was applied for 13 minutes. The final current readings, together with coating thickness measurements, were used to calculate the electrical resistance per nm of coating. Figure 5 below shows the effect of adding a range of crosslinkers on the electrical resistance per nm of the barrier coatings. Figure 5 clearly shows the improvement in resistance that can be gained by adding the crosslinker to the PFAC6 monomer. Furthermore, all of the crosslinkers tested gave non-smear/non-tacky coatings with concentration levels of 20% and above, with the exceptions of DVTMDS, DCHD, PFHDA and DVPFH which were non-smear at levels of 40% and above.

Example 3

Example of crosslinker concentration on water repellent coating.

Water repellent coatings were prepared in a 125 litre volume chamber using PFAC6 with different levels of divinyl adipate (DVA) and using helium as a carrier gas. The deposition process consisted of a 3 minute CW step with 300W power and a pulsed step with 150W power and an RF pulse duty cycle of 0.018%. Silicon wafer was used as the test substrates and the contact angle of the coated wafer was determined by applying a 3ul drop of deionised water onto the coated wafer and measuring the contact angle using a VCA Optima (AST products) with image analysis software. The variation of contact angle with (v/v)% DVA crosslinker is shown in Figure 4. It was also noted that for the 10 (v/v) % DVA concentration, the water drop left a mark on the coated wafer where it's circumference had been. This indicated that a higher level of crosslinker was required to give a more stable coating. This observation, in combination with the results in figure 4, suggest an optimum range of 20 – 40 (v/v) % DVA crosslinker, though this is likely to vary, typically within the range of from 10-60 (v/v) % for different monomers and crosslinkers and different chamber sizes.

Pulsed plasma polymerisation of perfluoro alkyl acrylate in the presence of divinyl adipate (DVA) was carried out in a 125 litre chamber. Figure 4 shows the effect of adding different liquid volume percentages of DVA crosslinker to perfluorooctyl acrylate on water contact angle on a silicon wafer substrate.

Example 4

Example of AFM measurement of monomer/crosslinker co-polymer coating

A barrier-style coating was prepared in a 22 litre volume chamber using PFAC6 with 10% DVA. The deposition process consisted of a 1 minute CW step with a CW power to flow ratio of 3.9 (W/microliter/min), and a pulsed step with a PW power/monomer flow ratio of 0.28 (W/microlitres/min). Figure 6 shows the representative topographical and phase contrast images were obtained from all the samples. High spatial resolution images show mainly the structures of areas between the raised features. The RMS roughness of the coating is 1.65 nm and the $\Delta z/d = 0.05$. As the $\Delta z/d$ value is < 0.15, then this indicates that the physical layer is substantially pinhole free.

Example 5

Example of FTIR/ATR measurement of monomer/crosslinker co-polymer coating

Two barrier-style coatings were prepared in a 22 litre volume chamber using PFAC6 only and PFAC6 with 10% DVA as described in example 4. The deposition process consisted of a 1 minute CW step with a CW power to flow ratio of 3.9 (W/microliter/min), and a pulsed step with a PW power/monomer flow ratio of 0.28 (W/microlitres/min). Figure 7 shows the representative FTIR/ATR spectrum obtained from both samples.

The FTIR/ATR intensity ratios of peaks attributed to stretching mode of CF_3 and C=O groups, $\text{CF}_3/\text{C=O}$, of the coating is indicative of sufficient cross linking in the coating to form a physical barrier. CF_3 refers to the terminal groups in the side chain of PFAC6.

Formation of the barrier coatings is believed to be caused by a mixture of cross linking and controlled fragmentation of the monomers during polymerisation. Cross linking is believed to be predominantly via the $\text{CF}_2\text{-CF}_3$ chain, whilst fragmentation is thought to be predominantly through loss of the C=O group during polymerisation and to a lesser extent shortening of the CF_2 chain. Cross linking effects the abundance of $-\text{CF}_3$ groups in the coating and controlled fragmentation controls the amount of C=O groups in the coating. The ratio of these two functional groups is an indication that sufficient cross-linking and fragmentation has taken place and can be measured by the ratio of the intensities of the corresponding FTIR/ATR peaks.

It has been shown that coatings with reduced $\text{CF}_3 / \text{C=O}$ ratios give higher resistance values in the electrical test, as described in Example 2, showing an improved coating performance on increased cross linking (for CF_3) and fragmentation (for C=O). If the FTIR/ATR intensity ratio of the peaks attributed to $-\text{CF}_3$ stretching and C=O stretching, $\text{CF}_3/\text{C=O}$, is less than $0.6e^{-0.1n}$ (where $n=6$ for PFAC6) the resistance of the coating at the electrical test is expected to be higher than 8MOhms.

The peak intensity ratio $\text{CF}_3/\text{C=O}$ the coating thickness and the final current readings during the electrical test are shown in table 2:

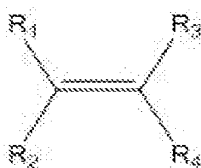
| | Thickness (nm) | Resistance water at 8V (Ohms) | in $\text{CF}_3/\text{C=O}$ ATR peak intensity ratio |
|------------------------------------|-------------------|-------------------------------------|--|
| PFAC6 | 1480 | 2.1×10^7 | 0.23 |
| PFAC6 and 10%DVA (in volume) | 1450 | 9.2×10^9 | 0.11 |

Table 2

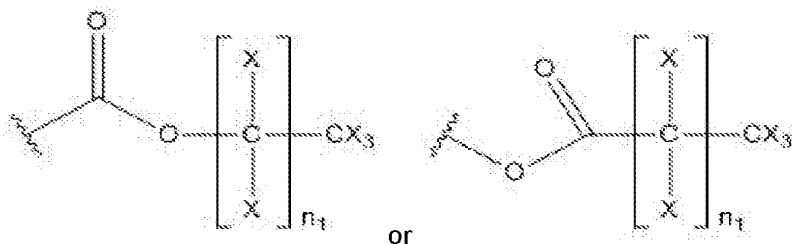
Claims:

1. An electronic or electrical device or component thereof comprising a protective cross-linked polymeric coating on a surface of the electronic or electrical device or component thereof; wherein the protective cross-linked polymeric coating is obtainable by exposing the electronic or electrical device or component thereof to a plasma comprising a monomer compound and a crosslinking reagent for a period of time sufficient to allow formation of the protective cross-linked polymeric coating on a surface thereof,

wherein the monomer compound has the following formula:

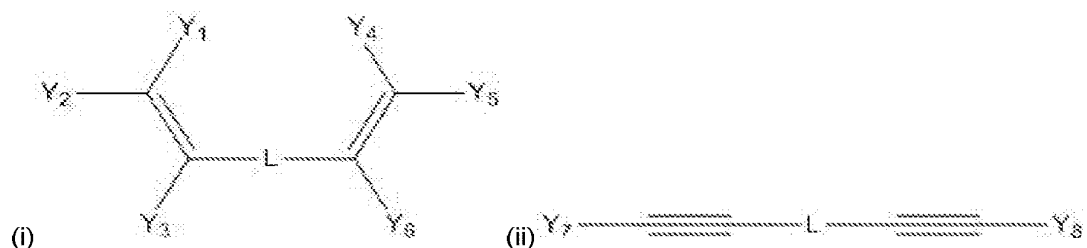


where R_1 , R_2 and R_4 are each independently selected from hydrogen, optionally substituted branched or straight chain C_1 - C_8 alkyl or halo alkyl or aryl optionally substituted by halo, and R_3 is selected from:



where each X is independently selected from hydrogen, a halogen, optionally substituted branched or straight chain C_1 - C_8 alkyl, halo alkyl or aryl optionally substituted by halo; and n_1 is an integer from 1 to 27; and wherein the crosslinking reagent comprises two or more unsaturated bonds attached by means of one or more linker moieties and has a boiling point of less than 500 °C at standard pressure.

2. An electronic or electrical device or component thereof according to claim 1, wherein the protective cross-linked polymeric coating is a physical barrier to mass and electron transport.
3. An electronic or electrical device or component thereof according to claim 1 or claim 2, wherein the protective cross-linked polymeric coating forms a liquid repellent surface defined by a static water contact angle (WCA) of at least 90°.
4. An electronic or electrical device or component thereof according to any preceding claim, wherein the crosslinking reagent has one of the following structures:



where Y_1 , Y_2 , Y_3 , Y_4 , Y_5 , Y_6 , Y_7 and Y_8 are each independently selected from hydrogen, optionally substituted cyclic, branched or straight chain C_1 - C_8 alkyl or aryl; and L is a linker moiety.

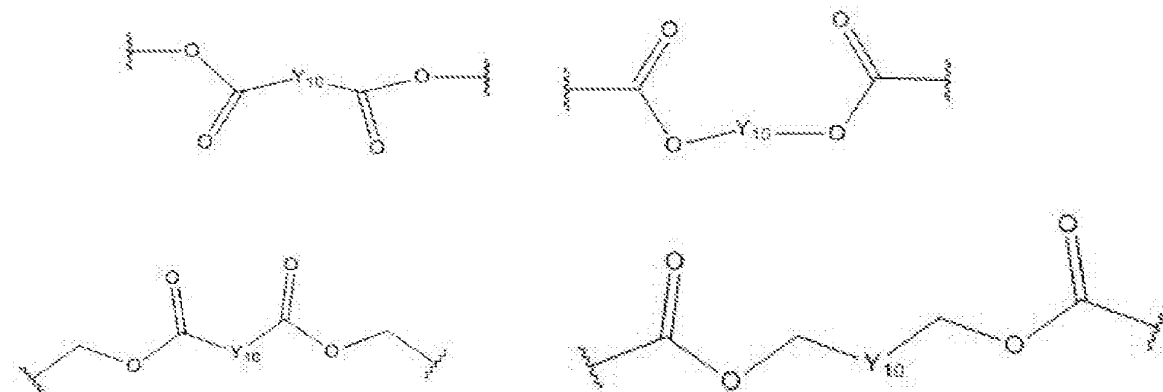
5. An electronic or electrical device or component thereof according to claim 4, wherein for compound (i) L has the following formula:



each Y_9 is independently selected from, a bond, $-O-$, $-O-C(O)-$, $-C(O)-O-$, $-Y_{11}-O-C(O)-$, $-C(O)-O-Y_{11}-$, $-OY_{11}-$, and $-Y_{11}O-$, where Y_{11} is an optionally substituted cyclic, branched or straight chain C_1 - C_8 alkylene; and

Y_{10} is selected from optionally substituted cyclic, branched or straight chain C_1 - C_8 alkylene and a siloxane group.

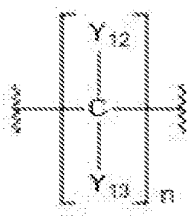
6. An electronic or electrical device or component thereof according to claim 5, wherein for compound (i) L has one of the following structures:



7. An electronic or electrical device or component thereof according to claim 5, wherein for compound (i) L has one of the following structures:

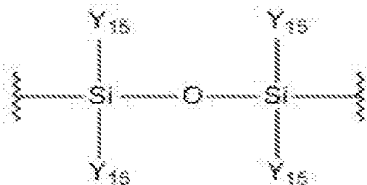


8. An electronic or electrical device or component thereof according to any of claims 5 to 7, wherein Y_{10} has the following formula:



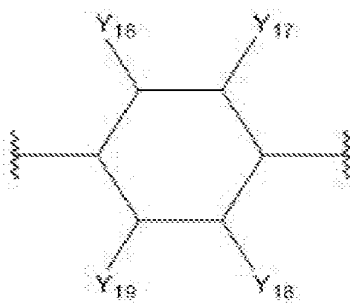
wherein each Y_{12} and Y_{13} is independently selected from H, halo, optionally substituted cyclic, branched or straight chain alkyl, or $-OY_{14}$, where Y_{14} is selected from optionally substituted branched or straight chain C_1 - C_8 alkyl or alkenyl, and n is an integer from 1 to 10.

9. An electronic or electrical device or component thereof according to claim 8, wherein each Y_{12} is H and each Y_{13} is H.
10. An electronic or electrical device or component thereof according to claim 8, wherein each Y_{12} is fluoro and each Y_{13} is fluoro.
11. An electronic or electrical device or component thereof according to any of claims 8 to 10, wherein n is from 4 to 6.
12. An electronic or electrical device or component thereof according to any of claims 5 to 7, wherein Y_{10} has the following formula:



Wherein each Y_{15} is independently selected from optionally substituted branched or straight chain C_1 - C_6 alkyl.

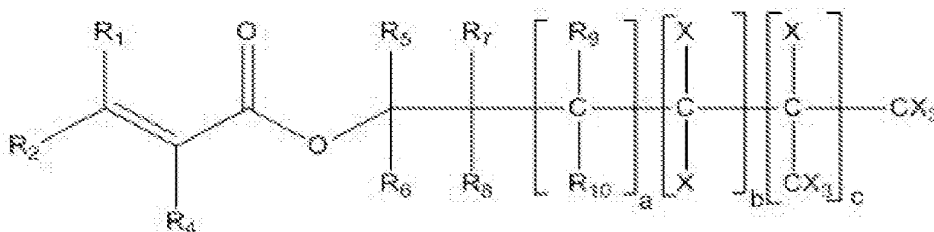
13. An electronic or electrical device or component thereof according to claim 12, wherein each Y_{15} is methyl, and each Y_9 is a bond.
14. An electronic or electrical device or component thereof according to any of claims 5 to 7, wherein Y_{10} has the following formula:



wherein Y_{16} to Y_{19} are each independently selected from H and optionally substituted branched or straight chain C_1 - C_8 alkyl or alkenyl.

15. An electronic or electrical device or component thereof according to claim 14, wherein Y_{18} is H or vinylene, and Y_{16} , Y_{17} and Y_{19} are each H.
16. An electronic or electrical device or component thereof according to any preceding claim wherein the crosslinking reagent is selected from Divinyl Adipate (DVA), 1,4 Butanediol divinyl ether (BDVE), 1,4 Cyclohexanedimethanol divinyl ether (CDDE), 1,7 -Octadiene (17OD), 1,2,4 -Trivinylcyclohexane (TVCH), 1,3-Divinyltetramethyldisiloxane (DVTMDS), Diallyl 1,4-cyclohexanedicarboxylate (DCHD), 1,6-Divinylperfluorohexane (DVPFH), 1H,1H,6H,6H-Perfluorohexanediol diacrylate (PFHDA) and glyoxal bis (diallyl acetal) (GBDA).
17. An electronic or electrical device or component thereof according to claim 4, wherein for compound (ii), L is selected from a branched or straight chain C_1 - C_8 alkylene or an ether group.
18. An electronic or electrical device or component thereof according to any preceding claim wherein the monomer is a compound of formula I(a):

I(a)

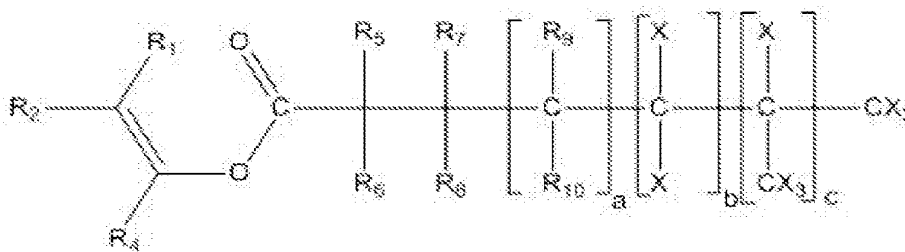


wherein each of R_1 , R_2 , R_4 , and R_5 to R_{10} is independently selected from hydrogen or an optionally substituted C_1 - C_8 branched or straight chain alkyl group; each X is independently selected from hydrogen or halogen; a is from 0-10; b is from 2 to 14; and c is 0 or 1;

; or

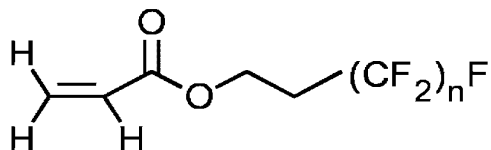
wherein the monomer is a compound of formula I(b):

I(b)



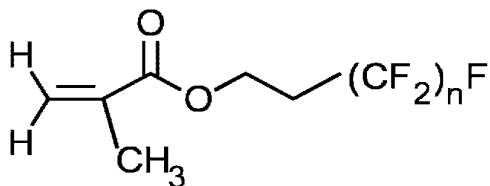
wherein each of R₁, R₂, R₄, and R₅ to R₁₀ is independently selected from hydrogen or an optionally substituted C₁-C₆ branched or straight chain alkyl group; each X is independently selected from hydrogen or halogen; a is from 0-10; b is from 2 to 14; and c is 0 or 1.

19. An electronic or electrical device or component thereof according to claim 18, wherein the halogen is fluorine.
20. An electronic or electrical device or component thereof according to claim 18 or claim 19, wherein each of R₁, R₂, R₄, and R₅ to R₁₀ is independently selected from hydrogen, methyl, ethyl, n-propyl, isopropyl, n-butyl, isobutyl, tert-butyl, sec-butyl, n-pentyl, neopentyl, n-hexyl, isohexyl, and 3-methylpentyl.
21. An electronic or electrical device or component thereof according to claim 20, wherein each of R₁, R₂, R₄, and R₅ to R₁₀ is independently selected from hydrogen or methyl.
22. An electronic or electrical device or component thereof according to any of claims 18 to 20, wherein a and c are each independently 0 or 1; and b is from 3 to 7.
23. An electronic or electrical device or component thereof according to any preceding claim wherein each X is H.
24. An electronic or electrical device or component thereof according to any of claims 1 to 22, wherein each X is F.
25. An electronic or electrical device or component thereof according to any of claims 18 to 24, wherein R₁ and R₂ are both hydrogen.
26. An electronic or electrical device or component thereof according to any of claims 18 to 25, wherein R₄ is hydrogen or methyl.
27. An electronic or electrical device or component thereof according to any of claims 18 to 26, wherein R₉ is hydrogen and R₁₀ is branched or straight chain C₁-C₆ alkyl group.
28. An electronic or electrical device or component thereof according to claim 27, wherein R₁₀ is methyl.
29. An electronic or electrical device or component thereof according to any of claims 18 to 28, wherein each of R₅ to R₈ is hydrogen.
30. An electronic or electrical device or component thereof according to any of claims 18 to 29 wherein each of each of R₁, R₂, R₄, and R₅ to R₁₀ is hydrogen, each X is H, a=0 and c=0.
31. An electronic or electrical device or component thereof according to any of claims 24 to 30, wherein the compound of formula I(a) has the following formula:



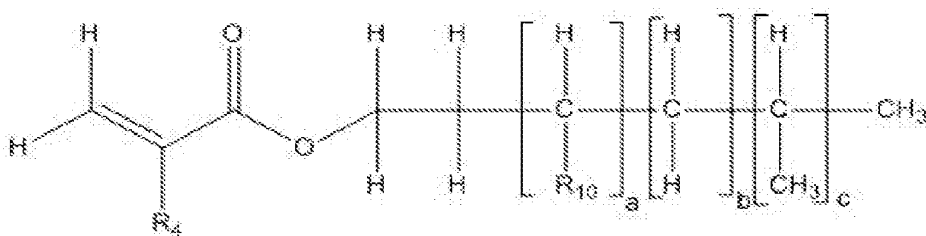
where n is from 2 to 10.

32. An electronic or electrical device or component thereof according to any of claims 24 to 30, wherein the compound of formula I(a) has the following formula:



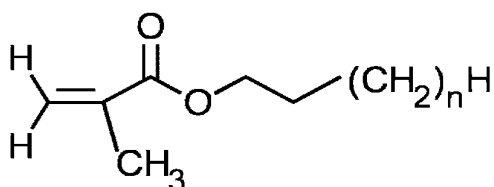
where n is from 2 to 10.

33. An electronic or electrical device or component thereof according to claim 31, wherein the compound of formula I(a) is selected from 1H,1H,2H,2H-perfluorohexyl acrylate (PFAC4), 1H,1H,2H,2H-perfluorooctyl acrylate (PFAC6), 1H,1H,2H,2H-perfluorodecyl acrylate (PFAC8) and 1H,1H,2H,2H-perfluorododecyl acrylate (PFAC10).
34. An electronic or electrical device or component thereof according to claim 32, wherein the compound of formula I(a) is selected from 1H,1H,2H,2H-perfluorohexyl methacrylate (PFMAC4), 1H,1H,2H,2H-perfluorooctyl methacrylate (PFMAC6) and 1H,1H,2H,2H-perfluorodecyl methacrylate (PFMAC8).
35. An electronic or electrical device or component thereof according to any of claims 18 to 23, wherein the compound of formula I(a) has the following formula:



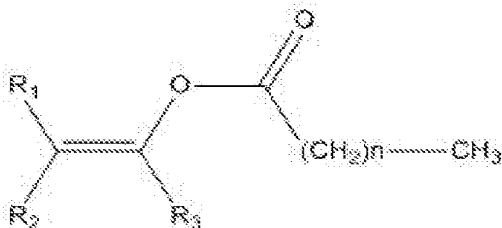
wherein a and c are each independently 0 or 1, b=3-7 and n is 4 to 10, where n=a+b+c+1.

36. An electronic or electrical device or component thereof according to any of claims 18 to 23, wherein the compound of formula I(a) has the following formula:



where n is 2 to 12.

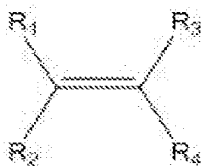
37. An electronic or electrical device or component thereof according to claim 35 or claim 36, wherein the compound of formula I(a) is selected from ethyl hexyl acrylate, hexyl acrylate, decyl acrylate, lauryl dodecyl acrylate and iso decyl acrylate.
38. An electronic or electrical device or component thereof according to any of claims 18 to 23, wherein the monomer is a compound of formula I(b) and has the following formula:



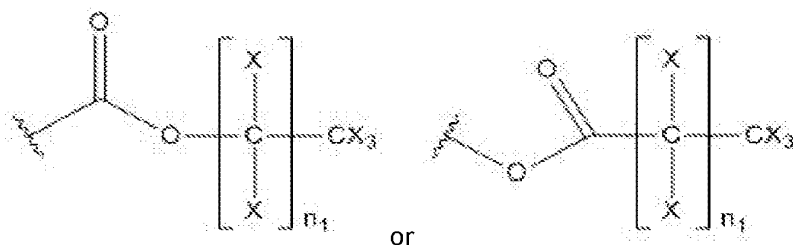
where n is from 4 to 14, optionally where R₁, R₂ and R₃ are each H.

39. An electronic or electrical device or component thereof according to any of the preceding claims, wherein the electronic or electrical device or component thereof is selected from mobile phones, smartphones, pagers, radios, sound and audio systems such as loudspeakers, microphones, ringers and/or buzzers, hearing aids, personal audio equipment such as personal CD, tape cassette or MP3 players, televisions, DVD players including portable DVD players, video recorders, digi and other set-top boxes, computers and related components such as laptop, notebook, tablet, phablet or palmtop computers, personal digital assistants (PDAs), keyboards, or instrumentation, games consoles, data storage devices, outdoor lighting systems, radio antennae and other forms of communication equipment, and printed circuit boards.
40. A method for treating an electronic or electrical device or component thereof comprising:
- exposing the electronic or electrical device or component thereof to a plasma comprising a monomer compound and a crosslinking reagent for a period of time sufficient to allow a protective cross-linked polymeric coating to form on a surface of the electronic or electrical device or component thereof;

wherein the monomer compound has the following formula:

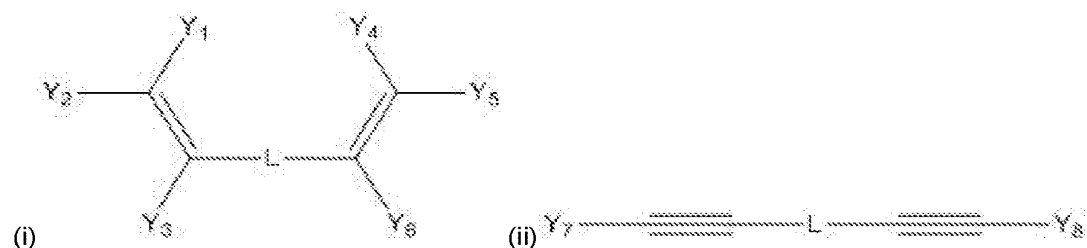


where R_1 , R_2 and R_4 are each independently selected from hydrogen, optionally substituted branched or straight chain C_1 - C_8 alkyl or halo alkyl or aryl optionally substituted by halo, and R_3 is selected from:



where each X is independently selected from hydrogen, optionally substituted branched or straight chain C_1 - C_8 alkyl, halo alkyl or aryl optionally substituted by halo; and n_1 is an integer from 1 to 27; and wherein the crosslinking reagent comprises two or more unsaturated bonds attached by means of one or more linker moieties and has a boiling point at standard pressure of less than 500°C .

41. A method according to claim 40, wherein the crosslinking reagent has one of the following structures:



where Y_1 , Y_2 , Y_3 , Y_4 , Y_5 , Y_6 , Y_7 and Y_8 are each independently selected from hydrogen, optionally substituted cyclic, branched or straight chain C_1 - C_8 alkyl or aryl; and L is a linker moiety.

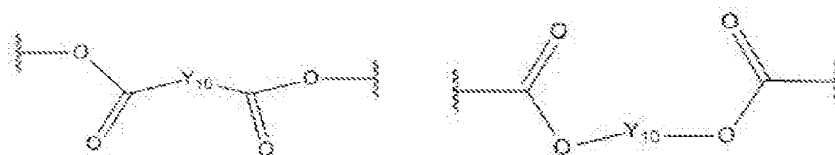
42. A method according to claim 41, wherein for compound (i) L has the following formula:

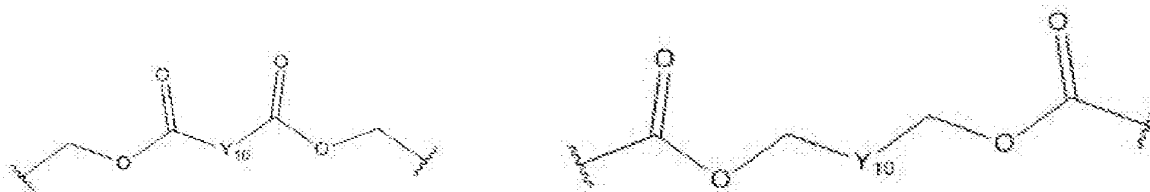


each Y_9 is independently selected from, a bond, $-\text{O}-$, $-\text{O}-\text{C}(\text{O})-$, $-\text{C}(\text{O})-\text{O}-$, $-\text{Y}_{11}-\text{O}-\text{C}(\text{O})-$, $-\text{C}(\text{O})-\text{O}-\text{Y}_{11}-$, $-\text{OY}_{11}-$, and $-\text{Y}_{11}\text{O}-$, where Y_{11} is an optionally substituted cyclic, branched or straight chain C_1 - C_8 alkylene; and

Y_{10} is selected from optionally substituted cyclic, branched or straight chain C_1 - C_8 alkylene and a siloxane group.

43. A method according to claim 42, wherein for compound (i) L has one of the following structures:

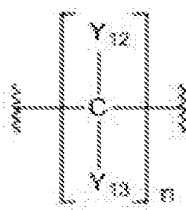




44. A method according to claim 42, wherein for compound (i) L has one of the following structures:

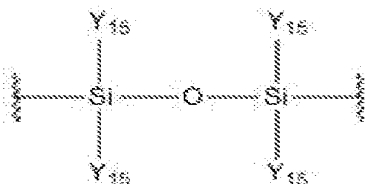


45. An method according to any of claims 42 to 44, wherein Y_{10} has the following formula:



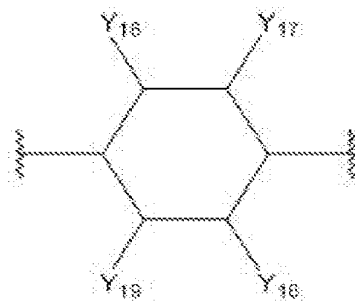
wherein each Y_{12} and Y_{13} is independently selected from H, halo, optionally substituted cyclic, branched or straight chain alkyl, or $-OY_{14}$, where Y_{14} is selected from optionally substituted branched or straight chain C_1 - C_8 alkyl or alkenyl, and n is an integer from 1 to 10.

46. A method according to claim 45, wherein each Y_{12} is H and each Y_{13} is H.
 47. A method according to claim 45, wherein each Y_{12} is fluoro and each Y_{13} is fluoro.
 48. A method according to any of claims 45 to 47, wherein n is from 4 to 6.
 49. A method according to any of claims 42 to 44, wherein Y_{10} has the following formula:



Wherein each Y_{15} is independently selected from optionally substituted branched or straight chain C_1 - C_6 alkyl.

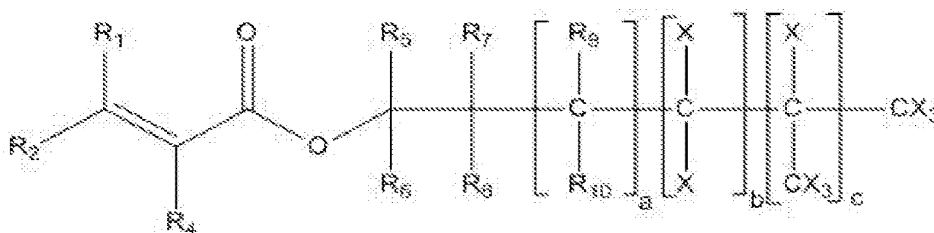
50. A method according to claim 49, wherein each Y_{15} is methyl, and each Y_9 is a bond.
 51. A method according to any of claims 42 to 44, wherein Y_{10} has the following formula:



wherein Y₁₆ to Y₁₉ are each independently selected from H and optionally substituted branched or straight chain C₁-C₈ alkyl or alkenyl.

52. An method according to claim 51, wherein Y₁₈ is vinylene or H, and Y₁₆, Y₁₇ and Y₁₉ are each H.
53. A method according to any of claims 40 to 52, wherein the crosslinking reagent is selected from Divinyl Adipate (DVA), 1,4 Butanediol divinyl ether (BDVE), 1,4 Cyclohexanedimethanol divinyl ether (CDDE), 1,7 -Octadiene (17OD), 1,2,4 -Trivinylcyclohexane (TVCH), 1,3-Divinyltetramethyldisiloxane (DVTMDS), Diallyl 1,4-cyclohexanedicarboxylate (DCHD), 1,6-Divinylperfluorohexane (DVPFH), 1H,1H,6H,6H-Perfluorohexanediol diacrylate (PFHDA) and glyoxal bis (diallyl acetal) (GBDA).
54. A method according to claim 41, wherein for compound (ii), L is selected from a branched or straight chain C₁-C₈ alkylene or an ether group.
55. A method according to any of claims 40 to 54 wherein the monomer is a compound of formula I(a):

I(a)

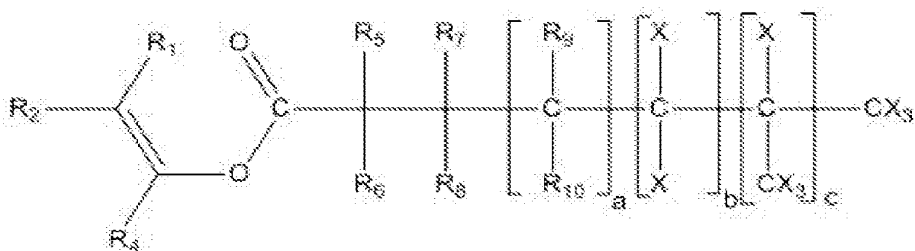


wherein each of R₁, R₂, R₄, and R₅ to R₁₀ is independently selected from hydrogen or an optionally substituted C₁-C₈ branched or straight chain alkyl group; each X is independently selected from hydrogen or halogen; a is from 0-10; b is from 2 to 14; and c is 0 or 1;

; or

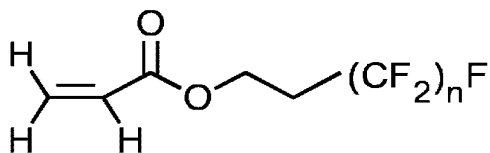
wherein the monomer is a compound of formula I(b):

I(b)



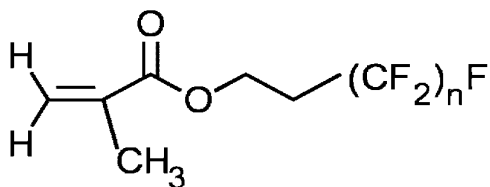
wherein each of R_1 , R_2 , R_4 , and R_5 to R_{10} is independently selected from hydrogen or an optionally substituted C_1 - C_6 branched or straight chain alkyl group; each X is independently selected from hydrogen or halogen; a is from 0-10; b is from 2 to 14; and c is 0 or 1.

56. A method according to claim 55, wherein the halogen is fluorine.
57. A method according to claim 55 or claim 56, wherein each of R_1 , R_2 , R_4 , and R_5 to R_{10} is independently selected from hydrogen, methyl, ethyl, *n*-propyl, isopropyl, *n*-butyl, isobutyl, *tert*-butyl, *sec*-butyl, *n*-pentyl, neopentyl, *n*-hexyl, isohexyl, and 3-methylpentyl.
58. A method according to claim 57, wherein each of R_1 , R_2 , R_4 , and R_5 to R_{10} is independently selected from hydrogen or methyl.
59. A method according to any of claims 55 to 58, wherein a and c are each independently 0 or 1; and b is from 3 to 7.
60. A method according to any preceding claim wherein each X is H.
61. A method according to any of claims 41 to 59, wherein each X is F.
62. A method according to any of claims 41 to 61, wherein R_1 and R_2 are both hydrogen.
63. A method according to any of claims 41 to 62, wherein R_4 is hydrogen or methyl.
64. A method according to any of claims 41 to 63, wherein R_9 is hydrogen and R_{10} is branched or straight chain C_1 - C_6 alkyl group.
65. A method according to claim 64, wherein R_{10} is methyl.
66. A method to any of claims 41 to 65, wherein each of R_5 to R_8 is hydrogen.
67. A method according to any of claims 41 to 66 wherein each of each of R_1 , R_2 , R_4 , and R_5 to R_{10} is hydrogen, each X is H, $a=0$ and $c=0$.
68. A method according to any of claims 61 to 67, wherein the compound of formula I(a) has the following formula:



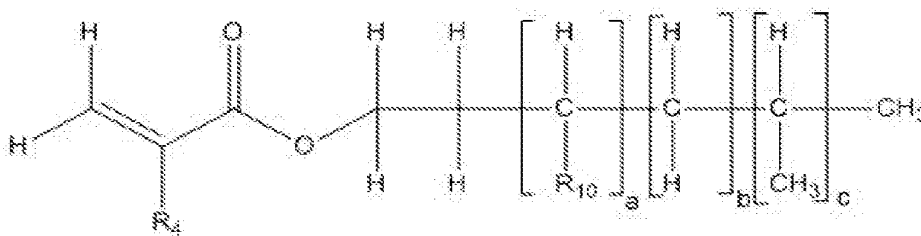
where n is from 2 to 10.

69. A method according to any of claims 61 to 67, wherein the compound of formula I(a) has the following formula:



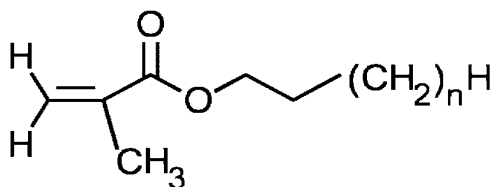
where n is from 2 to 10.

70. A method according to claim 68, wherein the compound of formula I(a) is selected from 1H,1H,2H,2H-perfluorohexyl acrylate (PFAC4), 1H,1H,2H,2H-perfluorooctyl acrylate (PFAC6), 1H,1H,2H,2H-perfluorodecyl acrylate (PFAC8) and 1H,1H,2H,2H-perfluorododecyl acrylate (PFAC10).
71. A method according to claim 69, wherein the compound of formula I(a) is selected from 1H,1H,2H,2H-perfluorohexyl methacrylate (PFMAC4), 1H,1H,2H,2H-perfluorooctyl methacrylate (PFMAC6) and 1H,1H,2H,2H-perfluorodecyl methacrylate (PFMAC8).
72. A method according to any of claims 41 to 61, wherein the compound of formula I(a) has the following formula:



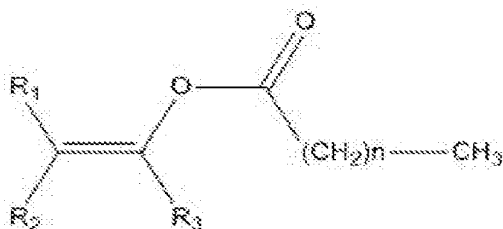
wherein a and c are each independently 0 or 1, b = 3-7 and n is 4 to 10, where n = a + b + c + 1.

73. A method according to any of claims 41 to 61, wherein the compound of formula I(a) has the following formula:



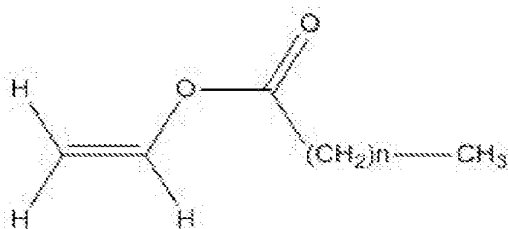
where n is 2 to 12.

74. A method according to claim 72 or claim 73, wherein the compound of formula I(a) is selected from ethyl hexyl acrylate, hexyl acrylate, decyl acrylate, lauryl dodecyl acrylate and iso decyl acrylate.
75. A method according to any of claims 41 to 60, wherein the monomer is a compound of formula I(b) and has the following formula:



where n is from 4 to 14.

76. A method according to claim 75 wherein the monomer is a compound of formula I(b) and has the following formula:



where n is from 4 to 14.

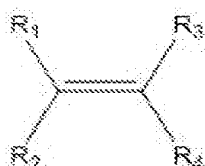
77. A method according to any of claims 40 to 76 wherein the electronic or electrical device or component thereof is placed in a plasma deposition chamber, a glow discharge is ignited within said chamber, and a voltage applied as a pulsed field.
78. A method according to claim 77 wherein applied voltage is at a power of from 40 to 500W.
79. A method according to claim 77 or claim 78 wherein the voltage is pulsed in a sequence in which the ratio of the time on : time off is in the range of from 1:500 to 1:1500.
80. A method according to any of claims 77 to 79 wherein the voltage is pulsed in a sequence where power is on for 20-50 μ s, and off for from 1000 μ s to 30000 μ s.
81. A method according to any of claims 77 to 80 wherein the voltage is applied as a pulsed field for a period of from 30 seconds to 90 minutes.
82. A method according to claim 81 wherein the voltage is applied as a pulsed field for from 5 to 60 minutes.
83. A method according to any one of claims 77 to 82, wherein in a preliminary step, a continuous power plasma is applied to the electronic or electrical device or component thereof.

84. A method according to claim 83, wherein the preliminary step is conducted in the presence of an inert gas.
85. A method according to any one of claims 77 to 84, wherein the monomer compound and/or the crosslinking reagent in gaseous form is fed into the plasma at a rate of from 80-300 mg/minute, whilst the pulsed voltage is applied.
86. A method according to any one of claims 77 to 85, wherein the plasma is created with a voltage at an average power of from 0.001 to 500w/m³.
87. A method according to any of claims 40 to 86, wherein the monomer compound is miscible with the crosslinking reagent.
88. A method according to any of claims 40 to 86, wherein the monomer compound is immiscible with the crosslinking reagent.
89. A method according to claim 87, wherein the step of exposing the electronic or electrical device or component thereof to a plasma occurs in a plasma chamber; and the monomer compound and crosslinking reagent are mixed prior to entering the chamber.
90. A method according to claim 87 or claim 88 wherein the step of exposing the electronic or electrical device or component thereof to a plasma occurs in a plasma chamber; and the monomer compound and crosslinking reagent are introduced separately into the chamber.
91. A method according to any of claims 40 to 90, wherein the crosslinking reagent is present in an amount of from 10 to 60 (v/v) % of the total volume of monomer compound and crosslinking reagent.

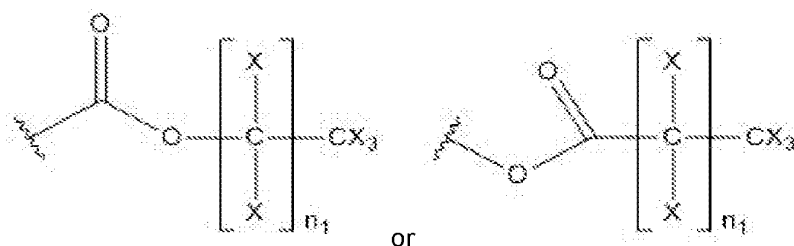
Abstract

The present invention provides an electronic or electrical device or component thereof comprising a cross-linked polymeric coating on a surface of the electronic or electrical device or component thereof; wherein the cross-linked polymeric coating is obtainable by exposing the electronic or electrical device or component thereof to a plasma comprising a monomer compound and a crosslinking reagent for a period of time sufficient to allow formation of the cross-linked polymeric coating on a surface thereof,

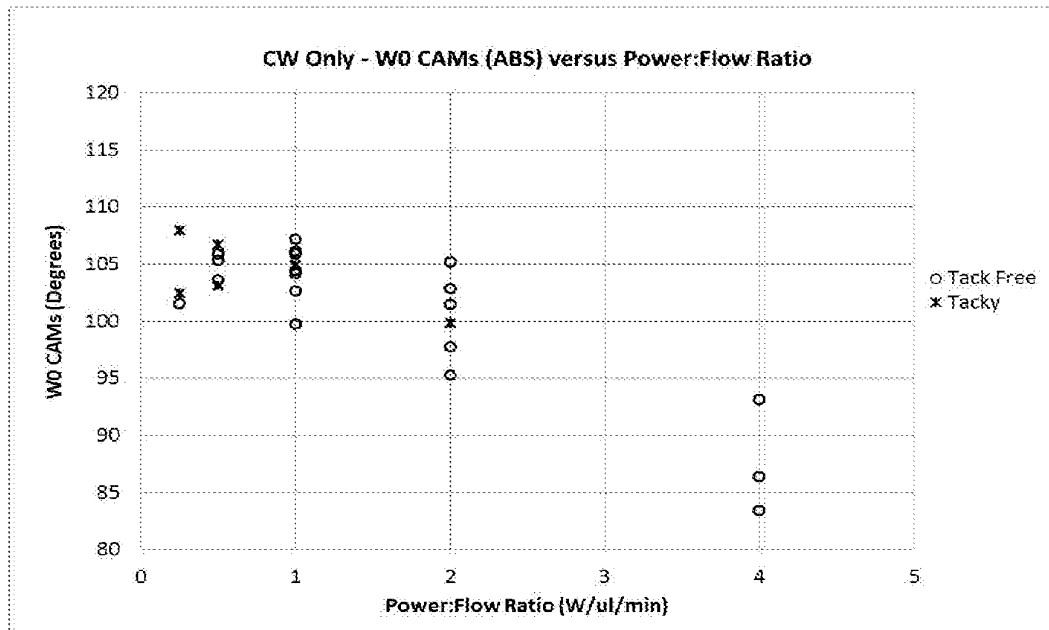
wherein the monomer compound has the following formula:



where R_1 , R_2 and R_4 are each independently selected from hydrogen, optionally substituted branched or straight chain C_1 - C_8 alkyl or halo alkyl or aryl optionally substituted by halo, and R_3 is selected from:



where each X is independently selected from hydrogen, a halogen, optionally substituted branched or straight chain C_1 - C_8 alkyl, halo alkyl or aryl optionally substituted by halo; and n_1 is an integer from 1 to 27; and wherein the crosslinking reagent comprises two or more unsaturated bonds attached by means of one or more linker moieties and has a boiling point at standard pressure of less than 500°C .

**Figure 1**

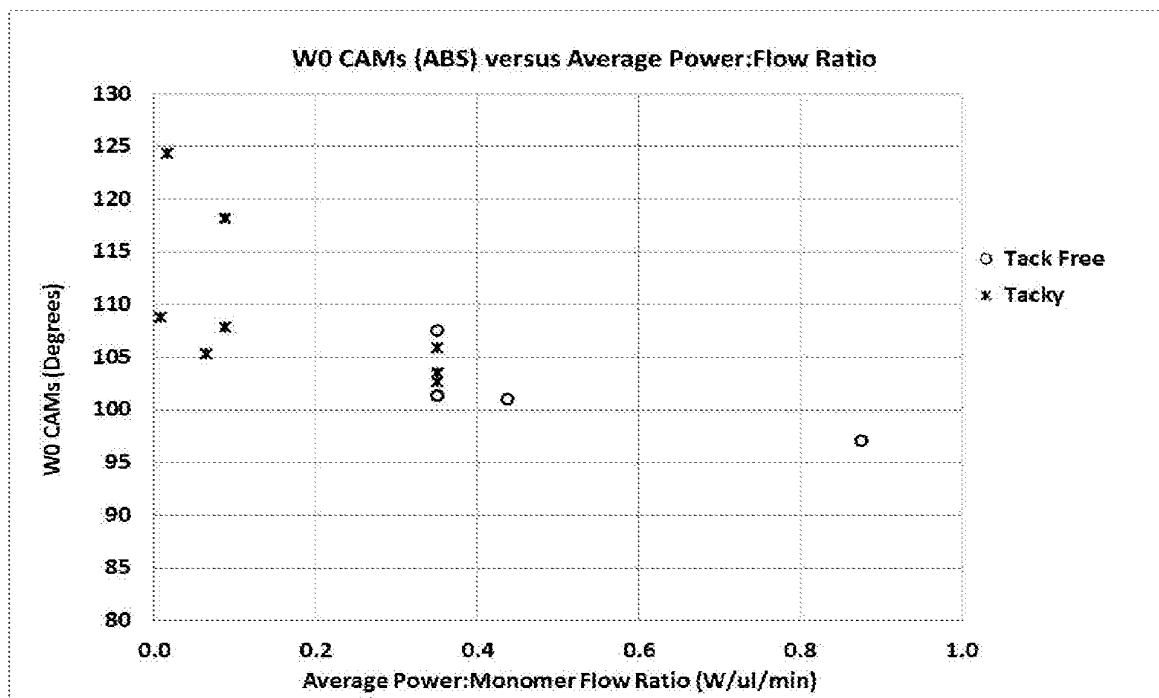
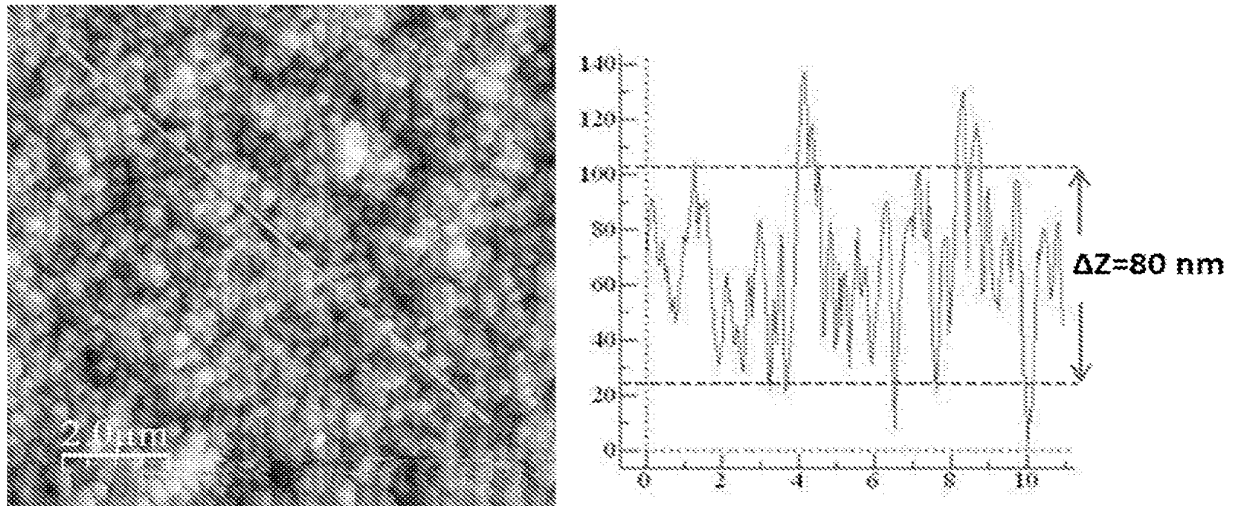
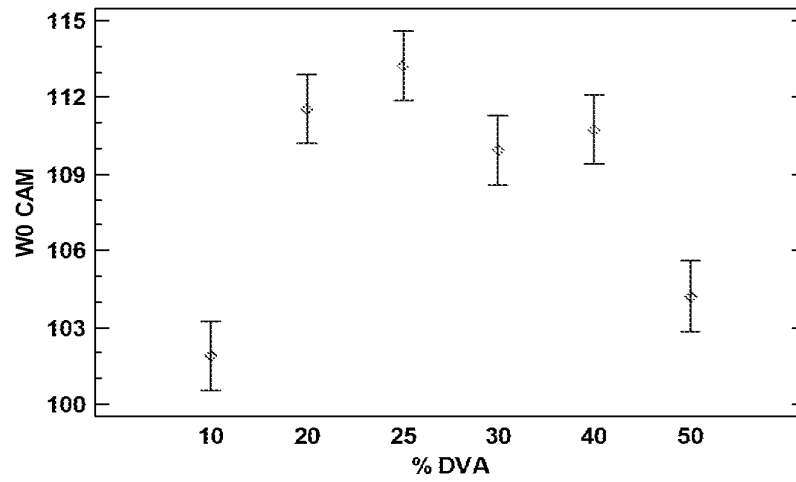


Figure 2

**Figure 3**

**Figure 4**

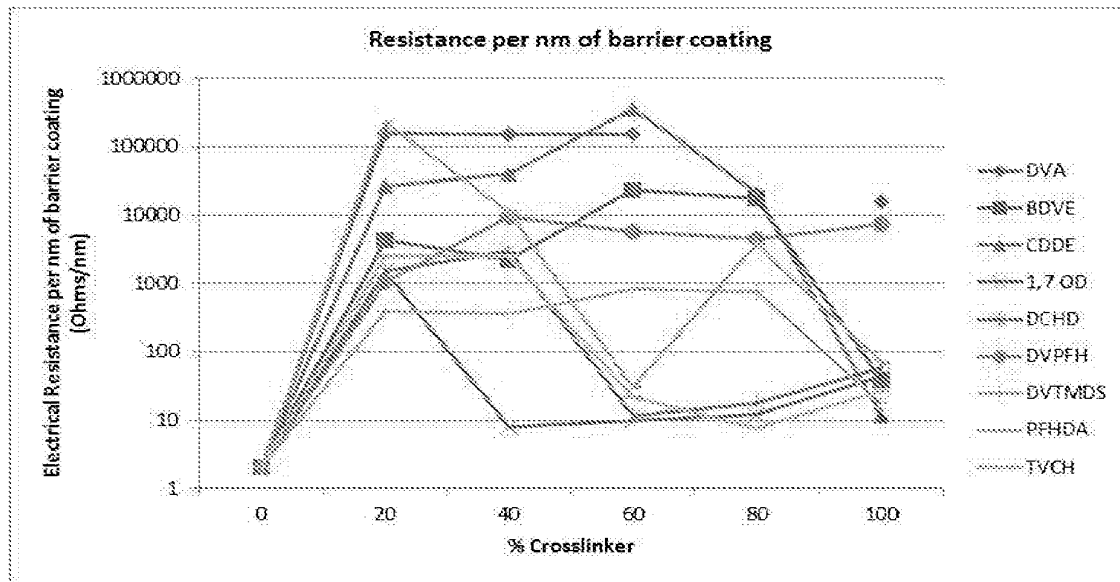


Figure 5

Figure 6

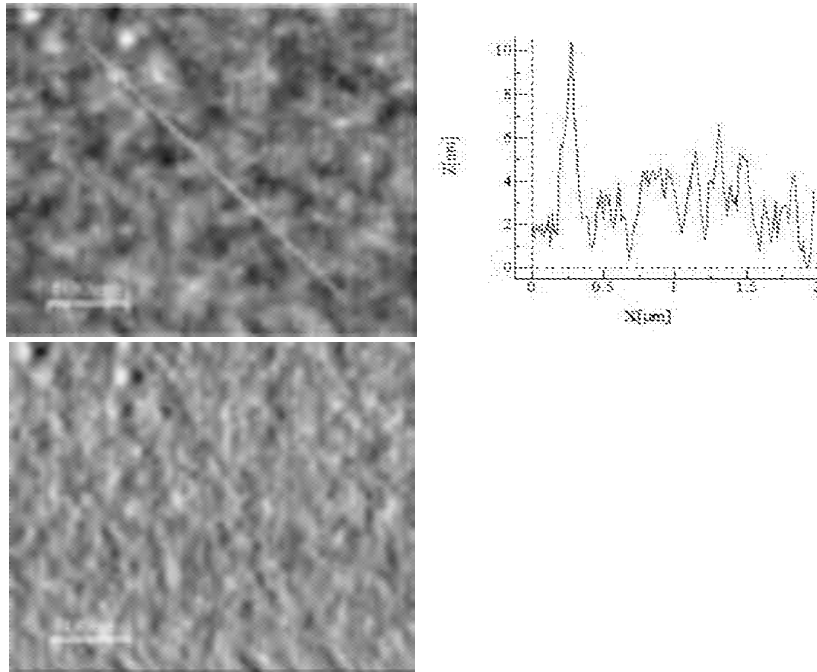
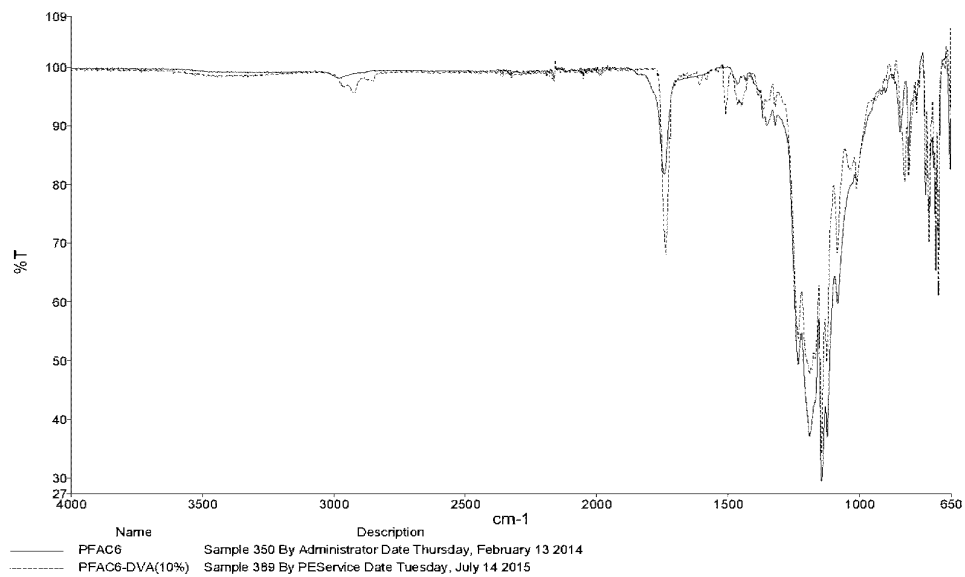


Figure 7



Electronic Patent Application Fee Transmittal

| | | | | |
|---|-------------------------------|-----------------|---------------|-----------------------------|
| Application Number: | 15735130 | | | |
| Filing Date: | 08-Dec-2017 | | | |
| Title of Invention: | COATINGS | | | |
| First Named Inventor/Applicant Name: | Stephen Richard COULSON | | | |
| Filer: | Paul John Parins/Sonja Talley | | | |
| Attorney Docket Number: | GBL0007/US | | | |
| Filed as Large Entity | | | | |
| Filing Fees for U.S. National Stage under 35 USC 371 | | | | |
| Description | Fee Code | Quantity | Amount | Sub-Total in USD(\$) |
| Basic Filing: | | | | |
| Pages: | | | | |
| Claims: | | | | |
| Miscellaneous-Filing: | | | | |
| Petition: | | | | |
| Patent-Appeals-and-Interference: | | | | |
| Post-Allowance-and-Post-Issuance: | | | | |
| Extension-of-Time: | | | | |

| Description | Fee Code | Quantity | Amount | Sub-Total in USD(\$) |
|-----------------------------------|----------|----------|--------|----------------------|
| Extension - 1 month with \$0 paid | 1251 | 1 | 200 | 200 |
| Miscellaneous: | | | | |
| Total in USD (\$) | | | | 200 |

Electronic Acknowledgement Receipt

| | |
|---|--------------------------------------|
| EFS ID: | 32913023 |
| Application Number: | 15735130 |
| International Application Number: | |
| Confirmation Number: | 7101 |
| Title of Invention: | COATINGS |
| First Named Inventor/Applicant Name: | Stephen Richard COULSON |
| Customer Number: | 33072 |
| Filer: | Paul John Parins/Sonja Talley |
| Filer Authorized By: | Paul John Parins |
| Attorney Docket Number: | GBL0007/US |
| Receipt Date: | 15-JUN-2018 |
| Filing Date: | 08-DEC-2017 |
| Time Stamp: | 15:25:26 |
| Application Type: | U.S. National Stage under 35 USC 371 |

Payment information:

| | |
|--|-----------------------|
| Submitted with Payment | yes |
| Payment Type | CARD |
| Payment was successfully received in RAM | \$200 |
| RAM confirmation Number | 061818INTEFSW15261300 |
| Deposit Account | |
| Authorized User | |

The Director of the USPTO is hereby authorized to charge indicated fees and credit any overpayment as follows:

| | | | | | |
|------------------------------|---|---|--|------------------|------------------|
| | | | | | |
| File Listing: | | | | | |
| Document Number | Document Description | File Name | File Size(Bytes)/ Message Digest | Multi Part /.zip | Pages (if appl.) |
| 1 | Applicant Response to Pre-Exam Formalities Notice | 2018-06-15-GBL0007US_Response_to_Notice_of_Innsufficiency.pdf | 104817 | no | 2 |
| | | | dd5a3e5142000676238de063b0dad3695a92e9ee | | |
| Warnings: | | | | | |
| Information: | | | | | |
| 2 | | 2018-06-15-GBL0007US_Preliminary_Amendment.pdf | 217014 | yes | 10 |
| | | | 8f754c0913495099afe3326be0b93545b7883330 | | |
| | Multipart Description/PDF files in .zip description | | | | |
| | Document Description | | Start | End | |
| | Preliminary Amendment | | 1 | 1 | |
| | Claims | | 2 | 8 | |
| | Applicant Arguments/Remarks Made in an Amendment | | 9 | 10 | |
| Warnings: | | | | | |
| Information: | | | | | |
| 3 | Other Reference-Patent/App/Search documents | PCT_GB2016_051688_As_Filed.pdf | 1601808 | no | 42 |
| | | | 580269b42951d2147958ac6f7f695c98d1951127 | | |
| Warnings: | | | | | |
| Information: | | | | | |
| 4 | Fee Worksheet (SB06) | fee-info.pdf | 30573 | no | 2 |
| | | | 63f6887f3ce37e3247d3a6e70d7b0e446007f731 | | |
| Warnings: | | | | | |
| Information: | | | | | |
| Total Files Size (in bytes): | | | 1954212 | | |

This Acknowledgement Receipt evidences receipt on the noted date by the USPTO of the indicated documents, characterized by the applicant, and including page counts, where applicable. It serves as evidence of receipt similar to a Post Card, as described in MPEP 503.

New Applications Under 35 U.S.C. 111

If a new application is being filed and the application includes the necessary components for a filing date (see 37 CFR 1.53(b)-(d) and MPEP 506), a Filing Receipt (37 CFR 1.54) will be issued in due course and the date shown on this Acknowledgement Receipt will establish the filing date of the application.

National Stage of an International Application under 35 U.S.C. 371

If a timely submission to enter the national stage of an international application is compliant with the conditions of 35 U.S.C. 371 and other applicable requirements a Form PCT/DO/EO/903 indicating acceptance of the application as a national stage submission under 35 U.S.C. 371 will be issued in addition to the Filing Receipt, in due course.

New International Application Filed with the USPTO as a Receiving Office

If a new international application is being filed and the international application includes the necessary components for an international filing date (see PCT Article 11 and MPEP 1810), a Notification of the International Application Number and of the International Filing Date (Form PCT/RO/105) will be issued in due course, subject to prescriptions concerning national security, and the date shown on this Acknowledgement Receipt will establish the international filing date of the application.

PATENT

In The United States Patent And Trademark Office

| | |
|---|---------------------------------|
| Inventor: Stephen Richard COULSON | Examiner: To Be Assigned |
| Serial No. 15/735,130 | Group Art Unit: 1762 |
| International Filing Date: June 8, 2016 | Confirmation No. 7101 |
| International App. No. PCT/GB2016/051688 | Docket No. GBL0007/US |
| 371(c): December 8, 2017 | |
| For: COATINGS | |

Commissioner for Patents
P.O. Box 1450
Alexandria, Virginia 22313-1450

I CERTIFY THAT ON 06/15/2018, THIS CORRESPONDENCE IS
BEING ELECTRONICALLY TRANSMITTED TO THE UNITED STATES
PATENT AND TRADEMARK OFFICE VIA THE OFFICE'S EFS-WEB

/Sonja A. Talley/
SONJA A. TALLEY

**RESPONSE TO NOTIFICATION OF INSUFFICIENCY UNDER 37 CFR 1.492 and/or
1.495 (DO/EO/US)**

Dear Sir or Madam:

This communication is responsive to the Notification of Insufficiency under 37 CFR 1.492 and/or 1.495 (DO/EO/US), which was mailed on March 16, 2018 in the above captioned application.

The two-month shortened-statutory period for response expired on May 16, 2018. Applicant respectfully petitions the Commissioner for Patents to extend the time for response for one month, from May 16, 2018 to June 16, 2018. It is respectfully submitted that this response is timely filed within the extended deadline. If any additional extension period is required in order for this paper to be timely filed, Applicant hereby requests an extension for such additional time period and authorizes the appropriate fees therefore to be charged to the Kagan Binder Deposit Account No. 50-1775 and to notify us of the same.

Payment in the amount of \$200.00 for a one-month extension of time is being provided by the electronic payment system of the EFS. In the event of failure of this payment by the electronic payment system of the EFS for any reason, the Commissioner is authorized to charge Kagan Binder Deposit Account No. 50-1775 and thereafter is requested to notify us of the same.

Remarks

According to the Notification, fees are due for multiple dependent claims and 70 total claims over 20.

Enclosed herewith is a Preliminary Amendment canceling claims so that the pending claims include one independent claim and 19 total claims. The claims are also amended to remove multiple dependencies.

Accordingly, no additional claims fees are due.

Respectfully Submitted,

Date: June 15, 2018

By: /Paul J. Parins/

Paul J. Parins, Reg. No. 54,358

Customer Number 33072

Phone: 651-275-9831

Fax: 651-351-2954

#119890



UNITED STATES PATENT AND TRADEMARK OFFICE

UNITED STATES DEPARTMENT OF COMMERCE
United States Patent and Trademark Office
Address: COMMISSIONER FOR PATENTS
P.O. Box 1450
Alexandria, Virginia 22313-1450
www.uspto.gov

| APPLICATION NUMBER | FILING OR 371(C) DATE | FIRST NAMED APPLICANT | ATTY. DOCKET NO./TITLE |
|--------------------|-----------------------|-------------------------|------------------------|
| 15/735,130 | 12/08/2017 | Stephen Richard COULSON | GBL0007/US |

CONFIRMATION NO. 7101

PUBLICATION NOTICE



OC000000100456356

33072
KAGAN BINDER, PLLC
SUITE 200, MAPLE ISLAND BUILDING
221 MAIN STREET NORTH
STILLWATER, MN 55082

Title:COATINGS

Publication No.US-2018-0171171-A1

Publication Date:06/21/2018

NOTICE OF PUBLICATION OF APPLICATION

The above-identified application will be electronically published as a patent application publication pursuant to 37 CFR 1.211, et seq. The patent application publication number and publication date are set forth above.

The publication may be accessed through the USPTO's publically available Searchable Databases via the Internet at www.uspto.gov. The direct link to access the publication is currently <http://www.uspto.gov/patft/>.

The publication process established by the Office does not provide for mailing a copy of the publication to applicant. A copy of the publication may be obtained from the Office upon payment of the appropriate fee set forth in 37 CFR 1.19(a)(1). Orders for copies of patent application publications are handled by the USPTO's Public Records Division. The Public Records Division can be reached by telephone at (571) 272-3150 or (800) 972-6382, by facsimile at (571) 273-3250, by mail addressed to the United States Patent and Trademark Office, Public Records Division, Alexandria, VA 22313-1450 or via the Internet.

In addition, information on the status of the application, including the mailing date of Office actions and the dates of receipt of correspondence filed in the Office, may also be accessed via the Internet through the Patent Electronic Business Center at www.uspto.gov using the public side of the Patent Application Information and Retrieval (PAIR) system. The direct link to access this status information is currently <https://portal.uspto.gov/pair/PublicPair>. Prior to publication, such status information is confidential and may only be obtained by applicant using the private side of PAIR.

Further assistance in electronically accessing the publication, or about PAIR, is available by calling the Patent Electronic Business Center at 1-866-217-9197.

Office of Data Management, Application Assistance Unit (571) 272-4000, or (571) 272-4200, or 1-888-786-0101

| PATENT APPLICATION FEE DETERMINATION RECORD Substitute for Form PTO-875 | | | | | | Application or Docket Number 15/735,130 | |
|--|---|------------------------------------|-----------------|--------------|--------------------|--|----------|
| APPLICATION AS FILED - PART I | | | | | | | |
| (Column 1) | | (Column 2) | | SMALL ENTITY | | OTHER THAN SMALL ENTITY | |
| FOR | NUMBER FILED | NUMBER EXTRA | RATE(\$) | FEE(\$) | | RATE(\$) | FEE(\$) |
| BASIC FEE (37 CFR 1.16(a), (b), or (c)) | N/A | N/A | N/A | | | N/A | 300 |
| SEARCH FEE (37 CFR 1.16(k), (i), or (m)) | N/A | N/A | N/A | | | N/A | 520 |
| EXAMINATION FEE (37 CFR 1.16(o), (p), or (q)) | N/A | N/A | N/A | | | N/A | 760 |
| TOTAL CLAIMS (37 CFR 1.16(j)) | 18 | minus 20 = * | | | OR | x 100 = | 0.00 |
| INDEPENDENT CLAIMS (37 CFR 1.16(h)) | 1 | minus 3 = * | | | | x 460 = | 0.00 |
| APPLICATION SIZE FEE (37 CFR 1.16(s)) | If the specification and drawings exceed 100 sheets of paper, the application size fee due is \$310 (\$155 for small entity) for each additional 50 sheets or fraction thereof. See 35 U.S.C. 41(a)(1)(G) and 37 CFR 1.16(s). | | | | | | 0.00 |
| MULTIPLE DEPENDENT CLAIM PRESENT (37 CFR 1.16(j)) | | | | | | | 0.00 |
| | | | TOTAL | | | TOTAL | 1580 |
| * If the difference in column 1 is less than zero, enter "0" in column 2. | | | | | | | |
| APPLICATION AS AMENDED - PART II | | | | | | | |
| (Column 1) | | (Column 2) | | (Column 3) | | SMALL ENTITY | |
| AMENDMENT A | CLAIMS REMAINING AFTER AMENDMENT | HIGHEST NUMBER PREVIOUSLY PAID FOR | PRESENT EXTRA | RATE(\$) | ADDITIONAL FEE(\$) | | RATE(\$) |
| | Total (37 CFR 1.16(i)) | * | Minus ** | = | x = | OR | x = |
| | Independent (37 CFR 1.16(h)) | * | Minus *** | = | x = | OR | x = |
| | Application Size Fee (37 CFR 1.16(s)) | | | | | OR | |
| | FIRST PRESENTATION OF MULTIPLE DEPENDENT CLAIM (37 CFR 1.16(j)) | | | | | OR | |
| | | | TOTAL ADD'L FEE | | OR | TOTAL ADD'L FEE | |
| (Column 1) | | (Column 2) | | (Column 3) | | SMALL ENTITY | |
| AMENDMENT B | CLAIMS REMAINING AFTER AMENDMENT | HIGHEST NUMBER PREVIOUSLY PAID FOR | PRESENT EXTRA | RATE(\$) | ADDITIONAL FEE(\$) | | RATE(\$) |
| | Total (37 CFR 1.16(i)) | * | Minus ** | = | x = | OR | x = |
| | Independent (37 CFR 1.16(h)) | * | Minus *** | = | x = | OR | x = |
| | Application Size Fee (37 CFR 1.16(s)) | | | | | OR | |
| | FIRST PRESENTATION OF MULTIPLE DEPENDENT CLAIM (37 CFR 1.16(j)) | | | | | OR | |
| | | | TOTAL ADD'L FEE | | OR | TOTAL ADD'L FEE | |
| * If the entry in column 1 is less than the entry in column 2, write "0" in column 3. ** If the "Highest Number Previously Paid For" IN THIS SPACE is less than 20, enter "20". *** If the "Highest Number Previously Paid For" IN THIS SPACE is less than 3, enter "3". The "Highest Number Previously Paid For" (Total or Independent) is the highest found in the appropriate box in column 1. | | | | | | | |



UNITED STATES PATENT AND TRADEMARK OFFICE

UNITED STATES DEPARTMENT OF COMMERCE
United States Patent and Trademark Office
Address: COMMISSIONER FOR PATENTS
P.O. Box 1450
Alexandria, Virginia 22313-1450
www.uspto.gov

| APPLICATION NUMBER | FILING or 371(c) DATE | GRP ART UNIT | FIL FEE REC'D | ATTY. DOCKET NO | TOT CLAIMS | IND CLAIMS |
|-----------------------|--------------------------|-----------------|---------------|-----------------|------------|------------|
| 15/735,130 | 12/08/2017 | 1762 | 1620 | GBL0007/US | 18 | 1 |

CONFIRMATION NO. 7101
UPDATED FILING RECEIPT

33072
KAGAN BINDER, PLLC
SUITE 200, MAPLE ISLAND BUILDING
221 MAIN STREET NORTH
STILLWATER, MN 55082



Date Mailed: 07/03/2018

Receipt is acknowledged of this non-provisional patent application. The application will be taken up for examination in due course. Applicant will be notified as to the results of the examination. Any correspondence concerning the application must include the following identification information: the U.S. APPLICATION NUMBER, FILING DATE, NAME OF APPLICANT, and TITLE OF INVENTION. Fees transmitted by check or draft are subject to collection. Please verify the accuracy of the data presented on this receipt. **If an error is noted on this Filing Receipt, please submit a written request for a Filing Receipt Correction. Please provide a copy of this Filing Receipt with the changes noted thereon. If you received a "Notice to File Missing Parts" for this application, please submit any corrections to this Filing Receipt with your reply to the Notice. When the USPTO processes the reply to the Notice, the USPTO will generate another Filing Receipt incorporating the requested corrections**

Inventor(s)

Stephen Richard COULSON, Abingdon, UNITED KINGDOM;
Delwyn EVANS, Abingdon, UNITED KINGDOM;
Angeliki SIOKOU, Abingdon, UNITED KINGDOM;
Clive TELFORD, Abingdon, UNITED KINGDOM;

Applicant(s)

P2i Ltd, Abingdon, UNITED KINGDOM

Power of Attorney: The patent practitioners associated with Customer Number 33072

Domestic Priority data as claimed by applicant

This application is a 371 of PCT/GB2016/051688 06/08/2016

Foreign Applications (You may be eligible to benefit from the **Patent Prosecution Highway** program at the USPTO. Please see <http://www.uspto.gov> for more information.)
EUROPEAN PATENT OFFICE (EPO) 15386017.6 06/09/2015

Permission to Access Application via Priority Document Exchange: Yes

Permission to Access Search Results: Yes

Applicant may provide or rescind an authorization for access using Form PTO/SB/39 or Form PTO/SB/69 as appropriate.

If Required, Foreign Filing License Granted: 03/14/2018

The country code and number of your priority application, to be used for filing abroad under the Paris Convention, is **US 15/735,130**

Projected Publication Date: Not Applicable

Non-Publication Request: No

Early Publication Request: No

Title

COATINGS

Preliminary Class

526

Statement under 37 CFR 1.55 or 1.78 for AIA (First Inventor to File) Transition Applications: No

PROTECTING YOUR INVENTION OUTSIDE THE UNITED STATES

Since the rights granted by a U.S. patent extend only throughout the territory of the United States and have no effect in a foreign country, an inventor who wishes patent protection in another country must apply for a patent in a specific country or in regional patent offices. Applicants may wish to consider the filing of an international application under the Patent Cooperation Treaty (PCT). An international (PCT) application generally has the same effect as a regular national patent application in each PCT-member country. The PCT process **simplifies** the filing of patent applications on the same invention in member countries, but **does not result** in a grant of "an international patent" and does not eliminate the need of applicants to file additional documents and fees in countries where patent protection is desired.

Almost every country has its own patent law, and a person desiring a patent in a particular country must make an application for patent in that country in accordance with its particular laws. Since the laws of many countries differ in various respects from the patent law of the United States, applicants are advised to seek guidance from specific foreign countries to ensure that patent rights are not lost prematurely.

Applicants also are advised that in the case of inventions made in the United States, the Director of the USPTO must issue a license before applicants can apply for a patent in a foreign country. The filing of a U.S. patent application serves as a request for a foreign filing license. The application's filing receipt contains further information and guidance as to the status of applicant's license for foreign filing.

Applicants may wish to consult the USPTO booklet, "General Information Concerning Patents" (specifically, the section entitled "Treaties and Foreign Patents") for more information on timeframes and deadlines for filing foreign patent applications. The guide is available either by contacting the USPTO Contact Center at 800-786-9199, or it can be viewed on the USPTO website at <http://www.uspto.gov/web/offices/pac/doc/general/index.html>.

For information on preventing theft of your intellectual property (patents, trademarks and copyrights), you may wish to consult the U.S. Government website, <http://www.stopfakes.gov>. Part of a Department of Commerce initiative, this website includes self-help "toolkits" giving innovators guidance on how to protect intellectual property in specific countries such as China, Korea and Mexico. For questions regarding patent enforcement issues, applicants may call the U.S. Government hotline at 1-866-999-HALT (1-866-999-4258).

LICENSE FOR FOREIGN FILING UNDER
Title 35, United States Code, Section 184
Title 37, Code of Federal Regulations, 5.11 & 5.15

GRANTED

The applicant has been granted a license under 35 U.S.C. 184, if the phrase "IF REQUIRED, FOREIGN FILING LICENSE GRANTED" followed by a date appears on this form. Such licenses are issued in all applications where the conditions for issuance of a license have been met, regardless of whether or not a license may be required as set forth in 37 CFR 5.15. The scope and limitations of this license are set forth in 37 CFR 5.15(a) unless an earlier license has been issued under 37 CFR 5.15(b). The license is subject to revocation upon written notification. The date indicated is the effective date of the license, unless an earlier license of similar scope has been granted under 37 CFR 5.13 or 5.14.

This license is to be retained by the licensee and may be used at any time on or after the effective date thereof unless it is revoked. This license is automatically transferred to any related applications(s) filed under 37 CFR 1.53(d). This license is not retroactive.

The grant of a license does not in any way lessen the responsibility of a licensee for the security of the subject matter as imposed by any Government contract or the provisions of existing laws relating to espionage and the national security or the export of technical data. Licensees should apprise themselves of current regulations especially with respect to certain countries, of other agencies, particularly the Office of Defense Trade Controls, Department of State (with respect to Arms, Munitions and Implements of War (22 CFR 121-128)); the Bureau of Industry and Security, Department of Commerce (15 CFR parts 730-774); the Office of Foreign Assets Control, Department of Treasury (31 CFR Parts 500+) and the Department of Energy.

NOT GRANTED

No license under 35 U.S.C. 184 has been granted at this time, if the phrase "IF REQUIRED, FOREIGN FILING LICENSE GRANTED" DOES NOT appear on this form. Applicant may still petition for a license under 37 CFR 5.12, if a license is desired before the expiration of 6 months from the filing date of the application. If 6 months has lapsed from the filing date of this application and the licensee has not received any indication of a secrecy order under 35 U.S.C. 181, the licensee may foreign file the application pursuant to 37 CFR 5.15(b).

SelectUSA

The United States represents the largest, most dynamic marketplace in the world and is an unparalleled location for business investment, innovation, and commercialization of new technologies. The U.S. offers tremendous resources and advantages for those who invest and manufacture goods here. Through SelectUSA, our nation works to promote and facilitate business investment. SelectUSA provides information assistance to the international investor community; serves as an ombudsman for existing and potential investors; advocates on behalf of U.S. cities, states, and regions competing for global investment; and counsels U.S. economic development organizations on investment attraction best practices. To learn more about why the United States is the best country in the world to develop technology, manufacture products, deliver services, and grow your business, visit <http://www.SelectUSA.gov> or call +1-202-482-6800.



UNITED STATES PATENT AND TRADEMARK OFFICE

UNITED STATES DEPARTMENT OF COMMERCE
United States Patent and Trademark Office
Address: COMMISSIONER FOR PATENTS
P.O. Box 1450
Alexandria, Virginia 22313-1450
www.uspto.gov

| | | |
|-----------------------------|-------------------------|------------------|
| U.S. APPLICATION NUMBER NO. | FIRST NAMED INVENTOR | ATTY. DOCKET NO. |
| 15/735,130 | Stephen Richard COULSON | GBL0007/US |

33072
KAGAN BINDER, PLLC
SUITE 200, MAPLE ISLAND BUILDING
221 MAIN STREET NORTH
STILLWATER, MN 55082

| | |
|-------------------------------|---------------|
| INTERNATIONAL APPLICATION NO. | |
| PCT/GB2016/051688 | |
| I.A. FILING DATE | PRIORITY DATE |
| 06/08/2016 | 06/09/2015 |

CONFIRMATION NO. 7101
371 ACCEPTANCE LETTER



Date Mailed: 07/03/2018

NOTICE OF ACCEPTANCE OF APPLICATION UNDER 35 U.S.C 371 AND 37 CFR 1.495

The applicant is hereby advised that the United States Patent and Trademark Office, in its capacity as a Designated / Elected Office (37 CFR 1.495), has ACCEPTED the above identified international application for national patentability examination in the United States Patent and Trademark Office.

The United States Application Number assigned to the application is shown above. A Filing Receipt will be issued for the present application in due course. **THE DATE APPEARING ON THE FILING RECEIPT AS THE "FILING DATE or 371(c) DATE" IS THE DATE ON WHICH THE LAST OF THE 35 U.S.C. 371 (c)(1) and (c)(2) REQUIREMENTS HAS BEEN RECEIVED IN THE OFFICE. THIS DATE IS SHOWN BELOW.** The filing date of the above identified application is the international filing date of the international application (Article 11(3) and 35 U.S.C. 363)

12/08/2017
DATE OF RECEIPT OF 35 U.S.C.
371(c)(1) and (c)(2) REQUIREMENTS

The following items have been received:

- Copy of the International Application filed on 12/08/2017
- Copy of the International Search Report filed on 12/08/2017
- Preliminary Amendments filed on 06/15/2018
- Information Disclosure Statements filed on 12/08/2017
- Request for Immediate Examination filed on 12/08/2017
- U.S. Basic National Fees filed on 12/08/2017
- Authorize Access to Search Results filed on 12/08/2017
- Priority Documents filed on 12/08/2017
- Power of Attorney filed on 12/08/2017
- Authorization to Permit Access filed on 12/08/2017
- Application Data Sheet (37 CFR 1.76) filed on 12/08/2017

Applicant is notified that the above-identified application contains the deficiencies noted below. No period for reply is set forth in this notice for correction of these deficiencies. However, if a deficiency relates to the inventor's oath or declaration, the applicant must file an oath or declaration in compliance with 37 CFR 1.63, or a substitute statement in compliance with 37 CFR 1.64, executed by or with respect to each actual inventor no later than the expiration of the time period set in the "Notice of Allowability" to avoid abandonment. See 37 CFR 1.495(c).

- Properly executed inventor's oath or declaration for the following inventor(s) has not been submitted: **Stephen Richard COULSON, Delwyn EVANS, Angeliki SIOKOU, and Clive TELFORD**

Applicant is reminded that any communications to the United States Patent and Trademark Office must be mailed to the address given in the heading and include the U.S. application no. shown above (37 CFR 1.5)

MAMYE I WAGSTAFF

Telephone: (571) 272-1057



UNITED STATES PATENT AND TRADEMARK OFFICE

UNITED STATES DEPARTMENT OF COMMERCE
United States Patent and Trademark Office
Address: COMMISSIONER FOR PATENTS
P.O. Box 1450
Alexandria, Virginia 22313-1450
www.uspto.gov

| APPLICATION NO. | FILING DATE | FIRST NAMED INVENTOR | ATTORNEY DOCKET NO. | CONFIRMATION NO. |
|----------------------------------|-------------|-------------------------|------------------------|------------------|
| 15/735,130 | 12/08/2017 | Stephen Richard COULSON | GBL0007/US | 7101 |
| 33072 | 7590 | 10/18/2018 | EXAMINER | |
| KAGAN BINDER, PLLC | | | BUIE-HATCHER, NICOLE M | |
| SUITE 200, MAPLE ISLAND BUILDING | | | ART UNIT | |
| 221 MAIN STREET NORTH | | | PAPER NUMBER | |
| STILLWATER, MN 55082 | | | 1767 | |
| | | | NOTIFICATION DATE | DELIVERY MODE |
| | | | 10/18/2018 | ELECTRONIC |

Please find below and/or attached an Office communication concerning this application or proceeding.

The time period for reply, if any, is set in the attached communication.

Notice of the Office communication was sent electronically on above-indicated "Notification Date" to the following e-mail address(es):

docketing@kaganbinder.com

DETAILED ACTION

Notice of Pre-AIA or AIA Status

The present application, filed on or after March 16, 2013, is being examined under the first inventor to file provisions of the AIA.

Response to Amendment

The amendment filed 06/15/2018 has been entered. **Claims 1-3, 5-16, and 31-34** are pending.

Specification

The disclosure is objected to because of the following informalities: on page 1, it is unknown what is the co-pending application.

The Brief Description of Drawings section is missing from the original specification.

Appropriate correction is required.

Drawings

The drawings are objected to because view numbers must be preceded by the abbreviation "Fig.". Corrected drawing sheets in compliance with 37 CFR 1.121(d) are required in reply to the Office action to avoid abandonment of the application. Any amended replacement drawing sheet should include all of the figures appearing on the immediate prior version of the sheet, even if only one figure is being amended. The figure or figure number of an amended drawing should not be labeled as "amended." If a drawing figure is to be canceled, the

appropriate figure must be removed from the replacement sheet, and where necessary, the remaining figures must be renumbered and appropriate changes made to the brief description of the several views of the drawings for consistency. Additional replacement sheets may be necessary to show the renumbering of the remaining figures. Each drawing sheet submitted after the filing date of an application must be labeled in the top margin as either “Replacement Sheet” or “New Sheet” pursuant to 37 CFR 1.121(d). If the changes are not accepted by the examiner, the applicant will be notified and informed of any required corrective action in the next Office action. The objection to the drawings will not be held in abeyance.

Claim Objections

Claims 6 and 7 are objected to because of the following informalities: both claims 6 and 7 do not end with periods. Appropriate correction is required.

Claims 12 and 13 are objected to because of the following informalities: the word “Wherein” should be lowercase in claim 12. Appropriate correction is required.

Claim 16 objected to because of the following informalities: the capital names used for the names of the compounds should be lowercase. The abbreviations are appropriate. Appropriate correction is required.

Claim Rejections - 35 USC § 112

The following is a quotation of 35 U.S.C. 112(b):

(b) CONCLUSION.—The specification shall conclude with one or more claims particularly pointing out and distinctly claiming the subject matter which the inventor or a joint inventor regards as the invention.

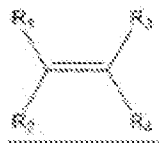
The following is a quotation of 35 U.S.C. 112 (pre-AIA), second paragraph:

The specification shall conclude with one or more claims particularly pointing out and distinctly claiming the subject matter which the applicant regards as his invention.

Claims 1-3, 5-16, and 31-34 are rejected under 35 U.S.C. 112(b) or 35 U.S.C. 112 (pre-AIA), second paragraph, as being indefinite for failing to particularly point out and distinctly claim the subject matter which the inventor or a joint inventor, or for pre-AIA the applicant regards as the invention.

Regarding claim 1, the instant claim recites, “wherein the protective cross-linked polymeric coating is obtainable by ...” on line 5. It is unknown by what other methods may produce the protective cross-linked polymeric coating.

Regarding claim 1, it is unknown how the following structure



relates to the instant claimed limitations.

Claim Rejections - 35 USC § 103

The following is a quotation of 35 U.S.C. 103 which forms the basis for all obviousness rejections set forth in this Office action:

A patent for a claimed invention may not be obtained, notwithstanding that the claimed invention is not identically disclosed as set forth in section 102, if the differences between the claimed invention and the prior art are such that the claimed invention as a whole would have been obvious before the effective filing date of the claimed invention to a person having ordinary skill in the art to which the claimed invention pertains. Patentability shall not be negated by the manner in which the invention was made.

The factual inquiries set forth in *Graham v. John Deere Co.*, 383 U.S. 1, 148 USPQ 459 (1966), that are applied for establishing a background for determining obviousness under 35 U.S.C. 103 are summarized as follows:

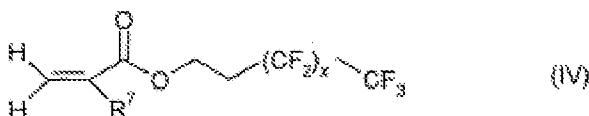
1. Determining the scope and contents of the prior art.
2. Ascertaining the differences between the prior art and the claims at issue.

3. Resolving the level of ordinary skill in the pertinent art.
4. Considering objective evidence present in the application indicating obviousness or nonobviousness.

This application currently names joint inventors. In considering patentability of the claims, the examiner presumes that the subject matter of the various claims was commonly owned as of the effective filing date of the claimed invention(s) absent any evidence to the contrary. Applicant is advised of the obligation under 37 CFR 1.56 to point out the inventor and effective filing dates of each claim that was not commonly owned as of the effective filing date of the later invention in order for the examiner to consider the applicability of 35 U.S.C. 102(b)(2)(C) for any potential 35 U.S.C. 102(a)(2) prior art against the later invention.

Claims 1-3, 5-6, 8-11, 16, and 31-34 are rejected under 35 U.S.C. 103 as being unpatentable over Coulson (WO 2007/083122 A1, referred to hereinafter as WO '122) in view of Coulson (US 2010/0203347 A1, referred to hereinafter as US '347).

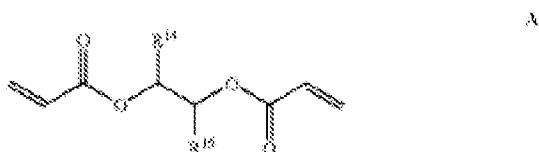
Regarding claims 1, 5-6, 8-10, 16, and 31-34, Coulson (WO '122) discloses an electronic or electrical device with a polymeric coating formed by exposing the device to pulsed plasma (P3/L20-CP4/3). The monomer used to formed the polymer includes a compound is of formula (IV):



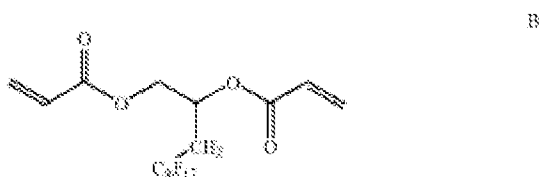
where R⁷ is includes H or C₁ alkyl, and in particular H and x is an integer of from 1 to 9 (P12/L14-22).

However, Coulson (WO '122) does not disclose the crosslinking reagent of the instant claims. Coulson (US '347) teaches a polymeric coating formed by exposing an item to plasma

comprising one or more organic monomeric compounds, at least one of which comprises two carbon-carbon double bonds (crosslinking reagents) [0029]. The crosslinking reagents include where Z is perfluoroalkyl group or optionally substituted alkyl groups, wherein the ester group is not required [0030-0031]. The compounds also include the following compound:



wherein each R¹⁴ and R¹⁵ is independently selected from hydrogen, alkyl or haloalkyl [0032, 0035]. One of the crosslinking reagents is of the following formula:



Since the crosslinking reagent meets the claimed limitations of the formula as recited in claim 5, it would be expected that the crosslinking reagent has a boiling point of less than 500°C, absent objective evidence to the contrary. The comonomers may include fluoro (meth)acrylates [0028]. Coulson (WO '122) and Coulson (US '347) are analogous art concerned with the same field of endeavor, namely plasma formed coatings. It would have been obvious to one of ordinary skill in the art before the effective filing date of the claimed invention to add the compound have two carbon-carbon double bonds per the teachings of Coulson (US '347), and the motivation to do so would have been as Coulson (US '347 suggests such polymeric layers are useful on the surface [0029]. The coatings have a high degree of liquid-proofing protection [0011]. Since the coating has a high degree of liquid-proofing protection, it would be expected that the protective cross-linked polymer coating forms a liquid repellent surface defined by a

static water contact angle (WCA) of at 90° or may be adjusted to achieve this static water contact angle. Due to two carbon-carbon double bonds, crosslinking would result, and the resulting coating is tougher and less flexible. In regards to the length of the carbon chain in the alkyl group for Y¹⁰, an obviousness rejection based on similarity in chemical structure and function entails the motivation of one skilled in the art to make a claimed compound in the expectation that compounds similar in structure will have similar properties. *In re Payne*, 606 F. 2d 303, 313, 203 USPQ 245, 254 (CCPA 979). See *In re Papesch*, 315.2d 381, 137 USPQ 43 (CCPA 1963) and *In re Dillon*, 919 F.2d 688, 16 USPQ2d 1897 (Fed. Cir. 1991). See MPEP 2144.09.

Regarding claim 2, Coulson (WO '122) does not disclose the protective cross-linked polymeric coating is a physical barrier to mass and electron transport. The Office realizes that all of the claimed effects or physical properties are not positively stated by the reference(s). However, the reference(s) teaches all of the claimed ingredients, claimed amounts, and substantially similar process of making. Therefore, the claimed effects and physical properties, i.e. the protective cross-linked polymeric coating is a physical barrier to mass and electron transport would implicitly be achieved by a composition with all the claimed ingredients, claimed amounts, and substantially similar process of making. See MPEP § 2112.01. If it is the applicant's position that this would not be the case: (1) evidence would need to be provided to support the applicant's position; and (2) it would be the Office's position that the application contains inadequate disclosure that there is no teaching as to how to obtain the claimed properties with only the claimed ingredients, claimed amounts, and substantially similar process of making.

Regarding claim 11, Coulson (US '347) teaches the formula of (V) wherein n is 1 to 10 [0032]. Therefore, similar results would be expected when n is 4 to 6. An obviousness rejection

based on similarity in chemical structure and function entails the motivation of one skilled in the art to make a claimed compound in the expectation that compounds similar in structure will have similar properties. *In re Payne*, 606 F. 2d 303, 313, 203 USPQ 245, 254 (CCPA 979). See *In re Papesch*, 315.2d 381, 137 USPQ 43 (CCPA 1963) and *In re Dillon*, 919 F.2d 688, 16 USPQ2d 1897 (Fed. Cir. 1991). See MPEP 2144.09.

Allowable Subject Matter

Claims 7 and 12-15 would be allowable if rewritten to overcome the rejection(s) under 35 U.S.C. 112(b) or 35 U.S.C. 112 (pre-AIA), 2nd paragraph, set forth in this Office action and to include all of the limitations of the base claim and any intervening claims.

The following is a statement of reasons for the indication of allowable subject matter: the closest prior art of record, Coulson (WO '122) does not teach or suggest each L has one of the structures as recited in claim 7, wherein Y¹⁰ meets the formula as recited in claim 12, wherein each Y¹⁵ is methyl and each Y⁹ is a bond, or Y¹⁰ has the formula as recited in claim 14.

Prior Art

The prior art made of record and not relied upon is considered pertinent to applicant's disclosure. Badyal et al. (US RE43,651 E) teaches a method of coating a surface with a polymer layer.

York et al. (WO 2004/065684 A2) teaches fibrous products.

Legein et al. (US 2016/0284518 A1) teaches low pressure plasma processes.

Petersen et al. (US 2014/0342103 A1) teaches atmospheric plasma polymerization.

Badyal et al. (EP 1557489 A1) teaches surface coatings.

Legein (US 8,852,693 B2) teaches a coated electronic device.

Sanyal et al. (US 2010/0021998 A1) teaches plasma polymerization.

Rauschnabel et al. (US 6,613,393 B1) teaches a method for applying wear protection layers.

Bongiovanni et al. "High performance UV-cured coatings for wood protection", Progress in Organic Coatings, 2002, 45, 359-363.

Morita et al. (WO 2006/129800 A1) teaches a finishing agent for photo lithography.

Correspondence

Any inquiry concerning this communication or earlier communications from the examiner should be directed to NICOLE M. BUIE-HATCHER whose telephone number is (571)270-3879. The examiner can normally be reached on M-F, 8 am - 4:30 pm.

Examiner interviews are available via telephone, in-person, and video conferencing using a USPTO supplied web-based collaboration tool. To schedule an interview, applicant is encouraged to use the USPTO Automated Interview Request (AIR) at <http://www.uspto.gov/interviewpractice>.

If attempts to reach the examiner by telephone are unsuccessful, the examiner's supervisor, Mark Eashoo can be reached on (571)272-1197. The fax phone number for the organization where this application or proceeding is assigned is 571-273-8300.

Information regarding the status of an application may be obtained from the Patent Application Information Retrieval (PAIR) system. Status information for published applications may be obtained from either Private PAIR or Public PAIR. Status information for unpublished applications is available through Private PAIR only. For more information about the PAIR

system, see <http://pair-direct.uspto.gov>. Should you have questions on access to the Private PAIR system, contact the Electronic Business Center (EBC) at 866-217-9197 (toll-free). If you would like assistance from a USPTO Customer Service Representative or access to the automated information system, call 800-786-9199 (IN USA OR CANADA) or 571-272-1000.

/NICOLE M. BUIE-HATCHER/
Primary Examiner, Art Unit 1767

| | | | | |
|--|---------------------------------------|--|--|-------------|
| <i>Notice of References Cited</i> | Application/Control No. 15/735,130 | | Applicant(s)/Patent Under Reexamination COULSON et al. | |
| | Examiner NICOLE M BUIE-HATCHER | | Art Unit 1767 | Page 1 of 1 |

U.S. PATENT DOCUMENTS

| * | | Document Number Country Code-Number-Kind Code | Date MM-YYYY | Name | CPC Classification | US Classification |
|---|---|--|-----------------|-----------------------|--------------------|-------------------|
| * | A | US-6613393-B1 | 09-2003 | Rauschnabel; Johannes | B05D1/62 | 204/192.12 |
| * | B | US-8852693-B2 | 10-2014 | Legein; Filip | B05D1/62 | 118/728 |
| * | C | US-RE43651-E | 09-2012 | Badyal; Jas P. S. | D06M10/02 | 427/487 |
| * | D | US-20100021998-A1 | 01-2010 | Sanyal; Suparna | C12N5/0068 | 435/366 |
| * | E | US-20100203347-A1 | 08-2010 | Coulson; Stephen | A43B7/12 | 428/500 |
| * | F | US-20140342103-A1 | 11-2014 | Petersen; Julien | B05D5/083 | 427/569 |
| * | G | US-20160284518-A1 | 09-2016 | Legein; Filip | H01J37/32146 | 1/1 |
| | H | | | | | |
| | I | | | | | |
| | J | | | | | |
| | K | | | | | |
| | L | | | | | |
| | M | | | | | |


FOREIGN PATENT DOCUMENTS

| * | | Document Number Country Code-Number-Kind Code | Date MM-YYYY | Country | Name | CPC Classification |
|---|---|--|-----------------|---------|---------------|--------------------|
| | N | WO-2004065684-A2 | 08-2004 | WO | York et al. | D06M10/025 |
| | O | EP-1557489-A1 | 07-2005 | EP | Badyal et al. | D06M10/02 |
| | P | WO-2006129800-A1 | 12-2006 | WO | Morita et al. | G03F7/0005 |
| | Q | | | | | |
| | R | | | | | |
| | S | | | | | |
| | T | | | | | |

NON-PATENT DOCUMENTS

| * | | Include as applicable: Author, Title Date, Publisher, Edition or Volume, Pertinent Pages) |
|---|---|--|
| | U | Bongiovanni et al. "High performance UV-cured coatings for wood protection", Progress in Organic Coatings, 2002, 45, 359-363. (Year: 2002) |
| | V | |
| | W | |
| | X | |

*A copy of this reference is not being furnished with this Office action. (See MPEP § 707.05(a).)
Dates in MM-YYYY format are publication dates. Classifications may be US or foreign.

| | | |
|---|--|--|
| <i>Search Notes</i>  | Application/Control No. 15/735,130 | Applicant(s)/Patent Under Reexamination COULSON et al. |
| | Examiner NICOLE M BUIE-HATCHER | Art Unit 1767 |

| | | |
|------------------------|-------------|-----------------|
| CPC - Searched* | | |
| Symbol | Date | Examiner |
| | | |

| | | |
|---|-------------|-----------------|
| CPC Combination Sets - Searched* | | |
| Symbol | Date | Examiner |
| | | |

| | | | |
|--------------------------------------|-----------------|-------------|-----------------|
| US Classification - Searched* | | | |
| Class | Subclass | Date | Examiner |
| | | | |

* See search history printout included with this form or the SEARCH NOTES box below to determine the scope of the search.

| | | |
|---------------------|-------------|-----------------|
| Search Notes | | |
| Search Notes | Date | Examiner |
| | | |

| | | | |
|----------------------------|------------------------------|-------------|-----------------|
| Interference Search | | | |
| US Class/CPC Symbol | US Subclass/CPC Group | Date | Examiner |
| | | | |

| | |
|--|--|
| | |
|--|--|

Session Began October 15, 2018 at 09:38 AM

Task Began October 15, 2018 09:38 AM

Explore substances by ID: dimethylpolysiloxane diacrylate (ID 1)

No answers

Task Began October 15, 2018 09:39 AM

Explore substances by ID: diacrylate dimethylpolysiloxane (ID 2)

No answers

Task Began October 15, 2018 09:39 AM

Explore references by patent: (ID 3)

Patent Number: us20180171171

Answer Type: References

Result Count: 1

Retrieve substance information in 1 reference (ID 4)

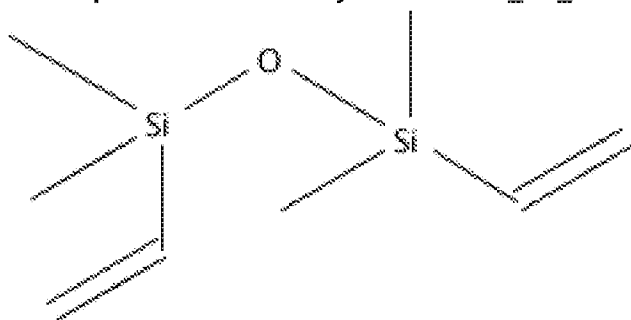
From ID: 3

Answer Type: Substances

Result Count: 17

Task Began October 15, 2018 09:41 AM

Explore substances by SIMILARITY_2D_EXACT



Candidates: 9

Candidates Selected (ID 6)

99 (most similar)

95-98

90-94

85-89

80-84

75-79

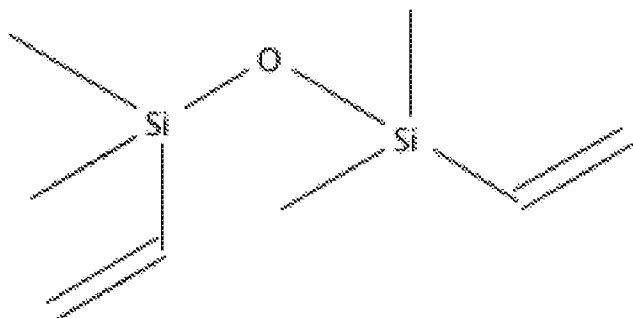
Answer Type: Substances

Result Count: 1536

Refine Answer set by structure (ID 7)

Structure: exact

From ID: 6



Limiters:

| | |
|---------------|------------|
| | Polymers |
| Answer Type: | Substances |
| Result Count: | 394 |

Retrieve reference information in 394 substances (ID 8)

| | |
|---------------|------------|
| From ID: | 7 |
| Answer Type: | References |
| Result Count: | 2 |

Task Began October 15, 2018 09:54 AM

Explore substances by property: (ID 9)

| | |
|---------------|------------------------------------|
| Property: | Experimental - Boiling Point, >500 |
| Answer Type: | Substances |
| Result Count: | 852 |

Task Began October 15, 2018 10:08 AM

Explore references by patent: (ID 10)

| | |
|----------------|---------------|
| Patent Number: | us20180171171 |
| Answer Type: | References |
| Result Count: | 1 |

Retrieve substance information in 1 reference (ID 11)

| | |
|---------------|------------|
| From ID: | 10 |
| Answer Type: | Substances |
| Result Count: | 17 |

Retrieve reference information in 17 substances (ID 12)

| | |
|---------------|------------|
| From ID: | 11 |
| Answer Type: | References |
| Result Count: | 668 |

Refine by research topic (ID 13)

| | |
|-----------------|---------------------|
| Research Topic: | crosslinked polymer |
| From ID: | 12 |
| Answer Type: | References |
| Result Count: | 139 |

Copyright © 2018 American Chemical Society. All Rights Reserved.

Copyright © 1994-2018 Sun Microsystems, Inc. All Rights Reserved. (Java runtime environment)

Copyright © 2018, Yahoo! Inc. All Rights Reserved. (YUI Base, Fonts, Reset CSS)

Copyright © 200-2018, The Dojo Foundation. All Rights Reserved

Copyright © 2018, Exadel, Inc. All Rights Reserved. (JBoss RichFaces)

Copyright © 2002 InfoChem GmbH. All Rights Reserved. (InfoChem's reaction classification program CLASSIFY)

The following copyright notice applies to Raphael only: Permission is hereby granted, free of charge, to any person obtaining a copy of this software and associated documentation files (the 'Software'), to deal in the Software without restriction, including without limitation the rights to use, copy, modify, merge, publish, distribute, sublicense, and/or sell copies of the Software, and to permit persons to whom the Software is furnished to do so, subject to the following conditions: The above copyright notice and this permission notice shall be included in all copies or substantial portions of the Software. THE SOFTWARE IS PROVIDED 'AS IS', WITHOUT WARRANTY OF ANY KIND, EXPRESS OR IMPLIED, INCLUDING BUT NOT LIMITED TO THE WARRANTIES OF MERCHANTABILITY, FITNESS FOR A PARTICULAR PURPOSE AND NONINFRINGEMENT. IN NO EVENT SHALL THE AUTHORS OR COPYRIGHT HOLDERS BE LIABLE FOR ANY CLAIM, DAMAGES OR OTHER LIABILITY, WHETHER IN AN ACTION OF CONTRACT, TORT OR OTHERWISE, ARISING FROM, OUT OF OR IN CONNECTION WITH THE SOFTWARE OR THE USE OR OTHER DEALINGS IN THE SOFTWARE.

The following copyright notice applies to Google Closure Templates only: Copyright 2009 Google Inc. Licensed under the Apache License, Version 2.0 (the 'License'); you may not use this file except in compliance with the License. You may obtain a copy of the License at <http://www.apache.org/licenses/LICENSE-2.0>. Unless required by applicable law or agreed to in writing, software distributed under the License is distributed on an 'AS IS' BASIS, WITHOUT WARRANTIES OR CONDITIONS OF ANY KIND, either express or implied. See the License for the specific language governing permissions and limitations under the License.

Copyright (c) 2010, 2012 Adobe Systems Incorporated, All Rights Reserved, (Source Sans Pro) Licensed under the SIL Open Font License, Version 1.1 (available at: <http://scripts.sil.org/OFL>)

Copyright 2005-2013 The Dojo Foundation (Dojo Toolkit) Licensed under the Apache License, Version 2.0 (the "License"); you may not use this file except in compliance with the License. You may obtain a copy of the License at: <http://www.apache.org/licenses/LICENSE-2.0>. Unless required by applicable law or agreed to in writing, software distributed under the License is distributed on an "AS IS" BASIS, WITHOUT WARRANTIES OR CONDITIONS OF ANY KIND, either express or implied. See the License for the specific language governing permissions and limitations under the License.

Copyright (c) 2008 Dmitry Baranovskiy (Raphael) Licensed under the MIT License (available at: <http://opensource.org/licenses/MIT>) Permission is hereby granted, free of charge, to any person obtaining a copy of this software and associated documentation files (the "Software"), to deal in the Software without restriction, including without limitation the rights to use, copy, modify, merge, publish, distribute, sublicense, and/or sell copies of the Software, and to permit persons to whom the Software is furnished to do so, subject to the following conditions: The above copyright notice and this permission notice shall be included in all copies or substantial portions of the Software. THE SOFTWARE IS PROVIDED "AS IS", WITHOUT WARRANTY OF ANY KIND, EXPRESS OR IMPLIED, INCLUDING BUT NOT LIMITED TO THE WARRANTIES OF MERCHANTABILITY, FITNESS FOR A PARTICULAR PURPOSE AND NONINFRINGEMENT. IN NO EVENT SHALL THE AUTHORS OR COPYRIGHT HOLDERS BE LIABLE FOR ANY CLAIM, DAMAGES OR OTHER LIABILITY, WHETHER IN AN ACTION OF CONTRACT, TORT OR OTHERWISE, ARISING FROM, OUT OF OR IN CONNECTION WITH THE SOFTWARE OR THE USE OR OTHER DEALINGS IN THE SOFTWARE.

CAPLUSSM: Copyright © 2018 American Chemical Society. All Rights Reserved. (The U.K. patent material in this product/service is U.K. Crown copyright and is made available with permission.

Copyright © Crown Copyright. The French (FR) patent material in this product/service is made available from Institut National de la Propriété Industrielle (INPI).)

CAS REGISTRYSM: Copyright © 2018 American Chemical Society. All Rights Reserved. (Some records contain information from GenBank[®]. See also: Benson D.A., Karsch-Mizrahi L., Lipman D.J., Ostel J., Rapp B.A., Wheeler D.L. Genbank. Nucl. Acids Res. 28(1):15-18 (2000). Property values tagged with IC are from the ZIC/VINITI data file provided by InfoChem.) CAS Registry is a service mark of the American Chemical Society. GenBank is a registered trademark of the U.S. Library of Medicine.

CASREACT[®]: Copyright © 2018 American Chemical Society. All Rights Reserved. CASREACT contains reactions from CAS and from: ZIC/VINITI database (1974-1999) provided by InfoChem; INPI data prior to 1998; Biotransformations database compiled under the direction of Professor Dr. Klaus Kieslich; organic reactions, portions copyright 1998-2008 John Wiley & Sons, Ltd., John Wiley and Sons, Inc., Organic Reactions Inc., and Organic Syntheses Inc. Reproduced under license. All Rights Reserved.

CHEMCATS[®]: Copyright © 2018 American Chemical Society. All Rights Reserved. Chemical supplier information is supplied on an "as is" basis. Full information regarding substance availability, price, etc., is provided when you request supplier information.

CHEMLIST[®]: Copyright © 2018 American Chemical Society. All Rights Reserved.

MEDLINE[®]: is produced by the U.S. National Library of Medicine. MEDLINE is a registered trademark of the U.S. National Library of Medicine.

Bibliographic Data

Application No: 15/735,130

Foreign Priority claimed: ☒ Yes ☐ No

35 USC 119 (a-d) conditions met: ☒ Yes ☐ No ☐ Met After Allowance

Verified and Acknowledged:

/NICOLE M. BUIE-
HATCHER/

Examiner's Signature

Initials

Title:

COATINGS

| FILING or 371(c) DATE | CLASS | GROUP ART UNIT | ATTORNEY DOCKET NO. |
|-----------------------|-------|----------------|---------------------|
| 12/08/2017 | 526 | 1767 | GBL0007/US |
| RULE | | | |

APPLICANTS

P2i Ltd, Abingdon, UNITED KINGDOM

INVENTORS

Stephen Richard COULSON Abingdon, UNITED KINGDOM

Delwyn EVANS Abingdon, UNITED KINGDOM

Angeliki SIOKOU Abingdon, UNITED KINGDOM

Clive TELFORD Abingdon, UNITED KINGDOM

CONTINUING DATA

This application is a 371 of PCT/GB2016/051688 06/08/2016

FOREIGN APPLICATIONS

EPO 15386017.6 06/09/2015

IF REQUIRED, FOREIGN LICENSE GRANTED**

03/14/2018

STATE OR COUNTRY

UNITED KINGDOM

ADDRESS

KAGAN BINDER, PLLC
SUITE 200, MAPLE ISLAND BUILDING
221 MAIN STREET NORTH
STILLWATER, MN 55082
UNITED STATES

FILING FEE RECEIVED

\$1,620

Doc code: IDS

PTO/SB/08a (03-15)

Doc description: Information Disclosure Statement (IDS) Filed

Approved for use through 07/31/2016. OMB 0651-0031
U.S. Patent and Trademark Office; U.S. DEPARTMENT OF COMMERCE

Under the Paperwork Reduction Act of 1995, no persons are required to respond to a collection of information unless it contains a valid OMB control number.

| | | | |
|---|------------------------|-------------------------|--|
| INFORMATION DISCLOSURE STATEMENT BY APPLICANT (Not for submission under 37 CFR 1.99) | Application Number | | |
| | Filing Date | | |
| | First Named Inventor | Stephen Richard COULSON | |
| | Art Unit | | |
| | Examiner Name | | |
| | Attorney Docket Number | GBL0007/US | |

| U.S.PATENTS | | | | | | |
|-------------------|---------|---------------|------------------------|------------|---|--|
| Examiner Initial* | Cite No | Patent Number | Kind Code ¹ | Issue Date | Name of Patentee or Applicant of cited Document | Pages,Columns,Lines where Relevant Passages or Relevant Figures Appear |
| | 1 | | | | | |

If you wish to add additional U.S. Patent citation information please click the Add button.

U.S.PATENT APPLICATION PUBLICATIONS

| Examiner Initial* | Cite No | Publication Number | Kind Code ¹ | Publication Date | Name of Patentee or Applicant of cited Document | Pages,Columns,Lines where Relevant Passages or Relevant Figures Appear |
|-------------------|---------|--------------------|------------------------|------------------|---|--|
| | 1 | | | | | |

If you wish to add additional U.S. Published Application citation information please click the Add button.

FOREIGN PATENT DOCUMENTS

| Examiner Initial* | Cite No | Foreign Document Number ³ | Country Code ²ⁱ | Kind Code ⁴ | Publication Date | Name of Patentee or Applicant of cited Document | Pages,Columns,Lines where Relevant Passages or Relevant Figures Appear | T ⁵ |
|-------------------|---------|--------------------------------------|----------------------------|------------------------|------------------|---|--|--------------------------|
| /N.M.B./ | 1 | 98/58117 | WO | A1 | 1998-12-23 | THE SECRETARY OF STATE FOR DEFENCE | | <input type="checkbox"/> |
| /N.M.B./ | 2 | 2003/097245 | WO | A2 | 2003-11-27 | SURFACE INNOVATIONS LIMITED | | <input type="checkbox"/> |
| /N.M.B./ | 3 | 03/101621 | WO | A2 | 2003-12-11 | SURFACE INNOVATIONS LIMITED | | <input type="checkbox"/> |

**INFORMATION DISCLOSURE
STATEMENT BY APPLICANT**
(Not for submission under 37 CFR 1.99)

| | | |
|------------------------|-------------------------|--|
| Application Number | | |
| Filing Date | | |
| First Named Inventor | Stephen Richard COULSON | |
| Art Unit | | |
| Examiner Name | | |
| Attorney Docket Number | GBL0007/US | |

| | | | | | | | | |
|----------|---|-------------|----|----|------------|---------------|--|--------------------------|
| /N.M.B./ | 4 | 2007/083122 | WO | A1 | 2007-07-26 | P2I LTD | | <input type="checkbox"/> |
| /N.M.B./ | 5 | 0834352 | EP | A1 | 1998-04-08 | CIBA-GEIGY AG | | <input type="checkbox"/> |
| /N.M.B./ | 6 | 2014/155099 | WO | A1 | 2014-10-02 | SEMBLANT LTD | | <input type="checkbox"/> |
| /N.M.B./ | 7 | 2015/128627 | WO | A1 | 2015-09-03 | P2I LTD | | <input type="checkbox"/> |
| /N.M.B./ | 8 | 2016/198855 | WO | A1 | 2016-12-15 | P2I LTD | | <input type="checkbox"/> |
| /N.M.B./ | 9 | 2016/198856 | WO | A1 | 2016-12-15 | P2I LTD | | <input type="checkbox"/> |

If you wish to add additional Foreign Patent Document citation information please click the Add button

NON-PATENT LITERATURE DOCUMENTS

| Examiner Initials* | Cite No | Include name of the author (in CAPITAL LETTERS), title of the article (when appropriate), title of the item (book, magazine, journal, serial, symposium, catalog, etc), date, pages(s), volume-issue number(s), publisher, city and/or country where published. | T ⁵ |
|--------------------|---------|---|--------------------------|
| /N.M.B./ | 1 | International Search Report and Written Opinion for International Application No. PCT/GB2016/051688, mailed November 8, 2016 (9 pages) | <input type="checkbox"/> |

If you wish to add additional non-patent literature document citation information please click the Add button

EXAMINER SIGNATURE

| | | | |
|--------------------|--------------------------|-----------------|------------|
| Examiner Signature | /NICOLE M. BUIE-HATCHER/ | Date Considered | 10/12/2018 |
|--------------------|--------------------------|-----------------|------------|

*EXAMINER: Initial if reference considered, whether or not citation is in conformance with MPEP 609. Draw line through a citation if not in conformance and not considered. Include copy of this form with next communication to applicant.

**INFORMATION DISCLOSURE
STATEMENT BY APPLICANT**
(Not for submission under 37 CFR 1.99)

| | |
|------------------------|-------------------------|
| Application Number | |
| Filing Date | |
| First Named Inventor | Stephen Richard COULSON |
| Art Unit | |
| Examiner Name | |
| Attorney Docket Number | GBL0007/US |

¹ See Kind Codes of USPTO Patent Documents at www.USPTO.GOV or MPEP 901.04. ² Enter office that issued the document, by the two-letter code (WIPO Standard ST.3). ³ For Japanese patent documents, the indication of the year of the reign of the Emperor must precede the serial number of the patent document. ⁴ Kind of document by the appropriate symbols as indicated on the document under WIPO Standard ST.16 if possible. ⁵ Applicant is to place a check mark here if English language translation is attached.

Session Began October 12, 2018 at 05:12 PM

Task Began October 12, 2018 05:13 PM

Explore references by patent: (ID 1)

Patent Number: us20180171171
Answer Type: References
Result Count: 1

Detailed display

From ID: 1
Type: hydrophobic coating for protection of an electronic or electric device

Retrieve substance information in 1 reference (ID 2)

From ID: 0
Answer Type: Substances
Result Count: 17

Retrieve reference information in 17 substances (ID 3)

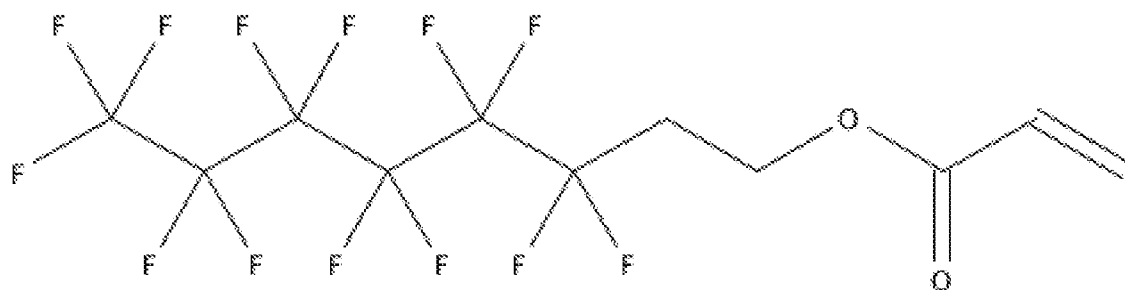
From ID: 2
Answer Type: References
Result Count: 755

Refine by research topic (ID 4)

Research Topic: plasma
From ID: 3
Answer Type: References
Result Count: 42

Prepared 6 reference answers from Answer set 4 for printing in PDF form

Task Began October 12, 2018 05:21 PM

Explore substances by SIMILARITY_2D_EXACT

Candidates: 9

Candidates Selected (ID 6)

99 (most similar)
Answer Type: Substances
Result Count: 1802

Retrieve reference information in 1,802 substances (ID 7)

From ID: 6
Answer Type: References
Result Count: 755

Refine by research topic (ID 8)

Research Topic: plasma polymerization
From ID: 7

Answer Type:

References

Result Count:

17

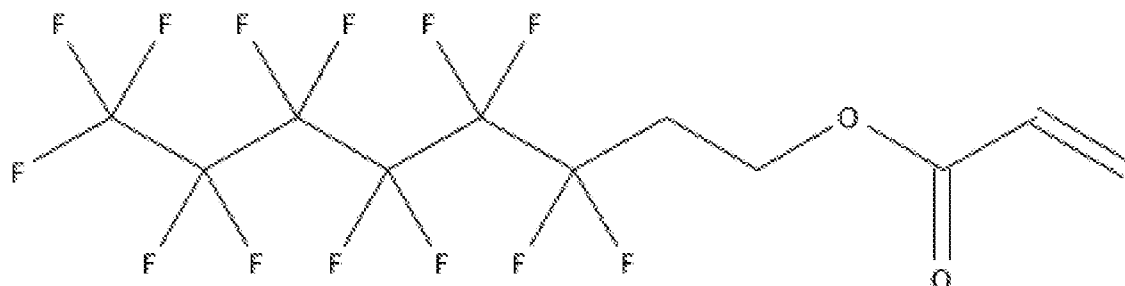
Refine Answer set by structure (ID 9)

Structure:

exact

From ID:

6



Limiters:

Polymers

Answer Type:

Substances

Result Count:

1800

Retrieve reference information in 1,800 substances (ID 10)

From ID:

9

Answer Type:

References

Result Count:

37

Prepared 9 reference answers from Answer set 10 for printing in PDF form**Task Began October 12, 2018 06:02 PM****Explore references by research topic: plasma polymerization of fluoroacrylates**

Research Topic:

plasma polymerization of fluoroacrylates

Result Count:

3

Candidates Selected (ID 11)

2 references were found containing the two concepts "plasma polymerization" and "fluoroacrylates".

Answer Type:

References

Result Count:

2

Task Began October 12, 2018 06:05 PM**Explore references by research topic: plasma fluoroacrylates crosslinked coating**

Research Topic:

plasma fluoroacrylates crosslinked coating

Result Count:

4

Candidates Selected (ID 12)

1150 references were found containing the concept "crosslinked coating", and either the concept "plasma" or the concept "fluoroacrylates".

Automatically removed 25 duplicate MEDLINE answer(s)

2 references were found containing the concept "plasma fluoroacrylates".

Automatically removed 25 duplicate MEDLINE answer(s)

Answer Type:

References

Result Count:

1127

Prepared 1 reference answer from Answer set 12 for printing in PDF form

Copyright © 1994-2018 Sun Microsystems, Inc. All Rights Reserved. (Java runtime environment)

Copyright © 2018, Yahoo! Inc. All Rights Reserved. (YUI Base, Fonts, Reset CSS)

Copyright © 200-2018, The Dojo Foundation. All Rights Reserved

Copyright © 2018, Exadel, Inc. All Rights Reserved. (JBoss RichFaces)

Copyright © 2002 InfoChem GmbH. All Rights Reserved. (InfoChem's reaction classification program CLASSIFY)

The following copyright notice applies to Raphael only: Permission is hereby granted, free of charge, to any person obtaining a copy of this software and associated documentation files (the 'Software'), to deal in the Software without restriction, including without limitation the rights to use, copy, modify, merge, publish, distribute, sublicense, and/or sell copies of the Software, and to permit persons to whom the Software is furnished to do so, subject to the following conditions: The above copyright notice and this permission notice shall be included in all copies or substantial portions of the Software. THE SOFTWARE IS PROVIDED 'AS IS', WITHOUT WARRANTY OF ANY KIND, EXPRESS OR IMPLIED, INCLUDING BUT NOT LIMITED TO THE WARRANTIES OF MERCHANTABILITY, FITNESS FOR A PARTICULAR PURPOSE AND NONINFRINGEMENT. IN NO EVENT SHALL THE AUTHORS OR COPYRIGHT HOLDERS BE LIABLE FOR ANY CLAIM, DAMAGES OR OTHER LIABILITY, WHETHER IN AN ACTION OF CONTRACT, TORT OR OTHERWISE, ARISING FROM, OUT OF OR IN CONNECTION WITH THE SOFTWARE OR THE USE OR OTHER DEALINGS IN THE SOFTWARE.

The following copyright notice applies to Google Closure Templates only: Copyright 2009 Google Inc. Licensed under the Apache License, Version 2.0 (the 'License'); you may not use this file except in compliance with the License. You may obtain a copy of the License at <http://www.apache.org/licenses/LICENSE-2.0> Unless required by applicable law or agreed to in writing, software distributed under the License is distributed on an 'AS IS' BASIS, WITHOUT WARRANTIES OR CONDITIONS OF ANY KIND, either express or implied. See the License for the specific language governing permissions and limitations under the License.

Copyright (c) 2010, 2012 Adobe Systems Incorporated, All Rights Reserved. (Source Sans Pro) Licensed under the SIL Open Font License, Version 1.1 (available at: <http://scripts.sil.org/OFL>)

Copyright 2005-2013 The Dojo Foundation (Dojo Toolkit) Licensed under the Apache License, Version 2.0 (the "License"); you may not use this file except in compliance with the License. You may obtain a copy of the License at: <http://www.apache.org/licenses/LICENSE-2.0> Unless required by applicable law or agreed to in writing, software distributed under the License is distributed on an "AS IS" BASIS, WITHOUT WARRANTIES OR CONDITIONS OF ANY KIND, either express or implied. See the License for the specific language governing permissions and limitations under the License.

Copyright (c) 2008 Dmitry Baranovskiy (Raphael) Licensed under the MIT License (available at: <http://opensource.org/licenses/MIT>) Permission is hereby granted, free of charge, to any person obtaining a copy of this software and associated documentation files (the "Software"), to deal in the Software without restriction, including without limitation the rights to use, copy, modify, merge, publish, distribute, sublicense, and/or sell copies of the Software, and to permit persons to whom the Software is furnished to do so, subject to the following conditions: The above copyright notice and this permission notice shall be included in all copies or substantial portions of the Software. THE SOFTWARE IS PROVIDED "AS IS", WITHOUT WARRANTY OF ANY KIND, EXPRESS OR IMPLIED, INCLUDING BUT NOT LIMITED TO THE WARRANTIES OF MERCHANTABILITY, FITNESS FOR A PARTICULAR PURPOSE AND NONINFRINGEMENT. IN NO EVENT SHALL THE AUTHORS OR COPYRIGHT HOLDERS BE LIABLE FOR ANY CLAIM, DAMAGES OR OTHER LIABILITY, WHETHER IN AN ACTION OF CONTRACT, TORT OR OTHERWISE, ARISING FROM, OUT OF OR IN CONNECTION WITH THE SOFTWARE OR THE USE OR OTHER DEALINGS IN THE SOFTWARE.

CAPLUSSM: Copyright © 2018 American Chemical Society. All Rights Reserved. (The U.K. patent material in this product/service is U.K. Crown copyright and is made available with permission.)

Copyright © Crown Copyright. The French (FR) patent material in this product/service is made available from Institut National de la Propriété Industrielle (INPI.)

CAS REGISTRYSM: Copyright © 2018 American Chemical Society. All Rights Reserved. (Some records contain information from GenBank®. See also: Benson D.A., Karsch-Mizrahi I., Lipman D.J., Ostel J., Rapp B.A., Wheeler D.L. Genbank. Nucl. Acids Res. 28(1):15-18 (2000). Property values tagged with IC are from the ZIC/VINITI data file provided by InfoChem.) CAS Registry is a service mark of the American Chemical Society. GenBank is a registered trademark of the U.S. Library of Medicine.

CASREACT® : Copyright © 2018 American Chemical Society. All Rights Reserved. CASREACT contains reactions from CAS and from: ZIC/VINITI database (1974-1999) provided by InfoChem; INPI data prior to 1988; Biotransformations database compiled under the direction of Professor Dr. Klaus Kieslich; organic reactions, portions copyright 1995-2005 John Wiley & Sons, Ltd., John Wiley and Sons, Inc., Organic Reactions Inc., and Organic Syntheses Inc. Reproduced under license. All Rights Reserved.

CHEMCATS® : Copyright © 2018 American Chemical Society. All Rights Reserved. Chemical supplier information is supplied on an "as is" basis. Full information regarding substance availability, price, etc., is provided when you request supplier information.

CHEMLIST® : Copyright © 2018 American Chemical Society. All Rights Reserved.

MEDLINE® : is produced by the U.S. National Library of Medicine. MEDLINE is a registered trademark of the U.S. National Library of Medicine.

| | | | |
|---|------------------------|-------------------------|------------|
| INFORMATION DISCLOSURE STATEMENT BY APPLICANT (Not for submission under 37 CFR 1.99) | Application Number | | 15735130 |
| | Filing Date | | 2017-12-08 |
| | First Named Inventor | Stephen Richard COULSON | |
| | Art Unit | 1767 | |
| | Examiner Name | BUIE-HATCHER, NICOLE M | |
| | Attorney Docket Number | GBL0007/US | |

| U.S.PATENTS | | | | | | |
|-------------------|---------|---------------|------------------------|------------|---|--|
| Examiner Initial* | Cite No | Patent Number | Kind Code ¹ | Issue Date | Name of Patentee or Applicant of cited Document | Pages, Columns, Lines where Relevant Passages or Relevant Figures Appear |
| | 1 | | | | | |

If you wish to add additional U.S. Patent citation information please click the Add button.

| U.S.PATENT APPLICATION PUBLICATIONS | | | | | | |
|-------------------------------------|---------|--------------------|------------------------|------------------|---|--|
| Examiner Initial* | Cite No | Publication Number | Kind Code ¹ | Publication Date | Name of Patentee or Applicant of cited Document | Pages, Columns, Lines where Relevant Passages or Relevant Figures Appear |
| | 1 | | | | | |

If you wish to add additional U.S. Published Application citation information please click the Add button.

| FOREIGN PATENT DOCUMENTS | | | | | | | | |
|--------------------------|---------|--------------------------------------|---------------------------|------------------------|------------------|---|--|--------------------------|
| Examiner Initial* | Cite No | Foreign Document Number ³ | Country Code ² | Kind Code ⁴ | Publication Date | Name of Patentee or Applicant of cited Document | Pages, Columns, Lines where Relevant Passages or Relevant Figures Appear | T ⁵ |
| | 1 | | | | | | | <input type="checkbox"/> |

If you wish to add additional Foreign Patent Document citation information please click the Add button

| NON-PATENT LITERATURE DOCUMENTS | | | |
|---------------------------------|---------|---|----------------|
| Examiner Initials* | Cite No | Include name of the author (in CAPITAL LETTERS), title of the article (when appropriate), title of the item (book, magazine, journal, serial, symposium, catalog, etc), date, pages(s), volume-issue number(s), publisher, city and/or country where published. | T ⁵ |
| | | | |

**INFORMATION DISCLOSURE
STATEMENT BY APPLICANT**
(Not for submission under 37 CFR 1.99)

| | |
|------------------------|-------------------------|
| Application Number | 15735130 |
| Filing Date | 2017-12-08 |
| First Named Inventor | Stephen Richard COULSON |
| Art Unit | 1767 |
| Examiner Name | BUIE-HATCHER, NICOLE M |
| Attorney Docket Number | GBL0007/US |

| | | |
|---|--|--------------------------|
| 1 | "Plasma treatment can apply liquid repellency to any material", Technical Textiles International, May 2005, pages 23-25 (3 pages). | <input type="checkbox"/> |
|---|--|--------------------------|

If you wish to add additional non-patent literature document citation information please click the Add button

EXAMINER SIGNATURE

| | | | |
|--------------------|--|-----------------|--|
| Examiner Signature | | Date Considered | |
|--------------------|--|-----------------|--|

*EXAMINER: Initial if reference considered, whether or not citation is in conformance with MPEP 609. Draw line through a citation if not in conformance and not considered. Include copy of this form with next communication to applicant.

¹ See Kind Codes of USPTO Patent Documents at www.USPTO.GOV or MPEP 901.04. ² Enter office that issued the document, by the two-letter code (WIPO Standard ST.3). ³ For Japanese patent documents, the indication of the year of the reign of the Emperor must precede the serial number of the patent document. ⁴ Kind of document by the appropriate symbols as indicated on the document under WIPO Standard ST.16 if possible. ⁵ Applicant is to place a check mark here if English language translation is attached.

**INFORMATION DISCLOSURE
STATEMENT BY APPLICANT**
(Not for submission under 37 CFR 1.99)

| | |
|------------------------|-------------------------|
| Application Number | 15735130 |
| Filing Date | 2017-12-08 |
| First Named Inventor | Stephen Richard COULSON |
| Art Unit | 1767 |
| Examiner Name | BUIE-HATCHER, NICOLE M |
| Attorney Docket Number | GBL0007/US |

CERTIFICATION STATEMENT

Please see 37 CFR 1.97 and 1.98 to make the appropriate selection(s):

☐ That each item of information contained in the information disclosure statement was first cited in any communication from a foreign patent office in a counterpart foreign application not more than three months prior to the filing of the information disclosure statement. See 37 CFR 1.97(e)(1).

OR

☐ That no item of information contained in the information disclosure statement was cited in a communication from a foreign patent office in a counterpart foreign application, and, to the knowledge of the person signing the certification after making reasonable inquiry, no item of information contained in the information disclosure statement was known to any individual designated in 37 CFR 1.56(c) more than three months prior to the filing of the information disclosure statement. See 37 CFR 1.97(e)(2).

☐ See attached certification statement.

☒ The fee set forth in 37 CFR 1.17 (p) has been submitted herewith.

☒ A certification statement is not submitted herewith.

SIGNATURE

A signature of the applicant or representative is required in accordance with CFR 1.33, 10.18. Please see CFR 1.4(d) for the form of the signature.

| | | | |
|------------|------------------|---------------------|------------|
| Signature | /Paul J. Parins/ | Date (YYYY-MM-DD) | 2018-11-21 |
| Name/Print | Paul J. Parins | Registration Number | 54,358 |

This collection of information is required by 37 CFR 1.97 and 1.98. The information is required to obtain or retain a benefit by the public which is to file (and by the USPTO to process) an application. Confidentiality is governed by 35 U.S.C. 122 and 37 CFR 1.14. This collection is estimated to take 1 hour to complete, including gathering, preparing and submitting the completed application form to the USPTO. Time will vary depending upon the individual case. Any comments on the amount of time you require to complete this form and/or suggestions for reducing this burden, should be sent to the Chief Information Officer, U.S. Patent and Trademark Office, U.S. Department of Commerce, P.O. Box 1450, Alexandria, VA 22313-1450. **DO NOT SEND FEES OR COMPLETED FORMS TO THIS ADDRESS. SEND TO: Commissioner for Patents, P.O. Box 1450, Alexandria, VA 22313-1450.**

Privacy Act Statement

The Privacy Act of 1974 (P.L. 93-579) requires that you be given certain information in connection with your submission of the attached form related to a patent application or patent. Accordingly, pursuant to the requirements of the Act, please be advised that: (1) the general authority for the collection of this information is 35 U.S.C. 2(b)(2); (2) furnishing of the information solicited is voluntary; and (3) the principal purpose for which the information is used by the U.S. Patent and Trademark Office is to process and/or examine your submission related to a patent application or patent. If you do not furnish the requested information, the U.S. Patent and Trademark Office may not be able to process and/or examine your submission, which may result in termination of proceedings or abandonment of the application or expiration of the patent.

The information provided by you in this form will be subject to the following routine uses:

1. The information on this form will be treated confidentially to the extent allowed under the Freedom of Information Act (5 U.S.C. 552) and the Privacy Act (5 U.S.C. 552a). Records from this system of records may be disclosed to the Department of Justice to determine whether the Freedom of Information Act requires disclosure of these records.
2. A record from this system of records may be disclosed, as a routine use, in the course of presenting evidence to a court, magistrate, or administrative tribunal, including disclosures to opposing counsel in the course of settlement negotiations.
3. A record in this system of records may be disclosed, as a routine use, to a Member of Congress submitting a request involving an individual, to whom the record pertains, when the individual has requested assistance from the Member with respect to the subject matter of the record.
4. A record in this system of records may be disclosed, as a routine use, to a contractor of the Agency having need for the information in order to perform a contract. Recipients of information shall be required to comply with the requirements of the Privacy Act of 1974, as amended, pursuant to 5 U.S.C. 552a(m).
5. A record related to an International Application filed under the Patent Cooperation Treaty in this system of records may be disclosed, as a routine use, to the International Bureau of the World Intellectual Property Organization, pursuant to the Patent Cooperation Treaty.
6. A record in this system of records may be disclosed, as a routine use, to another federal agency for purposes of National Security review (35 U.S.C. 181) and for review pursuant to the Atomic Energy Act (42 U.S.C. 218(c)).
7. A record from this system of records may be disclosed, as a routine use, to the Administrator, General Services, or his/her designee, during an inspection of records conducted by GSA as part of that agency's responsibility to recommend improvements in records management practices and programs, under authority of 44 U.S.C. 2904 and 2906. Such disclosure shall be made in accordance with the GSA regulations governing inspection of records for this purpose, and any other relevant (i.e., GSA or Commerce) directive. Such disclosure shall not be used to make determinations about individuals.
8. A record from this system of records may be disclosed, as a routine use, to the public after either publication of the application pursuant to 35 U.S.C. 122(b) or issuance of a patent pursuant to 35 U.S.C. 151. Further, a record may be disclosed, subject to the limitations of 37 CFR 1.14, as a routine use, to the public if the record was filed in an application which became abandoned or in which the proceedings were terminated and which application is referenced by either a published application, an application open to public inspections or an issued patent.
9. A record from this system of records may be disclosed, as a routine use, to a Federal, State, or local law enforcement agency, if the USPTO becomes aware of a violation or potential violation of law or regulation.

Electronic Patent Application Fee Transmittal

| | | | | |
|---|---------------------------------------|-----------------|---------------|-----------------------------|
| Application Number: | 15735130 | | | |
| Filing Date: | 08-Dec-2017 | | | |
| Title of Invention: | COATINGS | | | |
| First Named Inventor/Applicant Name: | Stephen Richard COULSON | | | |
| Filer: | Paul John Parins/Bethany Lichtscheidl | | | |
| Attorney Docket Number: | GBL0007/US | | | |
| Filed as Large Entity | | | | |
| Filing Fees for U.S. National Stage under 35 USC 371 | | | | |
| Description | Fee Code | Quantity | Amount | Sub-Total in USD(\$) |
| Basic Filing: | | | | |
| Pages: | | | | |
| Claims: | | | | |
| Miscellaneous-Filing: | | | | |
| Petition: | | | | |
| Patent-Appeals-and-Interference: | | | | |
| Post-Allowance-and-Post-Issuance: | | | | |
| Extension-of-Time: | | | | |

| Description | Fee Code | Quantity | Amount | Sub-Total in USD(\$) |
|---|----------|----------|--------|----------------------|
| Miscellaneous: | | | | |
| SUBMISSION- INFORMATION DISCLOSURE STMT | 1806 | 1 | 240 | 240 |
| Total in USD (\$) | | | | 240 |

Electronic Acknowledgement Receipt

| | |
|---|---------------------------------------|
| EFS ID: | 34374605 |
| Application Number: | 15735130 |
| International Application Number: | |
| Confirmation Number: | 7101 |
| Title of Invention: | COATINGS |
| First Named Inventor/Applicant Name: | Stephen Richard COULSON |
| Customer Number: | 33072 |
| Filer: | Paul John Parins/Bethany Lichtscheidl |
| Filer Authorized By: | Paul John Parins |
| Attorney Docket Number: | GBL0007/US |
| Receipt Date: | 21-NOV-2018 |
| Filing Date: | 08-DEC-2017 |
| Time Stamp: | 13:19:45 |
| Application Type: | U.S. National Stage under 35 USC 371 |

Payment information:

| | |
|--|-----------------------|
| Submitted with Payment | yes |
| Payment Type | CARD |
| Payment was successfully received in RAM | \$240 |
| RAM confirmation Number | 112318INTEFSW13203600 |
| Deposit Account | |
| Authorized User | |

The Director of the USPTO is hereby authorized to charge indicated fees and credit any overpayment as follows:

| File Listing: | | | | | |
|--|--|----------------------|--|------------------|------------------|
| Document Number | Document Description | File Name | File Size(Bytes)/ Message Digest | Multi Part /.zip | Pages (if appl.) |
| 1 | Information Disclosure Statement (IDS) Form (SB08) | GBL0007US_SIDS.pdf | 73743 | no | 4 |
| | | | 456ad696f9fe41ff85772448b10ebe379e526a82 | | |
| Warnings: | | | | | |
| Information: | | | | | |
| This is not an USPTO supplied IDS fillable form | | | | | |
| 2 | Non Patent Literature | Plasma_treatment.pdf | 595496 | no | 3 |
| | | | 5eaec8c580a621d9715f7a8b6ad643d1d6ffe70 | | |
| Warnings: | | | | | |
| Information: | | | | | |
| 3 | Fee Worksheet (SB06) | fee-info.pdf | 30379 | no | 2 |
| | | | 63be4e84c6a538ea190cc6fc5dc8e7bd7605655 | | |
| Warnings: | | | | | |
| Information: | | | | | |
| Total Files Size (in bytes): | | | 699618 | | |
| <p>This Acknowledgement Receipt evidences receipt on the noted date by the USPTO of the indicated documents, characterized by the applicant, and including page counts, where applicable. It serves as evidence of receipt similar to a Post Card, as described in MPEP 503.</p> <p><u>New Applications Under 35 U.S.C. 111</u> If a new application is being filed and the application includes the necessary components for a filing date (see 37 CFR 1.53(b)-(d) and MPEP 506), a Filing Receipt (37 CFR 1.54) will be issued in due course and the date shown on this Acknowledgement Receipt will establish the filing date of the application.</p> <p><u>National Stage of an International Application under 35 U.S.C. 371</u> If a timely submission to enter the national stage of an international application is compliant with the conditions of 35 U.S.C. 371 and other applicable requirements a Form PCT/DO/EO/903 indicating acceptance of the application as a national stage submission under 35 U.S.C. 371 will be issued in addition to the Filing Receipt, in due course.</p> <p><u>New International Application Filed with the USPTO as a Receiving Office</u> If a new international application is being filed and the international application includes the necessary components for an international filing date (see PCT Article 11 and MPEP 1810), a Notification of the International Application Number and of the International Filing Date (Form PCT/RO/105) will be issued in due course, subject to prescriptions concerning national security, and the date shown on this Acknowledgement Receipt will establish the international filing date of the application.</p> | | | | | |

In The United States Patent And Trademark Office

| | |
|---|---------------------------------------|
| Inventor: Stephen Richard COULSON | Examiner: Buie-Hatcher, Nicole |
| Serial No. 15/735,130 | Group Art Unit: 1767 |
| International Filing Date: June 8, 2016 | Confirmation No. 7101 |
| International App. No. PCT/GB2016/051688 | Docket No. GBL0007/US |
| 371(c): December 8, 2017 | |
| For: COATINGS | |

Mail Stop Amendment
 Commissioner for Patents
 P.O. Box 1450
 Alexandria, Virginia 22313-1450

I CERTIFY THAT ON MARCH 14, 2019, THIS CORRESPONDENCE
 IS BEING ELECTRONICALLY TRANSMITTED TO THE UNITED
 STATES PATENT AND TRADEMARK OFFICE VIA THE
 OFFICE'S EFS-WEB.


 BETHANY J. LICHTSCHEIDL

TRANSMITTAL

Dear Sir or Madam:

Enclosed herewith are the following in connection with the above-identified patent application:

- A. Declarations (37 CFR 1.63) for Utility Application Using an Application Data Sheet, executed by:
 - Stephen Richard Coulson,
 - Delwyn Evans,
 - Angeliki Siokou
- B. A Substitute Statement in Lieu of an Oath or Declaration for Utility Design Patent Application or Design Patent Application (35 U.S.C. 115(d) And 37 CFR 1.64), executed by:
 - assignee (Dr. Stephen Coulson) on behalf of Clive Telford.
- C. It is believed that no surcharge is required in filing these declarations as the \$140.00 Surcharge for filing the Declaration late was paid on the same day that the application was filed (December 8, 2017).

Date: March 14, 2019

Respectfully Submitted,

By: 

Paul J. Parins, Reg. No. 54,358

Customer Number 33072

Phone: 651-275-9831

Fax: 651-351-2954

#124098

DECLARATION (37 CFR 1.63) FOR UTILITY OR DESIGN APPLICATION USING AN
APPLICATION DATA SHEET (37 CFR 1.76)

| | |
|------------------------|-------------------------------------|
| Title of Invention | COATINGS |
| Attorney Docket No. | GBL0007/US (P2494US00-CD7QC) |

As the below named inventor, I hereby declare that:

This declaration is directed to:

☐ The attached application; and/or
☒ United States application or PCT international application no.
PCT/GB2016/051688 filed on June 8, 2016; and/or
☐ The application having the above-identified attorney docket number on the
specification as filed, or the above-identified title on the specification as filed.

The above-identified application was made or authorized to be made by me.

I believe that I am the original inventor or an original joint inventor of a claimed invention in the application.

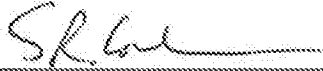
I hereby state that I have reviewed and understand the contents of the above-identified application,
including the claims.

I am aware of the duty to disclose information which is material to patentability as defined in 37 CFR 1.56.

I hereby acknowledge that any willful false statement made in this declaration is punishable under 18
U.S.C. 1001 by fine or imprisonment of not more than five (5) years, or both.

LEGAL NAME OF INVENTOR

Inventor: Stephen Richard COULSON Date (optional): 6.3.17

Signature: 

#117251

DECLARATION (37 CFR 1.63) FOR UTILITY OR DESIGN APPLICATION USING AN
APPLICATION DATA SHEET (37 CFR 1.76)

| | | | |
|------------------------|------------------------------|--|--|
| Title of Invention | COATINGS | | |
| Attorney Docket No. | GBL0007/US (P2494US00-CD7QC) | | |

As the below named inventor, I hereby declare that:

This declaration is directed to:

☐ The attached application; and/or
☒ United States application or PCT international application no. PCT/GB2016/051688 filed on June 8, 2016; and/or
☐ The application having the above-identified attorney docket number on the specification as filed, or the above-identified title on the specification as filed.

The above-identified application was made or authorized to be made by me.

I believe that I am the original inventor or an original joint inventor of a claimed invention in the application.


I hereby state that I have reviewed and understand the contents of the above-identified application, including the claims.

I am aware of the duty to disclose information which is material to patentability as defined in 37 CFR 1.56.

I hereby acknowledge that any willful false statement made in this declaration is punishable under 18 U.S.C. 1001 by fine or imprisonment of not more than five (5) years, or both.


LEGAL NAME OF INVENTOR

Inventor: Delwyn EVANS Date (optional): 27 FEB 2019

Signature: 

#117251

**DECLARATION (37 CFR 1.63) FOR UTILITY OR DESIGN APPLICATION USING AN
APPLICATION DATA SHEET (37 CFR 1.76)**

| | | | |
|--|-------------------------------------|----------------------------------|--|
| Title of Invention | COATINGS | | |
| Attorney Docket No. | GBL0007/US (P2494US00-CD7QC) | | |
| <p>As the below named inventor, I hereby declare that:</p> <p>This declaration is directed to: <input type="checkbox"/> The attached application; and/or</p> <p> <input checked="" type="checkbox"/> United States application or PCT international application no. PCT/GB2016/051688 filed on June 8, 2016; and/or</p> <p> <input type="checkbox"/> The application having the above-identified attorney docket number on the specification as filed, or the above-identified title on the specification as filed.</p> <p>The above-identified application was made or authorized to be made by me.</p> <p>I believe that I am the original inventor or an original joint inventor of a claimed invention in the application.</p> <p>I hereby state that I have reviewed and understand the contents of the above-identified application, including the claims.</p> <p>I am aware of the duty to disclose information which is material to patentability as defined in 37 CFR 1.56.</p> <p>I hereby acknowledge that any willful false statement made in this declaration is punishable under 18 U.S.C. 1001 by fine or imprisonment of not more than five (5) years, or both.</p> | | | |
| LEGAL NAME OF INVENTOR | | | |
| Inventor: Angeliki SIOKOU | | Date (optional): <u>27.02.19</u> | |
| Signature:  | | | |

#117251

**SUBSTITUTE STATEMENT IN LIEU OF AN OATH OR DECLARATION FOR UTILITY
OR DESIGN PATENT APPLICATION (35 U.S.C. 115(d) AND 37 CFR 1.64)**

| | | | |
|---|----------|---------|---------|
| Title of Invention | COATINGS | | |
| This statement is directed to: | | | |
| <input type="checkbox"/> The attached application, | | | |
| OR | | | |
| <input checked="" type="checkbox"/> United States application or PCT international application number <u>15/735,130</u> filed on <u>December 8, 2017</u> | | | |
| LEGAL NAME of inventor to whom this substitute statement applies: | | | |
| (E.g., Given Name (first and middle (if any)) and Family Name or Surname) | | | |
| Clive Telford | | | |
| Residence (except for a deceased or legally incapacitated inventor): | | | |
| City | State | Country | |
| Mailing Address (except for a deceased or legally incapacitated inventor): | | | |
| City | State | Zip | Country |
| I believe the above-named inventor or joint inventor to be the original inventor or an original joint inventor of a claimed invention in the application. | | | |
| The above-identified application was made or authorized to be made by me. | | | |
| I hereby acknowledge that any willful false statement made in this statement is punishable under 18 U.S.C. 1001 by fine or imprisonment of not more than five (5) years, or both. | | | |
| Relationship to the inventor to whom this substitute statement applies: | | | |
| <input type="checkbox"/> Legal Representative (for deceased or legally incapacitated inventor only). | | | |
| <input checked="" type="checkbox"/> Assignee. | | | |
| <input type="checkbox"/> Person to whom the inventor is under an obligation to assign. | | | |
| <input type="checkbox"/> Person who otherwise shows a sufficient proprietary interest in the matter (petition under 37 CFR 1.46 is required), or | | | |
| <input type="checkbox"/> Joint Inventor. | | | |

[Page 1 of 2]

This collection of information is required by 35 U.S.C. 115 and 37 CFR 1.63. The information is required to obtain or retain a benefit by the public which is to file (and by the USPTO to process) an application. Confidentiality is governed by 35 U.S.C. 122 and 37 CFR 1.11 and 1.14. This collection is estimated to take 1 minute to complete, including gathering, preparing, and submitting the completed application form to the USPTO. Time will vary depending upon the individual case. Any comments on the amount of time you require to complete this form and/or suggestions for reducing this burden, should be sent to the Chief Information Officer, U.S. Patent and Trademark Office, U.S. Department of Commerce, P.O. Box 1450, Alexandria, VA 22313-1450. DO NOT SEND FEES OR COMPLETED FORMS TO THIS ADDRESS. SEND TO: Commissioner for Patents, P.O. Box 1450, Alexandria, VA 22313-1450.

If you need assistance in completing the form, call 1-800-PTO-9199 and select option 2.

SUBSTITUTE STATEMENT

Circumstances permitting execution of this substitute statement:

- ☒ Inventor is deceased,
☐ Inventor is under legal incapacity,
☐ Inventor cannot be found or reached after diligent effort, or
☐ Inventor has refused to execute the oath or declaration under 37 CFR 1.63.

If there are joint inventors, please check the appropriate box below:


- ☒ An application data sheet under 37 CFR 1.76 (PTO/AIA/14 or equivalent) naming the entire inventive entity has been or is currently submitted.
 OR
☐ An application data sheet under 37 CFR 1.76 (PTO/AIA/14 or equivalent) has not been submitted. Thus, a Substitute Statement Supplemental Sheet (PTO/AIA/11 or equivalent) naming the entire inventive entity and providing inventor information is attached. See 37 CFR 1.64(b).

WARNING:

Petitioner/applicant is cautioned to avoid submitting personal information in documents filed in a patent application that may contribute to identity theft. Personal information such as social security numbers, bank account numbers, or credit card numbers (other than a check or credit card authorization form PTO-2038 submitted for payment purposes) is never required by the USPTO to support a petition or an application. If this type of personal information is included in documents submitted to the USPTO, petitioners/applicants should consider redacting such personal information from the documents before submitting them to the USPTO. Petitioner/applicant is advised that the record of a patent application is available to the public after publication of the application (unless a non-publication request in compliance with 37 CFR 1.213(a) is made in the application) or issuance of a patent. Furthermore, the record from an abandoned application may also be available to the public if the application is referenced in a published application or an issued patent (see 37 CFR 1.14). Checks and credit card authorization forms PTO-2038 submitted for payment purposes are not retained in the application file and therefore are not publicly available.

PERSON EXECUTING THIS SUBSTITUTE STATEMENT:

Name: **Dr. Stephen Coulson** Date (Optional): **6.3.19**

Signature: **APPLICANT NAME AND TITLE OF PERSON EXECUTING THIS SUBSTITUTE STATEMENT:**

If the applicant is a juristic entity, list the applicant name and the title of the signer:

P2i Ltd

Applicant Name:

Title of Person Executing
 This Substitute Statement: **CSO, Founder, President Information Security**

The signer, whose title is supplied above, is authorized to act on behalf of the applicant.

Residence of the signer (unless provided in an application data sheet, PTO/AIA/14 or equivalent):

City **Abingdon** State Country **GB**

Mailing Address of the signer (unless provided in an application data sheet, PTO/AIA/14 or equivalent)

9-12 North Central, 127 Olympic Avenue, Milton Park

City **Abingdon** State Zip **OX14 4SA** Country **GB**

Note: Use an additional PTO/AIA/02 form for each inventor who is deceased, legally incapacitated, cannot be found or reached after diligent effort, or has refused to execute the oath or declaration under 37 CFR 1.63.

Privacy Act Statement

The **Privacy Act of 1974 (P.L. 93-579)** requires that you be given certain information in connection with your submission of the attached form related to a patent application or patent. Accordingly, pursuant to the requirements of the Act, please be advised that: (1) the general authority for the collection of this information is 35 U.S.C. 2(b)(2); (2) furnishing of the information solicited is voluntary; and (3) the principal purpose for which the information is used by the U.S. Patent and Trademark Office is to process and/or examine your submission related to a patent application or patent. If you do not furnish the requested information, the U.S. Patent and Trademark Office may not be able to process and/or examine your submission, which may result in termination of proceedings or abandonment of the application or expiration of the patent.

The information provided by you in this form will be subject to the following routine uses:

1. The information on this form will be treated confidentially to the extent allowed under the Freedom of Information Act (5 U.S.C. 552) and the Privacy Act (5 U.S.C. 552a). Records from this system of records may be disclosed to the Department of Justice to determine whether disclosure of these records is required by the Freedom of Information Act.
2. A record from this system of records may be disclosed, as a routine use, in the course of presenting evidence to a court, magistrate, or administrative tribunal, including disclosures to opposing counsel in the course of settlement negotiations.
3. A record in this system of records may be disclosed, as a routine use, to a Member of Congress submitting a request involving an individual, to whom the record pertains, when the individual has requested assistance from the Member with respect to the subject matter of the record.
4. A record in this system of records may be disclosed, as a routine use, to a contractor of the Agency having need for the information in order to perform a contract. Recipients of information shall be required to comply with the requirements of the Privacy Act of 1974, as amended, pursuant to 5 U.S.C. 552a(m).
5. A record related to an International Application filed under the Patent Cooperation Treaty in this system of records may be disclosed, as a routine use, to the International Bureau of the World Intellectual Property Organization, pursuant to the Patent Cooperation Treaty.
6. A record in this system of records may be disclosed, as a routine use, to another federal agency for purposes of National Security review (35 U.S.C. 181) and for review pursuant to the Atomic Energy Act (42 U.S.C. 218(c)).
7. A record from this system of records may be disclosed, as a routine use, to the Administrator, General Services, or his/her designee, during an inspection of records conducted by GSA as part of that agency's responsibility to recommend improvements in records management practices and programs, under authority of 44 U.S.C. 2904 and 2906. Such disclosure shall be made in accordance with the GSA regulations governing inspection of records for this purpose, and any other relevant (i.e., GSA or Commerce) directive. Such disclosure shall not be used to make determinations about individuals.
8. A record from this system of records may be disclosed, as a routine use, to the public after either publication of the application pursuant to 35 U.S.C. 122(b) or issuance of a patent pursuant to 35 U.S.C. 151. Further, a record may be disclosed, subject to the limitations of 37 CFR 1.14, as a routine use, to the public if the record was filed in an application which became abandoned or in which the proceedings were terminated and which application is referenced by either a published application, an application open to public inspection or an issued patent.
9. A record from this system of records may be disclosed, as a routine use, to a Federal, State, or local law enforcement agency, if the USPTO becomes aware of a violation or potential violation of law or regulation.

Electronic Acknowledgement Receipt

| | |
|---|---------------------------------------|
| EFS ID: | 35420272 |
| Application Number: | 15735130 |
| International Application Number: | |
| Confirmation Number: | 7101 |
| Title of Invention: | COATINGS |
| First Named Inventor/Applicant Name: | Stephen Richard COULSON |
| Customer Number: | 33072 |
| Filer: | Paul John Parins/Bethany Lichtscheidl |
| Filer Authorized By: | Paul John Parins |
| Attorney Docket Number: | GBL0007/US |
| Receipt Date: | 14-MAR-2019 |
| Filing Date: | 08-DEC-2017 |
| Time Stamp: | 11:11:11 |
| Application Type: | U.S. National Stage under 35 USC 371 |

Payment information:

| | |
|------------------------|----|
| Submitted with Payment | no |
|------------------------|----|

File Listing:

| Document Number | Document Description | File Name | File Size(Bytes)/ Message Digest | Multi Part /.zip | Pages (if appl.) |
|-----------------|----------------------|--|--|------------------|------------------|
| 1 | Transmittal Letter | GBL0007US_Transmittal_for_Declarations.pdf | 212108 470f712ee11cbccf3c5990c6345f95e9e31729d6 | no | 2 |

Warnings:

Ex. 1028 - Favored - p. 282

| | | | | | |
|---|---------------------------|------------------------------------|--|----|---|
| Information: | | | | | |
| 2 | Oath or Declaration filed | GBL0007US_Declaration.pdf | 398658 | no | 3 |
| | | | dd44e5c87c549bb551dcb6c9c400982f43a4c68a | | |
| Warnings: | | | | | |
| Information: | | | | | |
| 3 | Oath or Declaration filed | GBL0007US_Substitute_Statement.pdf | 634125 | no | 3 |
| | | | 3eb370671cae47e410a2009cc6dfb7f38bed56b7 | | |
| Warnings: | | | | | |
| Information: | | | | | |
| Total Files Size (in bytes): | | | 1244891 | | |
| <p>This Acknowledgement Receipt evidences receipt on the noted date by the USPTO of the indicated documents, characterized by the applicant, and including page counts, where applicable. It serves as evidence of receipt similar to a Post Card, as described in MPEP 503.</p> <p><u>New Applications Under 35 U.S.C. 111</u> If a new application is being filed and the application includes the necessary components for a filing date (see 37 CFR 1.53(b)-(d) and MPEP 506), a Filing Receipt (37 CFR 1.54) will be issued in due course and the date shown on this Acknowledgement Receipt will establish the filing date of the application.</p> <p><u>National Stage of an International Application under 35 U.S.C. 371</u> If a timely submission to enter the national stage of an international application is compliant with the conditions of 35 U.S.C. 371 and other applicable requirements a Form PCT/DO/EO/903 indicating acceptance of the application as a national stage submission under 35 U.S.C. 371 will be issued in addition to the Filing Receipt, in due course.</p> <p><u>New International Application Filed with the USPTO as a Receiving Office</u> If a new international application is being filed and the international application includes the necessary components for an international filing date (see PCT Article 11 and MPEP 1810), a Notification of the International Application Number and of the International Filing Date (Form PCT/RO/105) will be issued in due course, subject to prescriptions concerning national security, and the date shown on this Acknowledgement Receipt will establish the international filing date of the application.</p> | | | | | |

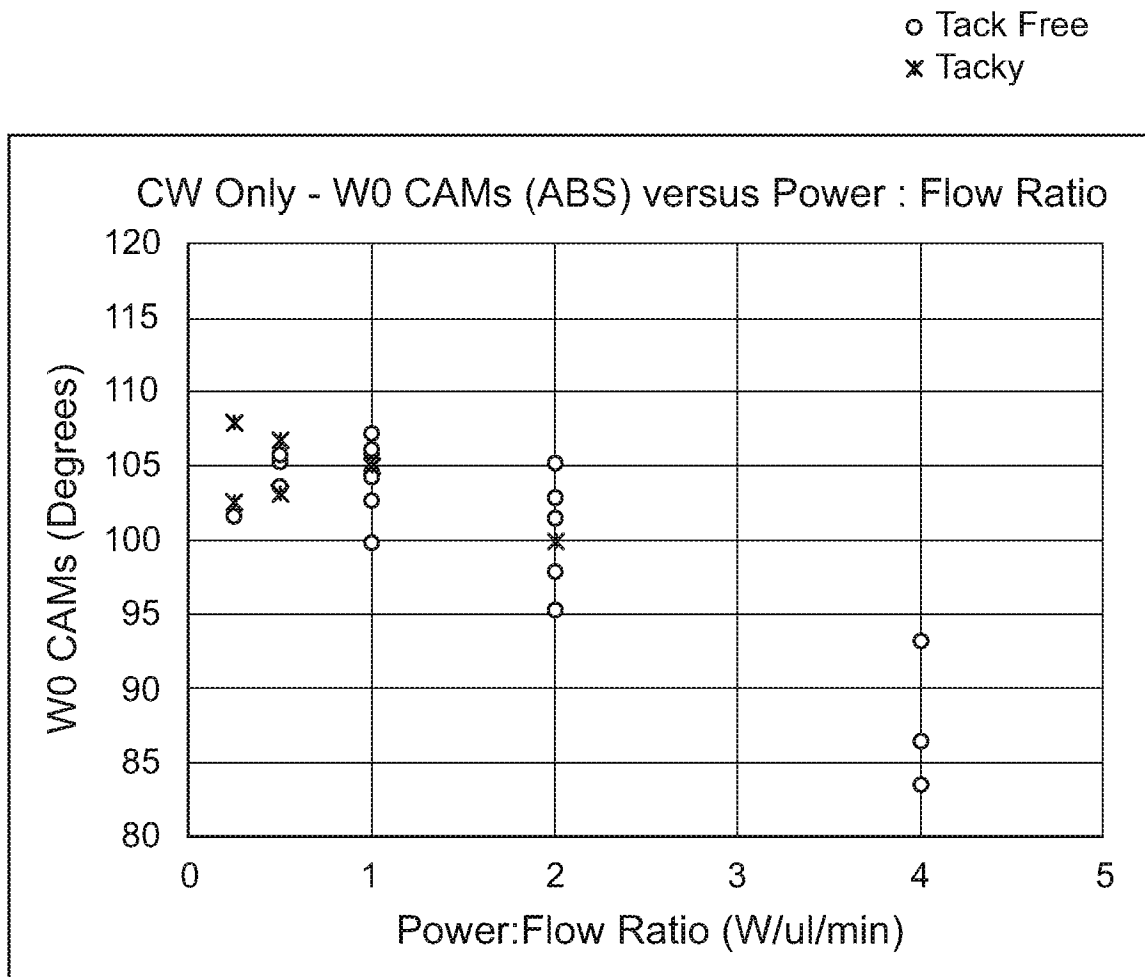


Fig. 1

○ Tack Free
× Tacky

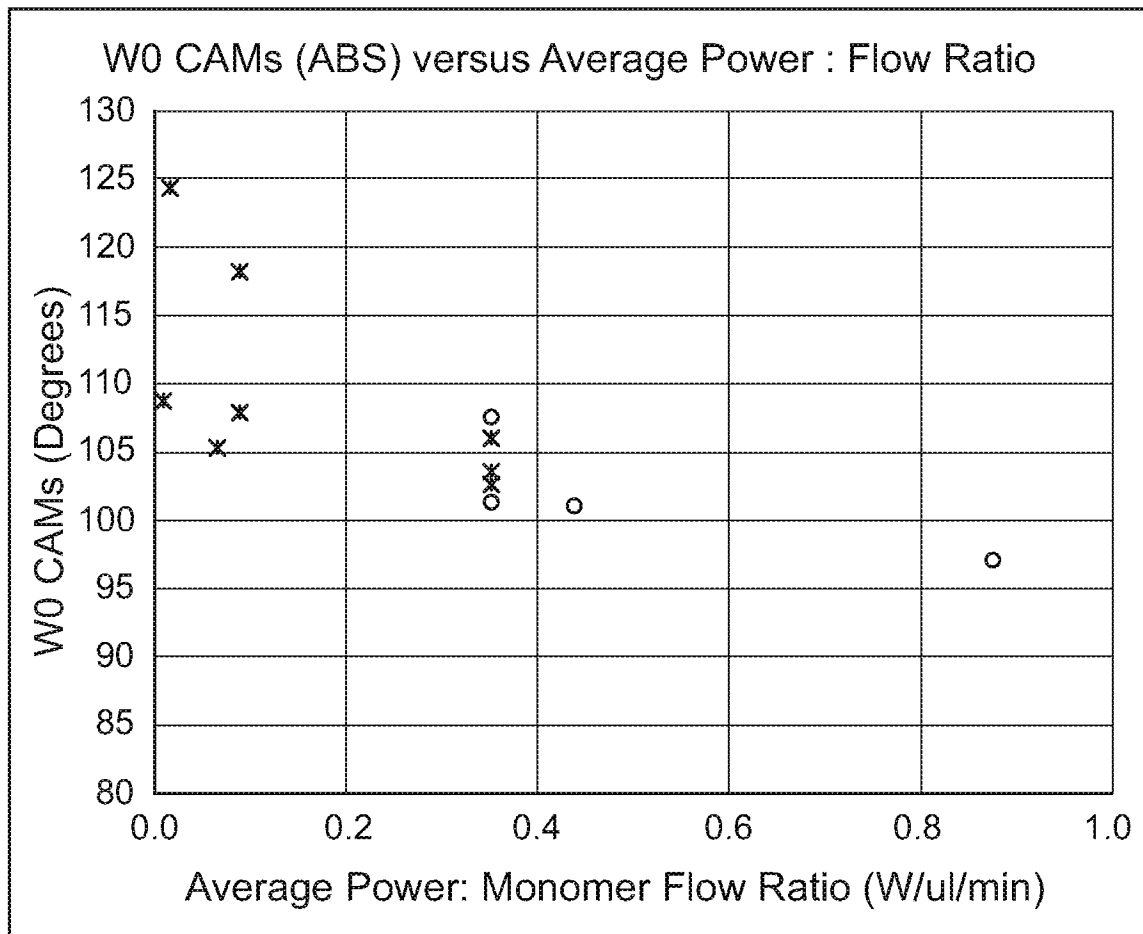


Fig. 2

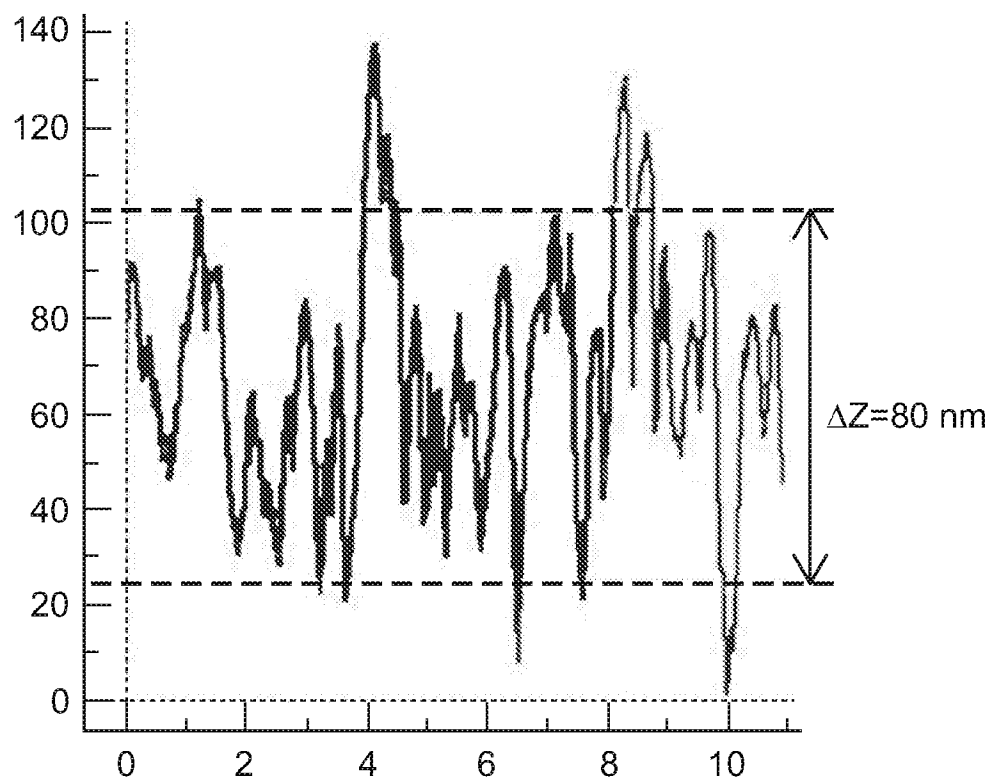
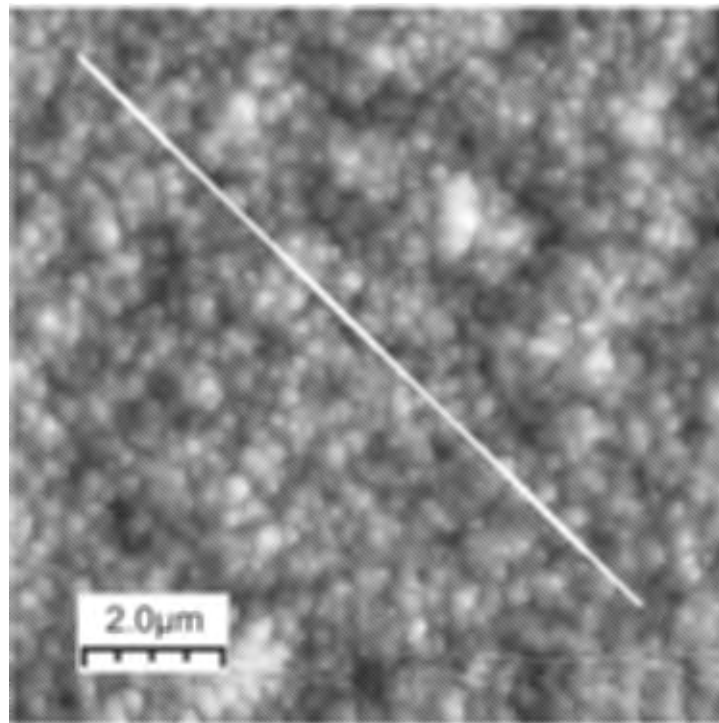


Fig. 3

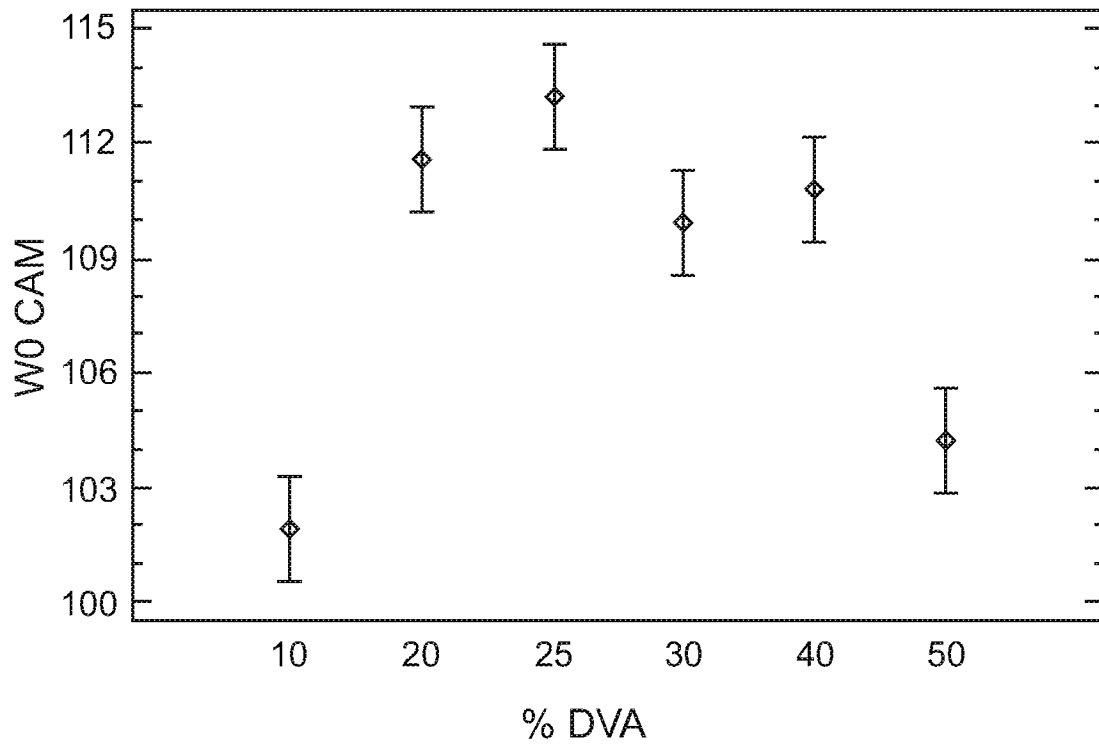


Fig. 4

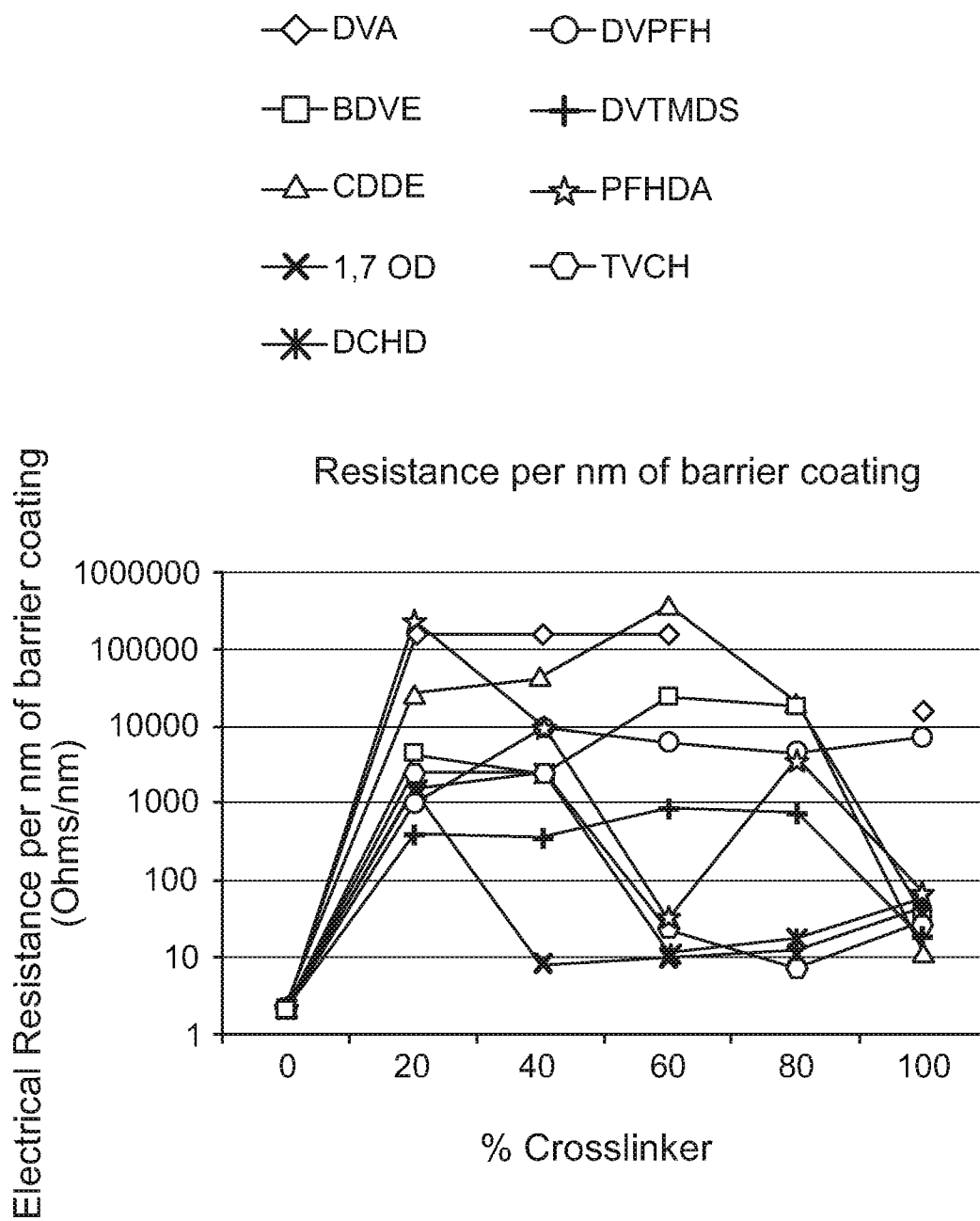


Fig. 5

6 / 6

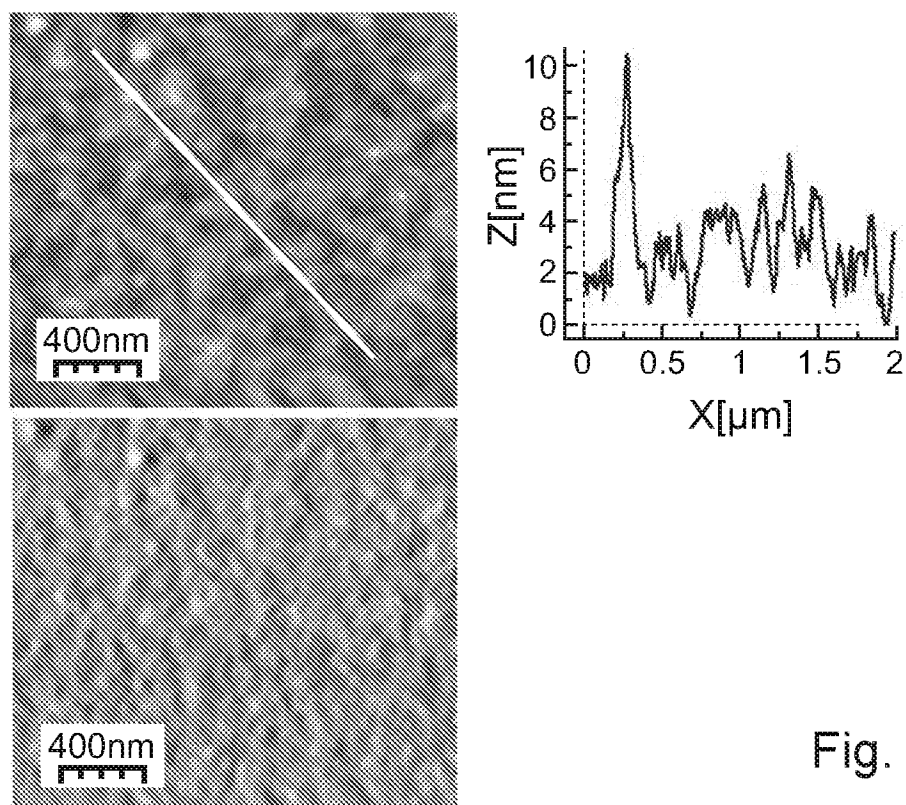
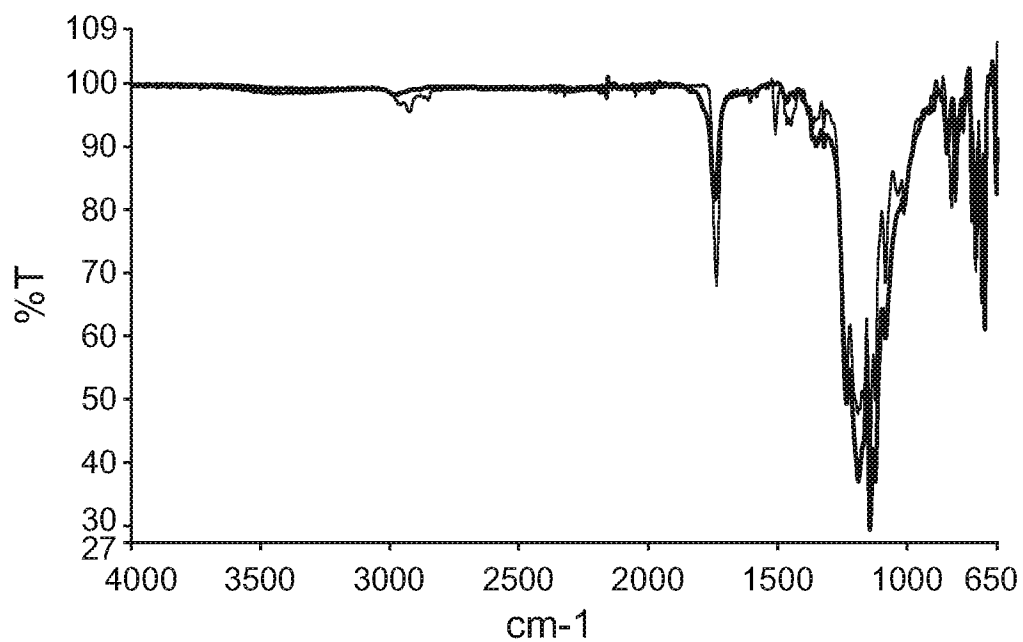


Fig. 6



| Name | Description |
|------------------|---|
| — PFAC6 | Sample 350 By Administrator Date Thursday, February 13 2014 |
| — PFAC6-DVA(10%) | Sample 389 By PEService Date Tuesday, July 14 2015 |

Fig. 7

| Electronic Patent Application Fee Transmittal | | | | |
|--|----------|---------------------------------------|--------|----------------------|
| Application Number: | | 15735130 | | |
| Filing Date: | | 08-Dec-2017 | | |
| Title of Invention: | | COATINGS | | |
| First Named Inventor/Applicant Name: | | Stephen Richard COULSON | | |
| Filer: | | Paul John Parins/Bethany Lichtscheidl | | |
| Attorney Docket Number: | | GBL0007/US | | |
| Filed as Large Entity | | | | |
| Filing Fees for U.S. National Stage under 35 USC 371 | | | | |
| Description | Fee Code | Quantity | Amount | Sub-Total in USD(\$) |
| Basic Filing: | | | | |
| Pages: | | | | |
| Claims: | | | | |
| Miscellaneous-Filing: | | | | |
| Petition: | | | | |
| Patent-Appeals-and-Interference: | | | | |
| Post-Allowance-and-Post-Issuance: | | | | |
| Extension-of-Time: | | | | |

| Description | Fee Code | Quantity | Amount | Sub-Total in USD(\$) |
|------------------------------------|----------|----------|--------|----------------------|
| Extension - 2 months with \$0 paid | 1252 | 1 | 600 | 600 |
| Miscellaneous: | | | | |
| Total in USD (\$) | | | | 600 |

Electronic Acknowledgement Receipt

| | |
|---|---------------------------------------|
| EFS ID: | 35447276 |
| Application Number: | 15735130 |
| International Application Number: | |
| Confirmation Number: | 7101 |
| Title of Invention: | COATINGS |
| First Named Inventor/Applicant Name: | Stephen Richard COULSON |
| Customer Number: | 33072 |
| Filer: | Paul John Parins/Bethany Lichtscheidl |
| Filer Authorized By: | Paul John Parins |
| Attorney Docket Number: | GBL0007/US |
| Receipt Date: | 18-MAR-2019 |
| Filing Date: | 08-DEC-2017 |
| Time Stamp: | 11:00:25 |
| Application Type: | U.S. National Stage under 35 USC 371 |

Payment information:

| | |
|--|-----------------------|
| Submitted with Payment | yes |
| Payment Type | CARD |
| Payment was successfully received in RAM | \$600 |
| RAM confirmation Number | 031819INTEFSW11013800 |
| Deposit Account | |
| Authorized User | |

The Director of the USPTO is hereby authorized to charge indicated fees and credit any overpayment as follows:

| | | | | | |
|------------------------------|---|--|--|------------------|------------------|
| | | | | | |
| File Listing: | | | | | |
| Document Number | Document Description | File Name | File Size(Bytes)/ Message Digest | Multi Part /.zip | Pages (if appl.) |
| 1 | | GBL0007US_Response_to_Non-Final_OA.pdf | 182316 | yes | 13 |
| | | | d1a8000f854fd5dc38c714c95a6dbbe39173fd87 | | |
| | Multipart Description/PDF files in .zip description | | | | |
| | Document Description | | Start | End | |
| | Applicant Arguments/Remarks Made in an Amendment | | 10 | 13 | |
| | Claims | | 3 | 9 | |
| | Amendment/Req. Reconsideration-After Non-Final Reject | | 1 | 2 | |
| Warnings: | | | | | |
| Information: | | | | | |
| 2 | Drawings-only black and white line drawings | GBL0007US_replacement_drawings.pdf | 178887 | no | 6 |
| | | | 2ac523b93f2e1ef05eb5f5646d4401cfec3a2eba | | |
| Warnings: | | | | | |
| Information: | | | | | |
| 3 | Fee Worksheet (SB06) | fee-info.pdf | 30658 | no | 2 |
| | | | 1e28cad9fa35fafad8b5e7122f8b7073acbf2419 | | |
| Warnings: | | | | | |
| Information: | | | | | |
| Total Files Size (in bytes): | | | 391861 | | |

This Acknowledgement Receipt evidences receipt on the noted date by the USPTO of the indicated documents, characterized by the applicant, and including page counts, where applicable. It serves as evidence of receipt similar to a Post Card, as described in MPEP 503.

New Applications Under 35 U.S.C. 111

If a new application is being filed and the application includes the necessary components for a filing date (see 37 CFR 1.53(b)-(d) and MPEP 506), a Filing Receipt (37 CFR 1.54) will be issued in due course and the date shown on this Acknowledgement Receipt will establish the filing date of the application.

National Stage of an International Application under 35 U.S.C. 371

If a timely submission to enter the national stage of an international application is compliant with the conditions of 35 U.S.C. 371 and other applicable requirements a Form PCT/DO/EO/903 indicating acceptance of the application as a national stage submission under 35 U.S.C. 371 will be issued in addition to the Filing Receipt, in due course.

New International Application Filed with the USPTO as a Receiving Office

If a new international application is being filed and the international application includes the necessary components for an international filing date (see PCT Article 11 and MPEP 1810), a Notification of the International Application Number and of the International Filing Date (Form PCT/RO/105) will be issued in due course, subject to prescriptions concerning national security, and the date shown on this Acknowledgement Receipt will establish the international filing date of the application.

Remarks

Claims 1-3, 5-16, and 31-34 are pending. With this Response, claims 1, 6, 8, 13, 15, and 16 are amended; and claims 5, 7, 12, and 14 are cancelled. Upon entry of the current amendments, claims 1-3, 6, 8-11, 13, 15, 16, and 31-34 are pending for consideration. The present amendments are fully supported by the application as originally filed and do not introduce new matter.

Applicant respectfully requests reconsideration and further examination of the application in view of the amendments above and remarks below.

Allowable Subject Matter

Applicant gratefully acknowledges the indication in the Office Action that claims 7 and 12-15 would be allowable if rewritten to overcome the rejection(s) under 35 U.S.C. 112(b) or 35 U.S.C. 112 (pre-AIA), 2nd paragraph, set forth in this Office action and to include all of the limitations of the base claim and any intervening claims. As explained below, claim 1 is amended to include subject matter of claims 7, 12, and 14 (using alternative “or” language), therefore claim 1 is considered allowable.

Claim 1 is amended to limit L for compound (i) to having the features of claim 5 in combination with 7 (i.e. “Formula A”), or to having the features of claim 5 in combination with claim 12 or claim 14 (i.e. “Formula B”).

The terms “Formula A” and “Formula B” have been added to refer to the optional features included in claim 1, in order to avoid unnecessary repetition of these features in the dependent claims. Hence the inclusion of these terms in claim 1 does not add matter.

Claim 1 has been amended to broaden the definitions of Y1 to Y8 and R1, R2, R4, and R5 to R10 to be independently selected from hydrogen or an optionally substituted _{C1-C6} branched or straight chain alkyl group. Basis for this amendment is found on page 5, lines 22 and 23, and on page 5, lines 5 and 6 of the PCT application as published.

Specification

According to the Office Action, the disclosure is objected to because of the following informalities:

- 1) On page 1, “it is unknown what is the co-pending application.” With this reply, the paragraph on page 1 that begins with “Monosaturated monomers” and ends with “(see co-pending application)” is deleted thereby rendering this objection moot.
- 2) A Brief Description of Drawings section is missing. With this reply, said section is added to the specification, thereby rendering this objection moot.

Accordingly, it is respectfully requested that the specification objections be withdrawn.

Drawing Objections

According to the Office Action, the drawings are objected to because each of the view numbers must be preceded by the abbreviation “Fig.”

With this reply, each of figures 1-7 are amended to replace “Figure” with “Fig.” as requested in the Office Action.

Accordingly, it is respectfully requested the outstanding drawing objections be withdrawn.

Claim Objections

Claims 6 and 7 are objected to because of the following informalities: both claims 6 and 7 do not end with periods. Claim 6 is amended to include a period and claim 7 is cancelled.

Accordingly, it is respectfully requested that the objection of claims 7 and 6 be withdrawn.

Claims 12 and 13 are objected to because of the following informalities: the word "Wherein" should be lowercase in claim 12. Claim 12 is canceled thereby rendering this objection moot.

Accordingly, it is respectfully requested that the objection of claims 12 and 13 be withdrawn.

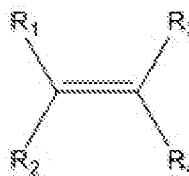
Claim 16 objected to because of the following informalities: the capital names used for the names of the compounds should be lowercase. The abbreviations are appropriate. Claim 16 is amended as suggested by the Office Action, thereby rendering this objection moot.

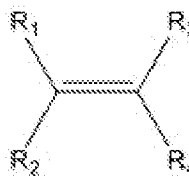
Accordingly, it is respectfully requested that the objection of claim 16 be withdrawn.

Rejection Under 35 U.S.C. §112(b)

Claims 1-3, 5-16, and 31-34 are rejected under 35 U.S.C. 112(b) or 35 U.S.C. 112 (pre-AIA), second paragraph, as being indefinite.

According to the Office Action, the phrase “wherein the protective cross-linked polymeric coating is obtainable by” in claim 1 is unclear. With this reply, the word “obtainable” in said phrase is replaced with “obtained,” thereby rendering this rejection moot.



According to the Office Action, the structure  in claim 1 is unclear. With this reply, said structure is deleted thereby rendering this rejection moot. Applicant notes that said structure was intended to be deleted with the Preliminary Amendment previously filed.

Accordingly, it is respectfully requested that the rejection of claims 1-3, 5-16, and 31-34 under 35 U.S.C. 112(b) or 35 U.S.C. 112(pre-AIA), second paragraph, as being indefinite, be withdrawn.

Rejection Under 35 U.S.C. §103

Claims 1-3, 5-6, 8-11, 16, and 31-34 are rejected under 35 U.S.C. 103 as being unpatentable over Coulson (WO 2007/083122 A1, referred to hereinafter as WO '122) in view of Coulson (US 2010/0203347 A1, referred to hereinafter as US '347).

Independent claim 1 is amended in manner to include the subject matter of claims 7, 12, or 14 (in an alternative manner), thereby rendering this rejection moot.

Accordingly, it is respectfully requested that the rejection of claims 1-3, 5-6, 8-11, 16, and 31-34, under 35 U.S.C. 103 as being unpatentable over Coulson (WO 2007/083122 A1, referred to hereinafter as WO '122) in view of Coulson (US 2010/0203347 A1, referred to hereinafter as US '347) be withdrawn.

Conclusion

In view of the above amendments and remarks, it is respectfully submitted that all of the pending claims are in condition for allowance. Approval of the application and allowance of the claims are earnestly solicited. In the event that a phone conference between the Examiner and the Applicants' undersigned attorney would help resolve any issues in the application, the Examiner is invited to contact said attorney at (651) 275-9831.

Respectfully Submitted,

Dated: March 18, 2019

By: Paul J. Parins/

Paul J. Parins, Reg. No. 54,358

Customer No. 33072

Phone: 651-275-9831

Facsimile: 651-351-2954

#124050

| | | |
|---|------------------------|-------------------------|
| INFORMATION DISCLOSURE STATEMENT BY APPLICANT (Not for submission under 37 CFR 1.99) | Application Number | 15735130 |
| | Filing Date | 2017-12-08 |
| | First Named Inventor | Stephen Richard COULSON |
| | Art Unit | 1767 |
| | Examiner Name | BUIE-HATCHER, NICOLE M |
| | Attorney Docket Number | GBL0007/US |

| U.S.PATENTS | | | | | | Remove |
|---|---------|--------------------|------------------------|------------------|---|--|
| Examiner Initial* | Cite No | Patent Number | Kind Code ¹ | Issue Date | Name of Patentee or Applicant of cited Document | Pages,Columns,Lines where Relevant Passages or Relevant Figures Appear |
| | 1 | 4822632 | A | 1989-04-18 | Williams et al. | |
| | 2 | 6764758 | B1 | 2004-07-20 | Grunze et al. | |
| If you wish to add additional U.S. Patent citation information please click the Add button. | | | | | | Add |
| U.S.PATENT APPLICATION PUBLICATIONS | | | | | | Remove |
| Examiner Initial* | Cite No | Publication Number | Kind Code ¹ | Publication Date | Name of Patentee or Applicant of cited Document | Pages,Columns,Lines where Relevant Passages or Relevant Figures Appear |
| | 1 | 20040028931 | A1 | 2004-02-12 | Bletsos et al. | |
| | 2 | 20120051018 | A1 | 2012-03-01 | Ollgaard | |
| | 3 | 20120171386 | A1 | 2012-07-05 | Bicker et al. | |
| | 4 | 20120321775 | A1 | 2012-12-20 | Parker | |

**INFORMATION DISCLOSURE
STATEMENT BY APPLICANT**
(Not for submission under 37 CFR 1.99)

| | |
|------------------------|-------------------------|
| Application Number | 15735130 |
| Filing Date | 2017-12-08 |
| First Named Inventor | Stephen Richard COULSON |
| Art Unit | 1767 |
| Examiner Name | BUIE-HATCHER, NICOLE M |
| Attorney Docket Number | GBL0007/US |

| | | | | |
|----|-------------|----|------------|------------------|
| 5 | 20140010969 | A1 | 2014-01-09 | Bicker et al. |
| 6 | 20090267489 | A1 | 2009-10-29 | Arita et al. |
| 7 | 20100189914 | A1 | 2010-07-29 | Coulson |
| 8 | 20120009231 | A1 | 2012-01-12 | Herbert et al. |
| 9 | 20140322525 | A1 | 2014-10-30 | Legein et al. |
| 10 | 20150291830 | A1 | 2015-10-15 | Galbreath et al. |
| 11 | 20040046165 | A1 | 2004-03-11 | Hunze et al. |

If you wish to add additional U.S. Published Application citation information please click the Add button.

Add

FOREIGN PATENT DOCUMENTS

Remove

| Examiner Initial* | Cite No | Foreign Document Number ³ | Country Code ^{2,i} | Kind Code ⁴ | Publication Date | Name of Patentee or Applicant of cited Document | Pages, Columns, Lines where Relevant Passages or Relevant Figures Appear | T ⁵ |
|-------------------|---------|--------------------------------------|-----------------------------|------------------------|------------------|---|--|----------------|
| | 1 | 2434368 | GB | A | 2007-07-25 | P2i Limited | | |
| | 2 | 2493264 | GB | A | 2013-01-30 | P2i Ltd | | |

**INFORMATION DISCLOSURE
STATEMENT BY APPLICANT**
(Not for submission under 37 CFR 1.99)

| | | |
|------------------------|-------------------------|------------|
| Application Number | | 15735130 |
| Filing Date | | 2017-12-08 |
| First Named Inventor | Stephen Richard COULSON | |
| Art Unit | 1767 | |
| Examiner Name | BUIE-HATCHER, NICOLE M | |
| Attorney Docket Number | GBL0007/US | |

| | | | | | | | |
|----|-------------|----|----|------------|---|--|--|
| 3 | 03063938 | WO | A1 | 2003-08-07 | ASTRAZENECA AB | | |
| 4 | 2005021833 | WO | A2 | 2005-03-10 | SURFACE INNOVATIONS LIMITED | | |
| 5 | 2009046093 | WO | A2 | 2009-04-09 | EXOGENESIS CORPORATION | | |
| 6 | 2009030435 | WO | A1 | 2009-03-12 | UNIV AUGSBURG | | |
| 7 | 2011029628 | WO | A1 | 2011-03-17 | SCHOTT AG | | |
| 8 | 2711153 | EP | A1 | 2014-03-26 | The Goodyear Tire and Rubber Company | | |
| 9 | 2005/089961 | WO | A1 | 2005-09-29 | THE SECRETARY OF STATE OF DEFENCE | | |
| 10 | 02/28548 | WO | A2 | 2002-04-11 | DOW CORNING CORPORATION | | |
| 11 | 2013/132250 | WO | A1 | 2013-09-12 | SEMBLANT LTD | | |
| 12 | 2012/158953 | WO | A2 | 2012-11-22 | GADGET IP LLC | | |
| 13 | 2014026967 | WO | A2 | 2014-02-20 | EUROPLASMA NV | | |

| | | | |
|---|------------------------|-------------------------|------------|
| INFORMATION DISCLOSURE STATEMENT BY APPLICANT (Not for submission under 37 CFR 1.99) | Application Number | | 15735130 |
| | Filing Date | | 2017-12-08 |
| | First Named Inventor | Stephen Richard COULSON | |
| | Art Unit | 1767 | |
| | Examiner Name | BUIE-HATCHER, NICOLE M | |
| | Attorney Docket Number | GBL0007/US | |

If you wish to add additional Foreign Patent Document citation information please click the Add button

NON-PATENT LITERATURE DOCUMENTS

| Examiner Initials* | Cite No | Include name of the author (in CAPITAL LETTERS), title of the article (when appropriate), title of the item (book, magazine, journal, serial, symposium, catalog, etc), date, pages(s), volume-issue number(s), publisher, city and/or country where published. | T ⁵ |
|--------------------|---------|---|----------------|
| | 1 | Search Report under Section 17(5) from UK Patent Application No. GB1403558.8, mailed on August 13, 2014 (4 pages) | |
| | 2 | International Search Report and Written Opinion from International Application No. PCT/GB2015/050521, mailed June 5, 2015 (16 pages) | |
| | 3 | Search Report under Section 17(5) from UK Patent Application No. GB1503054.7, mailed on November 30, 2015 (4 pages) | |

If you wish to add additional non-patent literature document citation information please click the Add button

EXAMINER SIGNATURE

| | | | |
|--------------------|--|-----------------|--|
| Examiner Signature | | Date Considered | |
|--------------------|--|-----------------|--|

***EXAMINER:** Initial if reference considered, whether or not citation is in conformance with MPEP 609. Draw line through a citation if not in conformance and not considered. Include copy of this form with next communication to applicant.

¹ See Kind Codes of USPTO Patent Documents at www.USPTO.GOV or MPEP 901.04. ² Enter office that issued the document, by the two-letter code (WIPO Standard ST.3). ³ For Japanese patent documents, the indication of the year of the reign of the Emperor must precede the serial number of the patent document. ⁴ Kind of document by the appropriate symbols as indicated on the document under WIPO Standard ST.16 if possible. ⁵ Applicant is to place a check mark here if English language translation is attached.

**INFORMATION DISCLOSURE
STATEMENT BY APPLICANT**
(Not for submission under 37 CFR 1.99)

| | |
|------------------------|-------------------------|
| Application Number | 15735130 |
| Filing Date | 2017-12-08 |
| First Named Inventor | Stephen Richard COULSON |
| Art Unit | 1767 |
| Examiner Name | BUIE-HATCHER, NICOLE M |
| Attorney Docket Number | GBL0007/US |

CERTIFICATION STATEMENT

Please see 37 CFR 1.97 and 1.98 to make the appropriate selection(s):

That each item of information contained in the information disclosure statement was first cited in any communication from a foreign patent office in a counterpart foreign application not more than three months prior to the filing of the information disclosure statement. See 37 CFR 1.97(e)(1).

OR

☐ That no item of information contained in the information disclosure statement was cited in a communication from a foreign patent office in a counterpart foreign application, and, to the knowledge of the person signing the certification after making reasonable inquiry, no item of information contained in the information disclosure statement was known to any individual designated in 37 CFR 1.56(c) more than three months prior to the filing of the information disclosure statement. See 37 CFR 1.97(e)(2).

See attached certification statement.

- ☒ The fee set forth in 37 CFR 1.17 (p) has been submitted herewith.
- ☒ A certification statement is not submitted herewith.

SIGNATURE

A signature of the applicant or representative is required in accordance with CFR 1.33, 10.18. Please see CFR 1.4(d) for the form of the signature.

| | | | |
|------------|------------------|---------------------|------------|
| Signature | /Paul J. Parins/ | Date (YYYY-MM-DD) | 2019-03-18 |
| Name/Print | Paul J. Parins | Registration Number | 54,358 |

This collection of information is required by 37 CFR 1.97 and 1.98. The information is required to obtain or retain a benefit by the public which is to file (and by the USPTO to process) an application. Confidentiality is governed by 35 U.S.C. 122 and 37 CFR 1.14. This collection is estimated to take 1 hour to complete, including gathering, preparing and submitting the completed application form to the USPTO. Time will vary depending upon the individual case. Any comments on the amount of time you require to complete this form and/or suggestions for reducing this burden, should be sent to the Chief Information Officer, U.S. Patent and Trademark Office, U.S. Department of Commerce, P.O. Box 1450, Alexandria, VA 22313-1450. DO NOT SEND FEES OR COMPLETED FORMS TO THIS ADDRESS. **SEND TO: Commissioner for Patents, P.O. Box 1450, Alexandria, VA 22313-1450.**

Privacy Act Statement

The Privacy Act of 1974 (P.L. 93-579) requires that you be given certain information in connection with your submission of the attached form related to a patent application or patent. Accordingly, pursuant to the requirements of the Act, please be advised that: (1) the general authority for the collection of this information is 35 U.S.C. 2(b)(2); (2) furnishing of the information solicited is voluntary; and (3) the principal purpose for which the information is used by the U.S. Patent and Trademark Office is to process and/or examine your submission related to a patent application or patent. If you do not furnish the requested information, the U.S. Patent and Trademark Office may not be able to process and/or examine your submission, which may result in termination of proceedings or abandonment of the application or expiration of the patent.

The information provided by you in this form will be subject to the following routine uses:

1. The information on this form will be treated confidentially to the extent allowed under the Freedom of Information Act (5 U.S.C. 552) and the Privacy Act (5 U.S.C. 552a). Records from this system of records may be disclosed to the Department of Justice to determine whether the Freedom of Information Act requires disclosure of these records.
2. A record from this system of records may be disclosed, as a routine use, in the course of presenting evidence to a court, magistrate, or administrative tribunal, including disclosures to opposing counsel in the course of settlement negotiations.
3. A record in this system of records may be disclosed, as a routine use, to a Member of Congress submitting a request involving an individual, to whom the record pertains, when the individual has requested assistance from the Member with respect to the subject matter of the record.
4. A record in this system of records may be disclosed, as a routine use, to a contractor of the Agency having need for the information in order to perform a contract. Recipients of information shall be required to comply with the requirements of the Privacy Act of 1974, as amended, pursuant to 5 U.S.C. 552a(m).
5. A record related to an International Application filed under the Patent Cooperation Treaty in this system of records may be disclosed, as a routine use, to the International Bureau of the World Intellectual Property Organization, pursuant to the Patent Cooperation Treaty.
6. A record in this system of records may be disclosed, as a routine use, to another federal agency for purposes of National Security review (35 U.S.C. 181) and for review pursuant to the Atomic Energy Act (42 U.S.C. 218(c)).
7. A record from this system of records may be disclosed, as a routine use, to the Administrator, General Services, or his/her designee, during an inspection of records conducted by GSA as part of that agency's responsibility to recommend improvements in records management practices and programs, under authority of 44 U.S.C. 2904 and 2906. Such disclosure shall be made in accordance with the GSA regulations governing inspection of records for this purpose, and any other relevant (i.e., GSA or Commerce) directive. Such disclosure shall not be used to make determinations about individuals.
8. A record from this system of records may be disclosed, as a routine use, to the public after either publication of the application pursuant to 35 U.S.C. 122(b) or issuance of a patent pursuant to 35 U.S.C. 151. Further, a record may be disclosed, subject to the limitations of 37 CFR 1.14, as a routine use, to the public if the record was filed in an application which became abandoned or in which the proceedings were terminated and which application is referenced by either a published application, an application open to public inspections or an issued patent.
9. A record from this system of records may be disclosed, as a routine use, to a Federal, State, or local law enforcement agency, if the USPTO becomes aware of a violation or potential violation of law or regulation.



(11) EP 2 711 153 A1

(12) EUROPEAN PATENT APPLICATION

(43) Date of publication:
26.03.2014 Bulletin 2014/13

(51) Int Cl:
B29C 33/64 (2006.01) B29C 33/58 (2006.01)
B29D 30/06 (2006.01) B05D 1/00 (2006.01)
C23C 16/46 (2006.01)

(21) Application number: 13184883.0

(22) Date of filing: 18.09.2013

(84) Designated Contracting States:
AL AT BE BG CH CY CZ DE DK EE ES FI FR GB
GR HR HU IE IS IT LI LT LU LV MC MK MT NL NO
PL PT RO RS SE SI SK SM TR
Designated Extension States:
BA ME

(72) Inventors:
• Siffer, Frederic Gerard Auguste
Akron, Ohio 44313 (US)
• Richards, Jimmy Lee
Rootstown, Ohio 44272 (US)

(30) Priority: 21.09.2012 US 201213623922

(74) Representative: Kutsch, Bernd
Goodyear S.A.
Patent Department
Avenue Gordon Smith
7750 Colmar-Berg (LU)

(71) Applicant: The Goodyear Tire & Rubber Company
Akron, Ohio 44316 (US)

(54) Method of coating a metal mold surface with a polymer coating, mold for rubber products and method of molding rubber products

(57) A method of treating a metal surface (1) of a mold for molding and curing rubber products is disclosed. The method comprises applying an atmospheric plasma comprising an ionized gas containing a polymerizable

precursor to a metal molding surface of the mold metal surface to form and bond a polymer layer of plasma polymerized precursor on the mold metal surface (1).

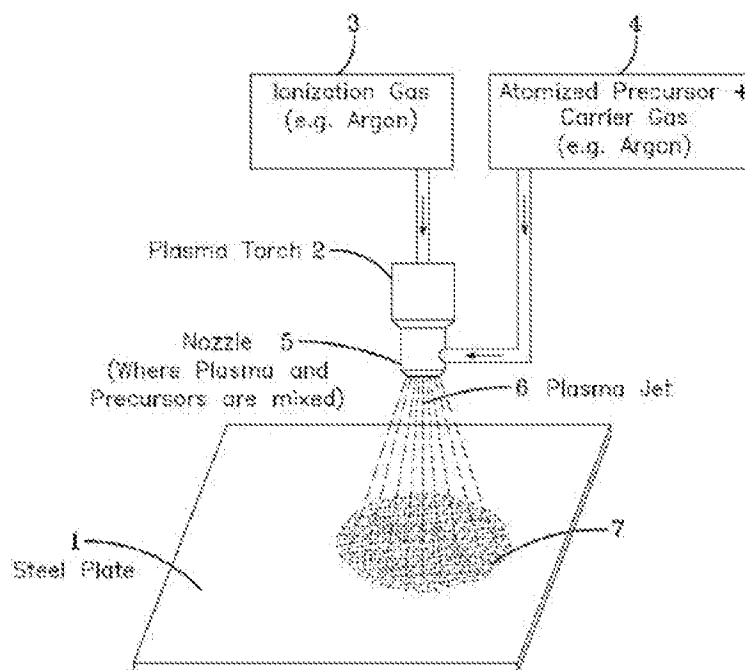


FIG-1

EP 2 711 153 A1

DescriptionField of the invention

5 [0001] This invention relates to preparation of a metal mold surface for molding rubber products by plasma jet directed polymer coating, a coated mold for rubber products and production of rubber products. Such rubber products are rubber tires and industrial products, particularly rubber tires.

Background

10 [0002] Rubber products, such as for example pneumatic rubber tires, are typically produced by molding and curing sulfur curable green, or uncured, rubber tires in a tire mold in which the uncured pneumatic rubber tire is pressed outwardly against a metal mold surface by means of an internal fluid expanding rubber bladder (e.g. by application of internal steam pressure to the bladder envelop). By such method the uncured rubber tire is pressed against and shaped

15 against the metal mold surface which defines the tire's tread pattern and configuration of its sidewalls and during which a portion of the rubber in proximity to the mold surface flows to permit the rubber to assume the contours of the mold surface. By application of heat to the mold, the tire is cured within the mold. The mold is then opened and the cured tire removed from the mold. Such pneumatic rubber tire molding and curing is well known to those having skill in such art.

20 [0003] It is important that such molded and sulfur cured rubber products such as tires be readily removable from their tire molds. A release coating is sometimes applied to the surface of the uncured tire to allow the cured tire to be suitably removed from the mold. It is both economically and physically desirable to provide a durable release coating on a tire mold surface itself to provide a releasability of the molded tire from the tire mold over repeated tire molding and curing cycles.

25 [0004] Further, by repeated tire molding and curing cycles, the mold surface of a metal tire mold may be subject to a buildup of materials, or debris, on its surface. Buildup of materials on the mold surface may sometimes be referred to mold fouling which may, for example, affect cosmetic appearance of the surface of the molded tire. Such fouled metal tire mold surface may be periodically cleaned to remove such mold fouling when appropriate. Such tendency for mold fouling is well known to those having skill in such art.

30 [0005] Therefore, in practice, it may be desired to promote releasability of sulfur cured rubber products such as tires, as well as various industrial product such as, for example belts, hoses, track pads and shoes, from their mold surfaces and, further, to retard, diminish or substantially prevent or release mold fouling of the surface of the rubber (e.g. tire) mold.

[0006] For this invention, it desired to evaluate a plasma induced polymer coating of a metal mold surface for molding rubber products (e.g. tires) to promote releasability of a cured rubber product.

Summary and Practice of the Invention

35 [0007] The invention relates to a method in accordance with claim 1, to a mold in accordance with claim 13 and to methods in accordance with claims 14 and 15 respectively.

[0008] Dependent claims refer to preferred embodiments of the invention.

40 [0009] In one aspect, the present invention is directed to a method of treating a metal (e.g. steel or aluminum) mold surface for molding and curing rubber products (e.g. tires) which comprises applying a thin polymer layer to the surface of the mold to facilitate removal of a rubber product having been cured with in such mold.

[0010] A plurality of such coatings on the metal mold surface may also be provided.

45 [0011] Further, a mold for rubber products (e.g. tires) is provided with a coated metal surface (e.g. steel or aluminum surface) prepared by said method.

[0012] In addition, a method of molding and curing a rubber product (e.g. pneumatic rubber tire) is provided.

50 [0013] In accordance with this invention, a method of treating a metal surface of a mold for molding and curing rubber products is provided which comprises directing an atmospheric plasma jet comprising an ionized gas containing a polymerizable precursor (e.g. atomized liquid polymerizable precursor) to a metal molding surface of the mold to form and bond a thin polymer layer of plasma polymerized precursor on said metal mold surface to promote release of a cured rubber product from said treated mold surface.

[0014] In one embodiment, said ionized gas for said plasma is electrically ionized by, for example, allowing said gas to pass between a set of positive and negative electrodes (e.g. an electric arc) to cause the gas to be in a high state of high energy environment to thereby promote polymerization of the introduced polymerizable precursor.

55 [0015] Such deposited polymer on said metal mold surface from said plasma is in a form of a polymer layer having a thickness, for example, in a range of from 10 nm to 500 nm, preferably 20 nm to 100 nm, and is provided from polymerizable precursors so long the resulting polymer has an ability to promote releasability of a cured rubber product from the deposited polymer layer on the metal mold surface.

[0016] Representative of such rubber products are pneumatic rubber tires and engineered (e.g. industrial) rubber products comprising for example belts (e.g. power and transmission belts), hoses, vehicular track pads and shoes.

[0017] It is to be appreciated that the atmospheric plasma jets may be in a form of pulse or continuous plasmas. The plasmas may be generated, for example, from dielectric barrier jets, microwave torches, radio-frequency torches, low frequency torches, current-carrying arc plasma jets, or by any other means of producing a suitable atmospheric plasma able to polymerize organic precursors of polymers known to one skilled in such art.

[0018] While there are additional methods of creating an atmospheric plasma jet, a current-carrying arc plasma jet, appears to be an acceptable means of applying the thin polymer layer to the metal mold surface.

[0019] In practice, to the ionized gas may be introduced a small particle sized (e.g. atomized) liquid polymerizable precursor (polymer precursor, namely a precursor to form a polymer) to form a plasma jet, expelled through and from a torch, comprised of the ionized gas and atomized precursor for which polymerization of the precursor is promoted by the high energy environment provided by the plasma.

[0020] The plasma jet is directed (e.g. directed from its torch) to a molding surface of the steel or aluminum mold to form and deposit a thin polymer layer (e.g. coating) from the polymerized precursor on said steel surface of the mold by polymerization of said polymerizable precursor.

[0021] In one embodiment, it is desired that the polymer layer is bonded to the steel or aluminum mold surface in a sense of chemical bonding of the polymerized coating to the surface of the steel or aluminum is created as a result of the high plasma energy environment.

[0022] In one embodiment, it is desired that the polymer layer resists significant adhesion of the rubber to thereby permit the molded and cured rubber to be releasable from the mold surface, (e.g. to permit removal of the molded rubber product from the mold surface).

[0023] In one embodiment, it is desired that the polymer comprising a crosslinked polymer in a sense of it has been observed that the plasma polymerized coatings usually exhibit some degree of crosslinking. In this manner, it is believed that a degree of crosslink density within the polymer coating promotes mechanical strength to withstand abrasion when taking cured tires out of the mold.

[0024] By atmospheric pressure plasma, it is meant that the pressure of the plasma is equal to or slightly above the ambient pressure of the surroundings. The pressure of the plasma may be desirably somewhat higher than ambient, such that the plasma pressure is sufficient to induce the desired flow rate through a plasma generator which contains an ionized gas promoting electric arc.

[0025] An ionizable gas (e.g. carrier gas for the polymerizable polymer precursor) may be, for example a noble gas such as, for example helium, argon, xenon and neon as well as, for example, oxygen, nitrogen, carbon dioxide, nitrous oxide and air. In one embodiment, such ionizable gas is argon.

[0026] A carrier gas to carry the atomized liquid polymerizable precursor to the plasma plume may be for example also a noble gas such as, for example, helium, argon, xenon and neon as well as, for example, oxygen, nitrogen, carbon dioxide, nitrous oxide and air. In one embodiment such carrier gas is argon.

[0027] Representative of such polymerizable precursors may be comprised of, for example, and not intended to be limiting, one or more of disiloxanes, disilazanes, perfluoroacrylates (e.g. perfluorooctylacrylate and perfluorooctylmethacrylate), fluorinated silanes, vinylidene fluoride.

[0028] For example, such polymerizable precursors may be a disiloxane such as, for example, hexamethyldisiloxane; a disilazane such as for example hexamethyldisilazane; fluorinated silane comprising, for example, 1 H, 1 H, 2H-perfluorooctyltriethoxysilane and other fluorinated compounds comprising at least one of 1 H, 1 H, 2H-perfluoro-1-alkylene where said alkylene group comprises of decene, octene, hexene or butene groups or 1 H, 1 H, 2H-perfluoro-1-alkanol where said alkanol group comprises of decanol, octanol, hexanol or butanol groups and fluorinated compounds such as, for example perfluoromethylcyclohexane, perfluorodimethylcyclohexane, hexafluorobenzene, perfluoromethyl vinyl ether, perfluoroethyl vinyl ether, perfluoropropyl vinyl ether, perfluorodimethylcyclobutane, perfluoromethylcyclopentane, perfluoromethyldecalin, tetrafluoroethylene, hexafluoropropylene and perfluorobutyl ethylene.

[0029] Such disiloxane, disilazane, fluorinated silane, vinylidene fluoride and fluorinated compounds may be combined, if desired and appropriate with said perfluoroacrylates to form a precursor for said plasma coating method and to thereby form a copolymer thereof.

[0030] The practice of the method of this invention is considered as being significant in a sense of being able to refresh the coating on the metal mold surface at a rubber product (e.g. pneumatic tire) manufacturing facility, particularly where a thin coating is desired to be applied by an energy and polymer precursor efficient method.

[0031] For a batch process wherein an object is exposed to an atmospheric plasma, the object may be exposed, for example, to the plasma for a period of from 1 to 100 seconds. In a continuous process, the exposure time may be characterized by a residence time expressed as the length or circumference of the object to be treated (e.g. in centimeters) with the plasma torch divided by the torch velocity (e.g. in cm/sec) multiplied by the number of plasma treatments applied to the object. The residence time in such a continuous process would then range, for example, from 0.5 to 100 seconds.

[0032] The flow rate of atomized precursor into the plasma generator necessary to obtain an effective amount of

polymerized or partially polymerized precursor onto the mold surface will depend on the desired face velocity in the plasma generator, i.e. the gas velocity (e.g. in cm/sec) exiting the plasma torch. Necessary flow rate may be determined by one skilled in the art without undue experimentation.

[0033] The following drawings are provided for further understanding of the invention and are not intended to be limiting.

The Drawings

[0034] A method is depicted for providing a thin polymer layer for enhancing releasability of sulfur cured rubber from a steel or aluminum surface, such as for example a metal surface of a mold for rubber products, such as for example tires and industrial products such as for example, belts, hoses, tracks and shoes, particularly a tire mold.

In the Drawings

[0035] In Figure 1 a steel plate 1, is sandblasted.

[0036] To a cylindrical torch 2 device is fed a stream 3 of argon gas in which the argon gas is caused to pass through between positive and negative electrodes to ionize the argon gas to a high energy level at a flow rate through the torch 2 device of about 800 liters/hour (l/hr).

[0037] To the torch 2 device is introduced a stream of atomized precursor 4 carried by a carrier gas 5, namely argon gas where the atomized precursor 4 stream, namely a polymer precursor, is introduced into the stream 3 of argon gas, at substantially ambient atmospheric pressure, in the nozzle 6 of the torch 2 device to create a plasma jet 7 comprising the stream 3 of ionized gas which contains the atomized precursor 4.

[0038] The plasma jet 7 is passed over the steel plate 1 to deposit a thin polymer coating 8 onto the steel plate 1 created by the atomized precursor 4 contained in the stream 3 of ionized argon gas.

[0039] The invention is further described with reference to the following examples.

EXAMPLE

[0040] In this example, treatments of sandblasted steel plates by an atmospheric plasma jet by which a polymerizable precursor is introduced are evaluated for providing a coating of polymer on the steel surface and compared to a steel plate without such treatment.

[0041] The plasma is provided as an electrical arc-formed ionized argon gas to which an atomized liquid polymerizable precursor is introduced. A polymerization of the precursor is promoted by the highly ionized argon gas in a sense being in a nature of what is believed to be a free radical polymerization process.

[0042] The liquid polymerizable precursor used was comprising 70 weight percent hexamethyldisiloxane (HMDSO) and 30 weight percent 1H,1H,2H,2H-perfluorooctyl acrylate (PFQA).

[0043] For this evaluation, 1 cm by 3 cm steel plates were positioned on a table and held stationary while the plasma jet was applied by the plasma torch device where the plasma jet was comprising the high energy ionized argon gas containing the atomized liquid polymerizable precursor for which one or more passes of the torch device, and thereby the jet, over the surface of the steel plates.

[0044] An electrical arc power of 70 Watts was provided through which the argon gas was passed within the torch device to cause its ionization.

[0045] The argon gas flow rate through the torch was about 800 liters/hour to which an atomized liquid polymerizable precursor is injected to form the plasma torch.

[0046] The plasma torch comprised of the ionized argon gas and polymerizable precursor is directed to and thereby applied to the surface of the steel plate in one and three passes. The period of exposure for each pass was about 15 seconds.

[0047] Three samples of the steel plate were evaluated and identified herein as Samples A, B and C.

[0048] The surface of all of the steel Samples had been pre-sandblasted with a fine grain sand.

[0049] Control Sample A was a steel plate without exposure by the plasma torch.

[0050] Experimental Sample B was a steel plate to which one pass of the plasma torch was applied to deposit the precursor in a form of a polymer layer on the steel plate.

[0051] Experimental Sample C was a steel plate to which three individual passes of the plasma torch was applied to deposit the precursor in a form of a polymer layer on the steel plate in a more homogeneous form therefore reducing the occurrence of pin holes.

[0052] To the steel plate Control Sample A and Experimental polymer coated Samples B and C were applied sulfur curable rubber blocks and the rubber blocks cured under conditions of elevated temperature and pressure against the Control and Experimental steel plates (for example at a temperature of about 155°C for about 35 minutes).

[0053] After cooling to room temperature (about 23°C), the energy needed to pull the cured rubber blocks from each

of the steel plate Control Sample A and polymer coated Experimental steel plates B and C was measured and reported in the following Table 1.

Table 1

| Sample | Coating Passes | Average Separation Energy, millijoules (mJ) |
|------------------|----------------|---|
| A (Control) | 0 | 1464 |
| B (Experimental) | 1 | 438 |
| C (Experimental) | 3 | 112 |

[0054] From Table 1 it can be seen that the energy needed to pull the cured rubber off of the steel plate drastically decreases when a plasma coating is applied as compared to the reference, or control, steel plate. Higher number of passes leads to a more homogeneous coating and therefore better release performances.

[0055] This is considered as being significant in a sense of that a thin plasma coated polymerized layer plays the role of a release coating.

[0056] Therefore, it is concluded that the level of performance of aforementioned plasma polymers as a release coating is similar to the one obtained with other more traditional release coatings. In addition, the plasma coating process can be done onsite using minimal amounts of energy, chemicals and handling while providing flexibility, on-demand mold coating as well as the possibility to automate the plasma coating process.

Claims

1. A method of treating a metal surface of a mold for molding and curing rubber products, the method comprising applying an atmospheric plasma comprising an ionized gas containing a polymerizable precursor to a metal molding surface of the mold metal surface to form and bond a polymer layer of plasma polymerized precursor on the mold metal surface.
2. The method of claim 1 wherein the metal mold surface comprises steel or aluminum.
3. The method of claim 1 or 2 wherein the mold is a mold for molding and curing pneumatic rubber tires or rubber products comprising belts, hoses, vehicular track pads or shoes.
4. The method of at least one of the previous claims wherein the polymerizable precursor is an atomized liquid polymerizable precursor contained in a carrier gas for the precursor and introduced into said ionized gas.
5. The method of claim 1 or 4, wherein the carrier gas for said polymerizable precursor and/or the ionized plasma gas comprises at least one of argon, helium, neon, xenon, oxygen, nitrogen, nitrous oxide and carbon dioxide.
6. The method of at least one of the previous claims wherein the precursor is a polymerizable monomer comprising at least one of disiloxanes, disilazanes, perfluoroacrylates, fluorinated silanes, vinylidene fluoride.
7. The method of at least one of the previous claims wherein the precursor is a polymerizable monomer comprising at least one of alkylsiloxane comprising hexamethyldisiloxane; disilazane comprising hexamethyldisilazane; fluorinated silane comprising 1 H, 1 H, 2H-perfluorooctyltriethoxysilane, 1 H, 1 H, 2H-perfluoro-1-alkylene wherein the alkylene group comprises decene, octene, hexene or butene groups, or 1 H, 1 H, 2H-perfluoro-1-alkanol wherein the alkanol group comprises of decanol, octanol, hexanol or butanol groups; vinylidene fluoride and fluorinated compounds comprising at least one of perfluoromethylcyclohexane, perfluorodimethylcyclohexane, hexafluorobenzene, perfluoromethyl vinyl ether, perfluoroethyl vinyl ether, perfluoropropyl vinyl ether, perfluorodimethylcyclobutane, perfluoromethylcyclopentane, perfluoromethyldecalin, tetrafluoroethylene, hexafluoropropylene and perfluorobutyl ethylene.
8. The method of at least one of the previous claims wherein the precursor is a perfluoroacrylate comprising at least one of perfluorooctylacrylate and perfluorooctylmethacrylate.
9. The method of claim 1 or 7 wherein the precursor is combined with a perfluoroacrylate.

10. The method of claim 9 wherein the perfluoroacrylate comprises at least one of perfluorooctylacrylate and perfluorooctylmethacrylate.
11. The method of at least one of the previous claims wherein the precursor is a disiloxane comprising hexamethyldisilazane.
12. The method of at least one of the previous claims wherein the precursor is a fluorinated compound comprising at least one of 1 H, 1 H, 2H-perfluorooctyltriethoxysilane, 1 H, 1 H, 2H-perfluoro-1-alkylene, 1 H, 1 H, 2H-perfluoro-1-alkanol, perfluoromethylcyclohexane, perfluorodimethylcyclohexane, hexafluorobenzene, perfluoromethyl vinyl ether, perfluoroethyl vinyl ether, perfluoropropyl vinyl ether, perfluorodimethylcyclobutane, perfluoromethylcyclopentane, perfluoromethyldecalin, tetrafluoroethylene, hexafluoropropylene and perfluorobutyl ethylene.
13. A mold for molding and curing rubber products prepared by the method of at least one of the previous claims.
14. A method of preparing a rubber product comprising molding and curing a rubber product in the mold of claim 13.
15. A method of preparing a pneumatic rubber tire comprising molding and curing a pneumatic rubber tire in the mold of claim 13.

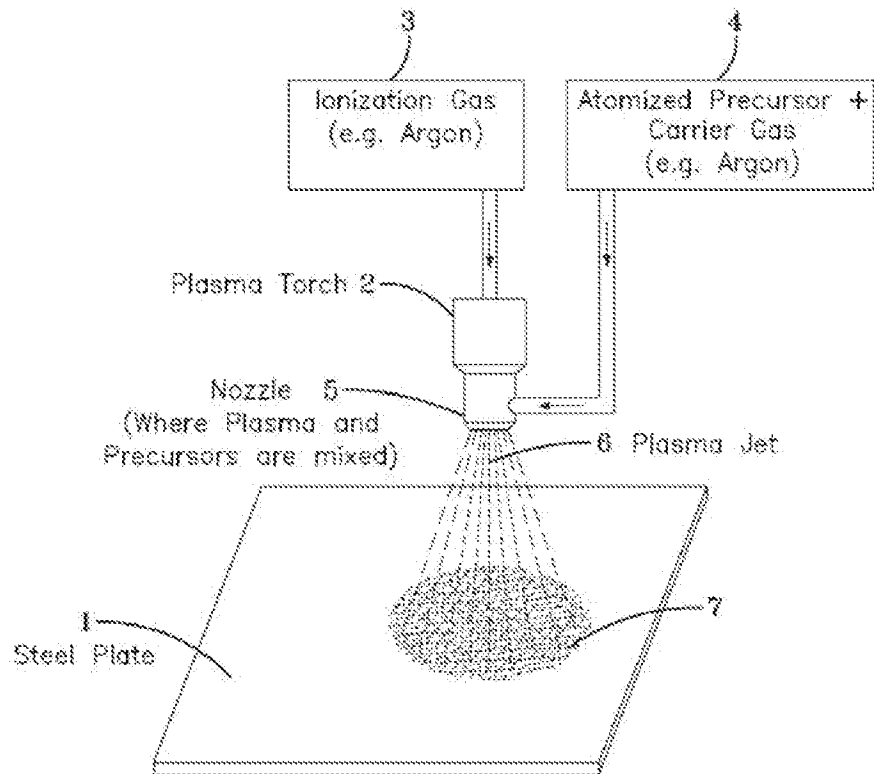


FIG-1



EUROPEAN SEARCH REPORT

 Application Number
EP 13 18 4883

| DOCUMENTS CONSIDERED TO BE RELEVANT | | | |
|---|---|--|---|
| Category | Citation of document with indication, where appropriate, of relevant passages | Relevant to claim | CLASSIFICATION OF THE APPLICATION (IPC) |
| X | US 2007/235890 A1 (PRYCE LEWIS HILTON G [US] ET AL) 11 October 2007 (2007-10-11) | 1-7, 13-15 | INV. B29C33/64 |
| Y | * paragraphs [0001], [0002], [0020] - [0023], [0050], [0051], [0052] - [0059]; figures 2A, 2B * | 3, 6-15 | B29C33/58 B29D30/06 B05D1/00 C23C16/46 |
| X | EP 0 374 080 A1 (GOODYEAR TIRE & RUBBER [US]) 20 June 1990 (1990-06-20) | 1-5, 13-15 | |
| Y | * page 2, line 10 - line 49; claims * | 3, 6-15 | |
| | * page 3, line 29 - page 4, line 7 * | | |
| X | WO 2009/033017 A1 (3M INNOVATIVE PROPERTIES CO [US]; DAVID MOSES M [US]; MAO GUOPING [US]) 12 March 2009 (2009-03-12) | 1, 2, 4-7 | |
| Y | * page 4, line 3 - page 5, line 12; claims; figures * | 3, 8-15 | |
| | * page 11, line 26 - page 13, line 16 * | | |
| | * abstract * | | |
| Y | WO 2011/111669 A1 (SHARP KK [JP]; HAYASHI HIDEKAZU; IMAOKU TAKAO) 15 September 2011 (2011-09-15) | 8-10 | TECHNICAL FIELDS SEARCHED (IPC) |
| | * the whole document * | | B29C B29D B05D C23C |
| Y, P | & US 2012/308678 A1 (HAYASHI HIDEKAZU [JP] ET AL) 6 December 2012 (2012-12-06) | 8-10 | |
| | * paragraphs [0086], [0105] * | | |
| Y, P | & US 2012/308678 A1 (HAYASHI HIDEKAZU [JP] ET AL) 6 December 2012 (2012-12-06) | 8-10 | |
| | * paragraphs [0086], [0105] * | | |
| Y | WO 2006/079359 A1 (PIRELLI [IT]; NANNI MARCO [IT]; LOPREVITE MASSIMO [IT]; BONGIO) 3 August 2006 (2006-08-03) | 12 | |
| | * paragraphs [0009], [0013]; claim 26 * | | |
| | -/- | | |
| The present search report has been drawn up for all claims. | | | |
| Place of search | | Date of completion of the search | Searcher |
| Munich | | 15 November 2013 | Kopp, Christian |
| CATEGORY OF CITED DOCUMENTS | | | |
| X : particularly relevant if taken alone Y : particularly relevant if combined with another document of the same category A : technological background G : non-written disclosure P : intermediate document | | T : theory or principle underlying the invention E : earlier patent document, but published on, or after the filing date O : document cited in the application L : document cited for other reasons S : member of the same patent family, corresponding document | |

EP 2 711 153 A1 (2013.11.15)



EUROPEAN SEARCH REPORT

 Application Number
 EP 13 18 4883

| DOCUMENTS CONSIDERED TO BE RELEVANT | | | |
|--|--|--|---|
| Category | Citation of document with indication, where appropriate, of relevant passages | Relevant to claim | CLASSIFICATION OF THE APPLICATION (IPC) |
| Y | EP 0 252 870 A2 (BATTELLE MEMORIAL INSTITUTE [US]) 13 January 1988 (1988-01-13) * page 3, line 5 - line 15 * | 11 | |
| A | EP 2 326 153 A2 (GOODYEAR TIRE & RUBBER [US]) 25 May 2011 (2011-05-25) * figures * | 1-15 | |
| | | | TECHNICAL FIELD SEARCHED (IPC) |
| | | | |
| The present search report has been drawn up for all claims | | | |
| Place of search Munich | | Date of completion of the search 15 November 2013 | Examiner Kopp, Christian |
| CATEGORY OF CITED DOCUMENTS X : particularly relevant if taken alone Y : particularly relevant if combined with another document of the same category A : technological background C : non-written disclosure P : intermediate document T : theory or principle underlying the invention E : earlier patent document, but published on, or after the filing date O : document cited in the application L : document cited for other reasons A : member of the same patent family, corresponding document | | | |

 2
 EP 2 711 153 A1

ANNEX TO THE EUROPEAN SEARCH REPORT
ON EUROPEAN PATENT APPLICATION NO.

EP 13 18 4883

This annex lists the patent family members relating to the patent documents cited in the above-mentioned European search report. The members are as contained in the European Patent Office EDP file on
The European Patent Office is in no way liable for these particulars which are merely given for the purpose of information.

15-11-2013

| Patent document cited in search report | Publication date | Patent family member(s) | Publication date |
|---|---------------------|----------------------------|---------------------|
| US 2007235890 A1 | 11-10-2007 | EP 2012991 A2 | 14-01-2009 |
| | | EP 2369038 A2 | 28-09-2011 |
| | | US 2007235890 A1 | 11-10-2007 |
| | | WO 2007120507 A2 | 25-10-2007 |
| EP 0374080 A1 | 20-06-1990 | DE 68905157 D1 | 08-04-1993 |
| | | DE 68905157 T2 | 23-09-1993 |
| | | EP 0374080 A1 | 20-06-1990 |
| | | JP 2701946 B2 | 21-01-1998 |
| | | JP H02214619 A | 27-08-1990 |
| WO 2009033017 A1 | 12-03-2009 | AT 534500 T | 15-12-2011 |
| | | CN 101795838 A | 04-08-2010 |
| | | CN 101795840 A | 04-08-2010 |
| | | EP 2197645 A1 | 23-06-2010 |
| | | EP 2197646 A1 | 23-06-2010 |
| | | JP 5161310 B2 | 13-03-2013 |
| | | JP 2010537867 A | 09-12-2010 |
| | | JP 2010537868 A | 09-12-2010 |
| | | US 2010239783 A1 | 23-09-2010 |
| | | US 2010308509 A1 | 09-12-2010 |
| | | WO 2009033017 A1 | 12-03-2009 |
| | | WO 2009033029 A1 | 12-03-2009 |
| WO 2011111669 A1 | 15-09-2011 | CN 102791453 A | 21-11-2012 |
| | | EP 2546042 A1 | 16-01-2013 |
| | | JP 4976593 B2 | 18-07-2012 |
| | | US 2012308678 A1 | 06-12-2012 |
| | | WO 2011111669 A1 | 15-09-2011 |
| WO 2006079359 A1 | 03-08-2006 | BR P10519967 A2 | 18-08-2009 |
| | | CN 101111355 A | 23-01-2008 |
| | | EP 1841576 A1 | 10-10-2007 |
| | | US 2009212467 A1 | 27-08-2009 |
| | | WO 2006079359 A1 | 03-08-2006 |
| EP 0252870 A2 | 13-01-1988 | AU 590829 B2 | 05-07-1990 |
| | | AU 7536787 A | 14-01-1988 |
| | | DE 3770705 D1 | 18-07-1991 |
| | | EP 0252870 A2 | 13-01-1988 |
| | | GR 3002125 T3 | 30-12-1992 |
| | | JP 56333429 A | 13-02-1988 |
| | | US 4778721 A | 18-10-1988 |
| EP 2326153 A2 | 25-05-2011 | EP 2326153 A2 | 25-05-2011 |
| | | US 2011121107 A1 | 26-05-2011 |

EP 13 18 4883

For more details about this annex : see Official Journal of the European Patent Office, No. 12/82

**ANNEX TO THE EUROPEAN SEARCH REPORT
ON EUROPEAN PATENT APPLICATION NO.**

EP 13 18 4883

This annex lists the patent family members relating to the patent documents cited in the above-mentioned European search report. The members are as contained in the European Patent Office EPO file on. The European Patent Office is in no way liable for these particulars which are merely given for the purpose of information.

15-11-2013

| Patent document cited in search report | Publication date | Patent family member(s) | Publication date |
|---|---------------------|----------------------------|---------------------|
| | | | |

EPO Form P422

For more details about this annex : see Official Journal of the European Patent Office, No. 12/82

(12) UK Patent Application (19) GB (11) 2 434 368 (13) A

(43) Date of A Publication 25.07.2007

(21) Application No: 0601113.4

(22) Date of Filing: 29.01.2006

(71) Applicant(s):
P2i Limited
(Incorporated in the United Kingdom)
Unit 14, Central 127, Milton Park,
ABINGDON, Oxfordshire, OX14 4SA,
United Kingdom

(72) Inventor(s):
Stephen Coulson

(74) Agent and/or Address for Service:
Greaves Brewster
Indigo House, Cheddar Business Park,
Wedmore Road, CHEDDAR, Somerset,
BS27 3EB, United Kingdom

(51) INT CL:
C08F 2/52 (2006.01) B29C 59/14 (2006.01)
C08J 7/16 (2006.01) D06M 10/02 (2006.01)
D06M 14/18 (2006.01)

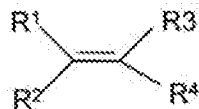
(52) UK CL (Edition X):
C3P PKN
C3W W227

(56) Documents Cited:
GB 2341864 A WO 2005/099961 A1
WO 2003/063938 A1

(58) Field of Search:
Other: ONLINE : CAS ONLINE, EPODOC, WPI.

(54) Abstract Title: **Polymeric coating of a laboratory consumable**

(57) A laboratory consumable having a polymeric coating, formed by exposing said consumable to pulsed plasma comprising a compound of formula (I)



where R^1 , R^2 and R^3 are independently selected from hydrogen, alkyl, haloalkyl or aryl optionally substituted by halo; and R^4 is a group $X-R^5$ where R^5 is an alkyl or haloalkyl group and X is a bond; a group of formula $-C(O)O(CH_2)_nY-$ where n is an integer of from 1 to 10 and Y is a bond or a sulphonamide group; or a group $-(O)_pR^6(O)_q(CH_2)_t-$ where R^6 is aryl optionally substituted by halo, p is 0 or 1, q is 0 or 1 and t is 0 or an integer of from 1 to 10, provided that where q is 1, t is other than 0, for a sufficient period of time to allow a polymeric layer to form on the surface of the laboratory consumable.

Consumables of this type show significantly reduced liquid sample retention, maximum sample recovery and ultra non-binding properties, as well as oil and water-repellency.

GB 2 434 368 A

Novel Products

The present invention relates to novel products in the form of laboratory consumables, which are treated to prevent retention
5 of reagents on the surface thereof, as well as to processes for their production.

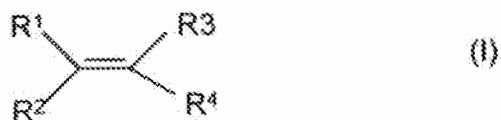
There is a need for laboratory equipment and in particular laboratory consumables, to be as non-binding to reagents or
10 samples as possible so as to avoid retention of these on the equipment. In particular, the biochemical and chemical reactions conducted for example for research or analysis, and in particular medical or forensic diagnostics, often use very small volumes of samples or reagents. Retention of these in
15 any significant proportion on the surface of laboratory equipment, which comes into direct contact with these, used to handle the samples or reagents used in these types of reaction can affect the results significantly. Furthermore, since many of the reagents are extremely costly, there are economic
20 implications as a result of reagent loss in this way.

Various approaches have been made to address this problem. These include processes aimed at creating ultra smooth surfaces or solid materials such as those used to produce pipette tips
25 in order to reduce retention. Other approaches have included processes aimed at adding additives to migrate through the polymer dip coating the tips in a liquid repellent media.

Plasma deposition techniques have been quite widely used for
30 the deposition of polymeric coatings onto a range of surfaces, and in particular onto fabric surfaces. This technique is recognised as being a clean, dry technique that generates little waste compared to conventional wet chemical methods. Using this method, plasmas are generated from organic
35 molecules, which are subjected to an electrical field. When this is done in the presence of a substrate, the radicals of

the compound in the plasma polymerise on the substrate. Conventional polymer synthesis tends to produce structures containing repeat units that bear a strong resemblance to the monomer species, whereas a polymer network generated using a plasma can be extremely complex. The properties of the resultant coating can depend upon the nature of the substrate as well as the nature of the monomer used and conditions under which it is deposited.

- 10 The applicants have found that by utilising a specific type of monomer under particular deposition conditions, laboratory consumables having ultra-non-binding nano-coatings thereon can be produced.
- 15 According to the present invention there is provided a laboratory consumable having a polymeric coating, formed by exposing said consumable to pulsed plasma comprising a compound of formula (I)



- 20 where R^1 , R^2 and R^3 are independently selected from hydrogen, alkyl, haloalkyl or aryl optionally substituted by halo; and R^4 is a group $\text{X}-\text{R}^5$ where R^5 is an alkyl or haloalkyl group and X is a bond; a group of formula $-\text{C}(\text{O})\text{O}(\text{CH}_2)_n\text{Y}-$ where n is an integer of from 1 to 10 and Y is a bond or a sulphonamide group; or a group $-(\text{O})_p\text{R}^6(\text{O})_q(\text{CH}_2)_t-$ where R^6 is aryl optionally substituted by halo, p is 0 or 1, q is 0 or 1 and t is 0 or an integer of from 1 to 10, provided that where q is 1, t is other
- 25 than 0, for a sufficient period of time to allow a polymeric layer to form on the surface of the laboratory consumable.
- 30

As used herein, the expression "in a gaseous state" refers to gases or vapours, either alone or in mixture, as well as aerosols.

- 5 As used herein, the term "laboratory consumable" refers to any piece of equipment, which comes into contact with liquid reagents, polar and apolar solvents or samples in the course of normal laboratory use, including use for example in medical environments such as hospitals and research laboratories.
- 10 Generally these will be single use disposable devices, although some may be used repeatedly, for example after washing or cleaning of some type.

- Particular examples of laboratory consumables include pipette
- 15 tips, filtration membranes, microplates (including 96 well plates), immunoassay products (such as lateral flow devices), centrifuge tubes (including microcentrifuge tubes), microtubes, specimen tubes, test tubes, blood collection tubes, flat based tubes, aseptically produced containers, general labware,
- 20 burettes, curvettes, needles, hypodermic syringes, sample vials/bottles, screw cap containers, or weighing bottles. These can be made from a wide variety of materials such as, thermosetting resins, thermoplastic resins polyolefins, acetals, polyamidic resins, acrylic resins (PMMA), hydrocarbons
- 25 or fluorocarbons such as polyethylene (PE), polypropylene (PP), polymethylpentene (PMP or TPX®), polystyrene (PS), polyvinyl chloride (PVC), polyoxymethylene (POM), nylon (PA6), polycarbonates (PC), polytetrafluoroethylene (PTFE), tetrafluoroethylene-perfluoropropylene (FEP), perfluoroalkoxy
- 30 (PFA), polyvinylidene fluoride (PVDF), ethylene-tetrafluoroethylene (ETFE), ethylene-chlorotrifluoroethylene (E-CTFE) and glass.

In a particular embodiment, the laboratory consumable is a pipette tip. By utilising the method defined above, highly advantageous pipette tips have been produced. In particular, the main features and benefits of using this approach are that
5 both the inside and the outside of the tip are enhanced, allowing much less binding, reducing loss of liquid and maximizing sample recovery.

Laboratory consumables treated in this way provide are water
10 and oil repellent, and also resistant to clogging. They may have useful "shake dry" properties also, reducing risk of contamination after washing.

Furthermore, the enhancement material is molecularly bound to
15 the surface and so there are no leachables; the modification becomes part of the device. The coated tip's surface displays much lower surface energies than polypropylene and so will bind less material. As a result, liquid retention by the pipette tip in use is virtually eliminated (as can be illustrated
20 photographically) meaning that the results obtained using these tips are more accurate and reliable, and reagent costs can be reduced, in some cases by more than 10%.

Similar advantages accrue in relation to other laboratory
25 consumables which are used to handle in particular small volumes of sample or reagent, such as microtitre plates, microplates (including 96 well plates), centrifuge tubes (including microcentrifuge tubes) and microtubes.

30 The fact that needles and syringes can be made so that virtually no liquid is retained therein is a significant advantage since it means that accurate dosing of materials, in particular medicines and the like, can be ensured.

35 Another type of laboratory consumable to benefit significantly from the modification of the invention are membranes such as

those used as filtration membranes or those used in immunoassay devices. These membranes are made of porous material. It is necessary for liquid to pass through the pores of the material. In the case of filtration membranes, particles above a particular size are retained on the surface. In the case of membranes used immunoassays, liquid and small particles such as gold or latex particles are required also to pass through the pores of the membrane. Although these membranes are able to fulfil these functions, they are generally made of a liquid absorbent material such as cellulose. This means that significant quantities of liquid will be retained within the membrane, as they will become saturated in use. This can be problematic, in particular where only small volumes of liquid are being used.

Membranes treated in accordance with the invention retain their porosity, as the coating layer deposited thereon is only molecules thick. Therefore, the liquid or even small particles can continue to pass through them, in particular when a positive pressure is applied to the liquid, or a negative pressure is applied to the other side of the membrane to draw the liquid through. However, larger particles will not. However, substantially all the liquid applied will pass through the membrane, since it will not adhere or be absorbed by the material of the membrane.

Precise conditions under which the plasma polymerization takes place in an effective manner will vary depending upon factors such as the nature of the polymer, the laboratory consumable etc. and will be determined using routine methods and/or the techniques.

Suitable plasmas for use in the method of the invention include non-equilibrium plasmas such as those generated by radiofrequencies (RF), microwaves or direct current (DC). They may operate at atmospheric or sub-atmospheric pressures as are

known in the art. In particular however, they are generated by radiofrequencies (Rf).

5 Various forms of equipment may be used to generate gaseous plasmas. Generally these comprise containers or plasma chambers in which plasmas may be generated. Particular examples of such equipment are described for instance in WO2005/089961 and WO02/28548, but many other conventional plasma generating apparatus are available.

10

In general, the item to be treated is placed within a plasma chamber together with the material to be deposited in gaseous state, a glow discharge is ignited within the chamber and a suitable voltage is applied, which may be pulsed.

15

The gas used within the plasma may comprise a vapour of the monomeric compound alone, but it may be combined with a carrier gas, in particular, an inert gas such as helium or argon. In particular helium is a preferred carrier gas as this can
20 minimises fragmentation of the monomer.

25

When used as a mixture, the relative amounts of the monomer vapour to carrier gas is suitably determined in accordance with procedures which are conventional in the art. The amount of monomer added will depend to some extent on the nature of the particular monomer being used, the nature of the laboratory disposable being treated, the size of the plasma chamber etc. Generally, in the case of conventional chambers, monomer is delivered in an amount of from 50-250mg/min, for example at a
30 rate of from 100-150mg/min. Carrier gas such as helium is suitably administered at a constant rate for example at a rate of from 5-90, for example from 15-30sccm. In some instances, the ratio of monomer to carrier gas will be in the range of from 100:1 to 1:100, for instance in the range of from 10:1 to
35 1:100, and in particular about 1:1 to 1:10. The precise ratio

selected will be so as to ensure that the flow rate required by the process is achieved.

In some cases, a preliminary continuous power plasma may be struck for example for from 2-10 minutes for instance for about 4 minutes, within the chamber. This may act as a surface pre-treatment step, ensuring that the monomer attaches itself readily to the surface, so that as polymerisation occurs, the coating "grows" on the surface. The pre-treatment step may be conducted before monomer is introduced into the chamber, in the presence of only the inert gas.

The plasma is then suitably switched to a pulsed plasma to allow polymerisation to proceed, at least when the monomer is present.

In all cases, a glow discharge is suitably ignited by applying a high frequency voltage, for example at 13.56MHz. This is suitably applied using electrodes, which may be internal or external to the chamber, but in the case of the larger chambers are internal.

Suitably the gas, vapour or gas mixture is supplied at a rate of at least 1 standard cubic centimetre per minute (sccm) and preferably in the range of from 1 to 100sccm.

In the case of the monomer vapour, this is suitably supplied at a rate of from 80-300mg/minute, for example at about 120mg per minute depending upon the nature of the monomer, whilst the pulsed voltage is applied.

Gases or vapours may be drawn or pumped into the plasma region. In particular, where a plasma chamber is used, gases or vapours may be drawn into the chamber as a result of a reduction in the pressure within the chamber, caused by use of an evacuating

pump, or they may be pumped or injected into the chamber as is common in liquid handling.

5 Polymerisation is suitably effected using vapours of compounds of formula (I), which are maintained at pressures of from 0.1 to 200mtorr, suitably at about 80-100mtorr.

The applied fields are suitably of power of from 40 to 500W, suitably at about 100W peak power, applied as a pulsed field.
10 The pulses are applied in a sequence which yields very low average powers, for example in a sequence in which the ratio of the time on : time off is in the range of from 1:500 to 1:1500. Particular examples of such sequence are sequences where power is on for 20-50 μ s, for example about 30 μ s, and off for from
15 1000 μ s to 30000 μ s, in particular about 20000 μ s. Typical average powers obtained in this way are 0.01W.

The fields are suitably applied from 30 seconds to 90 minutes, preferably from 5 to 60 minutes, depending upon the nature of
20 the compound of formula (I) and the laboratory consumable etc.

Suitably a plasma chamber used is of sufficient volume to accommodate multiple laboratory consumables, for example up to 500,000 pipette tips at the same time.

25 A particularly suitable apparatus and method for producing laboratory consumables in accordance with the invention is described in WO2005/089961, the content of which is hereby incorporated by reference.

30 In particular, when using high volume chambers of this type, the plasma is created with a voltage as a pulsed field, at an average power of from 0.001 to 500w/m³, for example at from 0.001 to 100w/m³ and suitably at from 0.005 to 0.5w/m³.

35

These conditions are particularly suitable for depositing good quality uniform coatings, in large chambers, for example in chambers where the plasma zone has a volume of greater than 500cm³, for instance 0.5m³ or more, such as from 0.5m³-10m³ and suitably at about 1m³. The layers formed in this way have good mechanical strength.

The dimensions of the chamber will be selected so as to accommodate the particular laboratory consumable being treated. For instance, generally cuboid chambers may be suitable for a wide range of applications, but if necessary, elongate or rectangular chambers may be constructed or indeed cylindrical, or of any other suitable shape.

The chamber may be a sealable container, to allow for batch processes, or it may comprise inlets and outlets for laboratory consumables, to allow it to be utilised in a continuous process. In particular in the latter case, the pressure conditions necessary for creating a plasma discharge within the chamber are maintained using high volume pumps, as is conventional for example in a device with a "whistling leak". However it will also be possible to process certain items at atmospheric pressure, or close to, negating the need for "whistling leaks"

The monomers used are selected from monomers of formula (I) as defined above. Suitable haloalkyl groups for R¹, R², R³ and R⁵ are fluoroalkyl groups. The alkyl chains may be straight or branched and may include cyclic moieties.

For R⁵, the alkyl chains suitably comprise 2 or more carbon atoms, suitably from 2-20 carbon atoms and preferably from 6 to 12 carbon atoms.

For R¹, R⁴ and R³, alkyl chains are generally preferred to have from 1 to 6 carbon atoms.

Preferably R^5 is a haloalkyl, and more preferably a perhaloalkyl group, particularly a perfluoroalkyl group of formula C_mF_{2m+1} where m is an integer of 1 or more, suitably from 1-20, and preferably from 4-12 such as 4, 6 or 8.

Suitable alkyl groups for R^1 , R^2 and R^3 have from 1 to 6 carbon atoms.

10 In one embodiment, at least one of R^1 , R^2 and R^3 is hydrogen. In a particular embodiment R^1 , R^2 , R^3 are all hydrogen. In yet a further embodiment however R^3 is an alkyl group such as methyl or propyl.

15 Where X is a group $-C(O)O(CH_2)_nY-$, n is an integer which provides a suitable spacer group. In particular, n is from 1 to 5, preferably about 2.

Suitable sulphonamide groups for Y include those of formula $-N(R^7)SO_2$ where R^7 is hydrogen or alkyl such as C_{1-4} alkyl, in particular methyl or ethyl.

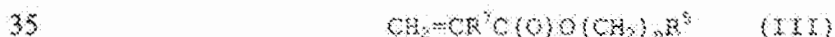
In one embodiment, the compound of formula (I) is a compound of formula (II)



where R^5 is as defined above in relation to formula (I).

30 In compounds of formula (II), X in formula (I) is a bond.

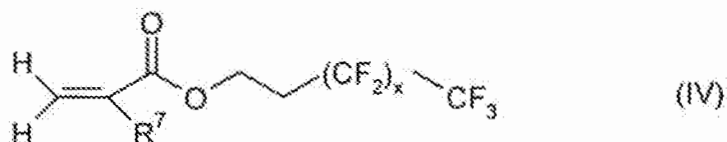
However in a preferred embodiment, the compound of formula (I) is an acrylate of formula (III)



II

where n and R¹ as defined above in relation to formula (I) and R² is hydrogen, C₁₋₁₀ alkyl, or C₁₋₁₀ haloalkyl. In particular R² is hydrogen or C₁₋₄ alkyl such as methyl. A particular example of a compound of formula (III) is a compound of formula (IV)

5

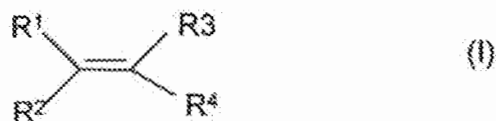


where R⁷ is as defined above, and in particular is hydrogen and x is an integer of from 1 to 9, for instance from 4 to 9, and preferably 7. In that case, the compound of formula (IV) is 1H,1H,2H,2H-heptafluorodecylacrylate.

10

In a further aspect, the invention provides a method for reducing liquid sample retention by a laboratory consumable, said method comprising exposing said consumable to a pulsed plasma comprising a compound of formula (I)

15



20

where R¹, R² and R³ are independently selected from hydrogen, alkyl, haloalkyl or aryl optionally substituted by halo; and R⁴ is a group X-R⁵ where R⁵ is an alkyl or haloalkyl group and X is a bond; a group of formula -C(O)O(CH₂)_nY- where n is an integer of from 1 to 10 and Y is a bond or a sulphonamide group; or a group -(O)_pR⁶(O)_q(CH₂)_t- where R⁶ is aryl optionally substituted by halo, p is 0 or 1, q is 0 or 1 and t is 0 or an integer of from 1 to 10, provided that where q is 1, t is other than 0,

25

in a gaseous state for a sufficient period of time to allow a polymeric layer to form on the surface of the laboratory consumable.

- 5 Suitably, the laboratory consumable is placed in a plasma deposition chamber, a glow discharge is ignited within said chamber, and a voltage applied as a pulsed field.

Suitable monomers and reaction conditions for use in this
10 method are as described above.

The invention will now be particularly described by way of example.

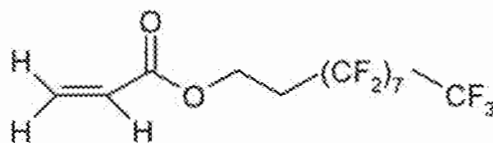
15 Example 1

Pipette Tips

A set of polypropylene pipette tips were placed into a plasma chamber with a processing volume of ~ 300 litres. The chamber was connected to supplies of the required gases or vapours, via
20 a mass flow controller and/or liquid mass flow meter and a mixing injector as appropriate.

The chamber was evacuated to between 3 - 10 mtorr base pressure
25 before allowing helium into the chamber at 20 sccm until a pressure of 80 mtorr was reached. A continuous power plasma was then struck for 4 minutes using RF at 13.56 MHz at 300 W.

After this period, 1H,1H,2H,2H-heptafluorodecylacrylate (CAS
30 # 27905-45-9) of formula



was brought into the chamber at a rate of 120 milli grams per minute and the plasma switched to a pulsed plasma at 30 micro seconds on-time and 20 milli seconds off-time at a peak power of 100 W for 40 minutes. On completion of the 40 minutes the
5 plasma power was turned off along with the processing gases and vapours and the chamber evacuated back down to base pressure. The chamber was then vented to atmospheric pressure and the pipette tips removed.

10 It was found that the pipette tips was covered with an ultra-non-binding, pin-hole free modification that had a surface energy much lower than a Teflon® (PTFE) coated surface. The coating was distributed both inside and outside the pipette tip and was molecularly attached.

15 The performance of the pipette tips was significantly enhanced, as liquid retention was virtually eliminated and maximum sample recovery was possible.

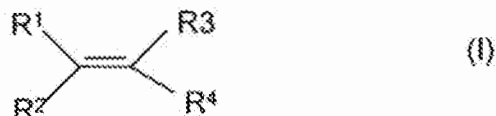
20 Example 2

Filter paper

By using the same method as outlined in Example 1, cellulose filter papers (Whatman number 1) were placed into the chamber and processed under the same conditions. It was found that
25 despite the fact the plasma enhanced paper appears the same, had the same handle and could not be differentiated either by appearance or feel; repelled water to an extent that the water formed beads on the surface which roll around effortlessly. Despite this, the droplets could be forced through the
30 microporous paper if sufficient force was applied as the process does not block up the pores. The paper remained un-wet but can still carry out its filtering capabilities.

Claims

1. A laboratory consumable having a polymeric coating,
formed by exposing said consumable to pulsed plasma comprising
5 a compound of formula (I)



- where R^1 , R^2 and R^3 are independently selected from hydrogen,
10 alkyl, haloalkyl or aryl optionally substituted by halo; and R^4
is a group $X-R^5$ where R^5 is an alkyl or haloalkyl group and X
is a bond; a group of formula $-C(O)O(CH_2)_nY-$ where n is an
integer of from 1 to 10 and Y is a bond or a sulphonamide
group; or a group $-(O)_pR^6(O)_q(CH_2)_t-$ where R^6 is aryl optionally
15 substituted by halo, p is 0 or 1, q is 0 or 1 and t is 0 or an
integer of from 1 to 10, provided that where q is 1, t is other
than 0, for a sufficient period of time to allow a polymeric
layer to form on the surface of the laboratory consumable.

- 20 2. A laboratory consumable according to claim 1 which is
selected from pipette tips, filtration membranes, microplates
immunoassay products, centrifuge tubes, microtubes, specimen
tubes, test tubes, blood collection tubes, flat based tubes,
aseptically produced containers, general labware, burettes,
25 curvettes, needles, hypodermic syringes, sample vials/bottles,
screw cap containers, or weighing bottles.

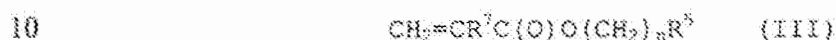
3. A laboratory consumable according to claim 2 which is a
pipette tip.

- 30 4. A laboratory consumable according to any one of the
preceding claims wherein the laboratory consumable is exposed
to the pulsed plasma within a plasma deposition chamber.

5. A laboratory consumable according to any one of the preceding claims wherein the compound of formula (I) is a compound of formula (II)



where R^5 is as defined in claim 1, or a compound of formula (III)

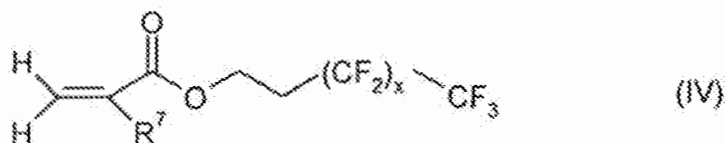


where n and R^5 as defined in claim 1 and R^7 is hydrogen, C_{1-10} alkyl, or C_{1-10} haloalkyl.

15 6. A laboratory consumable according to claim 5 wherein the compound of formula (I) is a compound of formula (III).

7. A laboratory consumable according to claim 6 wherein the compound of formula (III) is a compound of formula (IV)

20



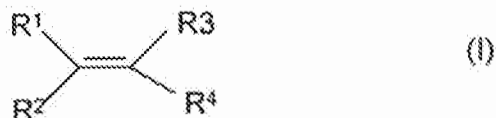
where R^7 is as defined in claim 4, and x is an integer of from 1 to 9.

25

8. A laboratory consumable according to claim 7 wherein the compound of formula (IV) is is 1H,1H,2H,2H-heptadecafluorodecylacrylate.

30

9. A method for reducing liquid sample retention by a laboratory consumable, said method comprising exposing said consumable to a pulsed plasma comprising a compound of formula (I)

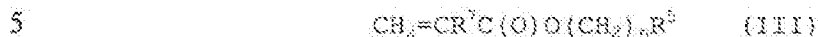


- 5 where R^1 , R^2 and R^3 are independently selected from hydrogen, alkyl, haloalkyl or aryl optionally substituted by halo; and R^4 is a group $X-R^5$ where R^5 is an alkyl or haloalkyl group and X is a bond; a group of formula $-C(O)O(CH_2)_nY-$ where n is an integer of from 1 to 10 and Y is a bond or a sulphonamide
- 10 group; or a group $-(O)_pR^6(O)_q(CH_2)_t-$ where R^6 is aryl optionally substituted by halo, p is 0 or 1, q is 0 or 1 and t is 0 or an integer of from 1 to 10, provided that where q is 1, t is other than 0,
- in a gaseous state for a sufficient period of time to allow a
- 15 polymeric layer to form on the surface of the laboratory consumable.
10. A method according to claim 9 wherein the laboratory consumable is placed in a plasma deposition chamber, a glow
- 20 discharge is ignited within said chamber, and a voltage applied as a pulsed field.
11. A method according to claim 9 or claim 10 wherein applied voltage is at a power of from 40 to 500W.
- 25 12. A method according to any one of claims 9 to 11 wherein the voltage is pulsed in a sequence in which the ratio of the time on : time off is in the range of from 1:500 to 1:1500.
- 30 13. A method according to claim 12 wherein the voltage is pulsed in a sequence where power is on for 20-50 μ s, and off for from 1000 μ s to 30000 μ s.

14. A method according to any one of claims 9 to 13 wherein the voltage is applied as a pulsed field at for a period of from 30 seconds to 90 minutes.
- 5 15. A method according to claim 14 wherein the voltage is applied as a pulsed field for from 5 to 60 minutes.
16. A method according to any one of claims 9 to 15, wherein in a preliminary step, a continuous power plasma is applied to
10 the laboratory consumable.
17. A method according to claim 16 wherein the preliminary step is conducted in the presence of an inert gas.
- 15 18. A method according to any one of claims 9 to 17 wherein the compound of formula (I) in gaseous form is fed into the plasma at a rate of from 80-300 mg/minute, whilst the pulsed voltage is applied.
- 20 19. A method according to any one of claims 9 to 18 wherein the plasma is created with a voltage at an average power of from 0.001 to 500w/m³.
20. A method according to claim 19 wherein the plasma is
25 created with a voltage at an average power of from 0.001 to 100w/m³.
21. A method according to claim 20 wherein the plasma is created with a voltage at an average power of from 0.005 to
30 0.5w/m³.
22. A method according to any one of claims 9 to 21 wherein wherein the compound of formula (I) is a compound of formula (II)
- 35



where R^5 is as defined in claim 1, or a compound of formula (III)

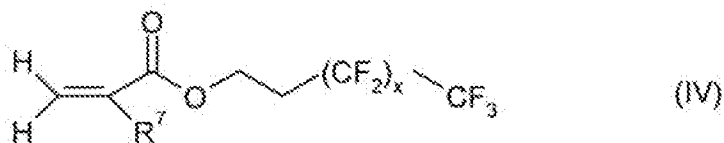


where n and R^5 as defined in claim 1 and R^7 is hydrogen, C_{1-10} alkyl, or C_{1-10} haloalkyl.

10 24. A method according to claim 23 wherein the compound of formula (I) is a compound of formula (III).

25. A method according to claim 24 wherein the compound of formula (III) is a compound of formula (IV)

15



where R^7 is as defined in claim 4, and x is an integer of from 1 to 9.

20

26. A method according to claim 25 wherein the compound of formula (IV) is 1H,1H,2H,2H-heptafluorodecyl acrylate.

27. A laboratory consumable substantially as hereinbefore
25 described with reference to the examples.

28. A method for reducing liquid sample retention by a laboratory consumable, substantially as hereinbefore described with reference to the Examples.

30



19

Application No: GB0601113.4

Examiner: Dr Albert Mthupha

Claims searched: 1-28

Date of search: 21 April 2006

Patents Act 1977: Search Report under Section 17**Documents considered to be relevant:**

| Category | Relevant to claims | Identity of document and passage or figure of particular relevance |
|----------|--------------------------|---|
| X | 1-2, 4-11, 14-17, 22-26. | GB2341864 A (SECRETARY OF STATE FOR DEFENCE), see whole document, note particularly page 1 lines 3-12. |
| X | 1-2, 4-11, 14-17, 22-26. | WO2005/089961 A1 (SECRETARY OF STATE OF DEFENCE), see whole document, note particularly page 1 lines 3-11. |
| X | 1-2, 4-11, 14-17, 22-26. | WO03/063938 A1 (ASTRAZENECA), see whole document, note particularly page 1 lines 7-11, page 2 lines 30-33, page 8 lines 10-18, Claims 1-6. |

Categories:

| | | | |
|---|---|---|---|
| X | Document indicating lack of novelty or inventive step | A | Document indicating technological background and/or state of the art. |
| Y | Document indicating lack of inventive step if combined with one or more other documents of same category. | P | Document published on or after the declared priority date but before the filing date of this invention. |
| & | Member of the same patent family | E | Patent document published on or after, but with priority date earlier than, the filing date of this application |

Field of Search:Search of GB, EP, WO & US patent documents classified in the following areas of the UKC^X:

Worldwide search of patent documents classified in the following areas of the IPC

The following online and other databases have been used in the preparation of this search report

ONLINE : CAS ONLINE, EPODOC, WPI.

(21) Application No: 1212808.8

(22) Date of Filing: 19.07.2012

(30) Priority Data:
(31) 1112516 (32) 21.07.2011 (33) GB

(71) Applicant(s):
P2i Ltd
(Incorporated in the United Kingdom)
127 (9-12 North) Milton Park, ABINGDON,
Oxfordshire, OX14 4SA, United Kingdom

(72) Inventor(s):
Stephen Coulson
Paul De Blaquiere
Delwyn Evans
Warren Lee

(74) Agent and/or Address for Service:
Greaves Brewster LLP
Copa House, Station Road, CHEDDAR, Somerset,
BS27 3AH, United Kingdom

(51) INT CL:
B05D 7/24 (2006.01) C08F 2/52 (2006.01)
C08J 7/04 (2006.01) D06M 10/10 (2006.01)
B05D 5/00 (2006.01)

(58) Documents Cited:
GB 2434379 A GB 1495480 A
EP 1087034 A1 EP 1074588 A2
WO 2008/010753 A2 WO 2007/015779 A2
WO 2006/020130 A1

(58) Field of Search:
INT CL B05D, C08F, C08J, D06M
Other: EPODOC; WPI

(54) Title of the Invention: Surface coatings
Abstract Title: Formation of a liquid repellent coating, using plasma polymerisation

(57) The plasma deposition process involves exposing a substrate to a monomer in a plasma polymerisation process. This allows a polymeric layer to form on the surface of the substrate. The process is subjected to at least one cycle of varying pressure. The preferred monomer is a mono-olefin, especially a compound of formula (I) or (II).

$\text{CH}_2=\text{CHR}^1$ (I)

$\text{CH}_2=\text{CR}^2\text{C}(\text{O})\text{O}(\text{CH}_2)_n\text{R}^1$ (II)

R¹ = alkyl or haloalkyl;

R² = H, 1-10C alkyl or 1-10C haloalkyl;

n = 1-10.

An especially preferred monomer is 1H,1H,2H,2H-heptadecafluorodecylacrylate. Preferably, pulsed plasma is used. The substrate is preferably fabric, metal, glass, ceramic, paper or polymer, especially an item of clothing.

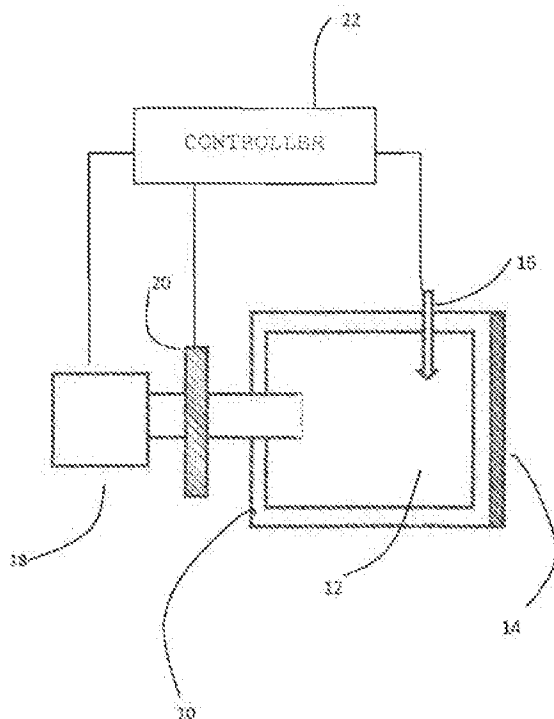


Fig. 1

GB 2493264 A

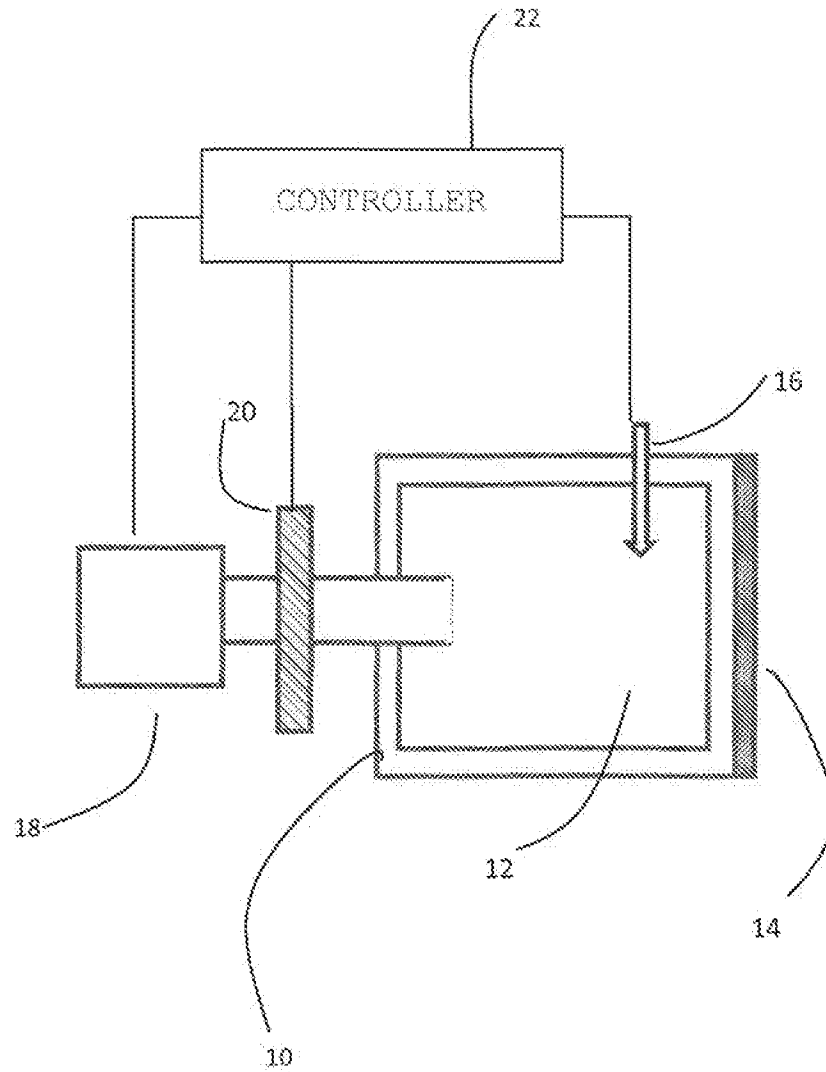


Fig 1

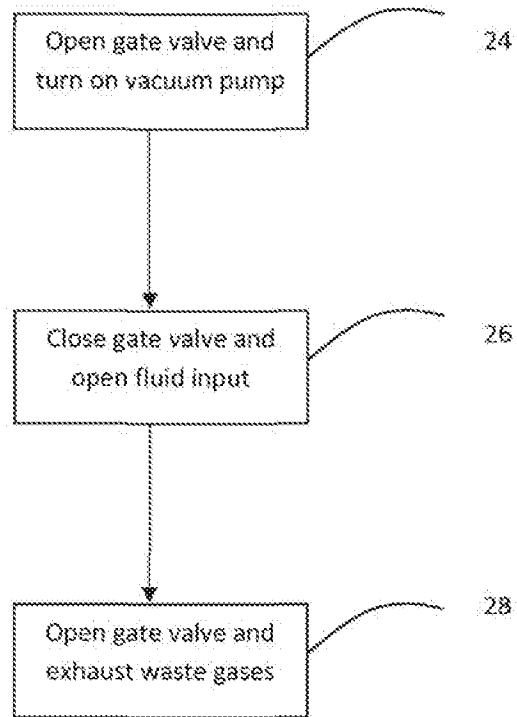


Fig 2

Surface Coatings

The present invention relates to the coating of surfaces, in particular to the production of oil and water repellent surfaces, as well as to coated articles obtained thereby.

It is known to apply water and oil repellent coatings by a wide variety of methods. Such coatings typically include fluorocarbon chains, with the degree of oil and water repellency being a function of the number and length of fluorocarbon groups or moieties that can be fitted into the available space.

Plasma deposition techniques have been quite widely used for the deposition of polymeric coatings onto a range of surfaces. This technique is recognised as being a clean, dry technique that generates little waste compared to conventional wet chemical methods. Using this method, plasmas are generated from small organic molecules, which are subjected to an ionising electrical field under low pressure conditions. When this is done in the presence of a substrate, the ions, radicals and excited molecules of the monomer in the plasma polymerise in the gas phase and react with a growing polymer film on the substrate. Conventional polymer synthesis tends to produce structures containing repeat units which bear a strong resemblance to the monomer species, whereas a polymer network, generated using a plasma can be extremely complex.

US patent 6,551,950 discloses use of plasma polymerisation to form an oil or water repellent surface using long chain hydrocarbons and fluorocarbons.

The applicants have discovered that by increasing the residency time of the monomer in the processing chamber, the quality of the coating and efficiency of its deposition can be increased.

Residency time is the average amount of time that a particle spends in a particular system. Residency time can be defined by the equation

$$\tau = C/Q \quad [1]$$

- 5 where τ is residence time, C is the capacity of the processing chamber and Q is the flow rate of the gas through the system at the pressure in the chamber.

10 From the above equation, it can be seen that for any given gas flow rate, an increase chamber size results in a longer residency time. Likewise, the slower the gas flow rate, the greater the residency time. Therefore, slow flow rates and large chambers will result in long residency times.

- 15 The longer a molecule remains in the processing chamber, the great the likelihood that it will undergo a deposition process, such as polymerisation, and attach to the surface.

20 A first aspect of the present invention provides a method of forming a liquid repellent coating on a surface of a substrate, said method comprising exposing said surface to a monomer in a plasma deposition process under conditions that maintain the monomer in situ for a period of time to allow a polymeric layer to form on the surface, wherein the conditions comprise at least
25 one cycle of varying pressure.

The use of variable pressure cycling increases the residency time during which molecules of the monomer are prevalent in the processing chamber. This is due to the cycling keeping the
30 pressure within the optimal range. It is desirable that the chamber remains within the optimal pressure range to maximise polymerisation.

In comparison to coating at static pressure, the use of variable
35 pressure cycling improves the evenness of the coating, reduces

the processing time and uses less monomer to produce a given thickness of coating.

5 The processing chamber may be provided with valve such as a gas exhaust gate, which is closed during introduction of the monomer. The exhaust gate may be adjusted to open, closed or partially open.

10 The at least one cycle may comprise continually introducing the monomer into the processing chamber (for example by injection) and allowing the pressure to rise. Exhaust gas may be expelled from the processing chamber at the end of each cycle.

15 The processing chamber may be at least partly evacuated at the beginning of each cycle; this enables it to reach the low pressure optimal for the polymer coating to form.

20 As gases are expelled at the end of each cycle, waste gases such as water vapour can be vented without affecting the low pressure achieved at the beginning of the cycle.

Whilst leaving the exhaust gate of the chamber closed throughout the process ensures that optimum pressure can easily be reached, a combination of monomer input and outgassing from the treated
25 product causes the pressure within the chamber to build up until it exceeds optimum levels. By contrast, if the gate remains open for the duration of the process, outgassing products can be effectively removed; this allows the pressure to be brought down to optimum levels. However, a significant amount of the input
30 monomer will simply flow through the chamber without undergoing polymerisation. The use of variable pressure cycling enables undesired outgassing products to be vented from the chamber but following venting the gate is closed to bring the chamber back to the bottom of the optimal pressure range. The closing of the
35 gate at a particular point in the cycle is important to maintain

the monomer in situ (increase its residency time) so polymerisation can occur.

5 The at least one cycle of varying pressure may comprise a time based cycle. For example the processing chamber may be at least partly evacuated after a predetermined amount of time in each cycle. Time cycling is particularly beneficial in that it allows for the monomer to be retained in situ so increasing residency time which assists in improving the coating process.

10

The at least one cycle of varying pressure may comprise a pressure based cycle. For example the processing chamber may be at least partly evacuated if the pressure falls outside the optimum range. The processing chamber may comprise pressure
15 sensors to determine the pressure within the chamber. Feedback from the pressure sensor may be used to adjust an exhaust gate of the processing chamber. For example, the exhaust gate may be closed if the pressure drops below the optimum range and the exhaust gate may be opened if the pressure rises above the
20 optimum range.

A pressure based system is suitable for low outgassing products, such as hearing aids and mobile telephones, as it produces a long cycle within the desired pressure range. For higher
25 outgassing products, such as shoes, pressure based cycles can be too short. However, a time based cycle can be designed which allows the cycle to be of good length, gives the monomer a good residency time, whilst still staying near the optimal pressure.

30 The variable pressure may be maintained below a maximum pressure. For example, the maximum pressure may be at or below 150 mTorr. Suitably, the maximum pressure may be at or below 125 mTorr.

35 Each cycle may be between 45 and 75 seconds. Each cycle may be approximately 60 seconds.

The method may comprise exposing the surface to two or more cycles of varying pressure and in particular up to four cycles. Said two or more cycles may comprise between 5 and 12 cycles. Alternatively, said two or more cycles may comprise 8 or 9 cycles.

In one embodiment, the deposition process is a gas process. The deposition process may be a plasma process, for example a plasma polymerisation process.

Where the deposition process is a plasma polymerisation process, the coating may be applied using a pulsed plasma.

The liquid repellent coating may comprise an oil or water repellent coating.

The polymeric layer may be uniform. However, it may also be advantageous to form a non uniform polymeric layer, for example where the coating is used in a bio array.

The liquid repellent coating is suitable for surfaces on a wide range of substrates, for example fabric, metal, glass, ceramics, paper or polymer substrates. Items such as clothing (including footwear), laboratory consumables (including pipette tips), filtration membranes, electronic devices (including mobile phones, audio equipments, laptop computers and hearing aids), microfluidic devices and photovoltaic modules (such as solar panels) can all suitably be treated using the method of this invention.

Plasma polymers are typically generated by subjecting a coating forming precursor to an ionising electric field under low pressure conditions. Deposition occurs when excited species generated by the action of the electric field upon the precursor (radicals, ions, excited molecules etc) polymerise in the gas

phase and react with the substrate surface to form a growing polymer film.

Suitable plasmas for use in the method described herein include
5 non-equilibrium plasmas such as those generated by radiofrequencies (RF), microwaves or direct current (DC). They may operate at atmospheric or sub-atmospheric pressures as are known in the art. In particular however, they are generated by radiofrequencies (RF).

10

Various forms of equipment may be used to generate gaseous plasmas. Generally these comprise containers or plasma chambers in which plasmas may be generated. Particular examples of such equipment are described for instance in WO2005/089961 and
15 WO02/28548, but many other conventional plasma generating apparatus are available.

In general, the item to be treated is placed within a plasma chamber together with the material to be deposited in gaseous
20 state, a glow discharge is ignited within the chamber and a suitable voltage is applied, which may be pulsed.

The gas used within the plasma may comprise a vapour of the monomeric compound alone, but it may be combined with a carrier
25 gas, in particular, an inert gas such as helium or argon. In particular helium is a preferred carrier gas as this can minimise fragmentation of the monomer.

When used as a mixture, the relative amount of the monomer
30 vapour to carrier gas is suitably determined in accordance with procedures which are conventional in the art. The amount of monomer added will depend to some extent on the nature of the particular monomer being used, the nature of the substrate being treated, the size of the plasma chamber etc. Generally, in the
35 case of conventional chambers, monomer is delivered in an amount of from 50-600mg/min, for example at a rate of from 100-

150mg/min. Carrier gas such as helium is suitably administered at a constant rate for example at a rate of from 5-90, for example from 15-30sccm. In some instances, the ratio of monomer to carrier gas will be in the range of from 100:1 to 1:100, for
5 instance in the range of from 10:1 to 1:100, and in particular about 1:1 to 1:10. The precise ratio selected will be so as to ensure that the flow rate required by the process is achieved.

In some cases, a preliminary continuous power plasma may be
10 struck for example for from 0.5-10 minutes for instance for about 4 minutes, within the chamber. This may act as a surface pre-treatment step, ensuring that the monomer attaches itself readily to the surface, so that as polymerisation occurs, the coating "grows" on the surface. The pre-treatment step may be
15 conducted before monomer is introduced into the chamber, in the presence of only the inert gas.

The plasma is then suitably switched to a pulsed plasma to allow polymerisation to proceed, at least when the monomer is present.

20 In all cases, a glow discharge is suitably ignited by applying a high frequency voltage, for example at 13.56MHz. This is suitably applied using electrodes, which may be internal or external to the chamber, but in the case of the larger chambers
25 are internal.

Suitably the gas, vapour or gas mixture is supplied at a rate of at least 1 standard cubic centimetre per minute (sccm) and preferably in the range of from 1 to 100sccm.

30 In the case of the monomer vapour, this is suitably supplied at a rate of from 80-300mg/minute, for example at about 120mg per minute depending upon the nature of the monomer, whilst the pulsed voltage is applied.

35

Gases or vapours may be drawn or pumped into the plasma region. In particular, where a plasma chamber is used, gases or vapours may be drawn into the chamber as a result of a reduction in the pressure within the chamber, caused by use of an evacuating
5 pump, or they may be pumped or injected into the chamber as is common in liquid handling.

Polymerisation is suitably effected using vapours of compounds of formula (I), which are kept within a pressure range of from
10 40 to 150mtorr, suitably at about 80-120mtorr.

The applied fields are suitably of power of from 0.2W to 20W, more suitably about 2W, applied as a pulsed field. These powers are suitable for use in a chamber having a volume of 50cm³. For
15 larger or smaller chambers, a suitable power giving the same power density can be used. For deposition of the polymeric layer, the pulses are applied in a sequence which yields very low average powers, suitably at a duty cycle of up to 10% (i.e. an on:off ratio of up to 10% on). More suitably, the duty cycle
20 is from 0.1% to 1%. The on pulses can be long or short.

The fields are suitably applied from 30 seconds to 90 minutes, preferably from 5 to 60 minutes, depending upon the nature of the compound of formula (I) and the item being treated etc.

25

Suitably a plasma chamber used is of sufficient volume to accommodate multiple items.

A particularly suitable apparatus and method for producing items
30 in accordance with the invention is described in WO2005/089961, the content of which is hereby incorporated by reference.

In particular, when using high volume chambers of this type, the plasma is created with a voltage as a pulsed field, at an
35 average power of from 0.001 to 500w/m², for example at from 0.001

to 100w/m² and suitably for the functional layer, at from 0.005 to 0.5w/m².

5 These conditions are particularly suitable for depositing good quality uniform coatings, in large chambers, for example in chambers where the plasma zone has a volume of greater than 500cm³, for instance 0.5m³ or more, such as from 0.5m³-10m³ and suitably at about 1m³. The layers formed in this way have good mechanical strength.

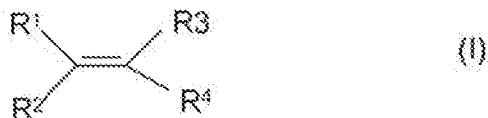
10

The dimensions of the chamber will be selected so as to accommodate the particular item being treated. For instance, generally cuboid chambers may be suitable for a wide range of applications, but if necessary, elongate or rectangular chambers 15 may be constructed or indeed cylindrical, or of any other suitable shape.

The chamber may be a sealable container, to allow for batch processes, or it may comprise inlets and outlets for the items, 20 for example yarn, to allow it to be utilised in a continuous process. In particular in the latter case, the pressure conditions necessary for creating a plasma discharge within the chamber are controlled using high volume pumps, as is conventional for example in a device with a "whistling leak". 25 However it will also be possible to process certain items at atmospheric pressure, or close to, negating the need for "whistling leaks"

The monomer may comprise a compound of formula (I)

30



where R¹, R² and R³ are independently selected from hydrogen, alkyl, haloalkyl or aryl optionally substituted by halo; and R⁴ is

a group $K-R^6$ where R^6 is an alkyl or haloalkyl group and X is a bond; a group of formula $-C(O)O(CH_2)_nY-$ where n is an integer of from 1 to 10 and Y is a bond or a sulphonamide group; or a group $-(O)_pR^6(O)_q(CH_2)_t-$ where R^6 is aryl optionally substituted by halo,
 5 p is 0 or 1, q is 0 or 1 and t is 0 or an integer of from 1 to 10, provided that where q is 1, t is other than 0.
 The monomers used are selected from monomers of formula (I) as defined above. Suitable haloalkyl groups for R^1 , R^2 , R^3 and R^5 are fluoroalkyl groups. The alkyl chains may be straight or
 10 branched and may include cyclic moieties.

For R^5 , the alkyl chains suitably comprise 2 or more carbon atoms, suitably from 2-20 carbon atoms and preferably from 6 to 12 carbon atoms.

15 For R^1 , R^2 and R^3 , alkyl chains are generally preferred to have from 1 to 6 carbon atoms.

Preferably R^5 is a haloalkyl, and more preferably a perhaloalkyl group, particularly a perfluoroalkyl group of formula C_mF_{2m+1}
 20 where m is an integer of 1 or more, suitably from 1-20, and preferably from 4-12 such as 4, 6 or 8.

Suitable alkyl groups for R^1 , R^2 and R^3 have from 1 to 6 carbon
 25 atoms.

In one embodiment, at least one of R^1 , R^2 and R^3 is hydrogen. In a particular embodiment R^1 , R^2 , R^3 are all hydrogen. In yet a further embodiment however R^3 is an alkyl group such as methyl or
 30 propyl.

Where X is a group $-C(O)O(CH_2)_nY-$, n is an integer which provides a suitable spacer group. In particular, n is from 1 to 5, preferably about 2.

35

Suitable sulphonamide groups for Y include those of formula -N(R⁷)SO₂ where R⁷ is hydrogen or alkyl such as C₁₋₄alkyl, in particular methyl or ethyl.

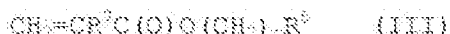
- 5 In one embodiment, the compound of formula (I) is a compound of formula (II)



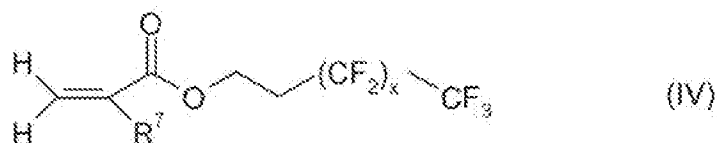
- 10 where R⁵ is as defined above in relation to formula (I).

In compounds of formula (II), X in formula (I) is a bond.

- 15 However in a preferred embodiment, the compound of formula (I) is an acrylate of formula (III)

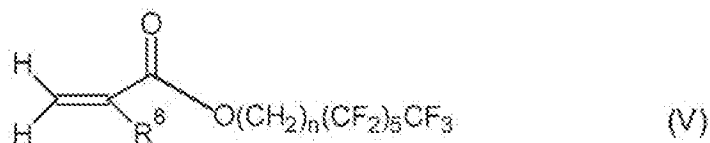


- 20 where n and R⁵ as defined above in relation to formula (I) and R⁶ is hydrogen, C₁₋₁₀alkyl, or C₁₋₁₀haloalkyl. In particular R⁶ is hydrogen or C₁₋₄alkyl such as methyl. A particular example of a compound of formula (III) is a compound of formula (IV)



- 25 where R⁷ is as defined above, and in particular is hydrogen and x is an integer of from 1 to 9, for instance from 4 to 9, and preferably 7. In that case, the compound of formula (IV) is 1H,1H,2H,2H-heptadecafluorodecylacrylate.

- 30 In one embodiment, the compound of formula (IV) is a compound of formula (V)



where R⁸ is hydrogen or methyl, ethyl or propyl group; and n = 1
 5 to 5.

A second aspect of the present invention provides a hydrophobic
 and/or oleophobic substrate which comprises a coating of a
 polymer which has been applied by the method.

10

The substrate may comprise, for example, a fabric or an item of
 clothing (including footwear) comprising said fabric.

Additionally, the substrate may comprise an electronic device,
 microfluidic device, laboratory consumable or photovoltaic

15

module.

Preferred features of the second aspect of the invention may be
 as described above in connection with the first aspect.

20

Throughout the description and claims of this specification, the
 words "comprise" and "contain" and variations of the words, for
 example "comprising" and "comprises", mean "including but not
 limited to", and do not exclude other moieties, additives,
 components, integers or steps.

25

Throughout the description and claims of this specification, the
 singular encompasses the plural unless the context otherwise
 requires. In particular, where the indefinite article is used,
 the specification is to be understood as contemplating plurality
 as well as singularity, unless the context requires otherwise.

Other features of the present invention will become apparent from the following example. Generally speaking the invention extends to any novel one, or any novel combination, of the features disclosed in this specification (including any
5 accompanying claims and drawings). Thus features, integers, characteristics, compounds, chemical moieties or groups described in conjunction with a particular aspect, embodiment or example of the invention are to be understood to be applicable to any other aspect, embodiment or example described herein
10 unless incompatible therewith.

Moreover unless stated otherwise, any feature disclosed herein may be replaced by an alternative feature serving the same or a similar purpose.

15 The invention will now be particularly described by way of example, with reference to the accompanying diagrammatic drawings in which:

Figure 1 illustrates the apparatus for carrying out the
20 invention; and

Figure 2 is a flow diagram showing the events within each variable pressure cycle.

The apparatus used for forming the polymeric coating is shown in
25 simplified form in Figure 1. A processing chamber 10 has a processing area 12 within it and an access door 14. Equipment for generating a plasma within the processing chamber are not shown.

30 A fluid input 16 is provided to allow monomer to be introduced into the chamber. Other fluids may also be introduced via this fluid input, for example a carrier gas or a pre-treatment gas.

The processing chamber 10 is connected to a vacuum pump 18 via a
35 gate valve 20. The gate valve 20 can be closed or opened (either partially or fully). When opened, the gate valve allows waste

gases to be exhausted and when used in conjunction with the vacuum pump enables the pressure within the processing area reduced.

- 5 The apparatus also has a controller 22 which controls the fluid input, gate valve and vacuum pump. The controller may be a microprocessor, PC or any other suitable device.

Figure 2 shows the sequence of events during a cycle. In a first step 24, the gate valve is opened and the vacuum pump partially evacuates the processing chamber. In particular the pressure in the processing chamber is brought down to a desired low pressure. The low pressure allows for increased residency time of monomer(s) in the processing chamber as it/they cannot escape from the valve and flow downstream with the evacuated gases. In a second step 26, the gate valve is closed (or at least partially closed) and the fluid input is opened, this allows the monomer to be introduced into the chamber. The pressure in the chamber gradually increases and in particular this is as a result of vaporisation of monomers introduced into the chamber. In a third step 28, the gate valve is opened so that the waste gases can be exhausted. The controller controls the actions of the vacuum pump, gate valve and fluid input during these steps in each cycle. The cycle is repeated to maintain the desired residency time of the monomer. The cycling rate can be selected either by the time taken to reach a given pressure or by the duration that is needed to provide monomer conversion to polymer. A typical number of cycles that is used is four. Further the use of cycling allows for the deposition rate to be moved within the chamber so that there is more uniformity of coating. In particular the deposition is moved from the edges of the chamber to the centre to improve coating.

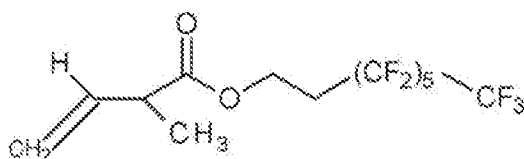
The cycles may be time based or pressure based. In the case of time based cycles, the controller further includes a clock or

counter which is used to control the length of each cycle and the timing of the events in each cycle.

If a pressure based cycle is used, one or more pressure sensors are provided in the processing area, with pressure data being output to the controller. In this case, each step occurs when the pressure within the processing area reaches a pre-determined value.

Example 1

In a first example, a hearing aid was coated using a monomer of formula VI below.



Formula VI

15

The plasma polymerisation coating was applied in an inductively coupled glow discharge reactor with a leak rate of better than 6×10^{-9} mol s⁻¹ and a monomer flow rate of 4mg/min or 3.2 mol s⁻¹. This was connected to a two stage Edwards rotary pump via a liquid nitrogen cold trap, a thermocouple pressure gauge, and a monomer tube containing the monomer. A 13.56 MHz radio frequency (RF) generator was used to power the electrical discharge.

20

A hearing aid having a textured AES plastic exterior surface was placed into the centre of the chamber, which was then evacuated down to 20mTorr.

25

The glow discharge was ignited the chamber subjected to continuous wave (CW) of 150W for 30s, during which the monomer was injected into the chamber in two doses 3 seconds apart.

30

A pulsed wave (PW) was then applied at 450W at a duty cycle of 35 microseconds on and 10 milliseconds off (0.35%), whilst the

monomer was injected into the chamber; 140 shots with 3 seconds between each shot.

During the PW phase, the pressure within the chamber was cycled as follows: Once the chamber pressure reached 50mTorr, the gate valve was closed for 60 seconds. After 60 seconds has elapsed, the gate was opened by a fixed percentage (between 50 to 100%). When pressure fell to 50mTorr, the gate was again closed for 60seconds and the cycle repeated. This was repeated for the duration of the PW cycle (i.e. approximately 420 seconds).

This method was repeated without pressure cycling. The conditions were exactly the same as above, except that the gate valve is left open for the duration of the continuous wave phase and PW phase.

The effect of the plasma deposition on the surface energy of the treated substrate was then determined. Water was applied to the surface of the treated hearing aid and the contact angle measured. The tests were carried out on the coated hearing aid surface and also after the coating had been subjected to 1,000 abrasions (using the same test as example 2). The results of coatings applied using pressure cycling and without pressure cycling are listed in the table below.

25

| Conditions | Initial contact angle (average) | Contact angle after 1,000 abrasions (average) |
|--------------------------|---------------------------------|---|
| With pressure cycling | 105.98 | 110.28 |
| Without pressure cycling | 103.63 | 99.03 |

Table 1

As seen from table 1, higher contact angles are achieved with pressure cycling (both initially and after abrasions). This

shows that the desired performance levels of the coating has been achieved more quickly with pressure cycling than without.

Example 2

- 5 In a second example, a mobile telephone was coated, which included various substrates of plastic, glass and metal. The same apparatus and monomer was used as described in example 1.

10 The chamber was evacuated to 20mTorr before igniting the glow discharge and subjecting the chamber to continuous wave (CW) of 150W for 30s, during which the monomer was injected into the chamber in two doses 3 seconds apart.

15 A pulsed wave (PW) was then applied at 300W at a duty cycle of 35 microseconds on and 10 milliseconds off (0.35%), whilst monomer was injected into the chamber; 140 shots with 3 seconds between each shot.

20 During the PW phase, the pressure within the chamber was cycled as follows: Once the chamber pressure reached 50mTorr, the gate was closed for 60 seconds. After 60 seconds has elapsed, the gate was opened by a fixed percentage (between 50 to 100%). When pressure fell to 50mTorr, the gate was gain closed for 60seconds and the cycle repeated. This was repeated for the duration of
25 the PW cycle (i.e. approximately 420 seconds).

This method was repeated without the pressure cycling.

30 The effect of the plasma deposition on the surface energy of the treated substrate was then determined. Water was applied to the surface of the treated hearing aid and the contact angle measured. The tests were carried out on the coated hearing aid surface and also after the coating had been subjected to 1,000 abrasions using the Taber 5750 linear abrader with a standard
35 Martindate abrasion fabric, 500g weight with a 1 inch abrasion

path. The results of coatings applied using pressure cycling and without pressure cycling are listed in the table below.

| Conditions | Initial contact angle (average) | Contact angle after 1,000 abrasions (average) |
|--------------------------|---------------------------------|---|
| Untreated | 89.04 | 77.4125 |
| Without pressure cycling | 91.78 | 113.3875 |
| With pressure cycling | 115.15 | 114.2375 |

Table 2

5

As with the previous example, the coating applied with pressure cycling results in a higher contact angle. The contact angle changes very little following the abrasions. As before, this is believed to be due to the coating forming more quickly.

10

Example 3

In a third example, a mobile telephone was coated, which included various substrates of plastic, glass and metal. The same apparatus and monomer was used as described in example 1.

15

The chamber was evacuated to 20mTorr before igniting the glow discharge and subjecting the chamber to continuous wave (CW) of 150W for 30s, during which the monomer was injected into the chamber in one dose.

20

A pulsed wave (PW) was then applied at 450W at a duty cycle of 35 microseconds on and 10 milliseconds off (0.35%), whilst monomer was injected into the chamber; 130 shots with 3 seconds between each shot.

25

During the PW phase, the pressure within the chamber was cycled by alternatively opening and closing the gate valve for periods of 60 seconds.

- 5 This method was repeated without the pressure cycling, during which the gate was left open for the duration of the CW and PW phases.

- 10 The effect of the plasma deposition on the surface energy of the treated substrate was then determined using the same methods outlined in example 2.

| Conditions | Average contact angle (interior) | Average contact angle (exterior) |
|--------------------------|----------------------------------|----------------------------------|
| Without pressure cycling | 88.52 | 113.47 |
| With pressure cycling | 113.62 | 121.42 |

Table 3

- 15 Table 3 shows greatly increased deposition on the inside of the telephone where pressure cycling is used, as compared to no cycling.

- 20 As shown by the examples, the use of variable pressure cycling speeds up the process of achieving the performance levels desired, i.e. it speeds up the processing time.

- 25 As shown in example 3, not only is an improvement seen on the outside of products but also on the inside of complex products. To achieve the same results with a non-cycling process would require a longer processing time and more monomer (although the same performance level may still not be reached).

By speeding up the processing time, pressure cycling also has the effect of using less monomer. Other advantages include improved evenness of coating. In addition, the desired performance levels occur across the whole processing chamber.

5

The increase in residency time has additional advantages, for example improved penetration of the coatings into nooks and crannies, for example in electronic devices. In addition, the increased residency time enables coatings to be formed from monomers which do not polymerise well in conventional conditions, for example shorter chain monomers such as 1H,1H,2H,2H-tridecafluoro-octyl acrylate.

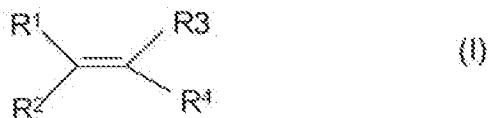
10

15

Claims

1. A method of forming a liquid repellent coating on a surface
5 of a substrate, said method comprising exposing said surface to
a monomer in a plasma deposition process under conditions that
maintain the monomer in situ for a period of time to allow a
polymeric layer to form on the surface, wherein the conditions
comprise at least one cycle of varying pressure.
- 10 2. A method according to claim 1, wherein the substrate is
exposed to a plasma in a processing chamber and the processing
chamber is at least partly evacuated at the beginning of each
cycle.
- 15 3. A method according to claim 1 or claim 2 wherein each cycle
comprises continually introducing the monomer into a processing
chamber and allowing the pressure to rise.
- 20 4. A method according to claim 2 or claim 3, wherein exhaust
gas is expelled from the processing chamber at the end of each
cycle.
- 25 5. A method according to any preceding claim wherein a valve
is used to control pressure in and out of the processing
chamber, with the valve being used to increase residency time of
the monomer in the chamber to allow the polymeric layer to form.
- 30 6. A method according to any preceding claim, wherein the
maximum pressure is at or below 150 mTorr and more particularly
is at or below 125 mTorr.
- 35 7. A method according to any preceding claim, wherein the at
least one cycle of varying pressure may comprise a time based
cycle and/or a pressure based cycle.

8. A method according to any preceding claim, wherein each cycle is between 45 and 75 seconds.
9. A method according to any preceding claim, wherein exposing the surface to at least one cycle of varying pressure comprises exposing the surface to two or more cycles of varying pressure and in particular said two or more cycles comprises between 5 and 12 cycles and more particularly 8 or 9 cycles.
10. A method according to any preceding claim wherein the monomer is a compound of formula (I)



- 15 where R^1 , R^2 and R^3 are independently selected from hydrogen, alkyl, haloalkyl or aryl optionally substituted by halo; and R^4 is a group $\text{X}-\text{R}^5$ where R^5 is an alkyl or haloalkyl group and X is a bond; a group of formula $-\text{C}(\text{O})\text{O}(\text{CH}_2)_n\text{Y}-$ where n is an integer of from 1 to 10 and Y is a bond or a sulphonamide group; or a group
- 20 $-(\text{O})_p\text{R}^6(\text{O})_q(\text{CH}_2)_t-$ where R^6 is aryl optionally substituted by halo, p is 0 or 1, q is 0 or 1 and t is 0 or an integer of from 1 to 10, provided that where q is 1, t is other than 0.

11. A method according to claim 10 wherein the compound of formula (I) is a compound of formula (II)



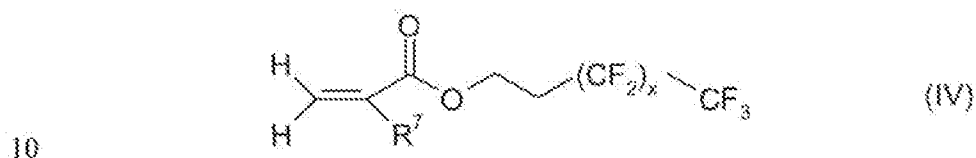
- where R^1 is as defined in claim 10, or a compound of formula (III)



where n and R^6 as defined in claim 10 and R^7 is hydrogen, C_{1-10} alkyl, or C_{1-10} haloalkyl.

12. A method according to claim 11 wherein the compound of
5 formula (I) is a compound of formula (III).

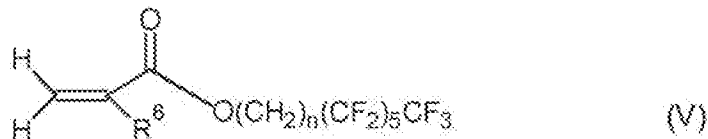
13. A method according to claim 12 wherein the compound of
formula (III) is a compound of formula (IV)



where R^7 is as defined in claim 11, and x is an integer of from 1
to 9.

14. A method according to claim 13 wherein the compound of
15 formula (IV) is 1H,1H,2H,2H-heptadecafluorodecylacrylate.

15. A method according to claim 13 wherein the compound of
20 formula (IV) is a compound of formula (V)



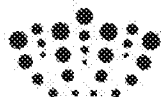
where R^8 is hydrogen or methyl, ethyl or propyl group; and $n = 1$
25 to 5.

16. A method according to any preceding claim, wherein the
plasma deposition process is a plasma polymerisation process and
the coating is applied using a pulsed plasma.

17. A method according to claim 16, wherein the pulsing sequence comprises a ratio of on:off of 1:200 to 1:1500.
- 5 18. A method according to any one of claims 16 or 17, wherein the pulsing sequence comprises power on for 20-50 μ s and off for from 500 μ s to 30000 μ s.
- 10 19. A method according to any preceding claim wherein the average power levels are between 1 W and 1 kW and more particularly between 300 W and 500 W.
- 15 20. A method according to any preceding claim, wherein in a preliminary step, a continuous power plasma is applied to the surface.
21. A method according to claim 20 wherein the preliminary step is conducted in the presence of an inert gas.
- 20 22. A method according to any preceding claim wherein the liquid repellent coating comprises an oil or water repellent coating.
- 25 23. A method according to any preceding claim, wherein the surface is a surface of a fabric, metal, glass, ceramics, paper or polymer substrate.
- 30 24. A method of forming a liquid repellent coating on a surface of a substrate, which method is substantially as herein described with reference to the accompanying illustrative drawings.
- 35 25. A liquid repellent substrate which comprises a coating of a polymer which has been applied by a method according to any one of the preceding claims.

26. A substrate according to claim 25 which comprises a fabric and in particular an item of clothing.

27. A liquid repellent substrate, which substrate is
5 substantially as herein described with reference to the accompanying illustrative drawings.



Application No: GB1212808.8

Examiner: Mr Robert Black

Claims searched: 1-24

Date of search: 23 November 2012

Patents Act 1977: Search Report under Section 17

Documents considered to be relevant:

| Category | Relevant to claims | Identity of document and passage or figure of particular relevance |
|----------|----------------------|---|
| Y | 1-7, 10-14 and 16-23 | EP 1087034 A1 (METALLE) see especially WPI abstract 2001-345753 and the EPODOC abstract |
| Y | 1-7, 10-14 and 16-23 | GB 2434379 A (P2i) see especially the abstract, page 11 lines 10-19 and page 16 lines 1-20 |
| Y | 1-5, 7 and 23 | WO 2007/015779 A2 (3M) see especially the abstract and claim 1 |
| Y | 1-7 and 23 | EP 1074588 A2 (BECTON) see especially claim 8, and paragraphs 0047 and 0048 |
| Y | 1-5, 7, 22 and 23 | WO 00/20130 A1 (MoD) see especially the abstract and page 13 lines 12-34 |
| Y | 1-7 and 23 | GB 1495480 A (AIRCO) see especially page 1 lines 10-14 and 45-56, page 1 line 85 to page 2 line 3, page 3 line 118 to page 4 line 10, page 4 lines 84-97, page 5 lines 16-31, and page 7 lines 8-24 |
| Y | 1-5, 7, 22 and 23 | WO 2009/010753 A2 (P2i) see especially the abstract, figure 1, page 4 lines 20 and 21, page 6 line 19 to page 7 line 11, page 9 lines 5-16, and page 12 lines 1-5 |

Categories:

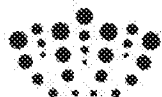
| | | | |
|---|---|---|--|
| X | Document indicating lack of novelty or inventive step | A | Document indicating technological background and/or state of the art. |
| Y | Document indicating lack of inventive step if combined with one or more other documents of same category. | P | Document published on or after the declared priority date but before the filing date of this invention. |
| & | Member of the same patent family | E | Patent document published on or after, but with priority date earlier than, the filing date of this application. |

Field of Search:

Search of GB, EP, WO & US patent documents classified in the following areas of the UKC^X :

Worldwide search of patent documents classified in the following areas of the IPC

B05D; C08F; C08I; D06M



The following online and other databases have been used in the preparation of this search report

EPODOC; WPI

International Classification:

| Subclass | Subgroup | Valid From |
|----------|----------|------------|
| B05D | 0007/24 | 01/01/2006 |
| C08F | 0002/52 | 01/01/2006 |
| C08J | 0007/04 | 01/01/2006 |
| D06M | 0010/10 | 01/01/2006 |
| B05D | 0005/00 | 01/01/2006 |

PATENT COOPERATION TREATY

From the INTERNATIONAL SEARCHING AUTHORITY

PCT

To:
Leland, Emma
Greaves Brewster LLP
European Patent Attorneys
Copa House
Station Road
GB-Cheddar, Somerset BS27 3AH
ROYAUME UNI

NOTIFICATION OF TRANSMITTAL OF
THE INTERNATIONAL SEARCH REPORT AND
THE WRITTEN OPINION OF THE INTERNATIONAL
SEARCHING AUTHORITY, OR THE DECLARATION

(PCT Rule 44.1)

| | |
|---|--|
| Applicant's or agent's file reference EL/P2310PC00 | Date of mailing (day/month/year) 5 June 2015 (05-06-2015) |
| International application No. PCT/GB2015/050521 | International filing date (day/month/year) 24 February 2015 (24-02-2015) |
| Applicant P2I LTD | |

1. ☒ The applicant is hereby notified that the international search report and the written opinion of the International Searching Authority have been established and are transmitted herewith.

Filing of amendments and statement under Article 19:

The applicant is entitled, if he so wishes, to amend the claims of the International Application (see Rule 46):

When? The time limit for filing such amendments is normally two months from the date of transmittal of the International Search Report.

How? Directly to the International Bureau of WIPO, 34 chemin des Colombettes
1211 Geneva 20, Switzerland, Facsimile No.: (41-22) 338.82.70

For more detailed instructions, see *PCT Applicant's Guide*, International Phase, paragraphs 9.004 - 9.011.

2. ☐ The applicant is hereby notified that no international search report will be established and that the declaration under Article 17(2)(a) to that effect and the written opinion of the International Searching Authority are transmitted herewith.

3. ☐ **With regard to any protest** against payment of (an) additional fee(s) under Rule 40.2, the applicant is notified that:

☐ the protest together with the decision thereon has been transmitted to the International Bureau together with any applicant's request to forward the texts of both the protest and the decision thereon to the designated Offices.

☐ no decision has been made yet on the protest; the applicant will be notified as soon as a decision is made.


4. Reminders

The applicant may submit comments on an informal basis on the written opinion of the International Searching Authority to the International Bureau. These comments will be made available to the public after international publication. The International Bureau will send a copy of such comments to all designated Offices unless an international preliminary examination report has been or is to be established.

Shortly after the expiration of 18 months from the priority date, the international application will be published by the International Bureau. If the applicant wishes to avoid or postpone publication, a notice of withdrawal of the international application, or of the priority claim, must reach the International Bureau before the completion of the technical preparations for international publication (Rules 90*bis*.1 and 90*bis*.3).

Within 19 months from the priority date, but only in respect of some designated Offices, a demand for international preliminary examination must be filed if the applicant wishes to postpone the entry into the national phase until 30 months from the priority date (in some Offices even later); otherwise, the applicant must, within 20 months from the priority date, perform the prescribed acts for entry into the national phase before those designated Offices. In respect of other designated Offices, the time limit of 30 months (or later) will apply even if no demand is filed within 19 months. For details about the applicable time limits, Office by Office, see www.wipo.int/pct/en/texts/time_limits.html and the *PCT Applicant's Guide*, National Chapters.

Within 19 months from the priority date, the applicant may request that a supplementary international search be carried out by a different International Searching Authority that offers this service (Rule 45*bis*.1). The procedure for requesting supplementary international search is described in the *PCT Applicant's Guide*, International Phase, paragraphs 8.006-8.032.

| | |
|--|--|
| Name and mailing address of the International Searching Authority  European Patent Office, P.B. 5818 Patentlaan 2 NL-2280 HV Rijswijk Tel. (+31-70) 340-2040 Fax: (+31-70) 340-3016 | Authorized officer VAN DER HOEVEN, Maria Tel: +31 (0)70 340-3342 |
|--|--|

PATENT COOPERATION TREATY

PCT

INTERNATIONAL SEARCH REPORT

(PCT Article 18 and Rules 43 and 44)

| | | |
|---|--|---|
| Applicant's or agent's file reference EL/P2310PC00 | FOR FURTHER ACTION see Form PCT/ISA/220 as well as, where applicable, item 5 below. | |
| International application No. PCT/GB2015/050521 | International filing date (<i>day/month/year</i>) 24 February 2015 (24-02-2015) | (Earliest) Priority Date (<i>day/month/year</i>) 28 February 2014 (28-02-2014) |
| Applicant P2I LTD | | |

This international search report has been prepared by this International Searching Authority and is transmitted to the applicant according to Article 18. A copy is being transmitted to the International Bureau.

This international search report consists of a total of 6 sheets.

☒ It is also accompanied by a copy of each prior art document cited in this report.

1. Basis of the report

a. With regard to the **language**, the international search was carried out on the basis of:

- ☒ the international application in the language in which it was filed
☐ a translation of the international application into _____, which is the language of a translation furnished for the purposes of international search (Rules 12.3(a) and 23.1(b))

b. ☐ This international search report has been established taking into account the **rectification of an obvious mistake** authorized by or notified to this Authority under Rule 91 (Rule 43.6**bis**(a)).

c. ☐ With regard to any **nucleotide and/or amino acid sequence** disclosed in the international application, see Box No. I.

2. ☒ **Certain claims were found unsearchable** (See Box No. II)

3. ☐ **Unity of invention is lacking** (see Box No. III)

4. With regard to the **title**,

- ☒ the text is approved as submitted by the applicant
☐ the text has been established by this Authority to read as follows:

5. With regard to the **abstract**,

- ☒ the text is approved as submitted by the applicant
☐ the text has been established, according to Rule 38.2, by this Authority as it appears in Box No. IV. The applicant may, within one month from the date of mailing of this international search report, submit comments to this Authority

6. With regard to the **drawings**,

- a. the figure of the **drawings** to be published with the abstract is Figure No. _____
☐ as suggested by the applicant
☐ as selected by this Authority, because the applicant failed to suggest a figure
☐ as selected by this Authority, because this figure better characterizes the invention
b. ☒ none of the figures is to be published with the abstract

INTERNATIONAL SEARCH REPORT

International application No.
PCT/GB2015/050521

Box No. II Observations where certain claims were found unsearchable (Continuation of item 2 of first sheet)

This international search report has not been established in respect of certain claims under Article 17(2)(a) for the following reasons:

1. ☐ Claims Nos.:
because they relate to subject matter not required to be searched by this Authority, namely:

2. ☒ Claims Nos.: 36, 37
because they relate to parts of the international application that do not comply with the prescribed requirements to such an extent that no meaningful international search can be carried out, specifically:
see FURTHER INFORMATION sheet PCT/ISA/210

3. ☐ Claims Nos.:
because they are dependent claims and are not drafted in accordance with the second and third sentences of Rule 6.4(a).

Box No. III Observations where unity of invention is lacking (Continuation of item 3 of first sheet)

This International Searching Authority found multiple inventions in this international application, as follows:

1. ☐ As all required additional search fees were timely paid by the applicant, this international search report covers all searchable claims.

2. ☐ As all searchable claims could be searched without effort justifying an additional fees, this Authority did not invite payment of additional fees.

3. ☐ As only some of the required additional search fees were timely paid by the applicant, this international search report covers only those claims for which fees were paid, specifically claims Nos.:

4. ☐ No required additional search fees were timely paid by the applicant. Consequently, this international search report is restricted to the invention first mentioned in the claims; it is covered by claims Nos.:

Remark on Protest

- ☐ The additional search fees were accompanied by the applicant's protest and, where applicable, the payment of a protest fee.
- ☐ The additional search fees were accompanied by the applicant's protest but the applicable protest fee was not paid within the time limit specified in the invitation.
- ☐ No protest accompanied the payment of additional search fees.

INTERNATIONAL SEARCH REPORT

International application No

PCT/GB2015/050521

A. CLASSIFICATION OF SUBJECT MATTER

INV. B05D3/04 B05D3/14 B05D7/22
ADD.

According to International Patent Classification (IPC) or to both national classification and IPC

B. FIELDS SEARCHED

Minimum documentation searched (classification system followed by classification symbols)

B05D

Documentation searched other than minimum documentation to the extent that such documents are included in the fields searched

Electronic data base consulted during the international search (name of data base and, where practicable, search terms used)

EPO-Internal, WPI Data

C. DOCUMENTS CONSIDERED TO BE RELEVANT

| Category* | Citation of document, with indication, where appropriate, of the relevant passages | Relevant to claim No. |
|-----------|--|-----------------------|
| X | US 4 822 632 A (WILLIAMS JOEL L [US] ET AL) 18 April 1989 (1989-04-18) examples I, II | 1-35 |
| X | US 2014/010969 A1 (BICKER MATTHIAS [DE] ET AL) 9 January 2014 (2014-01-09) example 1 | 1-35 |
| X | WO 2011/029628 A1 (SCHOTT AG [DE]; BICKER MATTHIAS [DE]; BAUCH HARTMUT [DE]; LOHMEYER MAN) 17 March 2011 (2011-03-17) page 21, line 20 - line 32 example 1 claims 1,8 | 1-35 |
| X | US 2012/321776 A1 (VETRECIN ROBERT [US]) 20 December 2012 (2012-12-20) examples 2,3 | 1-35 |
| | -/- | |

☒ Further documents are listed in the continuation of Box C.☒ See patent family annex.

* Special categories of cited documents:

"A" document defining the general state of the art which is not considered to be of particular relevance

"E" earlier application or patent but published on or after the international filing date

"L" document which may throw doubts on priority claim(s) or which is cited to establish the publication date of another citation or other special reason (as specified)

"O" document referring to an oral disclosure, use, exhibition or other means

"P" document published prior to the international filing date but later than the priority date claimed

"T" later document published after the international filing date or priority date and not in conflict with the application but cited to understand the principle or theory underlying the invention

"X" document of particular relevance; the claimed invention cannot be considered novel or cannot be considered to involve an inventive step when the document is taken alone

"Y" document of particular relevance; the claimed invention cannot be considered to involve an inventive step when the document is combined with one or more other such documents, such combination being obvious to a person skilled in the art

"&" document member of the same patent family

Date of the actual completion of the international search

28 May 2015

Date of mailing of the international search report

05/06/2015

Name and mailing address of the ISA/

European Patent Office, P.B. 5818 Patentlaan 2
NL - 2280 HV Rijswijk
Tel. (+31-70) 340-2040,
Fax: (+31-70) 340-3016

Authorized officer

Slembrouck, Igor

INTERNATIONAL SEARCH REPORT

International application No

PCT/GB2015/050521

C(Continuation). DOCUMENTS CONSIDERED TO BE RELEVANT

| Category* | Citation of document, with indication, where appropriate, of the relevant passages | Relevant to claim No. |
|-----------|---|-----------------------|
| A | <p>WO 2005/021833 A2 (SURFACE INNOVATIONS LTD [GB]; WARD LUKE [GB]; BADYAL JAS PAL [GB]) 10 March 2005 (2005-03-10) cited in the application -----</p> | 1 |

INTERNATIONAL SEARCH REPORT

Information on patent family members

International application No

PCT/GB2015/050521

| Patent document cited in search report | | Publication date | Patent family member(s) | Publication date |
|---|----|---------------------|----------------------------|---------------------|
| US 4822632 | A | 18-04-1989 | NONE | |
| ----- | | | | |
| US 2014010969 | A1 | 09-01-2014 | CA 2824074 A1 | 26-07-2012 |
| | | | DE 102011009057 A1 | 26-07-2012 |
| | | | EP 2666181 A1 | 27-11-2013 |
| | | | KR 20140007842 A | 20-01-2014 |
| | | | US 2014010969 A1 | 09-01-2014 |
| | | | WO 2012097972 A1 | 26-07-2012 |
| ----- | | | | |
| WO 2011029628 | A1 | 17-03-2011 | DE 102009041132 A1 | 14-04-2011 |
| | | | DK 2477755 T3 | 16-02-2015 |
| | | | EP 2477755 A1 | 25-07-2012 |
| | | | US 2012171386 A1 | 05-07-2012 |
| | | | WO 2011029628 A1 | 17-03-2011 |
| ----- | | | | |
| US 2012321776 | A1 | 20-12-2012 | AU 2012271807 A1 | 09-01-2014 |
| | | | CA 2839402 A1 | 20-12-2012 |
| | | | CN 103619493 A | 05-03-2014 |
| | | | EP 2720807 A1 | 23-04-2014 |
| | | | US 2012321776 A1 | 20-12-2012 |
| | | | WO 2012174054 A1 | 20-12-2012 |
| ----- | | | | |
| WO 2005021833 | A2 | 10-03-2005 | GB 2419358 A | 26-04-2006 |
| | | | WO 2005021833 A2 | 10-03-2005 |
| ----- | | | | |

FURTHER INFORMATION CONTINUED FROM PCT/ISA/ 210

Continuation of Box II.2

Claims Nos.: 36, 37

(1) claim 36 refers to a method of applying a coating whereby the characterising technical feature(s) should be disclosed by the illustrative drawings; this authority found no clear and unambiguous indication in the drawings that could possibly constitute a characterising technical feature for a method of applying a coating; furthermore, the passages of the description relating to the illustrative figures are disclosing so many technical details that it is impossible for the skilled reader to determine which one is essential to the definition of the invention; as a consequence, the subject matter of claim 36 is considered as lacking clarity (Article 6 PCT) and

The non-compliance with the substantive provisions is to such an extent that no meaningful search of claim 36 could be carried out at all (Article 17(2) PCT).

(2) claim 37 refers to a device whereby the characterising technical feature(s) should be disclosed by the illustrative drawings; this authority found no clear and unambiguous indication in the drawings that could possibly constitute a characterising technical feature for a device according to the invention; furthermore, the passages of the description relating to the illustrative figures are disclosing so many technical details that it is impossible for the skilled reader to determine which one is essential to the definition of the invention; as a consequence, the subject matter of claim 37 is considered as lacking clarity (Article 6 PCT) and

The non-compliance with the substantive provisions is to such an extent that no meaningful search of claim 37 could be carried out at all (Article 17(2) PCT).

The applicant's attention is drawn to the fact that claims relating to inventions in respect of which no international search report has been established need not be the subject of an international preliminary examination (Rule 66.1(e) PCT). The applicant is advised that the EPO policy when acting as an International Preliminary Examining Authority is normally not to carry out a preliminary examination on matter which has not been searched. This is the case irrespective of whether or not the claims are amended following receipt of the search report or during any Chapter II procedure. If the application proceeds into the regional phase before the EPO, the applicant is reminded that a search may be carried out during examination before the EPO (see EPO Guidelines C-IV, 7.2), should the problems which led to the Article 17(2) declaration be overcome.

PATENT COOPERATION TREATY

From the
INTERNATIONAL SEARCHING AUTHORITY

PCT

To:

see form PCT/ISA/220

WRITTEN OPINION OF THE INTERNATIONAL SEARCHING AUTHORITY (PCT Rule 43bis.1)

Date of mailing
(day/month/year) see form PCT/ISA/210 (second sheet)

Applicant's or agent's file reference
see form PCT/ISA/220

FOR FURTHER ACTION
See paragraph 2 below

International application No.
PCT/GB2015/050521

International filing date (day/month/year)
24.02.2015

Priority date (day/month/year)
28.02.2014

International Patent Classification (IPC) or both national classification and IPC
INV. B05D3/04 B05D3/14 B05D7/22

Applicant
P2I LTD

1. This opinion contains indications relating to the following items:

- ☒ Box No. I Basis of the opinion
- ☐ Box No. II Priority
- ☒ Box No. III Non-establishment of opinion with regard to novelty, inventive step and industrial applicability
- ☐ Box No. IV Lack of unity of invention
- ☒ Box No. V Reasoned statement under Rule 43bis.1(a)(i) with regard to novelty, inventive step and industrial applicability; citations and explanations supporting such statement
- ☐ Box No. VI Certain documents cited
- ☐ Box No. VII Certain defects in the international application
- ☒ Box No. VIII Certain observations on the international application

2. FURTHER ACTION

If a demand for international preliminary examination is made, this opinion will usually be considered to be a written opinion of the International Preliminary Examining Authority ("IPEA") except that this does not apply where the applicant chooses an Authority other than this one to be the IPEA and the chosen IPEA has notified the International Bureau under Rule 66.1bis(b) that written opinions of this International Searching Authority will not be so considered.

If this opinion is, as provided above, considered to be a written opinion of the IPEA, the applicant is invited to submit to the IPEA a written reply together, where appropriate, with amendments, before the expiration of 3 months from the date of mailing of Form PCT/ISA/220 or before the expiration of 22 months from the priority date, whichever expires later.

For further options, see Form PCT/ISA/220.

Name and mailing address of the ISA:



European Patent Office
P.B. 5818 Patentlaan 2
NL-2280 HV Rijswijk - Pays Bas
Tel. +31 70 340 - 2040
Fax: +31 70 340 - 3016

Date of completion of
this opinion

see form
PCT/ISA/210

Authorized Officer

Telephone No. +31 70 340-0



**WRITTEN OPINION OF THE
INTERNATIONAL SEARCHING AUTHORITY**

International application No.
PCT/GB2015/050521

Box No. I Basis of the opinion

1. With regard to the **language**, this opinion has been established on the basis of:

- ☒ the international application in the language in which it was filed.
- ☐ a translation of the international application into , which is the language of a translation furnished for the purposes of international search (Rules 12.3(a) and 23.1 (b)).

2. ☐ This opinion has been established taking into account the **rectification of an obvious mistake** authorized by or notified to this Authority under Rule 91 (Rule 43*bis*.1(a))

3. ☐ With regard to any **nucleotide and/or amino acid sequence** disclosed in the international application, this opinion has been established on the basis of a sequence listing:

a. ☐ forming part of the international application as filed:

- ☐ in the form of an Annex C/ST.25 text file.
- ☐ on paper or in the form of an image file.

b. ☐ furnished together with the international application under PCT Rule 13*ter*.1(a) for the purposes of international search only in the form of an Annex C/ST.25 text file.

c. ☐ furnished subsequent to the international filing date for the purposes of international search only:

- ☐ in the form of an Annex C/ST.25 text file (Rule 13*ter*.1(a)).
- ☐ on paper or in the form of an image file (Rule 13*ter*.1(b) and Administrative Instructions, Section 713).

4. ☐ In addition, in the case that more than one version or copy of a sequence listing has been filed or furnished, the required statements that the information in the subsequent or additional copies is identical to that forming part of the application as filed or does not go beyond the application as filed, as appropriate, were furnished.

5. Additional comments:

**WRITTEN OPINION OF THE
INTERNATIONAL SEARCHING AUTHORITY**

International application No.
PCT/GB2015/050521

Box No. III Non-establishment of opinion with regard to novelty, inventive step and industrial applicability

The questions whether the claimed invention appears to be novel, to involve an inventive step (to be non obvious), or to be industrially applicable have not been examined in respect of

☐ the entire international application

☒ claims Nos. 36, 37

because:

☐ the said international application, or the said claims Nos. relate to the following subject matter which does not require an international search (*specify*):

☐ the description, claims or drawings (*indicate particular elements below*) or said claims Nos. are so unclear that no meaningful opinion could be formed (*specify*):

☐ the claims, or said claims Nos. are so inadequately supported by the description that no meaningful opinion could be formed (*specify*):

☒ no international search report has been established for the whole application or for said claims Nos. 36, 37

☐ a meaningful opinion could not be formed without the sequence listing; the applicant did not, within the prescribed time limit:

☐ furnish a sequence listing in the form of an Annex C/ST.25 text file, and such listing was not available to the International Searching Authority in the form and manner acceptable to it; or the sequence listing furnished did not comply with the standard provided for in Annex C of the Administrative Instructions.

☐ furnish a sequence listing on paper or in the form of an image file complying with the standard provided for in Annex C of the Administrative Instructions, and such listing was not available to the International Searching Authority in the form and manner acceptable to it; or the sequence listing furnished did not comply with the standard provided for in Annex C of the Administrative Instructions.

☐ pay the required late furnishing fee for the furnishing of a sequence listing in response to an invitation under Rule 13*ter*.1(a) or (b).

☒ See Supplemental Box for further details

**WRITTEN OPINION OF THE
INTERNATIONAL SEARCHING AUTHORITY**

International application No.
PCT/GB2015/050521

Box No. V Reasoned statement under Rule 43bis.1(a)(i) with regard to novelty, inventive step or industrial applicability; citations and explanations supporting such statement

1. Statement

| | | |
|-------------------------------|-------------|-------------|
| Novelty (N) | Yes: Claims | <u>2-35</u> |
| | No: Claims | <u>1</u> |
| Inventive step (IS) | Yes: Claims | |
| | No: Claims | <u>1-35</u> |
| Industrial applicability (IA) | Yes: Claims | <u>1-35</u> |
| | No: Claims | |

2. Citations and explanations

see separate sheet

Box No. VIII Certain observations on the international application

The following observations on the clarity of the claims, description, and drawings or on the question whether the claims are fully supported by the description, are made:

see separate sheet

Reference is made to the following documents:

- D1 US 4 822 632 A (WILLIAMS JOEL L [US] ET AL) 18 April 1989
(1989-04-18)
- D2 US 2014/010969 A1 (BICKER MATTHIAS [DE] ET AL) 9 January 2014
(2014-01-09)
- D3 WO 2011/029628 A1 (SCHOTT AG [DE]; BICKER MATTHIAS [DE];
BAUCH HARTMUT [DE]; LOHMEYER MAN) 17 March 2011
(2011-03-17)
- D4 US 2012/321776 A1 (VETRECIN ROBERT [US]) 20 December 2012
(2012-12-20)
- D5 WO 2005/021833 A2 (SURFACE INNOVATIONS LTD [GB]; WARD LUKE
[GB]; BADYAL JAS PAL [GB]) 10 March 2005 (2005-03-10)cited in the
application

Re Item III

**Non-establishment of opinion with regard to novelty, inventive step and
industrial applicability**

(1) Claim 36 refers to a method of applying a coating whereby the characterising technical feature(s) should be disclosed by the illustrative drawings; this authority found no clear and unambiguous indication in the drawings that could possibly constitute a characterising technical feature for a method of applying a coating; furthermore, the passages of the description relating to the illustrative figures are disclosing so many technical details that it is impossible for the skilled reader to determine which one is essential to the definition of the invention; as a consequence, the subject matter of claim 36 is considered as lacking clarity (Article 6 PCT) and

The non-compliance with the substantive provisions is to such an extent that no meaningful search of claim 36 could be carried out at all (Article 17(2) PCT).

(2) Claim 37 refers to a device whereby the characterising technical feature(s) should be disclosed by the illustrative drawings; this authority found no clear and unambiguous indication in the drawings that could possibly constitute a characterising technical feature for a device according to the invention; furthermore, the passages of the description relating to the illustrative figures are disclosing so many technical

details that it is impossible for the skilled reader to determine which one is essential to the definition of the invention; as a consequence, the subject matter of claim 37 is considered as lacking clarity (Article 6 PCT) and

The non-compliance with the substantive provisions is to such an extent that no meaningful search of claim 37 could be carried out at all (Article 17(2) PCT).

Re Item VIII

Certain observations on the international application

Clarity (Article 6 PCT)

(3) The scope of claim 1 is not clear and lacks support according to Article 6 PCT for the following reasons:

(3.1) the term "housing" used in claim 1 is vague and unclear (Article 6 PCT);

actually, a plasma treatment chamber containing substrates could be regarded as a device according to claim 1, where the plasma treatment chamber would be regarded as a "housing" and the substrates would be regarded as the "components" according to the wording of claim 1;

this interpretation is also supported by the statement in the application's description on p. 6, l. 7-10, where the "device" is stipulated as comprising a "laboratory equipment"; said passage further stipulates that "articles of laboratory equipment (...) may be housed in a container"; this passage of the description support the vague interpretation of the terms "device", "housing" and "elements" used in claim 1;

(3.2) step (i) of claim 1 reads:

"applying liquid coating precursor to at least part of the internal surface of the housing and/or at least part of the one or more components",

it appears that the method of claim 1 may be directed to coat only "at least part of the one or more components";

(3.3) the term

"the pressure being sufficient to cause the liquid coating precursor to evaporate"

used in claim 1 constitutes an attempt to define the pressure in terms of the result to be achieved, which merely amounts to a statement of the underlying problem, without providing the technical features necessary for achieving this result; this attempt is also considered lacking clarity in that the liquid coating precursor may be applied:

- by spraying (i.e. in an atomised state; cf. claim 15),
- as multiple droplets (i.e. as liquid aggregates; cf. claim 16),
- within a porous material (i.e. the liquid coating precursor soaks a material; cf. claim 17),

those differences in applications result in differences of vacuum level needed to evaporate the liquid coating precursor; furthermore the term does not specify if the pressure should be sufficient to evaporate partly or completely the liquid coating precursor;

it is furthermore known that a reduction of the total pressure will necessarily lead to an increase partial pressure of any liquid subjected to said reduced total pressure, resulting in a partial evaporation of said liquid.

as a consequence, the term

"the pressure being sufficient to cause the liquid coating precursor to evaporate"

is considered as lacking clarity (Article 6 PCT) and can not be considered as possibly restricting the scope of claim 1;

(3.4) claim 1 therefore covers embodiments where :

- (i) components are coated with a liquid coating precursor,
- (ii) coated components of step (i) are subjected to reduced pressure in a plasma treatment chamber
- (iii) liquid coating precursor is subjected to plasma treatment;

those embodiments do not correspond to the scope of the invention as can be derived from the application; claim 1 is therefore considered as lacking support (Article 6 PCT).

(4) It is clear from the description's examples that

the fact that in step (ii) evaporation of the liquid coating precursor occurs is essential to the definition of the invention.

Since independent claim 1 does not contain this feature it does not meet the requirement following from Article 6 PCT taken in combination with Rule 6.3(b) PCT that any independent claim must contain all the technical features essential to the definition of the invention.

(5) In claim 1, step (ii) refers to a "closed housing";

in claim 20, it is stipulated that a vapour is introduced through an aperture in the housing;

in claim 21, it is stipulated that the liquid coating precursor is applied in step (i) whilst the housing is closed;

it appears from the examples, that the mobile phone enclosures (which are believed to correspond to the term "housing" used in the claims) has apertures;

the topological definition of a closed structure is that it has no part left open and that its internal surface does not contact its external surface;

it appears however that

the "closed housing" referred to in claim 1 may have apertures through which diffusion of vapours may occur (cf. claim 22, examples),

the apertures are not sealed at any moment of the method,

there is therefore a confusion around the term "closed" used in the claims, as it appears clearly that the housing may well be an open structure with apertures;

the subject matter of the claims is therefore considered as lacking clarity for this reason as well (Article 6 PCT).

Re Item V

Reasoned statement with regard to novelty, inventive step or industrial applicability; citations and explanations supporting such statement

Novelty (Article 33(3) PCT)

(6) In view of the clarity objections raised under Item VIII, this authority found that document D1 discloses (cf. document D1, passages cited in the search report):

a method of applying a coating to a device, the device comprising one or more components housed in a housing, the method comprising the steps of:

- (i) applying liquid coating precursor to least part of the one or more components ;
- (ii) applying below atmosphere pressure conditions to the closed housing, with the one or more components inside the housing; and
- (iii) initiation of the liquid coating precursor with a plasma, to thereby cause the coating to form from the liquid precursor on at least some of the internal surfaces of the device.

The subject matter of at least claim 1 is therefore not novel (Article 33(2) PCT).

(7) The attention of the applicant is brought to the fact that a similar objection against at least the subject matter of claim 1 could be raised on the basis of documents D2, D3 and D4 each considered independently.

Inventive Step (Article 33(3) PCT)

(8) At this stage of the procedure, this authority is of the opinion that if the subject matter of any of claims 2-35 should appear to be novel over the disclosure of any of documents D1-D4, it is not clear how the subject matter of claims 2-35 could eventually contribute to the inventive step of the present application: the technical features of claim 2-35 seem to represent nothing more than some out of many alternatives the person skilled in the art would select without the need of inventive skills, especially as no particular advantage can be derived from said technical features. Consequently, and without any acknowledgement concerning the novelty of said claims, it is considered at this stage of the procedure that the subject-matter of claims 2-35, if ever novel, lacks an inventive step (Article 33(3) PCT)).

Industrial Applicability (Article 33(4) PCT)

(9) The industrial applicability of the subject matter of claims 1-35 (method of applying a coating to a device) is obvious (Article 33(4) PCT).

Electronic Patent Application Fee Transmittal

| | | | | |
|---|---------------------------------------|-----------------|---------------|-----------------------------|
| Application Number: | 15735130 | | | |
| Filing Date: | 08-Dec-2017 | | | |
| Title of Invention: | COATINGS | | | |
| First Named Inventor/Applicant Name: | Stephen Richard COULSON | | | |
| Filer: | Paul John Parins/Bethany Lichtscheidl | | | |
| Attorney Docket Number: | GBL0007/US | | | |
| Filed as Large Entity | | | | |
| Filing Fees for U.S. National Stage under 35 USC 371 | | | | |
| Description | Fee Code | Quantity | Amount | Sub-Total in USD(\$) |
| Basic Filing: | | | | |
| Pages: | | | | |
| Claims: | | | | |
| Miscellaneous-Filing: | | | | |
| Petition: | | | | |
| Patent-Appeals-and-Interference: | | | | |
| Post-Allowance-and-Post-Issuance: | | | | |
| Extension-of-Time: | | | | |

| Description | Fee Code | Quantity | Amount | Sub-Total in USD(\$) |
|---|----------|----------|--------|----------------------|
| Miscellaneous: | | | | |
| SUBMISSION- INFORMATION DISCLOSURE STMT | 1806 | 1 | 240 | 240 |
| Total in USD (\$) | | | | 240 |

Electronic Acknowledgement Receipt

| | |
|---|---------------------------------------|
| EFS ID: | 35453134 |
| Application Number: | 15735130 |
| International Application Number: | |
| Confirmation Number: | 7101 |
| Title of Invention: | COATINGS |
| First Named Inventor/Applicant Name: | Stephen Richard COULSON |
| Customer Number: | 33072 |
| Filer: | Paul John Parins/Bethany Lichtscheidl |
| Filer Authorized By: | Paul John Parins |
| Attorney Docket Number: | GBL0007/US |
| Receipt Date: | 18-MAR-2019 |
| Filing Date: | 08-DEC-2017 |
| Time Stamp: | 15:21:19 |
| Application Type: | U.S. National Stage under 35 USC 371 |

Payment information:

| | |
|--|-----------------------|
| Submitted with Payment | yes |
| Payment Type | CARD |
| Payment was successfully received in RAM | \$240 |
| RAM confirmation Number | 031919INTEFSW15220300 |
| Deposit Account | |
| Authorized User | |

The Director of the USPTO is hereby authorized to charge indicated fees and credit any overpayment as follows:

| | | | | | |
|--|--|----------------------------|--|------------------|------------------|
| | | | | | |
| File Listing: | | | | | |
| Document Number | Document Description | File Name | File Size(Bytes)/ Message Digest | Multi Part /.zip | Pages (if appl.) |
| 1 | Information Disclosure Statement (IDS) Form (SB08) | GBL0007US_SIDS.pdf | 1044070 | no | 6 |
| | | | 8baa48276c651d906bc79b07cf8410e92d2abc63 | | |
| Warnings: | | | | | |
| Information: | | | | | |
| 2 | Foreign Reference | EP2711153A1.pdf | 1839209 | no | 11 |
| | | | 273f4666e3147230e78d058f4086ca67f021e5be | | |
| Warnings: | | | | | |
| Information: | | | | | |
| 3 | Foreign Reference | GB2434368A.pdf | 2735053 | no | 20 |
| | | | 702b3464b3a0e3421e053cd93f13359fb3de23fd | | |
| Warnings: | | | | | |
| Information: | | | | | |
| 4 | Foreign Reference | GB2493264A.pdf | 3598172 | no | 30 |
| | | | a4ee32791598c1fac8a32ef19f63ddb9e4ec066f | | |
| Warnings: | | | | | |
| Information: | | | | | |
| 5 | Non Patent Literature | NPL_1_GB_Search_Report.pdf | 1398326 | no | 4 |
| | | | c413532c08977af64faea9ca161d96d739822f79 | | |
| Warnings: | | | | | |
| The page size in the PDF is too large. The pages should be 8.5 x 11 or A4. If this PDF is submitted, the pages will be resized upon entry into the Image File Wrapper and may affect subsequent processing | | | | | |
| Information: | | | | | |
| 6 | Non Patent Literature | NPL_2_ISRWO.pdf | 1275001 | no | 16 |
| | | | fe50c4c810ddb3ef215915d1b884de576cb51b2 | | |
| Warnings: | | | | | |
| Ex. 1028 - Favored - p. 385 | | | | | |

The page size in the PDF is too large. The pages should be 8.5 x 11 or A4. If this PDF is submitted, the pages will be resized upon entry into the Image File Wrapper and may affect subsequent processing

Information:

| | | | | | |
|---|-----------------------|----------------------------|--|----|---|
| 7 | Non Patent Literature | NPL_3_GB_Search_Report.pdf | 310401 | no | 4 |
| | | | b9bd0825ccf8def34312675ba4a27982338b7ec6 | | |

Warnings:

The page size in the PDF is too large. The pages should be 8.5 x 11 or A4. If this PDF is submitted, the pages will be resized upon entry into the Image File Wrapper and may affect subsequent processing

Information:

| | | | | | |
|---|-------------------|------------------|--|----|----|
| 8 | Foreign Reference | WO03063938A1.pdf | 2895400 | no | 21 |
| | | | 378ea27fee4daf174f3631dce6ac971e93f57f50 | | |

Warnings:

Information:

| | | | | | |
|---|-------------------|------------------|--|----|----|
| 9 | Foreign Reference | WO2002028548.pdf | 895493 | no | 23 |
| | | | 2adf3502c0ae0c5b771be85abbfb71ca02e5ad95 | | |

Warnings:

Information:

| | | | | | |
|----|-------------------|--------------------|--|----|----|
| 10 | Foreign Reference | WO2005021833A2.pdf | 4660650 | no | 30 |
| | | | e045f75c5014592f618f9d5d50b566d30486fa87 | | |

Warnings:

Information:

| | | | | | |
|----|-------------------|--------------------|--|----|----|
| 11 | Foreign Reference | WO2009030435A1.pdf | 6162869 | no | 38 |
| | | | df502cdb45336421ad78165e707b8f72e14e4a22 | | |

Warnings:

Information:

| | | | | | |
|----|-------------------|--------------------|---|----|----|
| 12 | Foreign Reference | WO2009046093A2.pdf | 5660317 | no | 31 |
| | | | 6cf1a2a4492d245cddde8a7c00be5fb22d2767242 | | |

Warnings:

Information:

| | | | | | |
|------------------------------|----------------------|--------------------|---|----|----|
| 13 | Foreign Reference | WO2011029628A1.pdf | 9268672 | no | 62 |
| | | | 3b81efc196e17bbe58bb7f3fe79461a93fcf4c70 | | |
| Warnings: | | | | | |
| Information: | | | | | |
| 14 | Foreign Reference | WO2012158953.pdf | 961263 | no | 36 |
| | | | 643d47c6b265353102c52f09d0788b8c674b7a92 | | |
| Warnings: | | | | | |
| Information: | | | | | |
| 15 | Foreign Reference | WO2013132250.pdf | 1448452 | no | 35 |
| | | | 504c26eaa5100f6931b000001c12088ecf36bdba | | |
| Warnings: | | | | | |
| Information: | | | | | |
| 16 | Foreign Reference | WO2014026967A2.pdf | 6009473 | no | 46 |
| | | | fd42a9ec122303b1ac3efac89f699adee5787ab | | |
| Warnings: | | | | | |
| Information: | | | | | |
| 17 | Foreign Reference | WO2005089961.pdf | 1227145 | no | 29 |
| | | | 1472f0b0c75ae062271df78bfc9f88e8267dff909 | | |
| Warnings: | | | | | |
| Information: | | | | | |
| 18 | Fee Worksheet (SB06) | fee-info.pdf | 30379 | no | 2 |
| | | | 09b294e9188deda138c308ea0e3b111a92283867 | | |
| Warnings: | | | | | |
| Information: | | | | | |
| Total Files Size (in bytes): | | | 51420345 | | |

This Acknowledgement Receipt evidences receipt on the noted date by the USPTO of the indicated documents, characterized by the applicant, and including page counts, where applicable. It serves as evidence of receipt similar to a Post Card, as described in MPEP 503.

New Applications Under 35 U.S.C. 111

If a new application is being filed and the application includes the necessary components for a filing date (see 37 CFR 1.53(b)-(d) and MPEP 506), a Filing Receipt (37 CFR 1.54) will be issued in due course and the date shown on this Acknowledgement Receipt will establish the filing date of the application.

National Stage of an International Application under 35 U.S.C. 371

If a timely submission to enter the national stage of an international application is compliant with the conditions of 35 U.S.C. 371 and other applicable requirements a Form PCT/DO/EO/903 indicating acceptance of the application as a national stage submission under 35 U.S.C. 371 will be issued in addition to the Filing Receipt, in due course.

New International Application Filed with the USPTO as a Receiving Office

If a new international application is being filed and the international application includes the necessary components for an international filing date (see PCT Article 11 and MPEP 1810), a Notification of the International Application Number and of the International Filing Date (Form PCT/RO/105) will be issued in due course, subject to prescriptions concerning national security, and the date shown on this Acknowledgement Receipt will establish the international filing date of the application.

PATENT APPLICATION

IN THE UNITED STATES PATENT AND TRADEMARK OFFICE

| | |
|---|---------------------------------------|
| Inventor: Stephen Richard COULSON | Examiner: Buie-Hatcher, Nicole |
| Serial No. 15/735,130 | Group Art Unit: 1767 |
| International Filing Date: June 8, 2016 | Confirmation No. 7101 |
| International App. No. PCT/GB2016/051688 | Docket No. GBL0007/US |
| 371(c): December 8, 2017 | |
| For: COATINGS | |

Mail Stop Amendment
Commissioner for Patents
P.O. Box 1450
Alexandria, Virginia 22313-1450

I CERTIFY THAT ON **MARCH 18, 2019** THIS CORRESPONDENCE
IS BEING ELECTRONICALLY TRANSMITTED TO THE UNITED
STATES PATENT AND TRADEMARK OFFICE VIA THE
OFFICE'S EFS-WEB.

/Bethany Lichtscheidl/
BETHANY L. LICHTSCHEIDL

AMENDMENT AND RESPONSE

Dear Examiner Buie-Hatcher:

This communication is responsive to the non-final Office Action issued on October 18, 2018, in connection with the above-identified patent application.

The three-month shortened-statutory period for response expired on January 18, 2019. Applicant respectfully petitions the Commissioner for Patents to extend the time for response for two months, from January 18, 2019 to March 18, 2019. It is respectfully submitted that this response is timely filed within the extended deadline. If any additional extension period is required in order for this paper to be timely filed, Applicant hereby requests an extension for such additional time period and authorizes the appropriate fees therefore to be charged to the Kagan Binder Deposit Account No. 50-1775 and to notify us of the same.

Payment in the amount of \$600.00 for a two-month extension of time is being provided by the electronic payment system of the EFS. In the event of failure of this payment by the electronic payment system of the EFS for any reason, the Commissioner is authorized to charge Kagan Binder Deposit Account No. 50-1775 and thereafter is requested to notify us of the same.

Specification Amendments

Please delete the paragraph on page 1 that begins with “Monosaturated monomers” and ends with “(see co-pending application).”

Please add the following paragraph (section heading) on page 17 immediately prior to the paragraph beginning with “The present invention will now”:

Brief Description of Drawings

Claim Amendments

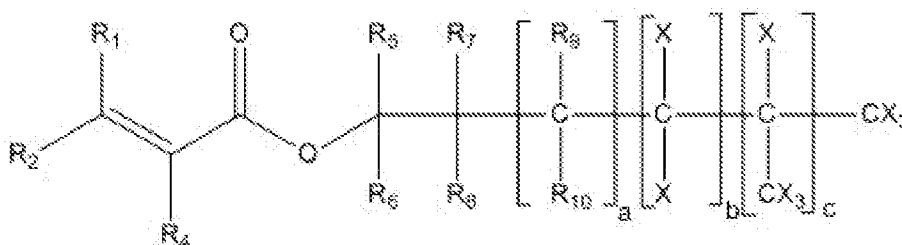
Please amend claims 1, 6, 8, 13, 15, and 16, and cancel claims 5, 7, 12, and 14 as follows:

1. (Currently Amended) An electronic or electrical device or electronic or electrical component thereof comprising a protective cross-linked polymeric coating on a surface of said device or component;

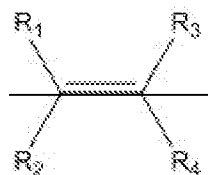
wherein the protective cross-linked polymeric coating is ~~obtainable~~ obtained by exposing said device or component to a plasma comprising a monomer compound and a crosslinking reagent for a period of time sufficient to allow formation of the protective cross-linked polymeric coating on a surface thereof,

wherein the monomer compound is a compound of formula I(a):

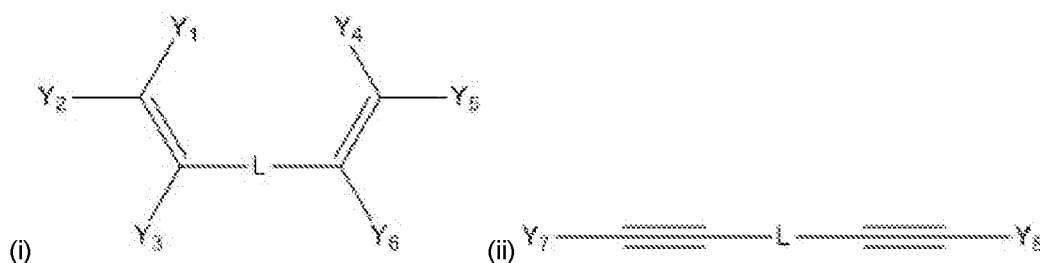
I(a)



wherein each of R₁, R₂, R₄ and R₅ to ~~R₈~~ R₁₀ ~~is hydrogen; each of R₄, R₉ and R₁₀ is independently hydrogen or methyl~~ is independently selected from hydrogen or an optionally substituted C₁-C₆ branched or straight chain alkyl group; each X is independently hydrogen or halogen; a is from 0-10; b is from 3 to 7; and c is 0 or 1 ; ~~[[:]~~



and wherein the crosslinking reagent comprises two or more unsaturated bonds attached by means of one or more linker moieties and has a boiling point of less than 500 °C at standard pressure, the crosslinking reagent having one of the following structures:



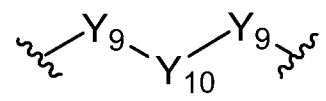
where Y₁, Y₂, Y₃, Y₄, Y₅, Y₆, Y₇ and Y₈ ~~are each hydrogen~~ are each independently selected from hydrogen, optionally substituted cyclic, branched or straight chain C₁-C₆ alkyl or aryl; and L is a linker moiety;

wherein for compound (i) L is of Formula A having one of the following structures:



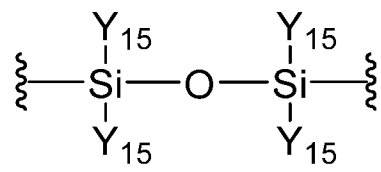
and Y₁₀ is selected from optionally substituted cyclic, branched or straight chain C₁-C₈ alkylene and a siloxane group;

or wherein for compound (i) L is of Formula B having the following formula:



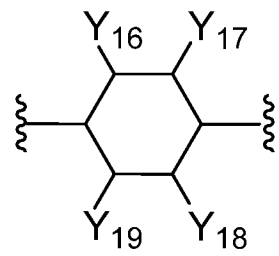
where each Y₉ is independently selected from, a bond, -O-, -O-C(O)-, -C(O)-O-, -Y₁₁-O-, C(O)-, -C(O)-O-Y₁₁-, -OY₁₁-, and -Y₁₁O-, where Y₁₁ is an optionally substituted cyclic, branched or straight chain C₁-C₈ alkylene; and

wherein Y₁₀ has the following formula:



and each Y_{15} is independently selected from optionally substituted branched or straight chain C_1 - C_6 alkyl;

or wherein Y_{10} has the following formula:



and Y_{16} to Y_{19} are each independently selected from H and optionally substituted branched or straight chain C_1 - C_8 alkyl or alkenyl.

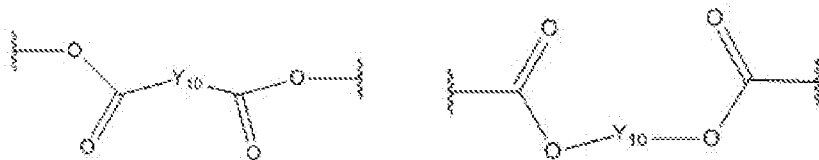
2. (Previously Presented) The device or component according to claim 1, wherein the protective cross-linked polymeric coating is a physical barrier to mass and electron transport.

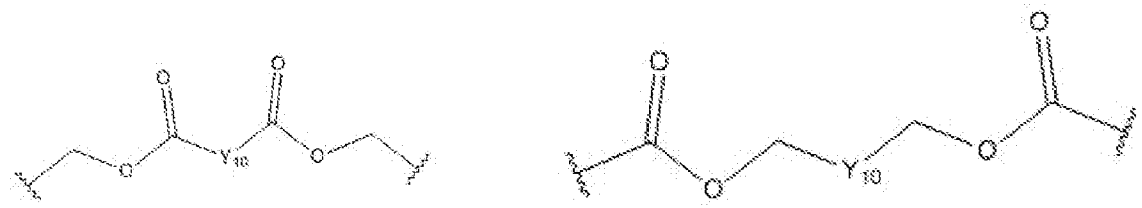
3. (Previously Presented) The device or component according to claim 1, wherein the protective cross-linked polymeric coating forms a liquid repellent surface defined by a static water contact angle (WCA) of at least 90° .

4. (Cancelled)

5. (Cancelled)

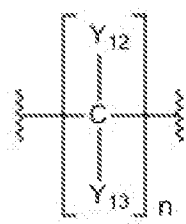
6. (Currently Amended) The device or component according to ~~claim 5~~ claim 1, wherein when for compound (i) L is of the formula B, L has one of the following structures:





7. (Cancelled)

8. (Currently Amended) The device or component according to ~~claim 5~~ claim 1, wherein when for compound (i) L is of Formula B, Y_{10} has the following formula:



wherein each Y_{12} and Y_{13} is independently selected from H, halo, optionally substituted cyclic, branched or straight chain alkyl, or $-OY_{14}$, where Y_{14} is selected from optionally substituted branched or straight chain C_1 - C_8 alkyl or alkenyl, and n is an integer from 1 to 10.

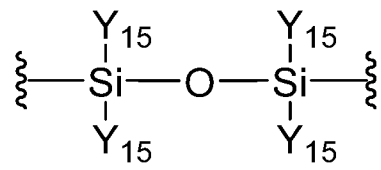
9. (Previously Presented) The device or component according to claim 8, wherein each Y_{12} is H and each Y_{13} is H.

10. (Previously Presented) The device or component according to claim 8, wherein each Y_{12} is fluoro and each Y_{13} is fluoro.

11. (Previously Presented) The device or component claim 8, wherein n is from 4 to 6.

12. (Cancelled)

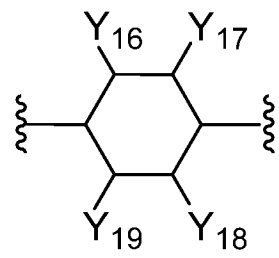
13. (Currently Amended) The device or component according to ~~claim 12~~ claim 1, wherein when for compound (i) L is of Formula B and Y₁₀ has the following formula:



each Y₁₅ is methyl, and each Y₉ is a bond.

14. (Cancelled)

15. (Currently Amended) The device or component according to ~~claim 14~~ claim 1, wherein when for compound (i) L is of Formula B and Y₁₀ has the following formula:

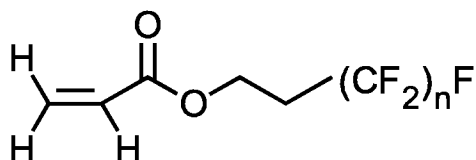


Y₁₈ is H or vinylene, and Y₁₆, Y₁₇ and Y₁₉ are each H.

16. (Currently Amended) The device or component according to claim 1, wherein the crosslinking reagent is selected from ~~Divinyl Adipate~~ divinyl adipate (DVA), ~~1,4-Butanediol~~ 1,4-butanediol divinyl ether (BDVE), ~~1,4-Cyclohexanedimethanol~~ 1,4-cyclohexanedimethanol divinyl ether (CDDE), ~~1,7-Octadiene~~ 1,7-octadiene (17OD), ~~1,2,4-trivinylcyclohexane~~ 1,2,4-trivinylcyclohexane (TVCH), ~~1,3-Divinyltetramethyldisiloxane~~ 1,3-divinyltetramethyldisiloxane (DVTMDS), ~~Diallyl~~ diallyl 1,4-cyclohexanedicarboxylate (DCHD), ~~1,6-Divinylperfluorohexane~~ 1,6-divinylperfluorohexane (DVPFH), ~~1H,1H,6H,6H-Perfluorohexanediol~~ perfluorohexanediol diacrylate (PFHDA) and glyoxal bis (diallyl acetal) (GBDA).

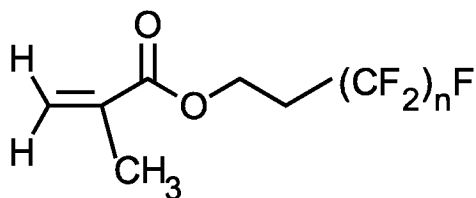
17-30. (Cancelled)

31. (Previously Presented) The device or component according to claim 1, wherein the compound of formula I(a) has the following formula:



where n is from 2 to 10.

32. (Previously Presented) The device or component ~~thereof~~ according to claim 1, wherein the compound of formula I(a) has the following formula:



where n is from 2 to 10.

33. (Previously Presented) The device or component according to claim 31, wherein the compound of formula I(a) is selected from 1H,1H,2H,2H-perfluorohexyl acrylate (PFAC4), 1H,1H,2H,2H-perfluorooctyl acrylate (PFAC6), 1H,1H,2H,2H-perfluorodecyl acrylate (PFAC8) and 1H,1H,2H,2H-perfluorododecyl acrylate (PFAC10).

34. (Previously Presented) The device or component ~~thereof~~ according to claim 32, wherein the compound of formula I(a) is selected from 1H,1H,2H,2H-perfluorohexyl methacrylate (PFMAC4), 1H,1H,2H,2H-perfluorooctyl methacrylate (PFMAC6) and 1H,1H,2H,2H-perfluorodecyl methacrylate (PFMAC8).

35-91. (Cancelled)

Drawing Amendments

Please replace each of original figures 1-7 with each of the respective, enclosed replacement sheets, which shall replace each of original figures 1-7.

Each of figures 1-7 are amended to replace “Figure,” which precedes each of the view numbers, with “Fig.” as requested in the Office Action.

Under the Paperwork Reduction Act of 1995, no persons are required to respond to a collection of information unless it displays a valid OMB control number.

| | | | | | | | | | | | | | |
|---|--|---|--------------|--|---------------|---------------------------|-----------------------|---------------------------------------|---|--|--|--|--|
| PATENT APPLICATION FEE DETERMINATION RECORD Substitute for Form PTO-875 | | | | Application or Docket Number 15/735,130 | | Filing Date 12/08/2017 | | <input type="checkbox"/> To be Mailed | | | | | |
| ENTITY: <input checked="" type="checkbox"/> LARGE <input type="checkbox"/> SMALL <input type="checkbox"/> MICRO | | | | | | | | | | | | | |
| APPLICATION AS FILED - PART I | | | | | | | | | | | | | |
| | | (Column 1) | (Column 2) | | | | | | | | | | |
| FOR | | NUMBER FILED | NUMBER EXTRA | | RATE (\$) | | FEE (\$) | | | | | | |
| <input type="checkbox"/> BASIC FEE (37 CFR 1.16(a), (b), or (c)) | | N/A | N/A | | N/A | | | | | | | | |
| <input type="checkbox"/> SEARCH FEE (37 CFR 1.16(k), (l), or (m)) | | N/A | N/A | | N/A | | | | | | | | |
| <input type="checkbox"/> EXAMINATION FEE (37 CFR 1.16(o), (p), or (q)) | | N/A | N/A | | N/A | | | | | | | | |
| TOTAL CLAIMS (37 CFR 1.16(i)) | | minus 20 = * | | x \$80 = | | | | | | | | | |
| INDEPENDENT CLAIMS (37 CFR 1.16(h)) | | minus 3 = * | | x \$420 = | | | | | | | | | |
| <input type="checkbox"/> APPLICATION SIZE FEE (37 CFR 1.16(s)) | | If the specification and drawings exceed 100 sheets of paper, the application size fee due is \$310 (\$155 for small entity) for each additional 50 sheets or fraction thereof. See 35 U.S.C. 41(a)(1)(G) and 37 CFR 1.16(s). | | | | | | | | | | | |
| <input type="checkbox"/> MULTIPLE DEPENDENT CLAIM PRESENT (37 CFR 1.16(j)) | | | | | | | | | | | | | |
| * If the difference in column 1 is less than zero, enter "0" in column 2. | | | | | TOTAL | | | | | | | | |
| APPLICATION AS AMENDED - PART II | | | | | | | | | | | | | |
| | | (Column 1) | (Column 2) | | (Column 3) | | | | | | | | |
| AMENDMENT | 03/18/2019 | CLAIMS REMAINING AFTER AMENDMENT | | HIGHEST NUMBER PREVIOUSLY PAID FOR | PRESENT EXTRA | RATE (\$) | ADDITIONAL FEE (\$) | | | | | | |
| | Total (37 CFR 1.16(i)) | * 15 | Minus | ** 20 | = 0 | x \$ 100 = | 0 | | | | | | |
| | Independent (37 CFR 1.16(h)) | * 1 | Minus | *** 3 | = 0 | x \$ 460 = | 0 | | | | | | |
| | <input type="checkbox"/> Application Size Fee (37 CFR 1.16(s)) | | | | | | | | | | | | |
| | <input type="checkbox"/> FIRST PRESENTATION OF MULTIPLE DEPENDENT CLAIM (37 CFR 1.16(j)) | | | | | | | | | | | | |
| | | | | | | | TOTAL ADD'L FEE | | 0 | | | | |
| | | (Column 1) | (Column 2) | | (Column 3) | | | | | | | | |
| AMENDMENT | | CLAIMS REMAINING AFTER AMENDMENT | | HIGHEST NUMBER PREVIOUSLY PAID FOR | PRESENT EXTRA | RATE (\$) | ADDITIONAL FEE (\$) | | | | | | |
| | Total (37 CFR 1.16(i)) | * | Minus | ** | = | x \$ 0 = | | | | | | | |
| | Independent (37 CFR 1.16(h)) | * | Minus | *** | = | x \$ 0 = | | | | | | | |
| | <input type="checkbox"/> Application Size Fee (37 CFR 1.16(s)) | | | | | | | | | | | | |
| | <input type="checkbox"/> FIRST PRESENTATION OF MULTIPLE DEPENDENT CLAIM (37 CFR 1.16(j)) | | | | | | | | | | | | |
| | | | | | | | TOTAL ADD'L FEE | | | | | | |
| * If the entry in column 1 is less than the entry in column 2, write "0" in column 3. | | | | | | | LIE | | | | | | |
| ** If the "Highest Number Previously Paid For" IN THIS SPACE is less than 20, enter "20". | | | | | | | /TERRANCE R LAWRENCE/ | | | | | | |
| *** If the "Highest Number Previously Paid For" IN THIS SPACE is less than 3, enter "3". | | | | | | | | | | | | | |
| The "Highest Number Previously Paid For" (Total or Independent) is the highest number found in the appropriate box in column 1. | | | | | | | | | | | | | |

This collection of information is required by 37 CFR 1.16. The information is required to obtain or retain a benefit by the public which is to file (and by the USPTO to process) an application. Confidentiality is governed by 35 U.S.C. 122 and 37 CFR 1.14. This collection is estimated to take 12 minutes to complete, including gathering, preparing, and submitting the completed application form to the USPTO. Time will vary depending upon the individual case. Any comments on the amount of time you require to complete this form and/or suggestions for reducing this burden, should be sent to the Chief Information Officer, U.S. Patent and Trademark Office, U.S. Department of Commerce, P.O. Box 1450, Alexandria, VA 22313-1450. DO NOT SEND FEES OR COMPLETED FORMS TO THIS ADDRESS. **SEND TO: Commissioner for Patents, P.O. Box 1450, Alexandria, VA 22313-1450.**

If you need assistance in completing the form, call 1-800-PTO-9199 and select option 2.

SCORE Placeholder Sheet for IFW Content

Application Number: 15735130

Document Date: 03/18/2019

The presence of this form in the IFW record indicates that the following document type was received in electronic format on the date identified above. This content is stored in the SCORE database.

Since this was an electronic submission, there is no physical artifact folder, no artifact folder is recorded in PALM, and no paper documents or physical media exist. The TIFF images in the IFW record were created from the original documents that are stored in SCORE.

- Drawing

At the time of document entry (noted above):

- USPTO employees may access SCORE content via eDAN using the Supplemental Content tab, or via the SCORE web page.
- External customers may access SCORE content via PAIR using the Supplemental Content tab.



UNITED STATES PATENT AND TRADEMARK OFFICE

UNITED STATES DEPARTMENT OF COMMERCE
United States Patent and Trademark Office
Address: COMMISSIONER FOR PATENTS
P.O. Box 1450
Alexandria, Virginia 22313-1450
www.uspto.gov

NOTICE OF ALLOWANCE AND FEE(S) DUE

33072 7590 05/15/2019
KAGAN BINDER, PLLC
SUITE 200, MAPLE ISLAND BUILDING
221 MAIN STREET NORTH
STILLWATER, MN 55082

EXAMINER

BUIE-HATCHER, NICOLE M

ART UNIT

PAPER NUMBER

1767

DATE MAILED: 05/15/2019

| APPLICATION NO. | FILING DATE | FIRST NAMED INVENTOR | ATTORNEY DOCKET NO. | CONFIRMATION NO. |
|-----------------|-------------|-------------------------|---------------------|------------------|
| 15/735,130 | 12/08/2017 | Stephen Richard COULSON | GBL0007/US | 7101 |

TITLE OF INVENTION: COATINGS

| APPLN. TYPE | ENTITY STATUS | ISSUE FEE DUE | PUBLICATION FEE DUE | PREV. PAID ISSUE FEE | TOTAL FEE(S) DUE | DATE DUE |
|----------------|---------------|---------------|---------------------|----------------------|------------------|------------|
| nonprovisional | UNDISCOUNTED | \$1000 | \$0.00 | \$0.00 | \$1000 | 08/15/2019 |

THE APPLICATION IDENTIFIED ABOVE HAS BEEN EXAMINED AND IS ALLOWED FOR ISSUANCE AS A PATENT. PROSECUTION ON THE MERITS IS CLOSED. THIS NOTICE OF ALLOWANCE IS NOT A GRANT OF PATENT RIGHTS. THIS APPLICATION IS SUBJECT TO WITHDRAWAL FROM ISSUE AT THE INITIATIVE OF THE OFFICE OR UPON PETITION BY THE APPLICANT. SEE 37 CFR 1.313 AND MPEP 1308.

THE ISSUE FEE AND PUBLICATION FEE (IF REQUIRED) MUST BE PAID WITHIN THREE MONTHS FROM THE MAILING DATE OF THIS NOTICE OR THIS APPLICATION SHALL BE REGARDED AS ABANDONED. THIS STATUTORY PERIOD CANNOT BE EXTENDED. SEE 35 U.S.C. 151. THE ISSUE FEE DUE INDICATED ABOVE DOES NOT REFLECT A CREDIT FOR ANY PREVIOUSLY PAID ISSUE FEE IN THIS APPLICATION. IF AN ISSUE FEE HAS PREVIOUSLY BEEN PAID IN THIS APPLICATION (AS SHOWN ABOVE), THE RETURN OF PART B OF THIS FORM WILL BE CONSIDERED A REQUEST TO REAPPLY THE PREVIOUSLY PAID ISSUE FEE TOWARD THE ISSUE FEE NOW DUE.

HOW TO REPLY TO THIS NOTICE:

I. Review the ENTITY STATUS shown above. If the ENTITY STATUS is shown as SMALL or MICRO, verify whether entitlement to that entity status still applies.

If the ENTITY STATUS is the same as shown above, pay the TOTAL FEE(S) DUE shown above.

If the ENTITY STATUS is changed from that shown above, on PART B - FEE(S) TRANSMITTAL, complete section number 5 titled "Change in Entity Status (from status indicated above)".

For purposes of this notice, small entity fees are 1/2 the amount of undiscounted fees, and micro entity fees are 1/2 the amount of small entity fees.

II. PART B - FEE(S) TRANSMITTAL, or its equivalent, must be completed and returned to the United States Patent and Trademark Office (USPTO) with your ISSUE FEE and PUBLICATION FEE (if required). If you are charging the fee(s) to your deposit account, section "4b" of Part B - Fee(s) Transmittal should be completed and an extra copy of the form should be submitted. If an equivalent of Part B is filed, a request to reapply a previously paid issue fee must be clearly made, and delays in processing may occur due to the difficulty in recognizing the paper as an equivalent of Part B.

III. All communications regarding this application must give the application number. Please direct all communications prior to issuance to Mail Stop ISSUE FEE unless advised to the contrary.

IMPORTANT REMINDER: Maintenance fees are due in utility patents issuing on applications filed on or after Dec. 12, 1980. It is patentee's responsibility to ensure timely payment of maintenance fees when due. More information is available at www.uspto.gov/PatentMaintenanceFees.

PART B - FEE(S) TRANSMITTAL

Complete and send this form, together with applicable fee(s), by mail or fax, or via EFS-Web.

By mail, send to: Mail Stop ISSUE FEE
 Commissioner for Patents
 P.O. Box 1450
 Alexandria, Virginia 22313-1450

By fax, send to: (571)-273-2885

INSTRUCTIONS: This form should be used for transmitting the ISSUE FEE and PUBLICATION FEE (if required). Blocks 1 through 5 should be completed where appropriate. All further correspondence including the Patent, advance orders and notification of maintenance fees will be mailed to the current correspondence address as indicated unless corrected below or directed otherwise in Block 1, by (a) specifying a new correspondence address; and/or (b) indicating a separate "FEE ADDRESS" for maintenance fee notifications.

CURRENT CORRESPONDENCE ADDRESS (Note: Use Block 1 for any change of address)

Note: A certificate of mailing can only be used for domestic mailings of the Fee(s) Transmittal. This certificate cannot be used for any other accompanying papers. Each additional paper, such as an assignment or formal drawing, must have its own certificate of mailing or transmission.

33072 7590 05/15/2019
KAGAN BINDER, PLLC
SUITE 200, MAPLE ISLAND BUILDING
221 MAIN STREET NORTH
STILLWATER, MN 55082

Certificate of Mailing or Transmission

I hereby certify that this Fee(s) Transmittal is being deposited with the United States Postal Service with sufficient postage for first class mail in an envelope addressed to the Mail Stop ISSUE FEE address above, or being transmitted to the USPTO via EFS-Web or by facsimile to (571) 273-2885, on the date below.

| | |
|--|-------------------------|
| | (Typed or printed name) |
| | (Signature) |
| | (Date) |

| APPLICATION NO. | FILING DATE | FIRST NAMED INVENTOR | ATTORNEY DOCKET NO. | CONFIRMATION NO. |
|-----------------|-------------|-------------------------|---------------------|------------------|
| 15/735,130 | 12/08/2017 | Stephen Richard COULSON | GBL0007/US | 7101 |

TITLE OF INVENTION: COATINGS

| APPLN. TYPE | ENTITY STATUS | ISSUE FEE DUE | PUBLICATION FEE DUE | PREV. PAID ISSUE FEE | TOTAL FEE(S) DUE | DATE DUE |
|----------------|---------------|---------------|---------------------|----------------------|------------------|------------|
| nonprovisional | UNDISCOUNTED | \$1000 | \$0.00 | \$0.00 | \$1000 | 08/15/2019 |

| EXAMINER | ART UNIT | CLASS-SUBCLASS |
|------------------------|----------|----------------|
| BUIE-HATCHER, NICOLE M | 1767 | 526-245000 |

1. Change of correspondence address or indication of "Fee Address" (37 CFR 1.363).

☐ Change of correspondence address (or Change of Correspondence Address form PTO/SB/122) attached.

☐ "Fee Address" indication (or "Fee Address" Indication form PTO/SB/47; Rev 03-09 or more recent) attached. **Use of a Customer Number is required.**

2. For printing on the patent front page, list

(1) The names of up to 3 registered patent attorneys or agents OR, alternatively,

1 _____

(2) The name of a single firm (having as a member a registered attorney or agent) and the names of up to 2 registered patent attorneys or agents. If no name is listed, no name will be printed.

2 _____

3 _____

3. ASSIGNEE NAME AND RESIDENCE DATA TO BE PRINTED ON THE PATENT (print or type)

PLEASE NOTE: Unless an assignee is identified below, no assignee data will appear on the patent. If an assignee is identified below, the document must have been previously recorded, or filed for recordation, as set forth in 37 CFR 3.11 and 37 CFR 3.81(a). Completion of this form is NOT a substitute for filing an assignment.

(A) NAME OF ASSIGNEE

(B) RESIDENCE: (CITY and STATE OR COUNTRY)

Please check the appropriate assignee category or categories (will not be printed on the patent) : ☐ Individual ☐ Corporation or other private group entity ☐ Government

4a. Fees submitted: ☐ Issue Fee ☐ Publication Fee (if required) ☐ Advance Order - # of Copies _____

4b. Method of Payment: (Please first reapply any previously paid fee shown above)

☐ Electronic Payment via EFS-Web ☐ Enclosed check ☐ Non-electronic payment by credit card (Attach form PTO-2038)

☐ The Director is hereby authorized to charge the required fee(s), any deficiency, or credit any overpayment to Deposit Account No. _____

5. **Change in Entity Status** (from status indicated above)

☐ Applicant certifying micro entity status. See 37 CFR 1.29

☐ Applicant asserting small entity status. See 37 CFR 1.27

☐ Applicant changing to regular undiscounted fee status.

NOTE: Absent a valid certification of Micro Entity Status (see forms PTO/SB/15A and 15B), issue fee payment in the micro entity amount will not be accepted at the risk of application abandonment.

NOTE: If the application was previously under micro entity status, checking this box will be taken to be a notification of loss of entitlement to micro entity status.

NOTE: Checking this box will be taken to be a notification of loss of entitlement to small or micro entity status, as applicable.

NOTE: This form must be signed in accordance with 37 CFR 1.31 and 1.33. See 37 CFR 1.4 for signature requirements and certifications.

Authorized Signature _____

Date _____

Typed or printed name _____

Registration No. _____



UNITED STATES PATENT AND TRADEMARK OFFICE

UNITED STATES DEPARTMENT OF COMMERCE
United States Patent and Trademark Office
Address: COMMISSIONER FOR PATENTS
P.O. Box 1450
Alexandria, Virginia 22313-1450
www.uspto.gov

| APPLICATION NO. | FILING DATE | FIRST NAMED INVENTOR | ATTORNEY DOCKET NO. | CONFIRMATION NO. |
|---|-------------|-------------------------|------------------------|------------------|
| 15/735,130 | 12/08/2017 | Stephen Richard COULSON | GBL0007/US | 7101 |
| 33072 | 7590 | 05/15/2019 | EXAMINER | |
| KAGAN BINDER, PLLC SUITE 200, MAPLE ISLAND BUILDING 221 MAIN STREET NORTH STILLWATER, MN 55082 | | | BUIE-HATCHER, NICOLE M | |
| | | | ART UNIT | PAPER NUMBER |
| | | | 1767 | |

DATE MAILED: 05/15/2019

Determination of Patent Term Adjustment under 35 U.S.C. 154 (b) (Applications filed on or after May 29, 2000)

The Office has discontinued providing a Patent Term Adjustment (PTA) calculation with the Notice of Allowance.

Section 1(h)(2) of the AIA Technical Corrections Act amended 35 U.S.C. 154(b)(3)(B)(i) to eliminate the requirement that the Office provide a patent term adjustment determination with the notice of allowance. See Revisions to Patent Term Adjustment, 78 Fed. Reg. 19416, 19417 (Apr. 1, 2013). Therefore, the Office is no longer providing an initial patent term adjustment determination with the notice of allowance. The Office will continue to provide a patent term adjustment determination with the Issue Notification Letter that is mailed to applicant approximately three weeks prior to the issue date of the patent, and will include the patent term adjustment on the patent. Any request for reconsideration of the patent term adjustment determination (or reinstatement of patent term adjustment) should follow the process outlined in 37 CFR 1.705.

Any questions regarding the Patent Term Extension or Adjustment determination should be directed to the Office of Patent Legal Administration at (571)-272-7702. Questions relating to issue and publication fee payments should be directed to the Customer Service Center of the Office of Patent Publication at 1-(888)-786-0101 or (571)-272-4200.

OMB Clearance and PRA Burden Statement for PTOL-85 Part B

The Paperwork Reduction Act (PRA) of 1995 requires Federal agencies to obtain Office of Management and Budget approval before requesting most types of information from the public. When OMB approves an agency request to collect information from the public, OMB (i) provides a valid OMB Control Number and expiration date for the agency to display on the instrument that will be used to collect the information and (ii) requires the agency to inform the public about the OMB Control Number's legal significance in accordance with 5 CFR 1320.5(b).

The information collected by PTOL-85 Part B is required by 37 CFR 1.311. The information is required to obtain or retain a benefit by the public which is to file (and by the USPTO to process) an application. Confidentiality is governed by 35 U.S.C. 122 and 37 CFR 1.14. This collection is estimated to take 30 minutes to complete, including gathering, preparing, and submitting the completed application form to the USPTO. Time will vary depending upon the individual case. Any comments on the amount of time you require to complete this form and/or suggestions for reducing this burden, should be sent to the Chief Information Officer, U.S. Patent and Trademark Office, U.S. Department of Commerce, P.O. Box 1450, Alexandria, Virginia 22313-1450. DO NOT SEND FEES OR COMPLETED FORMS TO THIS ADDRESS. SEND TO: Commissioner for Patents, P.O. Box 1450, Alexandria, Virginia 22313-1450. Under the Paperwork Reduction Act of 1995, no persons are required to respond to a collection of information unless it displays a valid OMB control number.

Privacy Act Statement

The Privacy Act of 1974 (P.L. 93-579) requires that you be given certain information in connection with your submission of the attached form related to a patent application or patent. Accordingly, pursuant to the requirements of the Act, please be advised that: (1) the general authority for the collection of this information is 35 U.S.C. 2(b)(2); (2) furnishing of the information solicited is voluntary; and (3) the principal purpose for which the information is used by the U.S. Patent and Trademark Office is to process and/or examine your submission related to a patent application or patent. If you do not furnish the requested information, the U.S. Patent and Trademark Office may not be able to process and/or examine your submission, which may result in termination of proceedings or abandonment of the application or expiration of the patent.

The information provided by you in this form will be subject to the following routine uses:

1. The information on this form will be treated confidentially to the extent allowed under the Freedom of Information Act (5 U.S.C. 552) and the Privacy Act (5 U.S.C. 552a). Records from this system of records may be disclosed to the Department of Justice to determine whether disclosure of these records is required by the Freedom of Information Act.
2. A record from this system of records may be disclosed, as a routine use, in the course of presenting evidence to a court, magistrate, or administrative tribunal, including disclosures to opposing counsel in the course of settlement negotiations.
3. A record in this system of records may be disclosed, as a routine use, to a Member of Congress submitting a request involving an individual, to whom the record pertains, when the individual has requested assistance from the Member with respect to the subject matter of the record.
4. A record in this system of records may be disclosed, as a routine use, to a contractor of the Agency having need for the information in order to perform a contract. Recipients of information shall be required to comply with the requirements of the Privacy Act of 1974, as amended, pursuant to 5 U.S.C. 552a(m).
5. A record related to an International Application filed under the Patent Cooperation Treaty in this system of records may be disclosed, as a routine use, to the International Bureau of the World Intellectual Property Organization, pursuant to the Patent Cooperation Treaty.
6. A record in this system of records may be disclosed, as a routine use, to another federal agency for purposes of National Security review (35 U.S.C. 181) and for review pursuant to the Atomic Energy Act (42 U.S.C. 218(c)).
7. A record from this system of records may be disclosed, as a routine use, to the Administrator, General Services, or his/her designee, during an inspection of records conducted by GSA as part of that agency's responsibility to recommend improvements in records management practices and programs, under authority of 44 U.S.C. 2904 and 2906. Such disclosure shall be made in accordance with the GSA regulations governing inspection of records for this purpose, and any other relevant (i.e., GSA or Commerce) directive. Such disclosure shall not be used to make determinations about individuals.
8. A record from this system of records may be disclosed, as a routine use, to the public after either publication of the application pursuant to 35 U.S.C. 122(b) or issuance of a patent pursuant to 35 U.S.C. 151. Further, a record may be disclosed, subject to the limitations of 37 CFR 1.14, as a routine use, to the public if the record was filed in an application which became abandoned or in which the proceedings were terminated and which application is referenced by either a published application, an application open to public inspection or an issued patent.
9. A record from this system of records may be disclosed, as a routine use, to a Federal, State, or local law enforcement agency, if the USPTO becomes aware of a violation or potential violation of law or regulation.

| | | | |
|-------------------------------|--|---------------------------------------|---------------------------------|
| Notice of Allowability | Application No. 15/735,130 | Applicant(s) COULSON et al. | |
| | Examiner NICOLE M BUIE-HATCHER | Art Unit 1767 | AIA (FITF) Status Yes |

-- The MAILING DATE of this communication appears on the cover sheet with the correspondence address--

All claims being allowable, PROSECUTION ON THE MERITS IS (OR REMAINS) CLOSED in this application. If not included herewith (or previously mailed), a Notice of Allowance (PTOL-85) or other appropriate communication will be mailed in due course. **THIS NOTICE OF ALLOWABILITY IS NOT A GRANT OF PATENT RIGHTS.** This application is subject to withdrawal from issue at the initiative of the Office or upon petition by the applicant. See 37 CFR 1.313 and MPEP 1308.

1. ☒ This communication is responsive to response filed 03/18/2019.
☐ A declaration(s)/affidavit(s) under **37 CFR 1.130(b)** was/were filed on ____.

2. ☐ An election was made by the applicant in response to a restriction requirement set forth during the interview on ____; the restriction requirement and election have been incorporated into this action.

3. ☒ The allowed claim(s) is/are See Continuation Sheet. As a result of the allowed claim(s), you may be eligible to benefit from the **Patent Prosecution Highway** program at a participating intellectual property office for the corresponding application. For more information, please see http://www.uspto.gov/patents/init_events/pph/index.jsp or send an inquiry to **PPHfeedback@uspto.gov**.

4. ☐ Acknowledgment is made of a claim for foreign priority under 35 U.S.C. § 119(a)-(d) or (f).

Certified copies:

a) ☐ All b) ☐ Some *c) ☐ None of the:

1. ☐ Certified copies of the priority documents have been received.

2. ☐ Certified copies of the priority documents have been received in Application No. ____.

3. ☐ Copies of the certified copies of the priority documents have been received in this national stage application from the International Bureau (PCT Rule 17.2(a)).

* Certified copies not received: ____.

Applicant has THREE MONTHS FROM THE "MAILING DATE" of this communication to file areply complying with the requirements noted below. Failure to timely comply will result in ABANDONMENT of this application.
THIS THREE-MONTH PERIOD IS NOT EXTENDABLE.

5. ☐ CORRECTED DRAWINGS (as "replacement sheets") must be submitted.
☐ including changes required by the attached Examiner's Amendment / Comment or in the Office action of Paper No./Mail Date ____.

Identifying indicia such as the application number (see 37 CFR 1.84(c)) should be written on the drawings in the front (not the back) of each sheet. Replacement sheet(s) should be labeled as such in the header according to 37 CFR 1.121(d).

6. ☐ DEPOSIT OF and/or INFORMATION about the deposit of BIOLOGICAL MATERIAL must be submitted. Note the attached Examiner's comment regarding REQUIREMENT FOR THE DEPOSIT OF BIOLOGICAL MATERIAL.

Attachment(s)

| | |
|--|---|
| 1. <input checked="" type="checkbox"/> Notice of References Cited (PTO-892) 2. <input checked="" type="checkbox"/> Information Disclosure Statements (PTO/SB/08), Paper No./Mail Date <u>20181121 / 20190318</u> . 3. <input type="checkbox"/> Examiner's Comment Regarding Requirement for Deposit of Biological Material _____. 4. <input checked="" type="checkbox"/> Interview Summary (PTO-413), Paper No./Mail Date. <u>20190430</u> . | 5. <input checked="" type="checkbox"/> Examiner's Amendment/Comment 6. <input checked="" type="checkbox"/> Examiner's Statement of Reasons for Allowance 7. <input type="checkbox"/> Other _____. |
|--|---|

| | |
|---|--|
| /NICOLE M. BUIE-HATCHER/ Primary Examiner, Art Unit 1767 | |
|---|--|

Continuation of 3. The allowed claim(s) is/are: 1-3,6,8-11,13,15-16 and 31-34

Notice of Pre-AIA or AIA Status

The present application, filed on or after March 16, 2013, is being examined under the first inventor to file provisions of the AIA.

EXAMINER'S AMENDMENT

An examiner's amendment to the record appears below. Should the changes and/or additions be unacceptable to applicant, an amendment may be filed as provided by 37 CFR 1.312. To ensure consideration of such an amendment, it **MUST** be submitted no later than the payment of the issue fee.

Authorization for this examiner's amendment was given in an interview with Paul Parins (Reg. No. 54,358) on 05/09/2019.

The application has been amended as follows:

In claim 1, line 23, replace "Formula A" with "formula A".

In claim 1, line 27, replace "Formula B" with "formula B".

In claim 8, line 1, replace "whereinwhen" with "wherein when".

In claim 8, line 2, replace "Formula B" with "formula A".

In claim 13, line 2, replace "Formula B" with "formula B".

In claim 15, line 2, replace "Formula B" with "formula B".

Reasons for Allowance

The following is an examiner's statement of reasons for allowance:

A) The closest prior art of record, Coulson (WO 2007/082122 A1) does not teach or suggest the crosslinking reagent comprises two or more unsaturated bonds attached by means of one or more linker moieties and has a boiling point of less than 500°C at standard pressure, the crosslinking reagent having one of the structures (i) or (ii).

B) Coulson (US 2010/0203347 A1) teaches a compound with more than one double bond [0030] and alkyne compounds comprise chains of carbon atoms, including one or more carbon-carbon triple bonds [0046]. However, Coulson (US '347) does not teach or suggest the crosslinking reagent comprises two or more unsaturated bonds attached by means of one or more linker moieties and has a boiling point of less than 500°C at standard pressure, the crosslinking reagent having one of the structures (i) or (ii).

Any comments considered necessary by applicant must be submitted no later than the payment of the issue fee and, to avoid processing delays, should preferably accompany the issue fee. Such submissions should be clearly labeled “Comments on Statement of Reasons for Allowance.”

Prior Art

The prior art made of record and not relied upon is considered pertinent to applicant's disclosure. Chen et al. (CN 103881013 A, Machine translation attached) teaches a fluorine containing hydrogel.

Correspondence

Any inquiry concerning this communication or earlier communications from the examiner should be directed to NICOLE M. BUIE-HATCHER whose telephone number is (571)270-3879. The examiner can normally be reached on M-F, 8 am - 4:30 pm.

Examiner interviews are available via telephone, in-person, and video conferencing using a USPTO supplied web-based collaboration tool. To schedule an interview, applicant is encouraged to use the USPTO Automated Interview Request (AIR) at <http://www.uspto.gov/interviewpractice>.

If attempts to reach the examiner by telephone are unsuccessful, the examiner's supervisor, Mark Eashoo can be reached on (571)272-1197. The fax phone number for the organization where this application or proceeding is assigned is 571-273-8300.

Information regarding the status of an application may be obtained from the Patent Application Information Retrieval (PAIR) system. Status information for published applications may be obtained from either Private PAIR or Public PAIR. Status information for unpublished applications is available through Private PAIR only. For more information about the PAIR system, see <http://pair-direct.uspto.gov>. Should you have questions on access to the Private PAIR system, contact the Electronic Business Center (EBC) at 866-217-9197 (toll-free). If you would like assistance from a USPTO Customer Service Representative or access to the automated information system, call 800-786-9199 (IN USA OR CANADA) or 571-272-1000.

/NICOLE M. BUIE-HATCHER/
Primary Examiner, Art Unit 1767

| | | | | |
|--|---------------------------------------|--|--|-------------|
| <i>Notice of References Cited</i> | Application/Control No. 15/735,130 | | Applicant(s)/Patent Under Reexamination COULSON et al. | |
| | Examiner NICOLE M BUIE-HATCHER | | Art Unit 1767 | Page 1 of 1 |

U.S. PATENT DOCUMENTS

| * | | Document Number Code-Number-Kind Code | Country Code | Date MM-YYYY | Name | CPC Classification | US Classification |
|---|---|--|-----------------|-----------------|------|--------------------|-------------------|
| | A | | | | | | |
| | B | | | | | | |
| | C | | | | | | |
| | D | | | | | | |
| | E | | | | | | |
| | F | | | | | | |
| | G | | | | | | |
| | H | | | | | | |
| | I | | | | | | |
| | J | | | | | | |
| | K | | | | | | |
| | L | | | | | | |
| | M | | | | | | |

FOREIGN PATENT DOCUMENTS

| * | | Document Number Code-Number-Kind Code | Country Code | Date MM-YYYY | Country | Name | CPC Classification |
|---|---|--|-----------------|-----------------|---------|-------------|--------------------|
| | N | CN-103881013-A | | 06-2014 | CN | Chen et al. | |
| | O | | | | | | |
| | P | | | | | | |
| | Q | | | | | | |
| | R | | | | | | |
| | S | | | | | | |
| | T | | | | | | |

NON-PATENT DOCUMENTS


| * | | Include as applicable: Author, Title Date, Publisher, Edition or Volume, Pertinent Pages) |
|---|---|---|
| | U | Machine translation of CN 103881013 A, retrived April 2019. (Year: 2019) |
| | V | |
| | W | |
| | X | |

*A copy of this reference is not being furnished with this Office action. (See MPEP § 707.05(a).)
Dates in MM-YYYY format are publication dates. Classifications may be US or foreign.

U.S. Patent and Trademark Office
PTO-892 (Rev. 01-2001)

Notice of References Cited

Part of Paper No. 20190430

| | | |
|---|--|--|
| <i>Search Notes</i>  | Application/Control No. 15/735,130 | Applicant(s)/Patent Under Reexamination COULSON et al. |
| | Examiner NICOLE M BUIE-HATCHER | Art Unit 1767 |

| CPC - Searched* | | |
|------------------|------------|----------|
| Symbol | Date | Examiner |
| C09D 133/16 | 05/09/2019 | NMB |
| B05D 1/62, 5/083 | 05/09/2019 | NMB |
| H01L 21/0274 | 05/09/2019 | NMB |

| CPC Combination Sets - Searched* | | |
|----------------------------------|------|----------|
| Symbol | Date | Examiner |
| | | |


| US Classification - Searched* | | | |
|-------------------------------|----------|------|----------|
| Class | Subclass | Date | Examiner |
| | | | |

* See search history printout included with this form or the SEARCH NOTES box below to determine the scope of the search.


| Search Notes | | |
|--------------------------------------|------------|----------|
| Search Notes | Date | Examiner |
| Inventor name search, PALM and EAST | 03/26/2019 | NMB |
| Global Dossier | 04/30/2019 | NMB |
| Scifinder | 04/30/2019 | NMB |
| EAST text search, see search history | 04/30/2019 | NMB |

| Interference Search | | | |
|---------------------|-----------------------|------------|----------|
| US Class/CPC Symbol | US Subclass/CPC Group | Date | Examiner |
| C09D | 133/16 | 05/09/2019 | NMB |
| B05D | 1/62 , 5/083 | 05/09/2019 | NMB |
| H01L | 21/0274 | 05/09/2019 | NMB |

| | |
|--|--|
| | |
|--|--|

| | | |
|---|--|--|
| <i>Search Notes</i>  | Application/Control No. 15/735,130 | Applicant(s)/Patent Under Reexamination COULSON et al. |
| | Examiner NICOLE M BUIE-HATCHER | Art Unit 1767 |


| | |
|--|--|
| | |
|--|--|

| | | | |
|--|--|--|--|
| Issue Classification  | Application/Control No. 15/735,130 | Applicant(s)/Patent Under Reexamination COULSON et al. | |
| | Examiner NICOLE M BUIE-HATCHER | Art Unit 1767 | |

| CPC | | | | | | |
|--------|---|------|---|-------|------|------------|
| Symbol | | | | | Type | Version |
| C09D | / | 133 | / | 16 | F | 2013-01-01 |
| C09D | / | 5 | / | 00 | I | 2013-01-01 |
| B05D | / | 5 | / | 08 | I | 2013-01-01 |
| B05D | / | 1 | / | 62 | I | 2013-01-01 |
| B05D | / | 5 | / | 083 | I | 2013-01-01 |
| H01J | / | 37 | / | 32082 | I | 2013-01-01 |
| H01J | / | 37 | / | 32467 | I | 2013-01-01 |
| H01J | / | 2237 | / | 332 | A | 2013-01-01 |
| H01J | / | 2237 | / | 3382 | A | 2013-01-01 |
| H01L | / | 21 | / | 02118 | A | 2013-01-01 |
| H01L | / | 21 | / | 02274 | A | 2013-01-01 |
| H01L | / | 21 | / | 56 | A | 2013-01-01 |
| H01L | / | 23 | / | 293 | A | 2013-01-01 |
| H01L | / | 23 | / | 564 | A | 2013-01-01 |
| H05K | / | 3 | / | 285 | A | 2013-01-01 |

| CPC Combination Sets | | | | |
|----------------------|------|-----|---------|---------|
| Symbol | Type | Set | Ranking | Version |
| / | | | | |

| | | | |
|--|--|------------------------------------|---------------------------|
| NONE (Assistant Examiner) _____ (Date) _____ | | Total Claims Allowed: 15 | |
| /NICOLE M. BUIE-HATCHER/ Primary Examiner, Art Unit 1767 (Primary Examiner) _____ (Date) _____ | | O.G. Print Claim(s) 1 | O.G. Print Figure none |

| | | | |
|--|--|--|--|
| Issue Classification  | Application/Control No. 15/735,130 | Applicant(s)/Patent Under Reexamination COULSON et al. | |
| | Examiner NICOLE M BUIE-HATCHER | Art Unit 1767 | |


| INTERNATIONAL CLASSIFICATION | | | | |
|------------------------------|---|-----|---|----|
| CLAIMED | | | | |
| C09D133/16 | / | 133 | / | 16 |
| C09D5/00 | / | 5 | / | 00 |
| B05D5/08 | / | 5 | / | 08 |
| B05D1/00 | / | 1 | / | 00 |
| H01L21/02 | / | 21 | / | 02 |
| H01L21/56 | / | 21 | / | 56 |
| H01L23/29 | / | 23 | / | 29 |
| H01L23/00 | / | 23 | / | 00 |
| H05K3/28 | / | 3 | / | 28 |

| NON-CLAIMED | | | | |
|-------------|---|----|---|----|
| H01J37/32 | / | 37 | / | 32 |

| US ORIGINAL CLASSIFICATION | |
|----------------------------|----------|
| CLASS | SUBCLASS |
| | |

| CROSS REFERENCES(S) | | | | | | |
|---------------------|-----------------------------------|--|--|--|--|--|
| CLASS | SUBCLASS (ONE SUBCLASS PER BLOCK) | | | | | |
| | | | | | | |

| | | | |
|---|-------------|------------------------------|-------------------|
| NONE | | Total Claims Allowed: | |
| (Assistant Examiner) | (Date) | 15 | |
| /NICOLE M. BUIE-HATCHER/ Primary Examiner, Art Unit 1767 | 09 May 2019 | O.G. Print Claim(s) | O.G. Print Figure |
| (Primary Examiner) | (Date) | 1 | none |

| | | |
|--|--|--|
| Issue Classification  | Application/Control No. 15/735,130 | Applicant(s)/Patent Under Reexamination COULSON et al. |
| | Examiner NICOLE M BUIE-HATCHER | Art Unit 1767 |

☐ Claims renumbered in the same order as presented by applicant
 ☐ CPA
 ☐ T.D.
 ☐ R.1.47

| CLAIMS | | | | | | | | | | | | | | | |
|--------|----------|-------|----------|-------|----------|-------|----------|-------|----------|-------|----------|-------|----------|-------|----------|
| Final | Original | Final | Original | Final | Original | Final | Original | Final | Original | Final | Original | Final | Original | Final | Original |
| 1 | 1 | 9 | 13 | - | 25 | - | 37 | - | 49 | - | 61 | - | 73 | - | 85 |
| 2 | 2 | - | 14 | - | 26 | - | 38 | - | 50 | - | 62 | - | 74 | - | 86 |
| 3 | 3 | 10 | 15 | - | 27 | - | 39 | - | 51 | - | 63 | - | 75 | - | 87 |
| - | 4 | 11 | 16 | - | 28 | - | 40 | - | 52 | - | 64 | - | 76 | - | 88 |
| - | 5 | - | 17 | - | 29 | - | 41 | - | 53 | - | 65 | - | 77 | - | 89 |
| 4 | 6 | - | 18 | - | 30 | - | 42 | - | 54 | - | 66 | - | 78 | - | 90 |
| - | 7 | - | 19 | 12 | 31 | - | 43 | - | 55 | - | 67 | - | 79 | - | 91 |
| 5 | 8 | - | 20 | 14 | 32 | - | 44 | - | 56 | - | 68 | - | 80 | | |
| 6 | 9 | - | 21 | 13 | 33 | - | 45 | - | 57 | - | 69 | - | 81 | | |
| 7 | 10 | - | 22 | 15 | 34 | - | 46 | - | 58 | - | 70 | - | 82 | | |
| 8 | 11 | - | 23 | - | 35 | - | 47 | - | 59 | - | 71 | - | 83 | | |
| - | 12 | - | 24 | - | 36 | - | 48 | - | 60 | - | 72 | - | 84 | | |

| | | | |
|--|--|------------------------------------|---------------------------|
| NONE (Assistant Examiner) _____ (Date) _____ | | Total Claims Allowed: 15 | |
| /NICOLE M. BUIE-HATCHER/ Primary Examiner, Art Unit 1767 (Primary Examiner) _____ (Date) 09 May 2019 | | O.G. Print Claim(s) 1 | O.G. Print Figure none |

EAST Search History

EAST Search History (Prior Art)

| Ref # | Hits | Search Query | DBs | Default Operator | Plurals | Time Stamp |
|-------|------|---|--|------------------|---------|------------------|
| S32 | 5 | ("9617353").PN. | US-PGPUB; USPAT; USOCR; FPRS; EPO; JPO; DERWENT | OR | OFF | 2019/03/26 17:33 |
| S33 | 13 | ("20040028931" "20040046165" "20090267489" "20100189914" "20120009231" "20120051018" "20120171386" "20120321775" "20140010969" "20140322525" "20150291830" "4822632" "6764758").PN. | US-PGPUB; USPAT | OR | ON | 2019/03/26 17:38 |
| S34 | 2 | ("6764758").PN. | US-PGPUB; USPAT; USOCR; FPRS; EPO; JPO; DERWENT | OR | OFF | 2019/03/26 17:56 |
| S35 | 14 | ("4822632" "20140010969" "20120321776" "20120051018" "20140010969" "20120321776" "20120171386" "4822632").pn. | US-PGPUB; USPAT; USOCR; FPRS; EPO; JPO; DERWENT | OR | ON | 2019/04/29 17:01 |
| S41 | 832 | (c09d133/16 b05d5/083).cpc. AND b05d1/62.cpc. | US-PGPUB; USPAT; USOCR; FPRS; EPO; JPO; DERWENT | OR | ON | 2019/04/30 10:23 |
| S40 | 0 | (c09d133/16 b05d5/083).cpc. SAME b05d1/62.cpc. | US-PGPUB; USPAT; USOCR; FPRS; EPO; JPO; DERWENT | OR | ON | 2019/04/30 10:23 |
| S42 | 12 | (c09d133/16 b05d5/083).cpc. AND h01I21/0274.cpc. | US-PGPUB; USPAT; USOCR; FPRS; EPO; JPO; DERWENT | OR | ON | 2019/04/30 10:25 |
| S44 | 75 | S43 AND (coulson-s\$ evans-d\$ siokou-a\$ | US- | OR | ON | 2019/04/30 |

| | | | | | | |
|-----|--------|---|--|----|----|---------------------|
| | | telford-c\$.in. | PGPUB; USPAT; USOCR; FPRS; EPO; JPO; DERWENT | | | 16:37 |
| S43 | 365816 | (((\$3fluor\$4 fluorin\$4) near5 (monomer\$3 acryl\$4 methacryl\$4)) or (\$3fluor\$4 fluorin\$4) near5 olefin or (\$3fluor\$4 fluorin\$4) near5 (alken\$2 alkenylene vinyl) \$3fluoroolefin\$2 \$3fluoroalkene\$2 \$3fluoroalkenyl\$2 \$3fluoromonomer\$2 \$3fluoroalkylene\$2 polyfluoroalkyl polyfluoroalkenyl \$3fluorocarbon\$2 \$3fluoro\$vinyl\$2 \$3fluoromethacryl\$4 \$3fluoroacryl\$4 | US- PGPUB; USPAT; FPRS; EPO; JPO; DERWENT | OR | ON | 2019/04/30 16:37 |
| S45 | 17 | S43 AND "p2i".as. | US- PGPUB; USPAT; USOCR; FPRS; EPO; JPO; DERWENT | OR | ON | 2019/04/30 16:45 |
| S46 | 2 | cn-103881013-\$.did. | US- PGPUB; USPAT; USOCR; FPRS; EPO; JPO; DERWENT | OR | ON | 2019/04/30 18:27 |

EAST Search History (Interference)

| Ref # | Hits | Search Query | DBs | Default Operator | Plurals | Time Stamp |
|-------|---------|---|------------------------|------------------|---------|---------------------|
| L3 | 232749 | (((\$3fluor\$4 fluorin\$4) near5 (monomer\$3 acryl\$4 methacryl\$4)) or (\$3fluor\$4 fluorin\$4) near5 olefin or (\$3fluor\$4 fluorin\$4) near5 (alken\$2 alkenylene vinyl) \$3fluoroolefin\$2 \$3fluoroalkene\$2 \$3fluoroalkenyl\$2 \$3fluoromonomer\$2 \$3fluoroalkylene\$2 polyfluoroalkyl polyfluoroalkenyl \$3fluorocarbon\$2 \$3fluoro\$vinyl\$2 \$3fluoromethacryl\$4 \$3fluoroacryl\$4 | US- PGPUB; USPAT | OR | ON | 2019/05/09 16:55 |
| L2 | 1250650 | crosslink\$6 (cross ADJ link\$6) curable cured curing vulcaniz\$6 | US- PGPUB; USPAT | OR | ON | 2019/05/09 16:55 |
| L1 | 29561 | (divinyl WITH adipate) (butanediol WITH divinyl WITH ether) (cyclohexanedimethanol WITH divinyl WITH ether) (octadiene) trivinylcyclohexane divinyltetramethyldisiloxane (diallyl WITH cyclohexanedicarboxylate) divinylperfluorohexane (perfluorohexanediol WITH diacrylate) (glyoxal WITH bis WITH diallyl WITH acetal) dialkyn\$2 ((di bis) NEAR2 alkyn\$2) | US- PGPUB; USPAT | OR | ON | 2019/05/09 16:55 |
| L4 | 129 | L1 SAME L2 SAME L3 | US- PGPUB; USPAT | OR | ON | 2019/05/09 17:02 |

| | | | | | | |
|----|----|-----------------------------------|-----------------|----|----|------------------|
| L6 | 29 | L1 SAME3 L2 SAME3 L3 SAME3 plasma | US-PGPUB; USPAT | OR | ON | 2019/05/09 17:03 |
| L5 | 3 | L1 SAME L2 SAME L3 SAME plasma | US-PGPUB; USPAT | OR | ON | 2019/05/09 17:03 |

5/ 9/ 2019 5:06:28 PM
C:\ Users\ nbuie\ Documents\ EAST\ Workspaces\ 15735130.wsp

Doc code: IDS

PTO/SB/08a (03-15)

Doc description: Information Disclosure Statement (IDS) Filed

Approved for use through 07/31/2016. OMB 0651-0031

U.S. Patent and Trademark Office; U.S. DEPARTMENT OF COMMERCE

Under the Paperwork Reduction Act of 1995, no persons are required to respond to a collection of information unless it contains a valid OMB control number.

INFORMATION DISCLOSURE STATEMENT BY APPLICANT (Not for submission under 37 CFR 1.99)

| | |
|------------------------|-------------------------|
| Application Number | 15735130 |
| Filing Date | 2017-12-08 |
| First Named Inventor | Stephen Richard COULSON |
| Art Unit | 1767 |
| Examiner Name | BUIE-HATCHER, NICOLE M |
| Attorney Docket Number | GBL0007/US |

U.S.PATENTS

Remove

| Examiner Initial* | Cite No | Patent Number | Kind Code ¹ | Issue Date | Name of Patentee or Applicant of cited Document | Pages,Columns,Lines where Relevant Passages or Relevant Figures Appear |
|-------------------|---------|---------------|------------------------|------------|---|--|
| /N.M.B./ | 1 | 4822632 | A | 1989-04-18 | Williams et al. | |
| /N.M.B./ | 2 | 6764758 | B1 | 2004-07-20 | Grunze et al. | |

If you wish to add additional U.S. Patent citation information please click the Add button.

Add

U.S.PATENT APPLICATION PUBLICATIONS

Remove

| Examiner Initial* | Cite No | Publication Number | Kind Code ¹ | Publication Date | Name of Patentee or Applicant of cited Document | Pages,Columns,Lines where Relevant Passages or Relevant Figures Appear |
|-------------------|---------|--------------------|------------------------|------------------|---|--|
| /N.M.B./ | 1 | 20040028931 | A1 | 2004-02-12 | Bletsos et al. | |
| /N.M.B./ | 2 | 20120051018 | A1 | 2012-03-01 | Ollgaard | |
| /N.M.B./ | 3 | 20120171386 | A1 | 2012-07-05 | Bicker et al. | |
| /N.M.B./ | 4 | 20120321775 | A1 | 2012-12-20 | Parker | |

INFORMATION DISCLOSURE STATEMENT BY APPLICANT (Not for submission under 37 CFR 1.99)

| | |
|------------------------|-------------------------|
| Application Number | 15735130 |
| Filing Date | 2017-12-08 |
| First Named Inventor | Stephen Richard COULSON |
| Art Unit | 1767 |
| Examiner Name | BUIE-HATCHER, NICOLE M |
| Attorney Docket Number | GBL0007/US |

| | | | | | |
|----------|----|-------------|----|------------|------------------|
| /N.M.B./ | 5 | 20140010969 | A1 | 2014-01-09 | Bicker et al. |
| /N.M.B./ | 6 | 20090267489 | A1 | 2009-10-29 | Arita et al. |
| /N.M.B./ | 7 | 20100189914 | A1 | 2010-07-29 | Coulson |
| /N.M.B./ | 8 | 20120009231 | A1 | 2012-01-12 | Herbert et al. |
| /N.M.B./ | 9 | 20140322525 | A1 | 2014-10-30 | Leguin et al. |
| /N.M.B./ | 10 | 20150291830 | A1 | 2015-10-15 | Galbreath et al. |
| /N.M.B./ | 11 | 20040046165 | A1 | 2004-03-11 | Hunze et al. |

If you wish to add additional U.S. Published Application citation information please click the Add button.

Add

FOREIGN PATENT DOCUMENTS

Remove

| Examiner Initial* | Cite No | Foreign Document Number ³ | Country Code ² | Kind Code ⁴ | Publication Date | Name of Patentee or Applicant of cited Document | Pages, Columns, Lines where Relevant Passages or Relevant Figures Appear | T ⁵ |
|-------------------|---------|--------------------------------------|---------------------------|------------------------|------------------|---|--|----------------|
| /N.M.B./ | 1 | 2434368 | GB | A | 2007-07-25 | P2i Limited | | |
| /N.M.B./ | 2 | 2493264 | GB | A | 2013-01-30 | P2i Ltd | | |

**INFORMATION DISCLOSURE
STATEMENT BY APPLICANT**
(Not for submission under 37 CFR 1.99)

| | | |
|------------------------|-------------------------|------------|
| Application Number | | 15735130 |
| Filing Date | | 2017-12-08 |
| First Named Inventor | Stephen Richard COULSON | |
| Art Unit | 1767 | |
| Examiner Name | BUIE-HATCHER, NICOLE M | |
| Attorney Docket Number | GBL0007/US | |

| | | | | | | | | |
|----------|----|-------------|----|----|------------|---|--|--|
| /N.M.B./ | 3 | 03063938 | WO | A1 | 2003-08-07 | ASTRAZENECA AB | | |
| /N.M.B./ | 4 | 2005021833 | WO | A2 | 2005-03-10 | SURFACE INNOVATIONS LIMITED | | |
| /N.M.B./ | 5 | 2009046093 | WO | A2 | 2009-04-09 | EXOGENESIS CORPORATION | | |
| /N.M.B./ | 6 | 2009030435 | WO | A1 | 2009-03-12 | UNIV AUGSBURG | | |
| /N.M.B./ | 7 | 2011029628 | WO | A1 | 2011-03-17 | SCHOTT AG | | |
| /N.M.B./ | 8 | 2711153 | EP | A1 | 2014-03-26 | The Goodyear Tire and Rubber Company | | |
| /N.M.B./ | 9 | 2005/089961 | WO | A1 | 2005-09-29 | THE SECRETARY OF STATE OF DEFENCE | | |
| /N.M.B./ | 10 | 02/28548 | WO | A2 | 2002-04-11 | DOW CORNING CORPORATION | | |
| /N.M.B./ | 11 | 2013/132250 | WO | A1 | 2013-09-12 | SEMBLANT LTD | | |
| /N.M.B./ | 12 | 2012/158953 | WO | A2 | 2012-11-22 | GADGET IP LLC | | |
| /N.M.B./ | 13 | 2014026967 | WO | A2 | 2014-02-20 | EUROPLASMA NV | | |

INFORMATION DISCLOSURE STATEMENT BY APPLICANT (Not for submission under 37 CFR 1.99)

| | |
|------------------------|-------------------------|
| Application Number | 15735130 |
| Filing Date | 2017-12-08 |
| First Named Inventor | Stephen Richard COULSON |
| Art Unit | 1767 |
| Examiner Name | BUIE-HATCHER, NICOLE M |
| Attorney Docket Number | GBL0007/US |

If you wish to add additional Foreign Patent Document citation information please click the Add button

NON-PATENT LITERATURE DOCUMENTS

| Examiner Initials* | Cite No | Include name of the author (in CAPITAL LETTERS), title of the article (when appropriate), title of the item (book, magazine, journal, serial, symposium, catalog, etc), date, pages(s), volume-issue number(s), publisher, city and/or country where published. | T ⁵ |
|--------------------|---------|---|----------------|
| /N.M.B./ | 1 | Search Report under Section 17(5) from UK Patent Application No. GB1403558.8, mailed on August 13, 2014 (4 pages) | |
| /N.M.B./ | 2 | International Search Report and Written Opinion from International Application No. PCT/GB2015/050521, mailed June 5, 2015 (16 pages) | |
| /N.M.B./ | 3 | Search Report under Section 17(5) from UK Patent Application No. GB1503054.7, mailed on November 30, 2015 (4 pages) | |

If you wish to add additional non-patent literature document citation information please click the Add button

EXAMINER SIGNATURE

| | | | |
|--------------------|--------------------------|-----------------|------------|
| Examiner Signature | /NICOLE M. BUIE-HATCHER/ | Date Considered | 04/29/2019 |
|--------------------|--------------------------|-----------------|------------|

*EXAMINER: Initial if reference considered, whether or not citation is in conformance with MPEP 609. Draw line through a citation if not in conformance and not considered. Include copy of this form with next communication to applicant.

¹ See Kind Codes of USPTO Patent Documents at www.USPTO.GOV or MPEP 901.04. ² Enter office that issued the document, by the two-letter code (WIPO Standard ST.3). ³ For Japanese patent documents, the indication of the year of the reign of the Emperor must precede the serial number of the patent document. ⁴ Kind of document by the appropriate symbols as indicated on the document under WIPO Standard ST.16 if possible. ⁵ Applicant is to place a check mark here if English language translation is attached.

Doc code: IDS

PTO/SB/08a (03-15)

Doc description: Information Disclosure Statement (IDS) Filed

Approved for use through 07/31/2016. OMB 0651-0031

U.S. Patent and Trademark Office; U.S. DEPARTMENT OF COMMERCE

Under the Paperwork Reduction Act of 1995, no persons are required to respond to a collection of information unless it contains a valid OMB control number.

| | | | | |
|---|------------------------|-------------------------|------------|--|
| INFORMATION DISCLOSURE STATEMENT BY APPLICANT (Not for submission under 37 CFR 1.99) | Application Number | | 15735130 | |
| | Filing Date | | 2017-12-08 | |
| | First Named Inventor | Stephen Richard COULSON | | |
| | Art Unit | 1767 | | |
| | Examiner Name | BUIE-HATCHER, NICOLE M | | |
| | Attorney Docket Number | GBL0007/US | | |

| U.S.PATENTS | | | | | | |
|-------------------|---------|---------------|------------------------|------------|---|--|
| Examiner Initial* | Cite No | Patent Number | Kind Code ¹ | Issue Date | Name of Patentee or Applicant of cited Document | Pages,Columns,Lines where Relevant Passages or Relevant Figures Appear |
| | 1 | | | | | |

If you wish to add additional U.S. Patent citation information please click the Add button.

| U.S.PATENT APPLICATION PUBLICATIONS | | | | | | |
|-------------------------------------|---------|--------------------|------------------------|------------------|---|--|
| Examiner Initial* | Cite No | Publication Number | Kind Code ¹ | Publication Date | Name of Patentee or Applicant of cited Document | Pages,Columns,Lines where Relevant Passages or Relevant Figures Appear |
| | 1 | | | | | |

If you wish to add additional U.S. Published Application citation information please click the Add button.

| FOREIGN PATENT DOCUMENTS | | | | | | | | |
|--------------------------|---------|--------------------------------------|---------------------------|------------------------|------------------|---|--|--------------------------|
| Examiner Initial* | Cite No | Foreign Document Number ³ | Country Code ² | Kind Code ⁴ | Publication Date | Name of Patentee or Applicant of cited Document | Pages,Columns,Lines where Relevant Passages or Relevant Figures Appear | T ⁵ |
| | 1 | | | | | | | <input type="checkbox"/> |

If you wish to add additional Foreign Patent Document citation information please click the Add button

| NON-PATENT LITERATURE DOCUMENTS | | | |
|---------------------------------|---------|---|----------------|
| Examiner Initials* | Cite No | Include name of the author (in CAPITAL LETTERS), title of the article (when appropriate), title of the item (book, magazine, journal, serial, symposium, catalog, etc), date, pages(s), volume-issue number(s), publisher, city and/or country where published. | T ⁵ |
| | | | |

**INFORMATION DISCLOSURE
STATEMENT BY APPLICANT**
(Not for submission under 37 CFR 1.99)

| | |
|------------------------|-------------------------|
| Application Number | 15735130 |
| Filing Date | 2017-12-08 |
| First Named Inventor | Stephen Richard COULSON |
| Art Unit | 1767 |
| Examiner Name | BUIE-HATCHER, NICOLE M |
| Attorney Docket Number | GBL0007/US |

| | | | |
|----------|---|--|--------------------------|
| /N.M.B./ | 1 | "Plasma treatment can apply liquid repellency to any material", Technical Textiles International, May 2005, pages 23-25 (3 pages). | <input type="checkbox"/> |
|----------|---|--|--------------------------|

If you wish to add additional non-patent literature document citation information please click the Add button

EXAMINER SIGNATURE

| | | | |
|--------------------|--------------------------|-----------------|------------|
| Examiner Signature | /NICOLE M. BUIE-HATCHER/ | Date Considered | 03/26/2019 |
|--------------------|--------------------------|-----------------|------------|

*EXAMINER: Initial if reference considered, whether or not citation is in conformance with MPEP 609. Draw line through a citation if not in conformance and not considered. Include copy of this form with next communication to applicant.

¹ See Kind Codes of USPTO Patent Documents at www.USPTO.GOV or MPEP 901.04. ² Enter office that issued the document, by the two-letter code (WIPO Standard ST.3). ³ For Japanese patent documents, the indication of the year of the reign of the Emperor must precede the serial number of the patent document. ⁴ Kind of document by the appropriate symbols as indicated on the document under WIPO Standard ST.16 if possible. ⁵ Applicant is to place a check mark here if English language translation is attached.



(12) 发明专利申请

(10) 申请公布号 CN 103881013 A

(43) 申请公布日 2014.06.25

(21) 申请号 201410100313.4

C08F 220/06 (2006.01)

(22) 申请日 2014.03.18

C08F 212/36 (2006.01)

(71) 申请人 南京工业大学

C08F 220/54 (2006.01)

地址 211816 江苏省南京市浦口区浦珠南路
30号

C08F 2/52 (2006.01)

(72) 发明人 陈苏 汤文岐 张强 周振方

(74) 专利代理机构 南京天华专利代理有限责任
公司 32218

代理人 徐冬涛 袁正英

(51) Int. Cl.

C08F 220/24 (2006.01)

C08F 220/28 (2006.01)

C08F 222/38 (2006.01)

C08F 220/20 (2006.01)

C08F 220/22 (2006.01)

C08F 226/10 (2006.01)

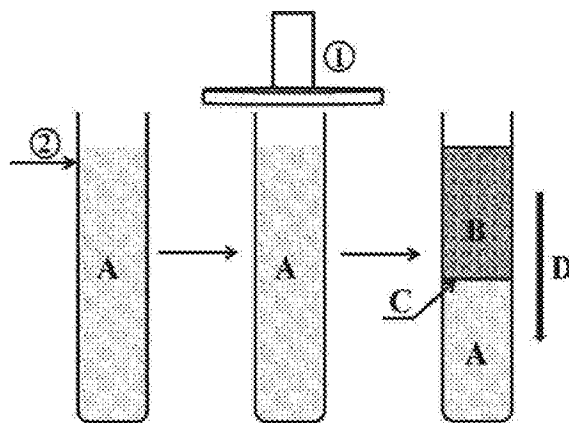
权利要求书1页 说明书4页 附图2页

(54) 发明名称

采用等离子前端聚合快速制备含氟水凝胶的
方法

(57) 摘要

本发明涉及一种采用等离子前端聚合快速制备含氟水凝胶的方法。其具体步骤如下：将活性单体、含氟单体、引发剂和交联剂按比例溶于有机溶剂中，配成均匀溶液；将配成均匀溶液混合移入反应器中，对反应器上端利用低温等离子体处理，使引发剂受热分解，引发聚合反应；关闭等离子体反应器，依靠聚合反应放出的热向未反应区域扩散，直到整个反应器内所有原料全部转化为聚合物；用去离子水漂洗，得到含氟水凝胶。该方法具有速度快、效率高、成本低、成型快等优点，避免了传统制备方法中工艺流程复杂、时间长等缺点，在工业生产中具有较高的应用价值。



1. 采用等离子前端聚合快速制备含氟水凝胶的方法,其具体步骤如下:
 - (1) 将活性单体、含氟单体、引发剂和交联剂按比例溶于有机溶剂中,配成均匀溶液;
 - (2) 将配成均匀溶液混合移入反应器中,对反应器上端利用低温等离子体处理,使引发剂受热分解,引发聚合反应;
 - (3) 关闭等离子体反应器,依靠聚合反应放出的热向未反应区域扩散,直到整个反应器内所有原料全部转化为聚合物;
 - (4) 取出所得的聚合物,用去离子水漂洗,得到含氟水凝胶。
2. 根据权利要求1所述的方法,其特征在于步骤(1)配成均匀溶液中,活性单体占溶液总质量的20~40%;含氟单体占溶液总质量的20~40%;交联剂占溶液总质量的0.5~5%;引发剂所占质量比为0.25~4%;有机溶剂占溶液总质量的15~45%。
3. 根据权利要求1所述的方法,其特征在于:所述活性单体为丙烯酸羟乙酯、丙烯酸羟丙酯、甲基丙烯酸羟乙酯、甲基丙烯酸羟丙酯、N,N'-二甲基丙烯酰胺,丙烯酰胺,双丙酮丙烯酰胺、N-羟甲基丙烯酰胺、N-异丙基丙烯酰胺、N-乙烯基吡咯烷酮、N-乙烯基己内酰胺、丙烯酸辛、丙烯酸或甲基丙烯酸中的至少一种。
4. 根据权利要求1所述的方法,其特征在于:含氟单体为甲基丙烯酸氟烷基酯、甲基丙烯酸全氟烷基乙基酯,甲基丙烯酸三氟乙酯、N-乙基全氟辛基磺酰胺基丙烯酸酯、全氟烷基丙烯酸酯、甲基丙烯酸全氟辛基乙酯、丙烯酸六氟丁酯、甲基丙烯酸六氟丁酯、甲基丙烯酸十二氟庚酯、三氟甲基丙烯酸或丙烯酸全氟烷基乙酯中的一种。
5. 根据权利要求1所述的方法,其特征在于:所述有机溶剂为:甘油、二甲亚砜、N,N'-二甲基甲酰胺或1-甲基-2-吡咯烷酮中的一种。
6. 根据权利要求1所述的方法,其特征在于:所述交联剂为N,N'-亚甲基双丙烯酰胺,二乙烯基苯或二甲基丙烯酸乙二醇酯中的一种。
7. 根据权利要求1所述的方法,其特征在于:所述引发剂为偶氮二异丁腈,偶氮二异庚腈,过氧化苯甲酰、过硫酸铵、过硫酸钾、叔丁基过氧化氢中的其中一种。
8. 根据权利要求1所述的方法,其特征在于:低温等离子体处理电压为30~80V;电流为1~3A;处理时间为5~60s。

采用等离子前端聚合快速制备含氟水凝胶的方法

技术领域

[0001] 本发明属于功能高分子材料制备技术领域,尤其涉及一种采用等离子前端聚合快速制备含氟水凝胶的方法。

背景技术

[0002] 水凝胶材料是一种能够在水中溶胀并保持大量水分且不被溶解的三维网络交联结构的新型高分子材料。根据不同的对环境刺激响应能力水凝胶材料可分为普通水凝胶和智能水凝胶。智能水凝胶能够感知外界环境的细微变化(如温度、pH 值、电、光、磁场、特定生物分子等的变化),以体积显著变化的形式反馈给人们,从而实现与外界环境的信息交流。

[0003] 含氟聚合物具有高度化学稳定性、极强的耐高低温性能、突出的不粘性、异常的润滑性、优异的电绝缘性、耐老化性、抗辐射性和极小的吸水性等优良的综合性能,广泛应用于航空航天、石油化工、机械、电子电器、建筑、纺织等诸多领域。

[0004] 前端聚合又被称为前沿聚合或者波聚合,是一种通过局部反应区域在聚合物单体中的移动而将聚合物单体转变为聚合物的一种新型自由基反应模式。前端聚合是对含有单体和引发剂的混合物进行局部加热,引发剂受热分解,引发单体发生聚合反应。而后撤离热源,聚合反应本身产生的热量向未反应区域扩散,继续引发聚合,从而产生反应热扩散耦合循环的狭窄反应区域,此区域称为聚合前端。聚合前端不断自发向前推进,直到容器内的单体聚合完全。

[0005] 由于前端聚合体系中所需的热量均来自于其本身,热扩散来自于对流传热,反应时间很短,因此它具有很好的节能环保等特性。大部分的前端聚合反应都是在无溶剂或少溶剂的条件下进行,这就意味着最终的产物无需从溶剂中分离,制备过程没有大量有机溶剂的使用和后处理,这就意味着保护环境,节省成本。前端聚合可以在高浓度单体和引发剂下反应而不会发生暴聚现象,反应快、效率高;相对于常规制备工艺,聚合前端的高温和快速移动能力能够有效地抑制相分离,得到具有特殊结构的聚合物。

[0006] 根据前端聚合反应的不同引发方式和反应机理,可以将前端聚合反应分为两类,即:热差前端聚合和等温前端聚合。随着研究的广泛深入,前端聚合家族又引进了光引发前端聚合。

[0007] 等离子体是由部分电子被剥夺后的原子及原子被电离后产生的正负电子组成的离子化气体状物质,广泛存在于宇宙中,被认为是除去固、液、气外物质存在的第四态。等离子技术在薄膜沉积、刻蚀、等离子引发聚合以及表面处理等方面具有广阔的应用前景。目前尚未有发现采用等离子体前端聚合制备智能水凝胶材料的报道。

发明内容

[0008] 本发明的目的在于提供一种采用等离子体前端聚合快速制备含氟水凝胶的方法。该方法具有速度快,效率高,成本低,成型快等优点,避免了传统制备方法中工艺流程复杂,

时间长等缺点,在工业生产中具有较高的应用价值。

[0009] 本发明的技术方案为:采用等离子前端聚合快速制备含氟水凝胶的方法,其具体步骤如下:

[0010] (1) 将活性单体、含氟单体、引发剂和交联剂按比例溶于有机溶剂中,配成均匀溶液;

[0011] (2) 将配成均匀溶液混合移入反应器中,对反应器上端利用低温等离子体处理,使引发剂受热分解,引发聚合反应;

[0012] (3) 关闭等离子体反应器,依靠聚合反应放出的热向未反应区域扩散,直到整个反应器内所有原料全部转化为聚合物;

[0013] (4) 取出所得的聚合物,用去离子水漂洗,得到含氟水凝胶。

[0014] 上述步骤(1)配成均匀溶液中,优选活性单体占溶液总质量的 20 ~ 40%;含氟单体占溶液总质量的 20 ~ 40%;交联剂占溶液总质量的 0.5 ~ 5%;引发剂所占质量比为 0.25 ~ 4%;有机溶剂占溶液总质量的 15 ~ 45%。

[0015] 优选上述活性单体为丙烯酸羟乙酯、丙烯酸羟丙酯、甲基丙烯酸羟乙酯、甲基丙烯酸羟丙酯、N,N'-二甲基丙烯酰胺、丙烯酰胺、双丙酮丙烯酰胺、N-羟甲基丙烯酰胺、N-异丙基丙烯酰胺、N-乙基吡咯烷酮、N-乙基己内酰胺、丙烯酸辛、丙烯酸或甲基丙烯酸中的至少一种。

[0016] 优选上述含氟单体为甲基丙烯酸氟烷基酯、甲基丙烯酸全氟烷基乙基酯、甲基丙烯酸三氟乙酯、N-乙基全氟辛基磺酰胺基丙烯酸酸乙酯、全氟烷基丙烯酸酯、甲基丙烯酸全氟辛基乙酯、丙烯酸六氟丁酯、甲基丙烯酸六氟丁酯、甲基丙烯酸十二氟庚酯、三氟甲基丙烯酸或丙烯酸全氟烷基乙酯中的一种。

[0017] 优选上述有机溶剂为:甘油、二甲亚砜、N,N'-二甲基甲酰胺或 1-甲基-2-吡咯烷酮中的一种。

[0018] 优选上述交联剂为 N,N'-亚甲基双丙烯酰胺、二乙烯基苯或二甲基丙烯酸乙二醇酯中的一种。

[0019] 优选上述引发剂为偶氮二异丁腈、偶氮二异庚腈、过氧化苯甲酰、过硫酸铵、过硫酸钾、叔丁基过氧化氢中的其中一种。

[0020] 优选低温等离子体处理电压为 30 ~ 80V;电流为 1 ~ 3A;处理时间为 5 ~ 60s。

[0021] 有益效果:

[0022] 1. 等离子前端聚合法与传统的聚合方法相比,所需的外供能量是短暂一次性的,聚合启动后即可停止供能,在聚合反应过程中不需要外加热源来维持整个体系的聚合,具有可控性,且聚合反应时间较短,节能,无污染。

[0023] 2. 等离子前端聚合体系中的热量产生于反应本身,热扩散源于对流传热,需要较少反应时间,因此是一种极佳的节能反应模式,在工业化生产中具有很高的应用价值。

附图说明

[0024] 图 1 为等离子体前端聚合制备水凝胶示意图;其中①为等离子体装置,②为反应器,A 为单体混合物,B 为聚合物,C 为前端端面,D 为前端传播方向;

[0025] 图 2 为实施例 1 等离子体前端聚合制备水凝胶的前端位置与时间变化关系图;

[0026] 图 3 为为实施例 1 等离子体前端聚合制备智能水凝胶的温度曲线图。

具体实施方式

[0027] 以下通过具体实施例说明本发明,但本发明并不仅限于这些实施例。

[0028] 实施例 1

[0029] 称取 60.0g 丙烯酸羟乙酯,4.25g N,N'-亚甲基双丙烯酰胺,100.0g 甲基丙烯酸氟烷烃酯溶于 135.0gN,N'-二甲基甲酰胺中配成均匀溶液,然后加入 0.75g 过硫酸钾,混合均匀后倒入玻璃反应器内,调节等离子体装置电压为 30V 电流为 3A,引发聚合反应,其中等离子体处理时间为 5s。关闭等离子体反应装置,借助放热反应的热自催化作用而完成单体聚合。用去离子水冲洗聚合物,得到智能水凝胶。如图 2 和 3 所示,从图中可以发现只存在纯粹的前端聚合,而没有自发本体聚合的发生。对水凝胶红外分析,1200 cm^{-1} 处为 C-F 的特征峰,2450 cm^{-1} 处为 O-H 的红外特征峰,说明得到的水凝胶为(丙烯酸羟乙酯-甲基丙烯酸氟烷烃酯)共聚物。

[0030] 实施例 2

[0031] 称取 40.0g 丙烯酸羟丙酯,0.5g 二甲基丙烯酸乙二醇酯,20g 甲基丙烯酸全氟烷基乙基酯溶于 38.0g N,N'-二甲基甲酰胺中配成均匀溶液,然后加入 1.5g 过硫酸铵,混合均匀后倒入玻璃反应器内,调节等离子体装置电压为 80V 电流为 1A,引发聚合反应,其中等离子体处理时间为 50s。关闭等离子体反应装置,借助放热反应的热自催化作用而完成单体聚合。用去离子水冲洗聚合物,得到智能水凝胶。对水凝胶红外分析,1190 cm^{-1} 处为 C-F 的特征峰,2445 cm^{-1} 处为 O-H 的红外特征峰,说明得到的水凝胶为(丙烯酸羟丙酯-甲基丙烯酸全氟烷基乙基酯)共聚物。

[0032] 实施例 3

[0033] 称取 20.0g N-乙烯基吡咯烷酮,1.5g 二甲基丙烯酸乙二醇酯,19.0g 甲基丙烯酸六氟丁酯溶于 7.5g 二甲亚砜中配成均匀溶液,然后加入 2.0g 过氧化苯甲酰,混合均匀后倒入玻璃反应器内,调节等离子体装置电压为 50V 电流为 3A,引发聚合反应,其中等离子体处理时间为 30s。关闭等离子体反应装置,借助放热反应的热自催化作用而完成单体聚合。用去离子水冲洗聚合物,得到智能水凝胶。对水凝胶红外分析,1185 cm^{-1} 处为 C-F 的特征峰,1440 cm^{-1} 处为内酰胺吸收峰,说明得到的水凝胶为(N-乙烯基吡咯烷酮-甲基丙烯酸六氟丁酯)共聚物。

[0034] 实施例 4

[0035] 称取 25.0g 丙烯酸,4.0g 二乙烯基苯,24.0g 甲基丙烯酸全氟辛基乙酯溶于 26.0g 甘油中配成均匀溶液,然后加入 1.0g 偶氮二异庚腈,混合均匀后倒入玻璃反应器内,调节等离子体装置电压为 30V 电流为 1A,引发聚合反应,其中等离子体处理时间为 60s。关闭等离子体反应装置,借助放热反应的热自催化作用而完成单体聚合。用去离子水冲洗聚合物,得到智能水凝胶。对水凝胶热重分析,物质质量和温度的关系可以说明得到的水凝胶为(丙烯酸-甲基丙烯酸全氟辛基乙酯)共聚物。

[0036] 实施例 5

[0037] 称取 100.0g N-异丙基丙烯酰胺,3.0g N,N'-亚甲基双丙烯酰胺,75.0g 甲基丙烯酸十二氟庚酯溶于 120.0g1-甲基-2-吡咯烷酮中配成均匀溶液,然后加入 2.0g 偶氮二异

丁腈,混合均匀后倒入玻璃反应器内,调节等离子体装置电压为 40V 电流为 2.5A,引发聚合反应,其中等离子体处理时间为 40s。关闭等离子体反应装置,借助放热反应的热自催化作用而完成单体聚合。用去离子水冲洗聚合物,得到智能水凝胶。对水凝胶热重分析,物质质量和温度的关系可以说明得到的水凝胶为 (N-异丙基丙烯酰胺-甲基丙烯酸十二氟庚酯) 共聚物。

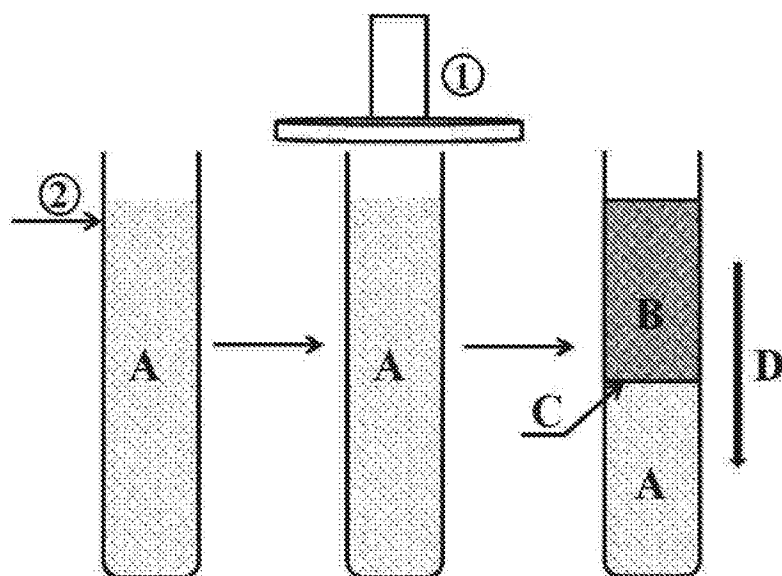


图 1

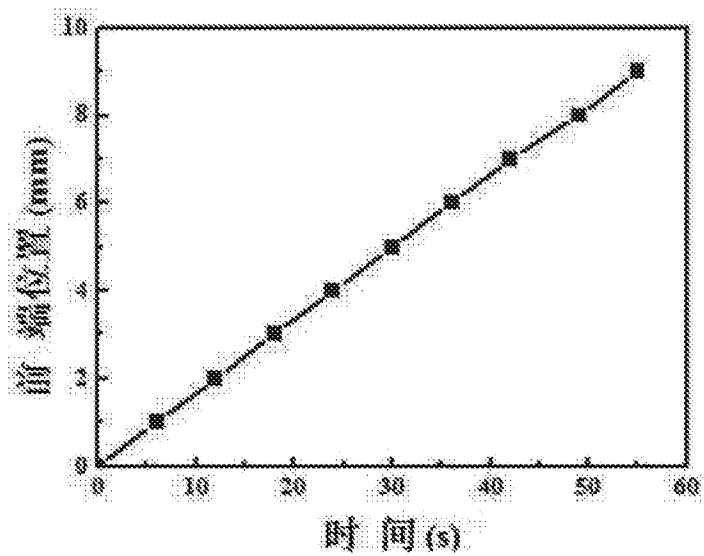


图 2

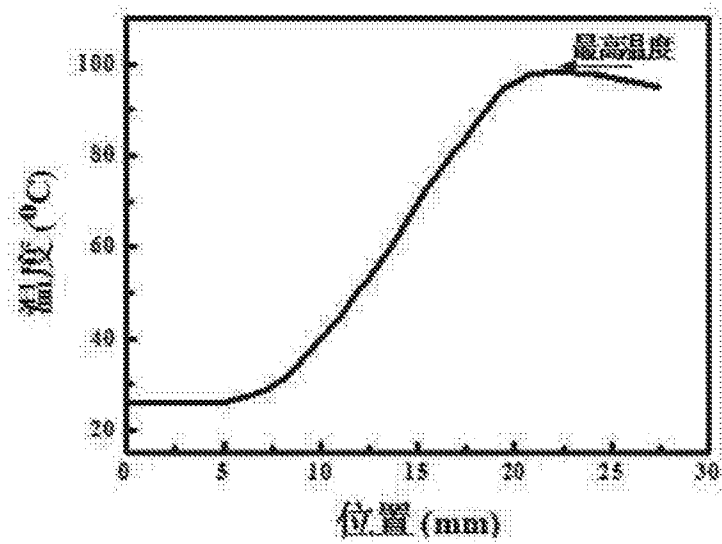


图 3

Session Began April 30, 2019 at 06:06 PM

Task Began April 30, 2019 06:06 PM

Explore references by research topic: plasma polymerization of fluoroacrylate and crosslinking monomer

Research Topic: plasma polymerization of fluoroacrylate and crosslinking monomer
Result Count: 9

Candidates Selected (ID 1)

4 references were found containing the two concepts "plasma polymerization" and "fluoroacrylate".

Automatically removed 4 duplicate MEDLINE answer(s)

173 references were found containing the two concepts "plasma polymerization" and "crosslinking monomer".

Automatically removed 4 duplicate MEDLINE answer(s)

50 references were found containing both of the concepts "fluoroacrylate" and "crosslinking monomer".

Automatically removed 4 duplicate MEDLINE answer(s)

Answer Type: References

Result Count: 223

Prepared 4 reference answers from Answer set 1 for printing in PDF form

Copyright © 2019 American Chemical Society. All Rights Reserved.

Copyright © 1994-2019 Sun Microsystems, Inc. All Rights Reserved. (Java runtime environment)

Copyright © 2019, Yahoo! Inc. All Rights Reserved. (YUI Base, Fonts, Reset CSS)

Copyright © 200-2019, The Dojo Foundation. All Rights Reserved

Copyright © 2019, Exadel, Inc. All Rights Reserved. (JBoss RichFaces)

Copyright © 2002 InfoChem GmbH. All Rights Reserved. (InfoChem's reaction classification program CLASSIFY)

The following copyright notice applies to Raphael only: Permission is hereby granted, free of charge, to any person obtaining a copy of this software and associated documentation files (the 'Software'), to deal in the Software without restriction, including without limitation the rights to use, copy, modify, merge, publish, distribute, sublicense, and/or sell copies of the Software, and to permit persons to whom the Software is furnished to do so, subject to the following conditions: The above copyright notice and this permission notice shall be included in all copies or substantial portions of the Software. THE SOFTWARE IS PROVIDED 'AS IS', WITHOUT WARRANTY OF ANY KIND, EXPRESS OR IMPLIED, INCLUDING BUT NOT LIMITED TO THE WARRANTIES OF MERCHANTABILITY, FITNESS FOR A PARTICULAR PURPOSE AND NONINFRINGEMENT. IN NO EVENT SHALL THE AUTHORS OR COPYRIGHT HOLDERS BE LIABLE FOR ANY CLAIM, DAMAGES OR OTHER LIABILITY, WHETHER IN AN ACTION OF CONTRACT, TORT OR OTHERWISE, ARISING FROM, OUT OF OR IN CONNECTION WITH THE SOFTWARE OR THE USE OR OTHER DEALINGS IN THE SOFTWARE.

The following copyright notice applies to Google Closure Templates only: Copyright 2009 Google Inc. Licensed under the Apache License, Version 2.0 (the 'License'); you may not use this file except in compliance with the License. You may obtain a copy of the License at <http://www.apache.org/licenses/LICENSE-2.0> Unless required by applicable law or agreed to in writing, software distributed under the License is distributed on an 'AS IS' BASIS, WITHOUT WARRANTIES OR CONDITIONS OF ANY KIND, either express or implied. See the License for the specific language governing permissions and limitations under the License.

Copyright (c) 2010, 2012 Adobe Systems Incorporated, All Rights Reserved, (Source Sans Pro) Licensed under the SIL Open Font License, Version 1.1 (available at: <http://scripts.sil.org/OFL>)

Copyright 2005-2013 The Dojo Foundation (Dojo Toolkit) Licensed under the Apache License, Version 2.0 (the "License"); you may not use this file except in compliance with the License. You may obtain a copy of the License at: <http://www.apache.org/licenses/LICENSE-2.0> Unless required by applicable law or agreed to in writing, software distributed under the License is distributed on an "AS IS" BASIS, WITHOUT WARRANTIES OR CONDITIONS OF ANY KIND, either express or implied. See the License for the specific language governing permissions and limitations under the License.

Copyright (c) 2008 Dmitry Baranovskiy (Raphael) Licensed under the MIT License (available at: <http://opensource.org/licenses/MIT>) Permission is hereby granted, free of charge, to any person obtaining a copy of this software and associated documentation files (the "Software"), to deal in the Software without restriction, including without limitation the rights to use, copy, modify, merge, publish, distribute, sublicense, and/or sell copies of the Software, and to permit persons to whom the Software is furnished to do so, subject to the following conditions: The above copyright notice and this permission notice shall be included in all copies or substantial portions of the Software. THE SOFTWARE IS PROVIDED "AS IS", WITHOUT WARRANTY OF ANY KIND, EXPRESS OR IMPLIED, INCLUDING BUT NOT LIMITED TO THE WARRANTIES OF MERCHANTABILITY, FITNESS FOR A PARTICULAR PURPOSE AND NONINFRINGEMENT. IN NO EVENT SHALL THE AUTHORS OR COPYRIGHT HOLDERS BE LIABLE FOR ANY CLAIM, DAMAGES OR OTHER LIABILITY, WHETHER IN AN ACTION OF CONTRACT, TORT OR OTHERWISE, ARISING FROM, OUT OF OR IN CONNECTION WITH THE SOFTWARE OR THE USE OR OTHER DEALINGS IN THE SOFTWARE.

CAPLUSSM: Copyright © 2019 American Chemical Society. All Rights Reserved. (The U.K. patent material in this product/service is U.K. Crown copyright and is made available with permission.

Copyright © Crown Copyright. The French (FR) patent material in this product/service is made available from Institut National de la Propriété Industrielle (INPI).)

CAS REGISTRYSM: Copyright © 2019 American Chemical Society. All Rights Reserved. (Some records contain information from GenBank[®]. See also: Benson D.A., Karsch-Mizrahi L., Lipman D.J., Ostel J., Rapp B.A., Wheeler D.L. Genbank. Nucl. Acids Res. 28(1):15-18 (2000). Property values tagged with IC are from the ZIC/VINITI data file provided by InfoChem.) CAS Registry is a service mark of the American Chemical Society. GenBank is a registered trademark of the U.S. Library of Medicine.

CASREACT[®]: Copyright © 2019 American Chemical Society. All Rights Reserved. CASREACT contains reactions from CAS and from: ZIC/VINITI database (1974-1999) provided by InfoChem; INPI data prior to 1986; Biotransformations database compiled under the direction of Professor Dr. Klaus Kieslich; organic reactions, portions copyright 1998-2006 John Wiley & Sons, Ltd., John Wiley and Sons, Inc., Organic Reactions Inc., and Organic Syntheses Inc. Reproduced under license. All Rights Reserved.

CHEMCATS[®]: Copyright © 2019 American Chemical Society. All Rights Reserved. Chemical supplier information is supplied on an "as is" basis. Full information regarding substance availability, price, etc., is provided when you request supplier information.

CHEMLIST[®]: Copyright © 2019 American Chemical Society. All Rights Reserved.

MEDLINE[®]: is produced by the U.S. National Library of Medicine. MEDLINE is a registered trademark of the U.S. National Library of Medicine.



UNITED STATES PATENT AND TRADEMARK OFFICE

UNITED STATES DEPARTMENT OF COMMERCE
United States Patent and Trademark Office
Address: COMMISSIONER FOR PATENTS
P.O. Box 1450
Alexandria, Virginia 22313-1450
www.uspto.gov

| APPLICATION NUMBER | FILING or 371(c) DATE | GRP ART UNIT | FIL FEE REC'D | ATTY. DOCKET NO | TOT CLAIMS | IND CLAIMS |
|-----------------------|--------------------------|-----------------|---------------|-----------------|------------|------------|
| 15/735,130 | 12/08/2017 | 1767 | 1620 | GBL0007/US | 18 | 1 |

CONFIRMATION NO. 7101
CORRECTED FILING RECEIPT

33072
KAGAN BINDER, PLLC
SUITE 200, MAPLE ISLAND BUILDING
221 MAIN STREET NORTH
STILLWATER, MN 55082
UNITED STATES OF AMERICA



Date Mailed: 07/02/2019

Receipt is acknowledged of this non-provisional utility patent application. The application will be taken up for examination in due course. Applicant will be notified as to the results of the examination. Any correspondence concerning the application must include the following identification information: the U.S. APPLICATION NUMBER, FILING DATE, NAME OF FIRST INVENTOR, and TITLE OF INVENTION. Fees transmitted by check or draft are subject to collection.

Please verify the accuracy of the data presented on this receipt. If an error is noted on this Filing Receipt, please submit a written request for a corrected Filing Receipt, including a properly marked-up ADS showing the changes with strike-through for deletions and underlining for additions. If you received a "Notice to File Missing Parts" or other Notice requiring a response for this application, please submit any request for correction to this Filing Receipt with your reply to the Notice. When the USPTO processes the reply to the Notice, the USPTO will generate another Filing Receipt incorporating the requested corrections provided that the request is grantable.

Inventor(s)

Stephen Richard COULSON, Abingdon, UNITED KINGDOM;
Delwyn EVANS, Abingdon, UNITED KINGDOM;
Angeliki SIOKOU, Abingdon, UNITED KINGDOM;
Clive TELFORD, Abingdon, UNITED KINGDOM, Deceased;

Applicant(s)

P2i Ltd, Abingdon, UNITED KINGDOM

Power of Attorney: The patent practitioners associated with Customer Number 33072

Domestic Priority data as claimed by applicant

This application is a 371 of PCT/GB2016/051688 06/08/2016

Foreign Applications (You may be eligible to benefit from the **Patent Prosecution Highway** program at the USPTO. Please see <http://www.uspto.gov> for more information.)
EUROPEAN PATENT OFFICE (EPO) 15386017.6 06/09/2015

Permission to Access Application via Priority Document Exchange: Yes

Permission to Access Search Results: Yes

Applicant may provide or rescind an authorization for access using Form PTO/SB/39 or Form PTO/SB/69 as appropriate.

If Required, Foreign Filing License Granted: 03/14/2018

The country code and number of your priority application, to be used for filing abroad under the Paris Convention, is **US 15/735,130**

Projected Publication Date: Not Applicable

Non-Publication Request: No

Early Publication Request: No
Title

COATINGS

Preliminary Class

526

Statement under 37 CFR 1.55 or 1.78 for AIA (First Inventor to File) Transition Applications: No

PROTECTING YOUR INVENTION OUTSIDE THE UNITED STATES

Since the rights granted by a U.S. patent extend only throughout the territory of the United States and have no effect in a foreign country, an inventor who wishes patent protection in another country must apply for a patent in a specific country or in regional patent offices. Applicants may wish to consider the filing of an international application under the Patent Cooperation Treaty (PCT). An international (PCT) application generally has the same effect as a regular national patent application in each PCT-member country. The PCT process **simplifies** the filing of patent applications on the same invention in member countries, but **does not result** in a grant of "an international patent" and does not eliminate the need of applicants to file additional documents and fees in countries where patent protection is desired.

Almost every country has its own patent law, and a person desiring a patent in a particular country must make an application for patent in that country in accordance with its particular laws. Since the laws of many countries differ in various respects from the patent law of the United States, applicants are advised to seek guidance from specific foreign countries to ensure that patent rights are not lost prematurely.

Applicants also are advised that in the case of inventions made in the United States, the Director of the USPTO must issue a license before applicants can apply for a patent in a foreign country. The filing of a U.S. patent application serves as a request for a foreign filing license. The application's filing receipt contains further information and guidance as to the status of applicant's license for foreign filing.

Applicants may wish to consult the USPTO booklet, "General Information Concerning Patents" (specifically, the section entitled "Treaties and Foreign Patents") for more information on timeframes and deadlines for filing foreign patent applications. The guide is available either by contacting the USPTO Contact Center at 800-786-9199, or it can be viewed on the USPTO website at <http://www.uspto.gov/web/offices/pac/doc/general/index.html>.

For information on preventing theft of your intellectual property (patents, trademarks and copyrights), you may wish to consult the U.S. Government website, <http://www.stopfakes.gov>. Part of a Department of Commerce initiative, this website includes self-help "toolkits" giving innovators guidance on how to protect intellectual property in specific countries such as China, Korea and Mexico. For questions regarding patent enforcement issues, applicants may call the U.S. Government hotline at 1-866-999-HALT (1-866-999-4258).

LICENSE FOR FOREIGN FILING UNDER
Title 35, United States Code, Section 184
Title 37, Code of Federal Regulations, 5.11 & 5.15

GRANTED

The applicant has been granted a license under 35 U.S.C. 184, if the phrase "IF REQUIRED, FOREIGN FILING LICENSE GRANTED" followed by a date appears on this form. Such licenses are issued in all applications where the conditions for issuance of a license have been met, regardless of whether or not a license may be required as set forth in 37 CFR 5.15. The scope and limitations of this license are set forth in 37 CFR 5.15(a) unless an earlier license has been issued under 37 CFR 5.15(b). The license is subject to revocation upon written notification. The date indicated is the effective date of the license, unless an earlier license of similar scope has been granted under 37 CFR 5.13 or 5.14.

This license is to be retained by the licensee and may be used at any time on or after the effective date thereof unless it is revoked. This license is automatically transferred to any related applications(s) filed under 37 CFR 1.53(d). This license is not retroactive.

The grant of a license does not in any way lessen the responsibility of a licensee for the security of the subject matter as imposed by any Government contract or the provisions of existing laws relating to espionage and the national security or the export of technical data. Licensees should apprise themselves of current regulations especially with respect to certain countries, of other agencies, particularly the Office of Defense Trade Controls, Department of State (with respect to Arms, Munitions and Implements of War (22 CFR 121-128)); the Bureau of Industry and Security, Department of Commerce (15 CFR parts 730-774); the Office of Foreign Assets Control, Department of Treasury (31 CFR Parts 500+) and the Department of Energy.

NOT GRANTED

No license under 35 U.S.C. 184 has been granted at this time, if the phrase "IF REQUIRED, FOREIGN FILING LICENSE GRANTED" DOES NOT appear on this form. Applicant may still petition for a license under 37 CFR 5.12, if a license is desired before the expiration of 6 months from the filing date of the application. If 6 months has lapsed from the filing date of this application and the licensee has not received any indication of a secrecy order under 35 U.S.C. 181, the licensee may foreign file the application pursuant to 37 CFR 5.15(b).

SelectUSA

The United States represents the largest, most dynamic marketplace in the world and is an unparalleled location for business investment, innovation, and commercialization of new technologies. The U.S. offers tremendous resources and advantages for those who invest and manufacture goods here. Through SelectUSA, our nation works to promote and facilitate business investment. SelectUSA provides information assistance to the international investor community; serves as an ombudsman for existing and potential investors; advocates on behalf of U.S. cities, states, and regions competing for global investment; and counsels U.S. economic development organizations on investment attraction best practices. To learn more about why the United States is the best country in the world to develop technology, manufacture products, deliver services, and grow your business, visit <http://www.SelectUSA.gov> or call +1-202-482-6800.

PART B - FEE(S) TRANSMITTAL

Complete and send this form, together with applicable fee(s), by mail or fax, or via EFS-Web.

By mail, send to: Mail Stop ISSUE FEE
Commissioner for Patents
P.O. Box 1450
Alexandria, Virginia 22313-1450

By fax, send to: (571)-273-2885

INSTRUCTIONS: This form should be used for transmitting the ISSUE FEE and PUBLICATION FEE (if required). Blocks 1 through 5 should be completed where appropriate. All further correspondence including the Patent, advance orders and notification of maintenance fees will be mailed to the current correspondence address as indicated unless corrected below or directed otherwise in Block 1, by (a) specifying a new correspondence address; and/or (b) indicating a separate "FEE ADDRESS" for maintenance fee notifications.

CURRENT CORRESPONDENCE ADDRESS (Note: Use Block 1 for any change of address)

33072 7590 08/15/2019
KAGAN BINDER, PLLC
SUITE 200, MAPLE ISLAND BUILDING
221 MAIN STREET NORTH
STILLWATER, MN 55082

Note: A certificate of mailing can only be used for domestic mailings of the Fee(s) Transmittal. This certificate cannot be used for any other accompanying papers. Each additional paper, such as an assignment or formal drawing, must have its own certificate of mailing or transmission.

CERTIFICATE UNDER 37 CFR 1.8

I hereby certify that this correspondence is being transmitted via the United States Patent and Trademark Office electronic filing system on
August 12, 2019
By: Bethany Lichtscheidl/
Name: Bethany Lichtscheidl

| APPLICATION NO. | FILING DATE | FIRST NAMED INVENTOR | ATTORNEY DOCKET NO. | CONFIRMATION NO. |
|-----------------|-------------|-------------------------|---------------------|------------------|
| 15/735,130 | 12/08/2017 | Stephen Richard COULSON | GBL0007/US | 7101 |

TITLE OF INVENTION: COATINGS

| APPLN. TYPE | ENTITY STATUS | ISSUE FEE DUE | PUBLICATION FEE DUE | PREV. PAID ISSUE FEE | TOTAL FEE(S) DUE | DATE DUE |
|----------------|---------------|---------------|---------------------|----------------------|------------------|------------|
| nonprovisional | UNDISCOUNTED | \$1000 | \$0.00 | \$0.00 | \$1000 | 08/15/2019 |

| EXAMINER | ART UNIT | CLASS-SUBCLASS |
|------------------------|----------|----------------|
| BUIE-HATCHER, NICOLE M | 1767 | 526-245000 |

1. Change of correspondence address or indication of "Fee Address" (37 CFR 1.363).

☐ Change of correspondence address (for Change of Correspondence Address form PTO/SB/122) attached.

☐ "Fee Address" indication (or "Fee Address" Indication form PTO/SB/47; Rev 03-09 or more recent) attached. Use of a Customer Number is required.

2. For printing on the patent front page, list

(1) The names of up to 3 registered patent attorneys or agents OR, alternatively,

(2) The name of a single firm (having as a member a registered attorney or agent) and the names of up to 2 registered patent attorneys or agents. If no name is listed, no name will be printed.

1. Kagan Binder, PLLC

2.

3.

3. ASSIGNEE NAME AND RESIDENCE DATA TO BE PRINTED ON THE PATENT (print or type)

PLEASE NOTE: Unless an assignee is identified below, no assignee data will appear on the patent. If an assignee is identified below, the document must have been previously recorded, or filed for recordation, as set forth in 37 CFR 3.11 and 37 CFR 3.81(a). Completion of this form is NOT a substitute for filing an assignment.

(A) NAME OF ASSIGNEE

(B) RESIDENCE (CITY and STATE OR COUNTRY)

P2i Ltd

Abingdon, United Kingdom

Please check the appropriate assignee category or categories (will not be printed on the patent): ☐ Individual ☒ Corporation or other private group entity ☐ Government

4a. Fees submitted: ☒ Issue Fee ☐ Publication Fee (if required) ☐ Advance Order - # of Copies

4b. Method of Payment: (Please first reapply any previously paid fee shown above)

☒ Electronic Payment via EFS-Web ☐ Enclosed check ☐ Non-electronic payment by credit card (Attach form PTO-2038)

☒ The Director is hereby authorized to charge the required fee(s), any deficiency, or credit any overpayment to Deposit Account No. 50-1775

5. Change in Entity Status (from status indicated above)

☐ Applicant certifying micro entity status. See 37 CFR 1.29.

☐ Applicant asserting small entity status. See 37 CFR 1.27

☐ Applicant changing to regular undiscounted fee status.

NOTE: Absent a valid certification of Micro Entity Status (see forms PTO/SB/15A and 15B), issue fee payment in the micro entity amount will not be accepted at the risk of application abandonment.

NOTE: If the application was previously under micro entity status, checking this box will be taken to be a notification of loss of entitlement to micro entity status.

NOTE: Checking this box will be taken to be a notification of loss of entitlement to small or micro entity status, as applicable.

NOTE: This form must be signed in accordance with 37 CFR 1.31 and 1.33. See 37 CFR 1.4 for signature requirements and certifications.

Authorized Signature

Date August 12, 2019

Typed or printed name Paul J. Parins

Registration No. 54,358

| Electronic Patent Application Fee Transmittal | | | | |
|--|----------|---------------------------------------|--------|----------------------|
| Application Number: | | 15735130 | | |
| Filing Date: | | 08-Dec-2017 | | |
| Title of Invention: | | COATINGS | | |
| First Named Inventor/Applicant Name: | | Stephen Richard COULSON | | |
| Filer: | | Paul John Parins/Bethany Lichtscheidl | | |
| Attorney Docket Number: | | GBL0007/US | | |
| Filed as Large Entity | | | | |
| Filing Fees for U.S. National Stage under 35 USC 371 | | | | |
| Description | Fee Code | Quantity | Amount | Sub-Total in USD(\$) |
| Basic Filing: | | | | |
| Pages: | | | | |
| Claims: | | | | |
| Miscellaneous-Filing: | | | | |
| Petition: | | | | |
| Patent-Appeals-and-Interference: | | | | |
| Post-Allowance-and-Post-Issuance: | | | | |
| UTILITY APPL ISSUE FEE | 1501 | 1 | 1000 | 1000 |

| Description | Fee Code | Quantity | Amount | Sub-Total in USD(\$) |
|--------------------|----------|----------|--------|----------------------|
| Extension-of-Time: | | | | |
| Miscellaneous: | | | | |
| Total in USD (\$) | | | | 1000 |

Electronic Acknowledgement Receipt

| | |
|---|---------------------------------------|
| EFS ID: | 36847528 |
| Application Number: | 15735130 |
| International Application Number: | |
| Confirmation Number: | 7101 |
| Title of Invention: | COATINGS |
| First Named Inventor/Applicant Name: | Stephen Richard COULSON |
| Customer Number: | 33072 |
| Filer: | Paul John Parins/Bethany Lichtscheidl |
| Filer Authorized By: | Paul John Parins |
| Attorney Docket Number: | GBL0007/US |
| Receipt Date: | 12-AUG-2019 |
| Filing Date: | 08-DEC-2017 |
| Time Stamp: | 15:05:07 |
| Application Type: | U.S. National Stage under 35 USC 371 |

Payment information:

| | |
|--|------------------|
| Submitted with Payment | yes |
| Payment Type | CARD |
| Payment was successfully received in RAM | \$1000 |
| RAM confirmation Number | E20198BF05367563 |
| Deposit Account | |
| Authorized User | |

The Director of the USPTO is hereby authorized to charge indicated fees and credit any overpayment as follows:

| File Listing: | | | | | |
|--|-----------------------------|-------------------------|---|------------------|------------------|
| Document Number | Document Description | File Name | File Size(Bytes)/ Message Digest | Multi Part /.zip | Pages (if appl.) |
| 1 | Issue Fee Payment (PTO-85B) | GBL0007US_Issue_Fee.pdf | 297603 | no | 1 |
| | | | 80f2dbd1b1fb615e227e0e21f7a15084e49e c76c | | |
| Warnings: | | | | | |
| Information: | | | | | |
| 2 | Fee Worksheet (SB06) | fee-info.pdf | 30014 | no | 2 |
| | | | 4e40642dec0dccc86f1d6acaa6b3f005b4f13f 1fa | | |
| Warnings: | | | | | |
| Information: | | | | | |
| Total Files Size (in bytes): | | | 327617 | | |
| <p>This Acknowledgement Receipt evidences receipt on the noted date by the USPTO of the indicated documents, characterized by the applicant, and including page counts, where applicable. It serves as evidence of receipt similar to a Post Card, as described in MPEP 503.</p> <p><u>New Applications Under 35 U.S.C. 111</u> If a new application is being filed and the application includes the necessary components for a filing date (see 37 CFR 1.53(b)-(d) and MPEP 506), a Filing Receipt (37 CFR 1.54) will be issued in due course and the date shown on this Acknowledgement Receipt will establish the filing date of the application.</p> <p><u>National Stage of an International Application under 35 U.S.C. 371</u> If a timely submission to enter the national stage of an international application is compliant with the conditions of 35 U.S.C. 371 and other applicable requirements a Form PCT/DO/EO/903 indicating acceptance of the application as a national stage submission under 35 U.S.C. 371 will be issued in addition to the Filing Receipt, in due course.</p> <p><u>New International Application Filed with the USPTO as a Receiving Office</u> If a new international application is being filed and the international application includes the necessary components for an international filing date (see PCT Article 11 and MPEP 1810), a Notification of the International Application Number and of the International Filing Date (Form PCT/RO/105) will be issued in due course, subject to prescriptions concerning national security, and the date shown on this Acknowledgement Receipt will establish the international filing date of the application.</p> | | | | | |



UNITED STATES PATENT AND TRADEMARK OFFICE

UNITED STATES DEPARTMENT OF COMMERCE
United States Patent and Trademark Office
Address: COMMISSIONER FOR PATENTS
P.O. Box 1450
Alexandria, Virginia 22313-1450
www.uspto.gov

| APPLICATION NO. | ISSUE DATE | PATENT NO. | ATTORNEY DOCKET NO. | CONFIRMATION NO. |
|-----------------|------------|------------|---------------------|------------------|
| 15/735,130 | 09/24/2019 | 10421876 | GBL0007/US | 7101 |

33072 7590 09/04/2019

KAGAN BINDER, PLLC
SUITE 200, MAPLE ISLAND BUILDING
221 MAIN STREET NORTH
STILLWATER, MN 55082

ISSUE NOTIFICATION

The projected patent number and issue date are specified above.

Determination of Patent Term Adjustment under 35 U.S.C. 154 (b) (application filed on or after May 29, 2000)

The Patent Term Adjustment is 0 day(s). Any patent to issue from the above-identified application will include an indication of the adjustment on the front page.

If a Continued Prosecution Application (CPA) was filed in the above-identified application, the filing date that determines Patent Term Adjustment is the filing date of the most recent CPA.

Applicant will be able to obtain more detailed information by accessing the Patent Application Information Retrieval (PAIR) WEB site (<http://pair.uspto.gov>).

Any questions regarding the Patent Term Extension or Adjustment determination should be directed to the Office of Patent Legal Administration at (571)-272-7702. Questions relating to issue and publication fee payments should be directed to the Application Assistance Unit (AAU) of the Office of Data Management (ODM) at (571)-272-4200.

APPLICANT(s) (Please see PAIR WEB site <http://pair.uspto.gov> for additional applicants):

Stephen Richard COULSON, Abingdon, UNITED KINGDOM;
P2i Ltd, Abingdon, UNITED KINGDOM
Delwyn EVANS, Abingdon, UNITED KINGDOM;
Angeliki SIOKOU, Abingdon, UNITED KINGDOM;
Clive TELFORD, Abingdon, UNITED KINGDOM, Deceased;

The United States represents the largest, most dynamic marketplace in the world and is an unparalleled location for business investment, innovation, and commercialization of new technologies. The USA offers tremendous resources and advantages for those who invest and manufacture goods here. Through SelectUSA, our nation works to encourage and facilitate business investment. To learn more about why the USA is the best country in the world to develop technology, manufacture products, and grow your business, visit SelectUSA.gov.