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APPLICATION NUMBER: 09/458,919 FILING DATE: December 10, 1999 PATENT NUMBER: 6,455,451 ISSUE DATE: September 24, 2002

> By Authority of the Under Secretary of Commerce for Intellectual Property and Director of the United States Patent and Trademark Office

JOHN A BURSON Certifying Officer

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PATENT APPLICATION SERIAL NO.

U.S. DEPARTMENT OF COMMERCE PATENT AND TRADEMARK OFFICE FEE RECORD SHEET

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PTO-1556 (5/87) ^{*U.S.} GPO: 1998-433-214/80404

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Bib Data Sheet

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UNITED STATES DEPARTMENT OF COMMERCE Patent and Trademark Office

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| SERIAL NUMBER 09/458,919 | FILING DATE 12/10/1999 RULE _ | CLASS 502 | GROUP ART UN 1754 | D | ATTORNEY OCKET NO. PP-1222NP |
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| TITLE PRESSABLE LITHIU | M DISILICATE GLASS (| CERAMICS | μ | - - | · · · · |
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IN THE UNITED STATES PATENT AND TRADEMARK OFFICE

Case Docket No. JPP-1222NP

Box Patent Application Assistant Commissioner for Patents Washington, D.C. 20231

"Express Mail" mailing label number EJ464338021US Date of deposit <u>December 10, 1999</u> I hereby certify that this paper or fee is being deposited with the United States Postal Service "Express Mail Post Office to Addressee" service under 37 CFR 1.10 on the date indicated above and is addressed to the Commissioner of Patents and Trademarks, Washington, D.C. 20231. Suzanne McHugh

(Typed or printer name of person mailing paper or fee)

Sir:

Transmitted herewith for filing is the patent application of :

INVENTOR(S): Dmitri Brodkin, Carlino Panzera, Paul Panzera

FOR: PRESSABLE LITHIUM DISILICATE GLASS CERAMICS

Enclosed are:

[X] <u>34</u> pages of specification, including claims and abstract.

[1] sheet(s) of drawing(s).

[] Declaration and Power of Attorney.

[] Information Disclosure Statement.

[] An associate power of attorney.

[] An assignment of the invention.

[] A certified copy of a ______ application.

[X] A verified statement to establish small entity under 37 CFR 1.9 and 37 CFR 1.27.

The filing fee has been calculated as shown below:

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| FOR: | # FILED | # EXTRA | RATE | FEE | RATE | FEE |
| BASIC FEE | /////////////////////////////////////// | /////////////////////////////////////// | \$380 | \$380 | \$760 | |
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| INDEP. CLAIMS (3) | 18 | 15 | x 39 | \$585 | x 78 | |
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[X] Please charge my Deposit Account No. 50-0718 in the amount of \$1,658.00. A duplicate copy of this sheet is enclosed.

[] A check in the amount of \$_____ is enclosed to cover the filing fee.

[X] The Commissioner is hereby authorized to charge payment of the following fees associated with this communication or credit any overpayment to Deposit Account No. 50-0718. A duplicate of this sheet is attached.

[X] Any additional filing fees required under 37 CFR 1.16.

[X] Any patent application processing fees under 37 CFR 1.17.

Please file this application and conduct all future correspondence with Applicants' attorney identified below.

Respectfully Submitted,

Ann M. Knab Applicants' Attorneys

Bv Ann M. Knab

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PRESSABLE LITHIUM DISILICATE GLASS CERAMICS

5 CROSS-REFERENCE TO RELATED APPLICATIONS

This application claims priority to U.S. Provisional Application No. 60/153,916, filed September 14, 1999, U.S. Provisional Application No. 60/122,558, filed March 2, 1999, and U.S. Provisional Application No. 60/111,872, filed December 11, 1998, all which are incorporated herein by reference.

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FIELD OF INVENTION

This invention relates generally to glass-ceramics comprising lithium disilicate and more specifically to glass-ceramics for use in the manufacture of dental restorations and methods of manufacture thereof.

BACKGROUND OF THE INVENTION

The use of lithium disilicate glass ceramics for use in dental restorations has been suggested in the prior art. U.S. Patent No. 4,189,325 to Barret et al. is directed to a glassceramic comprising lithium disilicate for use in dental restorations. The glass ceramic requires the presence of Nb₂O₅ and Pt as nucleation agents. Barrett et al. introduced dental restorations made from castable lithium disilicate glass-ceramics in the Li₂O-CaO-Al₂O₃-SiO₂ system nucleated by Pt and Nb₂O₅. According to Barret et al., dental restorations are made by casting a melt into an investment mold, and devitrifying thereafter.

U.S. Patent No. 4,515,634 to Wu et al. is directed to a castable glass-ceramic composition wherein the glass is melted and cast into a shape and is crystallized after it has been shaped. Therefore, the crystallization process is performed by the technician making the restoration, not the manufacturer of the dental material. Wu set al. suggests one way to improve properties of castable lithium disilicate dental restorations within the same Li₂O-





 $CaO-Al_2O_3-SiO_2$ system as described by Barrett et al. is by utilization of P_2O_5 as a nucleating agent. Both Barrett et al. and Wu et al. describe castable compositions having CaO as an essential ingredient believed to improve chemical durability of the resulting glass-ceramics. Chemical durability is one of the major issues that the Wu and Barrett inventions fail to address. For example, total alkali leaching rates for materials presented in Wu's examples were four to five times higher than those for commercial dental porcelain.

Castable dental ceramics as described in Barret et al. and Wu et al. employ melting glass ingots supplied by a manufacturer and casting dental articles into a refractory investment mold. Following the casting process, the cast articles are devitrified (crystallized) by the required heat-treatment steps. This process is very similar to casting metals whereby a heat-treatment step follows the casting process to increase hardness and strength.

U.S. Patent Nos. 5,507,981 and 5,702,514 to Petticrew teach lithium disilicate compositions for use in dental restorations, but the method described therein implies forming glass into the shape of a dental restoration at temperatures much higher than the melting temperature of lithium disilicate and heat-treating the dental restoration after forming to convert the glass into a glass-ceramic.

German Patent Application No. DE19647739 to Schweiger et al. is directed to lithium disilicate compositions for use in dental restorations. The glass-ceramic bodies or blanks used to press dental restorations are defined as "sinterable glass ceramics" which are produced from the starting amorphous glass powder by simultaneous sintering and powder crystallizing, which process is also known as surface crystallization. The glass must be in powder form to be crystallized. Additionally, the lithium disilicate compositions therein require the presence of La₂O₃, MgO and ZnO.

Many of the lithium disilicate compositions in the prior art require casting of the glass into the desired shape and crystallizing thereafter. The glass must be formed into the finally desired shape and thereafter heat treated to crystallize into a lithium disilicate phase. This may result in structural and other problems, since the microstructure is not formed by the dental materials manufacturer, but by the technician fabricating the dental restoration.

GC Corporation, et al. Exhibit 1006, p.8

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Overprocessing by a technician may change the microstructure of the material to something not preferred or desired by the dental materials manufacturer. Moreover, some of the prior art compositions require the forming of the glass ceramics by surface crystallization, limiting the forming and compositional possibilities of the material.

It is desirable to provide a lithium disilicate glass-ceramic which is pressable after the lithium disilicate is formed. It is beneficial to provide a lithium disilicate glass ceramic for use in the fabrication of dental restorations wherein crystallization is carried out by the dental materials manufacturer in the most controlled manner. It is beneficial to provide translucent lithium disilicate glass ceramics having high strength and good presssability.

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SUMMARY OF THE INVENTION

This invention is directed to lithium disilicate (Li₂Si₂O₅) based glass-ceramics comprising silica, lithium oxide, alumina, potassium oxide and phosphorus pentoxide in addition to other components listed below. The glass-ceramics are useful in the fabrication of single and multi-unit dental restorations including but not limited to orthodontic appliances, bridges, space maintainers, tooth replacement appliances, splints, crowns, partial crowns, dentures, posts, teeth, jackets, inlays, onlays, facing, veneers, facets, implants, abutments, cylinders, and connectors made by heat pressing into refractory investment molds produced using lost wax techniques. The glass-ceramics have good pressability, i.e., the ability to be formed into dental articles by heat pressing, also known as hot pressing, or injection molding, using commercially available equipment.

In accordance with one embodiment directed to the process of making the glassceramics, the compositions herein are melted at about 1200° to about 1600°C and preferably in the range of about 1300° to about 1400°C for a period of time, preferably for about 4 hours and thereafter quenched (e.g., water quenched or roller quenched) or cast into steel molds, or alternately, cooled to the crystallization temperature.

The resulting glass is heat-treated to form a glass-ceramic via a one or two step heattreatment cycle preferably in the temperature range of about 400° to about 1100°C. This



crystallization heat treatment may comprise a nucleation step and a crystal growth step. Depending on the composition, the first, nucleation step, may be carried out in the range of about 450°C to about 700°C and preferably in the range of about 500°C to about 650°C for about 0.5 to about 4 hours and the second, crystal growth step, may be carried out in the range of about 800°C to about 1000°C and preferably in the range of about 830°C to about 930°C for about 0.5 to about 48 hours. The most preferable heat treatment comprises about a one hour soak at about 645°C and a subsequent four hour soak at about 850°C.

The resulting glass ceramics are then pulverized into powder and used to form pressable pellets and/or blanks of desired shapes, sizes and structures. Additives may be mixed with the powder prior to forming into pellets or blanks. These pellets and blanks may be used for pressing cores or other frameworks or shapes for dental restorations. The blank or pellet may be subjected to viscous deformation at a temperature in the range of about 800° to about 1200°C, and more preferably in the range of about 850° to about 950°C, and most preferably at less than about 930°C, under vacuum and with the application of pressure of between about 2 to about 8 bar (0.2 to 0.8 MPa) and preferably no greater than about 6 bar (0.6 MPa) to obtain a dental restoration. Moreover, it is possible that the blanks may be machined to a dental restoration of desired geometry.

BRIEF DESCRIPTION OF THE DRAWINGS

Features of the present invention are disclosed in the accompanying drawing, wherein:

Figure 1 is perspective view of a plunger system in a pressing furnace for use in the fabrication of dental restorations in accordance with the invention.

25 DESCRIPTION OF THE INVENTION

As will be appreciated, the present invention provides glass-ceramic compositions comprising a glassy matrix and lithium disilicate ($Li_2Si_2O_5$). The glass-ceramics are useful for the fabrication of dental restorations. The compositions of the lithium disilicate glass-

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ceramics comprise inter alia, silica, lithium oxide, alumina, potassium oxide and phosphorus pentoxide in the ranges given in Table 1 below. The glass-ceramic compositions of the invention have a combination of properties useful for dental restorations. The glass-ceramics have good pressability, i.e., the ability to be formed into dental articles by heat pressing, also known as hot pressing, or injection molding, using commercially available equipment.

Pressable ceramics employ some form of hot-pressing or injection-molding of the <u>glass-ceramic</u> materials, which are typically in the form of pellets. The pellets contain one or more crystalline phases and their morphology as well as volume fraction are not significantly altered in the course of pressing. One reason for this is that the pressing temperature is typically lower than the melting temperature of the crystalline phases. This is a major advantage because microstructure is formed in the controlled conditions by the manufacturer of the glass-ceramic materials, e.g., pellets. Following pressing, the resulting dental article does not require crystallization heat-treatment.

The glass-ceramic pellets can be formed by a number of processes: (1) Glass can be cast into the shape of pellet. Pellets are taken from the mold and crystallized. These pellets can not be shaded by the addition of pigments. (2) Glass can be crystallized in bulk and subsequently milled into powder. Pigments and other additives can be added to the powder. The powder is formed into a pellet and partially or fully sintered. Pigments, if added, create color centers that impart a certain color to a translucent body of the dental article pressed from the pellet. The mechanism of crystallization in the two processes described above is volume crystallization.

Alternatively, surface crystallization may be utilized to crystallize a portion of the glass into one or more crystal phases. This involves milling glass into powder. Pigments and other additives can be added to the powder. This glass powder (amorphous, not crystalline)

25 is formed into a pellet. The glass pellet is sintered and crystallized in the same firing cycle. Not all glass-ceramic materials can be crystallized and sintered simultaneously. Only certain materials and compositions prone to surface crystallization can be processed this way. The glass-ceramics processed from glass powder via simultaneous sintering and crystallization are

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sometimes called "sintered" glass-ceramics. Another term that can be used is "sinterable" glass-ceramics.

The compositions herein are prepared by mixing, in the desired proportions, the oxides and/or compounds that decompose to form the oxides, followed by fusing the ingredients to obtain the compositions in Table 1. Convenient raw material include lithium carbonate, silica, alumina, carbonates of K, Na, Ca, ammonium phosphate, tricalcium aluminate, aluminum phosphate or aluminum metaphosphate and if necessary, Ta_2O_5 , CeO₂, Tb₄O₇, titanium dioxide, and zirconium dioxide.

| | | | Table | 1 | | |
|--------------------------------|--------------------------|-------------------------|-------------------------|-------------------------|---------------------------|------------------------|
| Oxide, wt% | Range 1 | Range 2 | Range 3 | Range 4 | Range 5 | Range 6 |
| SiO ₂ | about 62 to about 85 | about 64 to about 70 | about 62-85 | about 64-70 | about 64 to about 70 | about 62 to abo 76 |
| B ₂ O ₃ | about 0 toabout 4.9 | about 0 - about 2.7 | about 0-4.9 | about 0-2.7 | about 0.5 - about 3.0 | about 0 - about |
| Al ₂ O ₃ | about 1.5 to about 10 | about 1.5 to about 6.0 | about 5.1-10 | about 5.2-9.0 | about 1.5 to about 6.0 | about 1.5 to ab 10 |
| F | about 0 to about 1.5 | about 0 to about 1.5 | about 0 to about 1.5 | about 0 to about 1.5 | about 0 to about 1.5 | about 0 to about 1.5 |
| ZnO | about 0 to about 5 | about 0 to about 2 | about 0-5 / | about 0-2 | | about 0 to about |
| CaO | about 0 to about 7 | about 0 to about 0.9 | about 0-7 | about 0-0.9 | about 0 to about 0.9 | about 0 to abou |
| MgO | about 0 to about 2 | about 0 to about 2 | about 0 to about 2 | about 0 to about 2 | | about 0 to about |
| BaO | about 0 to about 7 | about 0 to about 7 | about 0 to about 7 | about 0 to about 7 | about 0 to about 7 | about 0 to about |
| SrO | about 0 to about 1 | about 0 to about 1 | about 0 to about 1 | about 0 to about 1 | about 0 to about 1 | about 0 to about |
| Cs ₂ O | about 0 to about 5 | about 0 to about 5 | about 0, to about 5 | about 0 to about 5 | about 0 to about 5 | about 0 to about |
| Li ₂ O | about 8 to about 19 | about 10 to about 15 | about/8-19 | about 10-15 | about 10 to about 15 | about 8 to about |
| K ₂ O | about 2.5 to about 7 | about 2.5 to about 5 | about 0-7 | about 0-5 | about 2.2 to about 5 | about 0 to about |
| Na ₂ O | about 0 to about 5 | about 0 to about 3 | about 0-5 | about 0-3 | about 0.5 to about 3 | about 0 to abo |
| TiO ₂ | about 0 to about 2 | about 0 to about 2 | about 0 to about 2 | about 0 to about 2 | about 0 to about 2 | about 0 to abo |
| ZrO ₂ | about 0 to about 3 | about 0 to about 3 | about 0 to about 3 | about 0 to about 3 | about 0 to about 3 | about 0 to abo |
| P ₂ O ₅ | about 0.5 to about 12 | about 2 to about 7 | about 0.5-12 | about 2-7 | about 2 to about 7 | about 0.3 to ab 7.0 |
| SnO ₂ | about 0 to about 1 | about 0/to about 1 | about 0 to about 1 | about 0 to about 1 | about 0 to about 1 | about 0 to abo |
| Sb ₂ O ₃ | about 0 to about 1 | about 0 to about 1 | about 0 to about 1 | about 0 to about 1 | about 0 to about 1 | about 0 to abo |
| Y ₂ O ₃ | about 0 to about 3 | about 0 to about 3 | about 0 to about 3 | about 0 to about 3 | about 0 to about 3 | about 0 to abo |
| CeO ₂ | about 0 to about 1 | about 0 to about 1 | about 0 to about 1 | about 0 to about 1 | about 0 to about 1 | about 0 to abo |
| Eu ₂ O ₃ | about 0 to about 1 | about 0 to about 1 | about 0 to about 1 | about 0 to about 1 | about 0 to about 1 | about 0 to abo |
| Tb ₄ O ₇ | about 0 to about 1 / | about 0 to about 1 | about 0 to abo |
| Nb ₂ O ₅ | about 0 to about 2 | about 0 to about 2 | about 0 to about 2 | about 0 to about 2 | about 0 to about 2 | about 0 to abo |
| Ta ₂ O ₅ | about 0 to about 2 | about 0 to about 2 | about 0 to about 2 | about 0 to about 2 | about 0 to about 2 | about 0.5 to al 8.0 |

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The compositions in Table 1 are melted at about 1200° to about 1600°C and preferably in the range of about 1300° to about 1400°C for a period of time, preferably for about 4 hours and thereafter quenched (e.g., water quenched or roller quenched), cast into steel molds, or alternately, cooled to the crystallization temperature. If the melt is cooled to the crystallization temperature, it may remain in the same furnace.

The resulting glass is heat-treated to form glass-ceramics using a one or a two step heat-treatment cycle preferably in the temperature range of about 400° to about 1100°C. This crystallization heat-treatment may comprise a nucleation step and a crystal growth step. Depending on the composition, the first, nucleation step, may be carried out in the range of about 450°C to about 700°C and preferably in the range of about 500°C to about 650°C for about 0.5 to about 4 hours and the second, crystal growth step, may be carried out in the range of about 800°C to about 1000°C and preferably in the range of about 830°C to about 930°C for about 0.5 to about 48 hours. The most preferable heat treatment comprises about a one hour soak at about 645°C and a subsequent four hour soak at about 850°C.

The glass ceramics comprise lithium disilicate. The resulting glass ceramics are then pulverized into powder sieved to –200 mesh to provide powder with average particle sizes of about 30 to about 40 microns. Pigments, fluorescing agents, opacifying agents, and the like may be added to the powder in a wide range in an amount between about 0 and about 6 wt% and preferably in the amount of between about 0 and about 5 wt% and most preferably in the amount of about 0% to about 3 wt%. Moreover, reinforcing agents may be added to the powder in an amount of to about 30 vol % and more preferably in an amount of from about 0 to about 30 vol % and more preferably in an amount of from about 0 to about 30 vol %. The reinforcing agents may include fibers, whiskers, and particulate fillers and may be fabricated of any known material, preferably a glass or ceramic material.

The powders may be used to form and fuse pressable pellets and/or blanks of desired shapes, sizes and structures. These pellets and blanks may be used for pressing cores or other frameworks or shapes for dental restorations. The blank or pellet may be subjected to viscous deformation at a temperature in the range of about 800° to about 1200°C, and more

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preferably in the range of about 850° to about 950°C, and most preferably at less than about 930°C, under vacuum and with the application of pressure of between about 2 to about 8 bar (0.2 - 0.8 MPa) and preferably no greater than about 6 bar (0.6 MPa) to obtain a dental restoration. Moreover, it is possible that the blanks may be machined to a dental restoration of desired geometry using commercially available milling equipment such as the Maho HGF 500 5 Axis CNC Milling Machine available from Fraunhofer Institut Produktionstechnologie, Germany.

In an alternative method herein, the compositions in Table 1 are melted at about 1200° to about 1600°C and preferably in the range of about 1300° to about 1400°C for a period of time, preferably for about 4 hours and thereafter water quenched or cast into steel molds.

The quenched glass is comminuted to a powder. Pigments, fluorescing agents, opacifying agents, and the like may be added to the powder in a wide range in an amount between about 0 and about 6 wt% and preferably in the amount of between about 0 and about 5 wt% and most preferably in the amount of about 0% to about 3 wt%. Moreover, reinforcing agents may be added to the powder in an amount of from about 0 to about 30 vol % and more preferably in an amount of from about 0 to about 20 vol %. The reinforcing agents may include fibers, whiskers, and particulate fillers and may be fabricated of any known material, preferably a ceramic material.

The powder is compacted into a pellet or starting blank. The blank is thereafter simultaneously sintered and crystallized. Heat treatment may be one or more cycles in the temperature range of about 400° to about 1100°C. The crystallization may comprise a nucleation step and a crystal growth step. Depending on the composition, the first, nucleation step, may be carried out in the range of about 450°C to about 700°C and preferably in the

25 range of about 500°C to about 650°C for about 0.5 to about 4 hours and the second, crystal growth step, may be carried out in the range of about 800°C to about 1000°C and preferably in the range of about 830°C to about 930°C for about 0.5 to about 48 hours. The most preferable heat treatment comprises about a one hour soak at about 645°C and a subsequent

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four hour soak at about 850°C.

The blank or pellet may be subjected to viscous deformation at a temperature in the range of about 800° to about 1200°C, and more preferably in the range of about 850° to about 950°C, and most preferably at less than about 930°C, under vacuum and with the application of pressure of about between about 2 to about 8 bar (0.2 - 0.8 MPa) and preferably no greater than 6 bar (0.6 MPa) to obtain a dental restoration. Moreover, it is possible that the blanks may be machined to a dental restoration of desired geometry.

To achieve the required combination of properties, namely sufficient strength, formability below 950°C, by heat-pressing using commercially available dental presses such as the Autopress® Plus available from Jeneric/Pentron, Wallingford, CT, translucency and chemical durability, the optimal chemical combinations and crystallization treatment of the present invention are necessary. The best properties are obtained when the lithium metasilicate (Li₂SiO₃) and silica phases are nearly absent, the volume fraction of Li₃PO₄ is less than about 5% and the volume fraction of lithium disilicate (Li₂Si₂O₅) is between about 35% and about 60%. High aspect ratio morphology of the lithium disilicate phase is important and is believed to enhance mechanical properties, i.e., strength and fracture toughness of the glass-ceramic.

Li₂O and SiO₂ are instrumental in crystallizing the required amount of the lithium dislicate phase in the compositions of the present invention. Additionally, BaO and Cs₂O stabilize the residual glass and boost the refractive index of the residual glass to match that of lithium disilicate. Al₂O₃ and to a lesser extent, B₂O₃, if less than 3%, yield chemically durable glass-ceramics that exhibit a sufficiently low solubility. Alkali (Na, K, Cs) and alkaline earth metal (Ca, Ba) oxides are required to lower processing temperatures of the glass-ceramic. However some of them affect chemical durability more than others. With

25 respect to the following group of alkali metals and alkaline earth metals of potassium, calcium, sodium, and barium; potassium is associated with the smallest decrease in chemical durability of lithium containing glasses, with calcium being next, and sodium and barium affecting chemical durability the most. However, the best combination of properties is

GC Corporation, et al. Exhibit 1006, p.15

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achieved when those oxides are used in combination to achieve the so-called "mixed alkali effect." F lowers the viscosity of the glass-matrix and enhances formability at temperatures below about 950°C. Y₂O₃ in combination with Ce₂O₃, Eu₂O₃ and Tb₄O₇ modify the refractive index as well as impart fluorescence. Nb₂O₅ and Ta₂O₅ modify the refractive index as well as aid nucleation and chemical durability of the resulting glass ceramics.

The following Table 2 illustrates examples of the compositions of the invention.

| _/ | | | | | | | Table | 2 | | | | | 1 | | |
|--------------------------------|-------|-------|-------|-------|-------|-------|-------|-------|----------|-----------|-----------|--------|-----------|-----------|--------|
| Oxide, wt% | Ex. 1 | Ex. 2 | Ex. 3 | Ex. 4 | Ex. 5 | Ex. 6 | Ex. 7 | Ex. 8 | Ex. 9 | Ex. 10 | Ex. 11 | Ex. 12 | Ex. 13 | Ex. 14 | E 1 |
| SiO ₂ | 68.8 | 70.0 | 70.1 | 65.4 | 70.7 | 70.9 | 68.1 | 69.5 | 72 | 69.0 | 68.8 | 63 | 67.2 | 68.9 | |
| B ₂ O ₃ | 1.2 | 1.0 | | | | | | 1.3 | 2.9 | | 1.3 | 1.0 | 1.2 | 0.9 | |
| Al ₂ O ₃ | 4.8 | 4.9 | 5.2 | 8.1 | 5.3 | 5.3 | 4.7 | 4.8 | 5.1 | 6.0 | 4.8 | 5 | 4.7 | 4.7 | |
| F | | | | 1.4 | 0.2 | | | | | | 7 | | | | |
| ZnO | | | | | | | | | | / | / | | | | - |
| CaO | 0.9 | 0.5 | 2.2 | | | | 2.0 | 2.0 | | | 1.0 | 1 | 1 | 0.5 | |
| MgO | | | | | | | | | | 7 | | | | | |
| BaO | 2.8 | 1.4 | | 3.8 | 5.4 | 5.4 | | | 2.0 | 2.7 | 2.8 | 2.7 | 2.7 | 1.4 | |
| SrO | | | | | • | | | | 7- | | | | | | |
| Cs ₂ O | | | | 4.1 | 4.9 | 4.9 | | / | | | | | | | |
| Li ₂ O | 14.4 | 14.7 | 14.7 | 13.4 | 11.0 | 10.8 | 14.3 | 14,6 | 13.0 | 15.3 | 14.4 | 15 | 14.1 | 14.3 | |
| K ₂ O | 2.5 | 2.5 | 4.6 | 1.7 | | | 4.4 | 4.5 | 2.0 | 2.1 | 2.2 | 2.2 | 2.2 | 2.0 | |
| Na ₂ O | 1.4 | 1.4 | | | | | / | | | | 1.5 | 1.4 | 1.4 | 1.3 | |
| TiO ₂ | | | | | | | 7- | | | | | | | | - |
| ZrO ₂ | | | | | | | / | | | | | | | | - |
| P ₂ O ₅ | 3.3 | 3.6 | 3.4 | 2.7 | 2.7 | 2.7 | 3.3 | 3.4 | 3.0 | 4.0 | 3.3 | 3.5 | 3.2 | 3.5 | |
| SnO ₂ | | | | | | 7- | | | | | | | | | - |
| Sb ₂ O ₃ | | | | | / | | | | | | | | | ****** | - |
| Y ₂ O ₃ | | | | | | | | | 0.5 | 0.8 | | | ****** | | C |
| CeO ₂ | | | | | / | | 0.4 | | 0.5 | | | 0.4 | 0.4 | 0.3 | |
| Eu ₂ O ₃ | | | | / | | | | | 0.5 | | | | | ****** | |
| Tb ₄ O ₇ | | | | | | | 0.8 | | | | | 0.9 | | 0.3 | • |
| Nb ₂ O ₅ | | | | / | | | | | ****** | | | | | | - |

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|---|------|------|------|-----|------|--------------------|--------------------|-----|--------------------|------|--------------------|
| Ta ₂ O ₅ | | | | 2.0 | | | | 1.9 | 2.0 | 1.8 | |
| Three- Point Flexural Strength per ISO 6872, | | | | | | 430 <u>+</u> 70 | 420 <u>+</u> 60 | | 440 <u>+</u> 60 | | 400 <u>+</u> 50 |
| MPa As- pressed Opacity (relativeo pacity | | | | | | 50 | 42 | | 25 | | 34 |
| units) CTE (25°C- 500°C), 10 ⁻⁶ °C ⁻¹ | 10.4 | | | | | 8.7 | 10.1- 10.6 | | 10.5 | 10.0 | 9.9 |

The following examples illustrate the invention.

Examples

Glass-ceramic compositions of the present invention were utilized to make glassceramic pellets. These pellets were used to make rectangular bars and rods for measuring the flexural strength by heat-pressing these pellets into the cavity of the refractory investment mold. The process was the same as that used to make dental restorations. The three-point flexure strength was greater or equal to 300 MPa. This strength is sufficient for multi-unit restorations such as anterior bridges. The compositions have a significant amount of glass phase, i.e., approximately 50% glass phase which enhances the pressability thereof.

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Moreover, the closeness of refractive indices of the matrix glass (\sim 1.5) and that of the crystallized phase - lithium disilicate - (\sim 1.55) allows for the possibility of translucent glass-ceramics. Specifically in the present invention, the refractive index of the glass matrix is increased to match that of the lithium disilicate phase by adding small amounts of heavy ions such as, but not limited to, Sr, Y, Nb, Cs, Ba, Ta, Ce, Eu and Tb.

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The following examples in Table 3 illustrate the effect of different additions on translucency, strength and reactivity with investment of the resulting glass ceramics. The composition of Example 11 (Table 2) was selected as a control composition for the purposes of this study. This composition was modified by adding 0.13 mole% of CeO₂, or 0.06 mole% of Tb₄O₇, or 0.26 mole% of Ta₂O₅, or La₂O₃, or Y₂O₃; and combinations of the latter with CeO₂. Opacity was measured on the pressed disk using an optical densitometer. Reactivity

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with investment was evaluated qualitatively by visual inspection of disks and copings prior to and after sand-blasting of the reaction layer. Surfaces of the disks were inspected for pittings under low-magnification (8x) stereomicroscope. Compositions comprising combinations of Ta_2O_5 , and CeO₂ were found to have the best combination of high translucency (low opacity) and low reactivity with the investment.

| Oxide, wt% | Ex, | Ex, | Ex. | Ex. |
|---|--------------------|-------|------|------|------|------|------|------|--------------------|-------------|
| | 11* | 16 | 17 | 18 | 19 | 20 | 21 | 22 | 13* | 23 |
| SiO ₂ | 68.8 | 68.12 | 68.5 | 68.2 | 67.5 | 67.8 | 67.9 | 68.0 | 67.2 | 67.5 |
| B ₂ O ₃ | 1.3 | 1.2 | 1.2 | 1.2 | 1.2 | 1.2 | 1.2 | 1.2 | 1.2 | 1.2 |
| Al ₂ O ₃ | 4.8 | 4.7 | 4.8 | 4.7 | 4.7 | 4.7 | 4.7 | 4.7 | 4.7 | 4.7 |
| CaO | 1.0 | 1.0 | 1.0 | 1.0 | 1.0 | 1.0 | 1.0 | 1.0 | 1.0 | 1.0 |
| BaO | 2.8 | 2.7 | 2.8 | 2.7 | 2.7 | 2.7 | 2.7 | 2.7 | 2.7 | 2.7 |
| Li ₂ O | 14.4 | 14.3 | 14.3 | 14.3 | 14.1 | 14.2 | 14.2 | 14.2 | 14.1 | 14.1 |
| K ₂ O | 2.2 | 2.2 | 2.2 | 2.2 | 2.2 | 2.2 | 2.2 | 2.2 | 2.2 | 2.2 |
| Na ₂ O | 1.5 | 1.4 | 1.5 | 1.4 | 1.4 | 1.4 | 1.4 | 1.4 | 1.4 | 1.4 |
| P ₂ O ₅ | 3.3 | 3.3 | 3.3 | 3.3 | 3.3 | 3.3 | 3.3 | 3.3 | 3.2 | 3.3 |
| Y ₂ O ₃ | ~~~~~ | 1.04 | | | | | 1.04 | | | |
| CeO ₂ | | | 0.39 | | | | 0.39 | 0.39 | 0.39 | 0.39 |
| Tb ₄ O ₇ | | | | 0.85 | | | | 0.85 | | |
| Ta ₂ O ₅ | | | | | 2.02 | * | | | 2.01 | |
| La2O3 | | | | | | 1.5 | | | | 1.49 |
| 3-pt Flexure Strength per ISO 6872, MPa | 420 <u>+</u> 60 | | | | | | | | 440 <u>+</u> 60 | |
| As-Pressed Rod 3- | 370± | | | | | | | | 370 <u>+</u> | |
| pt Flexure Strength, MPa | 40 | | | | | | | | 30 | |
| As-pressed Opacity (relative opacity units) | 42 | 35 | 39 | 37 | 27 | 25 | 31 | 32 | 25 | 24 |
| Reactivity with | | Medi | Lowe | High | Medi | High | Lowe | Medi | Lowe | The |
| investment material | | um | r | er | um | er | r | um | r | High est |

Table 3

*Correspond to compositions in Table 2.

Pellets of the compositions of Examples 2, 11, 13 and 14 were used to press a variety of dental articles in the AutoPress® dental press (Jeneric/Pentron, Wallingford, CT) at pressing cycles carried out under vacuum and involving heating from 700°C to 920°C and holding the temperature for 20 minutes prior to initiation of the pressing cycle. Pressure of 0.5 MPa was applied for 7 minutes through a mold-plunger assembly schematically shown in Figure 1. The plunger assembly used to press the pellets into dental restorations may be the system set forth in copending commonly assigned application Serial No. 09/431,659, filed

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GC Corporation, et al. Exhibit 1006, p.18



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with investment was evaluated qualitatively by visual inspection of disks and copings prior to and after sand-blasting of the reaction layer. Surfaces of the disks were inspected for pittings under low-magnification (8x) stereomicroscope. Compositions comprising combinations of Ta_2O_5 , and CeO₂ were found to have the best combination of high translucency (low opacity) and low reactivity with the investment.

| Oxide, wt% | Ex. | Ex. | Ex. | Ex. | Ex. | Ex. | Ex. | Ex. | Ex. | Ex. |
|---|--------------------|------------|-----------|------------|------------|------------|-----------|------------|--------------------|--------------------|
| Online, intro | 11* | 16 | 17 | 18 | 19 | 20 | 21 | 22 | 13* | 23 |
| SiO ₂ | 68.8 | 68.12 | 68.5 | 68.2 | 67.5 | 67.8 | 67.9 | 68.0 | 67.2 | 67.5 |
| B ₂ O ₃ | 1.3 | 1.2 | 1.2 | 1.2 | 1.2 | 1.2 | 1.2 | 1.2 | 1.2 | 1.2 |
| Al ₂ O ₃ | 4.8 | 4.7 | 4.8 | 4.7 | 4.7 | 4.7 | 4.7 | 4.7 | 4.7 | 4.7 |
| CaO | 1.0 | 1.0 | 1.0 | 1.0 | 1.0 | 1.0 | 1.0 | 1.0 | 1.0 | 1.0 |
| BaO | 2.8 | 2.7 | 2.8 | 2.7 | 2.7 | 2.7 | 2.7 | 2.7 | 2.7 | 2.7 |
| Li ₂ O | 14.4 | 14.3 | 14.3 | 14.3 | 14.1 | 14.2 | 14.2 | 14.2 | 14.1 | 14.1 |
| K ₂ O | 2.2 | 2.2 | 2.2 | 2.2 | 2.2 | 2.2 | 2.2 | 2.2 | 2.2 | 2.2 |
| Na ₂ O | 1.5 | 1.4 | 1.5 | 1.4 | 1.4 | 1.4 | 1.4 | 1.4 | 1.4 | 1.4 |
| P ₂ O ₅ | 3.3 | 3.3 | 3.3 | 3.3 | 3.3 | 3.3 | 3.3 | 3.3 | 3.2 | 3.3 |
| Y ₂ O ₃ | | 1.04 | | | | | 1.04 | | | |
| CeO ₂ | | | 0.39 | | | | 0.39 | 0.39 | 0.39 | 0.39 |
| Tb ₄ O ₇ | | | | 0.85 | | | | 0.85 | | |
| Ta ₂ O ₅ | | | | | 2.02 | | | | 2.01 | |
| La2O3 | | | | | | 1.5 | | | | 1.49 |
| 3-pt Flexure Strength per ISO 6872, MPa | 420 <u>+</u> 60 | | | | | | | | 440 <u>+</u> 60 | |
| As-Pressed Rod 3- pt Flexure Strength, | 370 <u>+</u> 40 | | | | | | | | 370 <u>+</u> 30 | |
| MPa | | | | | | | | | | 24 |
| As-pressed Opacity (relative opacity units) | 42 | 35 | 39 | 37 | 27 | 25 | 31 | 32 | 25 | |
| Reactivity with investment material | | Medi um | Lowe r | High er | Medi um | High er | Lowe r | Medi um | Lowe r | The High est |

Table 3

*Correspond to compositions in Table 2.

Pellets of the compositions of Examples 2, 11, 13 and 14 were used to press a variety of dental articles in the AutoPress® dental press (Jeneric/Pentron, Wallingford, CT) at pressing cycles carried out under vacuum and involving heating from 700°C to 920°C and holding the temperature for 20 minutes prior to initiation of the pressing cycle. Pressure of 0.5 MPa was applied for 7 minutes through a mold-plunger assembly schematically shown in Figure 1. The plunger assembly used to press the pellets into dental restorations may be the system set forth in copending commonly assigned application Serial No. 09/431,659, filed $e_{i1}|_{c_{i1},c_{i2},c_{i1},c_{i2}}$

November 1, 1999, which is hereby incorporated by reference. Disks of compositions of examples 11 and 13 were pressed as described above and chemical solubility was measured according to ISO 6872 and found to be significantly lower than the acceptable limit of 100 ug/cm^2 .

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The glass-ceramics of the present invention have the capability to be pressed into a single or multi-unit dental restorations at temperatures below about 950°C using already existing, commercially available equipment such as the Autopress[®]available from Jeneric/Pentron, Wallingford, CT. Pressability or ability to flow and be pressed into complex shapes of dental restorations at these temperatures is achieved due to the presence of a sufficient amount of the residual glass in the resulting glass-ceramic, in the range of about 15%-60% by volume. The glass-ceramics of the present invention have the capability to be shaded by admixing pigments to the glass-ceramic powder by methods commonly used for dental porcelains.

As will be appreciated, the present invention provides a simple and effective method for producing lithium disilicate glass-ceramic compositions and dental restorations therefrom. While various descriptions of the present invention are described above, it should be understood that the various features can be used singly or in any combination thereof. Therefore, this invention is not to be limited to only the specifically preferred embodiments depicted herein.

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Further, it should be understood that variations and modifications within the spirit and scope of the invention may occur to those skilled in the art to which the invention pertains. Accordingly, all expedient modifications readily attainable by one versed in the art from the disclosure set forth herein that are within the scope and spirit of the present invention are to be included as further embodiments of the present invention. The scope of the present invention is accordingly defined as set forth in the appended claims.

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up to about 1% CeO₂; up to about 1% Eu₂O₃; up to about 1% Tb₄O₇; up to about 2% Nb₂O₅; and up to about 2% Ta₂O₅.



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A dental restoration comprising the glass-ceramic of claim 1.

A method of making a lithium disilicate dental restoration comprising: melting a starting glass composition at temperatures within the range of about 1200 to about 1600°C;

quenching the glass melt;

subjecting the quenched glass to one or more heat treatments in the temperature range of from about 400° to about 1100°C to convert the glass into a glass-ceramic;

comminuting the glass ceramic to a powder;

compacting the powder to a starting blank;

sintering the blank; and

pressing the blank into the dental restoration.

A method of making a lithium disilicate dental product comprising: melting a starting glass composition at temperatures within the range of about 1200 to about 1600°C;

quenching the glass melt;

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subjecting the quenched glass to one or more heat treatments in the temperature range of from about 400° to about 1100°C to convert the glass into a glass-ceramic;

comminuting the glass ceramic to a powder;

compacting the powder to a starting blank; and sintering the blank. The method of claim s wherein the blank is manufactured to a desired ape and structure. The method of claim 6 wherein the blank is sintered to full density. The method of claim & wherein crystallization of lithium disilicate is 9. effected in the glass after the querching step The method of claims wherein the powder is sieved to the required mesh size prior to the compacting step. The method of claims further including adding additives to the powder prior to the compacting step. The method of claim-1 wherein the additives comprise pigments, 2112. fluorescing agents, opacifying agents. The method of claim JZ wherein the additives comprise reinforcing The method of claim 3 wherein the reinforcing agents comprise one of fibers, whiskers, particles and mixtures thereof. put The method of claim 3 wherein the reinforcing agents are fabricated of ceramic material. 16

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The method of claims wherein the sintering of the starting blank comprises heating to a temperature in the range of about 800° to about 1000°C.

The method of claim, wherein the sintering of the starting blank comprises heating to a temperature in the range of about 850° to about 950°C.

18. The method of claim 6 wherein the sintered starting blank is subjected to viscous deformation at a temperature in the range of from about 850° to about 950°C under vacuum and at a pressure not exceeding about 6 bar to obtained the desired shape.

Just of desired shape

dental restoration of desired shape.

The method of claim, of wherein the dental product is a dental core and is provided with one or more coatings.

The method of claim 20 wherein the one or more coatings is selected from a ceramic, a sintered ceramic, a glass ceramic, a porcelain, a glass, a glaze, a composite and mixtures thereof.

The method of claim 20 wherein the one or more coatings has a firing temperature in the range of about 700° to about 900°C and a coefficient of thermal expansion (measured from room temperature to its transition temperature) of within about $\pm 2.0 \times 10^{-6}$ / °C of the dental product (measured at the same temperature range).

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The dental restoration of claim formed into a component selected

GC Corporation, et al. Exhibit 1006, p.24

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from orthodontic appliances, bridges, space maintainers, tooth replacement appliances, splints, crowns, partial crowns, dentures, posts, teeth, jackets, inlays, onlays, facing, veneers, facets, implants, abutments, cylinders, and connector.

A blank for the manufacture of a dental restoration comprising the 5 ceramic of claim 1. glass The method of claims wherein the glass-ceramic comprises in weight percent: about 62 to about 85% SiO₂; 10 about 1.5 to about 10% Al₂O₃; about 8 to about 19% Li₂O; about 2.5 to about 7% K₂O; and about 0.5 to about 12 % P_2O_5 . 15 The method of claim 25 wherein the glass-ceramic further comprises *2*6. in weight percent: up to about 4.9% B₂O₃; up to about 1.5% F; 20 up to about 5% ZnO; up to about 7% CaO; up to about 2% MgO; up to about 7% BaO; up to about 1% SrO; 25 up to about 5% Cs₂O; up to about 5% Na₂O; up to about 2% TiO₂; up to about 3% ZrO₂;

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up to about 1% SnO₂; up to about 1% Sb₂O₃; up to about 1% Sb₂O₃; up to about 3% Y₂O₃; up to about 1% CeO₂; up to about $1\% Eu_2O_3$; up to about 1% Tb₂O₇; up to about 2% Nb₂O₅; and up to about 2% Ta₂O₅. A method of making a lithium disilicate dental product comprising: 27. melting a starting glass comprising a composition at temperatures within the range of about 1200 to about 1600°C; cooling the glass melt to the crystallization temperature to form a glassceramic; quenching the glass-ceramic; comminuting the glass ceramic to a powder; compacting the powder to a starting blank; and sintering the blank. The method of $\operatorname{claim}(2)$ wherein the sintering imparts sufficient 28. strength for further handling of the plank. UA 34 The method of claim wherein the powder is sieved to the required mesh size prior to the compacting step.

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powder prior to the compacting step.

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The method of claim *f* further including adding additives to the





The method of claim 30 wherein the additives comprise pigments, uorescing agents, opacifying agents.

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The method of claim wherein the additives comprise reinforcing

The method of claim $\frac{32}{2}$ wherein the reinforcing agents comprise one of fibers, whiskers, particles and mixtures thereof.

The method of claim 22 wherein the reinforcing agents are fabricated of ceramic material.

The method of claim 24 wherein the sintering of the starting blank comprises heating to a temperature in the range of about 800 to about 1000°C.



The method of claim 27 wherein the sintering of the starting blank 3 ¹316. comprises heating to a temperature in the range of about 850 to about 950°C.



The method of claim 29 wherein the sintered starting blank is

subjected to viscous deformation at a temperature in the range of from about 850 to about 950°C under vacuum and at a pressure not exceeding about 7 bar to obtained the desired shape.

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The method of claim 37 wherein the sintered blank is machined to a dental restoration of desired shape.

The method of claimer wherein the dental product is a core and is



provided with one or more coatings.

40. The method of claim 27 wherein the one or more coatings is selected from a ceramic, a sintered ceramic, a glass ceramic, a porcelain, a glass, a glaze, a composite and mixtures thereof.

W W M. The method of claim 39 wherein the one or more coatings has a firing temperature in the range of about 700 to about 900°C and a coefficient of thermal expansion (measured from room temperature to its transition temperature) of within $about \pm 2.0 \times 10^{-6}$ / °C of the dental product (measured at the same temperature range).

The method of claim 9 wherein the glass-ceramic comprises in weight percent:

about 62 to about 85% SiO₂; about 1.5 to about 10% Al₂O₃; about 8 to about 19% Li₂O; about 2.5 to about 7% K₂O; and about 0.5 to about 12 % P₂O₅.

about 2 to about 7 % P_2O_5 .

A glass-ceramic composition comprising in weight percent: about 64 to about 70% SiO₂;
about 1.5 to about 6 Al₂O₃;
about 10 to about 15% Li₂O;
about 2.5 to about 5% K₂O; and

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The glass-ceramic composition of claim 43 whereby the glass-ceramic is pressable.



45. The glass-ceramic composition of claim 43 further comprising in weight percent:

up to about 1.5% F; up to about 7% BaO; up to about 1% SrO; up to about 5% Cs₂O; up to about 2.7% B₂O₃; up to about 2% ZnO; up to about 0.9% CaO; up to about 2% MgO; up to about 3% Na2Q up to about 2% up to about 3%/ZrO2; up to about 1% SnO2; up to about 1% Sb₂O₃; up to about 3% Y2O3; up/to about 1% CeO2; up to about 1% Eu₂O₃; up to about 1% Tb₄O₇; up to about 2% Nb₂O₅; and up to about 2% Ta₂O₅.

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A dental restoration comprising the glass-ceramic of claim

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A method of making a lithium disilicate dental restoration comprising: melting a starting glass composition comprising in weight percent about 64 to about 70% SiO₂; about 1.5 to about 6 Al₂O₃;



about 10 to about 15% Li₂O;

about 2.5 to about 5% K_2O ; and

about 2 to about 7 % P_2O_5 ; at temperatures within the range of about 1200° to about 1600°C;

quenching the glass melt;

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subjecting the quenched glass to one or more heat treatments in the temperature range of from about 400° to about 1100°C to convert the glass into a glass-ceramic;

comminuting the glass ceramic to a powder;

compacting the powder to a starting blank;

sintering the blank; and

pressing the blank into the dental restoration.

48. A glass-ceramic composition comprising in weight percent: about 62 to about 85% SiO₂; about 5.1 to about 10 Al₂O₃; about 8 to about 19% Li₂O; and about 0.5 to about 12 % P₂O₅.

49. The glass-ceramic composition of claim 48 whereby the glass-ceramic is pressable.

50. The glass-teramic composition of claim 48 further comprising in weight percent: up to about 7% K₂O;

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up to about 1.5% F; up to about 7% BaO; up to about 1% SrO;

up to about 5% Cs₂O; up to about 4.9% B₂O₃; up to about 5% ZnO; up to about ∇ % CaO; up to about 2% MgO; up to about 5% Na₂O; up to about 2% TiO₂; up to about 3% ZrO2; up to about 1% \$nO₂; up to about 1% Sb₂O₃; up to about 3% YO3; up to about 1% CeO2; up to about $1\% Eu_2O_3$; up to about 1% Tb_4Q_7 ; up to about 2% Nb₂O₃; and up to about 2% Ta₂O₅.

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A dental restoration comprising the glass-ceramic of claim 48.

A method of making a lithium disilicate dental restoration comprising: melting a starting glass composition comprising in weight percent about 62 to about 85% SiO₂;

about 5.1 to about 10 Al_2O_3 ;

about 8 to about 19% Li_2O ; and

about 0.5 to about 12 % P_2O_5 ; at temperatures within the range of about 1200° to about 1600°C;

quenching the glass melt;

subjecting the quenched glass to one or more heat treatments in the

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temperature range of from about 400° to about 1100°C to convert the glass into a glass-ceramic;

comminuting the glass ceramic to a powder; compacting the powder to a starting blank;

sintering the blank; and

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pressing the blank into the dental restoration.

53. A glass-ceramic composition comprising in weight percent: about 64 to about 70% SiO_2 ; about 5.2 to about 9 Al_2O_3 ;

about10 to about 15% Li₂O; and

about 2 to about 7 % P_2O_5

The glass-ceramic composition of claim 39 whereby the glass-ceramic is pressable.

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 $\sqrt{35}$. The glass-ceramic composition of claim 53 further comprising in

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weight percent:

up to about 1.5% F; up to about 7% BaO; up to about 1% SrO; up to about 5% Cs₂O; up to about 5% K₂O; up to about 2.7% B₂Ø₃; up to about 2.9% ZnO; up to about 0.9% CaO; up to about 0.9% CaO; up to about 2% MgO; up to about 3% Na₂O;





up to about 2% TiO_2 ; up to about 3% ZrO_2 ; up to about 1% SnO_2 , up to about 1% Sb_2O_3 ; up to about 1% Sb_2O_3 ; up to about 3%/ Y_2O_3 ; up to about 1% CeO_2 ; up to about 1% CeO_2 ; up to about 1% Tb_4O_7 ; up to about 2% Nb_2O_5 ; and up to about 2% Ta_2O_5 .

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A dental restoration comprising the glass-ceramic of claim 53.

A method of making a lithium disilicate dental restoration comprising: melting a starting glass composition comprising in weight percent about 64 to about 70% SiO₂; about 5.2 to about 9 Al₂O₃;

about10 to about 15% Li2O; and

about 2 to about 7 % P_2O_5 ; at temperatures within the range of about 1200° to about 1600°C;

quenching the glass melt;

subjecting the quenched glass to one or more heat treatments in the temperature range of from about 400° to about 1100°C to convert the glass into a glass-ceramic;

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comminuting the glass ceramic to a powder;

compacting the powder to a starting blank;

sintering the blank; and

pressing the blank into the dental restoration.





A glass-ceramic composition comprising in weight percent:

about 64 to about 70% SiO_2 ;

about 1.5 to about 6 Al₂O₃;

about 10 to about 15% Li₂O;

about 2 to about 7 % P_2O_5 ;

about 2.2 to about 5% K₂O;

about 0.5 to about 3% Na₂O; and

about 0.5 to about $3\% B_2O_3$.

is pressable.

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The glass-ceramic composition of claim 58 further comprising in

The glass-ceramic composition of claim s whereby the glass-ceramic

weight percent:

up to about 1.5% F; up to about 7% BaO; up to about 1% SrO; up to about 5% Cs₂O; up to about 0.9% CaO; up to about 2% TiO₂; up to about 3% ZrO₂; up to about 1% SnO₂; up to about 1% Sb₂O₃; up to about 3% Y₂O₃; up to about 1% CeO₂; up to about 1% Eu₂O₃;

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up to about 1% Tb_4O_7 ; up to about 2% Nb_2O_5 ; and up to about 2% Ta_2O_5 .

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A dental restoration comprising the glass-ceramic of claim

42.1 A method of making a lithium disilicate dental restoration comprising: melting a starting glass composition comprising in weight percent about 64 to about 70% SiO₂;

about 1.5 to about 6 Al₂O₃;

about 10 to about 15% Li₂O;

about 2 to about 7 % P_2O_5 ;

about 2.2 to about 5% K₂O;

about 0.5 to about 3% Na₂O; and

about 0.5 to about 3% B_2O_3 ; at temperatures within the range of about 1200° to about 1600°C;

quenching the glass melt;

subjecting the quenched glass to one or more heat treatments in the temperature range of from about 400° to about 1100°C to convert the glass into a glass-ceramic;

comminuting the glass ceramic to a powder;

compacting the powder to a starting blank;

sintering the blank; and

pressing the blank into the dental restoration.

63. A glass-ceramic composition comprising in weight percent: about 62 to about 76% SiO_2 ; about 1.5 to about 10 Al_2O_3 ,

GC Corporation, et al. Exhibit 1006, p.35





about 8 to about 19% Li_2O ; about .3 to about 7% P_2O_5 ; and about .5 to about 8% Ta_2O_5 .

64. The glass-ceramic composition of claim 63 whereby the glass-ceramic is pressable.

65. The glass-ceramic composition of claim 63 further comprising in weight percent:

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up to about $\frac{5}{8}$ B₂O₃. up to about \$% Na₂O; up to about 7% K₂O; up to about 1 5% F; up to about 5% ZnO; up to about 2% MgO; up to about 7% BaO; up to about 1% SrO; up to about 5% Cs₂O; up to about 7% CaO; up to about 2% TiO₂; up to about 3% ZrO₂; up to about 1% SnO₂; up to about 1% \$b₂O₃; up to about $3\% \Psi_2O_3$; up to about 1% deO₂; up to about $1\% Eu_2O_3$; up to about 1% Tb₄O₇; and up to about $2\% Nb_2O_5$.

> GC Corporation, et al. Exhibit 1006, p.36

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A dental restoration comprising the glass-ceramic of claim 63.

67. A method of making a dental restoration comprising:

melting a starting glass composition comprising in weight percent about 62 to about 76% SiO₂;

about 1.5 to about 10 Al₂O₃;

about 8 to about 19% Li₂O;

66.

about .3 to about 7% P2O5; and

about .5 to about 8% Ta₂O₅; at temperatures within the range of about 1200° to about 1600°C;

quenching the glass melt;

subjecting the quenched glass to one or more heat treatments in the

temperature range of from about 400° to about 1100°C to convert the glass into a glass-ceramic;

comminuting the glass ceramic to a powder;

compacting the powder to a starting blank;

sintering the blank; and

pressing the blank into the dental restoration.

A glass-ceramic composition comprising in weight percent: about 68.8% SiO₂; about 1.3% B₂O₃; about 4.8% Al₂O₃; about 1.0% CaO;

about 2.8% BaO; about 14.4% Li₂O;

about 2.2 % K₂O;

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GC Corporation, et al. Exhibit 1006, p.37

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about 1.5% Na₂O; and about 3.3% P₂O₅.



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The glass-ceranic of claim 68 having a flexural strength of equal to or

The glass-ceramic composition of claim 18 whereby the glass-ceramic

is pressable.

A dental restoration comprising the glass-ceramic of claim

A method of making a dental restoration comprising:

melting a starting glass composition comprising in weight percent

about 68.8% SiO₂;

about 1.3% B₂O₃; about 4.8% Al₂O₃; about 1.0% CaO; about 2.8% BaO;

about 14.4% Li2O;

about 2.2 % K₂O;

about 1.5% Na₂O; and

about 3.3% P₂O₅; at temperatures within the range of about 1200° to about 1600°C;

quenching the glass melt;

subjecting the quenched glass to one or more heat treatments in the temperature range of from about 400° to about 1100°C to convert the glass into a glass-ceramic;

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comminuting the glass ceramic to a powder;



compacting the powder to a starting blank; sintering the blank; and pressing the blank into the dental restoration.

A glass-ceramic composition comprising in weight percent:

| • |
|---|
| about 67.2% SiO ₂ ; |
| about 1.2% B ₂ O ₃ ; |
| about 4.7% Al ₂ O ₃ ; |
| about 1.0% CaO; |
| about 2.7% BaO; |
| about 14.1% Li ₂ O; |
| about 2.2 % K ₂ O; |
| about 1.4% Na ₂ O; |
| about 3.2% P ₂ O ₅ ; |
| about 0.4% CeO ₂ ; and |
| about 2% Ta ₂ O ₅ . |
| |

74. The glass-ceranic of claim 73 having a flexural strength of equal to or greater than about 440 MPa.

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The glass-ceramic composition of claim, 25 whereby the glass-ceramic

is pressable.

A dental restoration comprising the glass-ceramic of claim

A method of making a dental restoration comprising: melting a starting glass composition comprising in weight percent about 68.8% SiO₂;





| about 1.3% B ₂ O ₃ ; |
|---|
| about 4.8% Al ₂ O ₃ ; |
| about 1.0% CaO; |
| about 2.8% BaO; |
| |

about 14.4% Li₂O; about 2.2 % K₂O;

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about 2.2 /0 K2O,

about 1.5% Na₂O; and

about 3.3% P_2O_5 ; at temperatures within the range of about 1200° to about 1600°C;

quenching the glass melt;

subjecting the quenched glass to one or more heat treatments in the

temperature range of from about 400 to about 1100°C to convert the glass into a glass-ceramic;

comminuting the glass ceramic to a powder;

compacting the powder to a starting blank;

sintering the blank; and

pressing the blank into the dental restoration.





Abstract

This invention is directed to lithium disilicate (Li₂Si₂O₅) based glass-ceramics comprising silica, lithium oxide, alumina, potassium oxide and phosphorus pentoxide. The glass-ceramics are useful in the fabrication of single and multi-unit dental restorations (e.g. anterior bridges) made by heat pressing into refractory investment molds produced using lost wax techniques. The glass-ceramics have good pressability, i.e., the ability to be formed into dental articles by heat-pressing using commercially available equipment. In accordance with one embodiment directed to the process of making the glass-ceramics, the compositions herein are melted at about 1200° to about 1600°C, thereafter quenched (e.g., water quenched or roller quenched) or cast into steel molds, or alternately, cooled to the crystallization temperature. The resulting glass is heat-treated to form a glass-ceramic via a one or two step heat-treatment cycle preferably in the temperature range of about 400° to about 1100°C. The resulting glass ceramics are then pulverized into powder and used to form pressable pellets and/or blanks of desired shapes, sizes and structures which are later pressed into dental restorations.

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| | Approved us Patent and Trademark Office; U yder the Paperwork Reduction Act of 1995, no persons are required to respond to a collection of information unless | PTO/SB/10 (12-97) se through 9/30/00. OMB 0651-0031 J.S. DEPARTMENT OF COMMERCE it displays a valid OMB control number. |
| | STATEMENT CLAIMING SMALL ENTITY STATUS (37 CFR 1.9(f) & 1.27(c))SMALL BUSINESS CONCERN | Docket Number (Optional) JPP-1222NP |
| | Applicant, Patentee, orldentifier. Dmitri Brodkin, Carlino Panzera, 1 Application or Patent No. | Paul Panzera |
| | Title: <u>PRESSABLE LITHIUM DISILICATE GLASS CERAMICS</u> | identified below: |
| | NAME OF SMALL BUSINESSCONCERN Jeneric/Pentron Incorporat | ed |
| | ADDRESSOFSMALLBUSINESSCONCERN 53 North Plains Industria Wallingford, CT 06492 | 1 Road |
| | I hereby state that the above identified small business concern qualifies as a small business concern qualifies as a small business CFR Part 121 for purposes of paying reduced fees to the United States Patent and Trader of employees of the concern, including those of its affiliates, does not exceed 500 persons. F (1) the number of employees of the business concern is the average over the previous fiscal year employed on a full-time, part-time, or temporary basis during each of the pay periods of the are affiliates of each other when either, directly or indirectly, one concern controls or has the a third party or parties controls or has the power to control both. | nark Office, in that the number for purposes of this statement, ar of the concern of the persons e fiscal year, and (2) concerns |
| | I hereby state that rights under contract or law have been conveyed to and remain wi identified above with regard to the invention described in: | th the small business concern |
| | It is specification filed herewith with title as listed above. ☐ the application identified above. ☐ the patent identified above. | |
| | If the rights held by the above identified small business concern are not exclusive organization having rights in the invention must file separate statements as to their status to the invention are held by any person, other than the inventor, who would not qualify as a 37 CFR 1.9(c) if that person made the invention, or by any concern which would not qualit under 37 CFR 1.9(d), or a nonprofit organization under 37 CFR 1.9(e). | as small entities, and no rights an independent inventor under |
| - 1 | | |

Each person, concern, or organization having any rights in the invention is listed below: no such person, concern, or organization exists. each such person, concern, or organization is listed below.

<u> contat ctoashoo</u>

Separate statements are required from each named person, concern or organization having rights to the invention stating their status as small entities. (37 CFR 1.27)

I acknowledge the duty to file, in this application or patent, notification of any change in status resulting in loss of entitlement to small entity status prior to paying, or at the time of paying, the earliest of the issue fee or any maintenance fee due after the date on which status as a small entity is no longer appropriate. (37 CFR 1.28(b))

| NAME OF PERSON SIGNING Ann M. Knab |
|---|
| TITLE OF PERSON IF OTHER THAN OWNER Assistant General Counsel |
| ADDRESS OF PERSON SIGNING Wallingford, CT 06492 |
| SIGNATURE DATE DATE |
| \bigvee |

Burden Hour Statement: This form is estimated to take 0.2 hours to complete. Time will vary depending upon the needs of the individual case. Any comments on the amount of time you are required to complete this form should be sent to the Chief Information Officer, Patent and Trademark Office, Washington, DC 20231. DO NOT SEND FEES OR COMPLETED FORMS TO THIS ADDRESS. SEND TO: Assistant Commissioner for Patents, Washington, DC 20231.





FIGURE 1



FIGURE 1

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|--|--|--|---|--|---|---|
| | APPLICATION NUMBER | FILING/RECEIPT DATE | | ST NAMED APPLICANT | <u>, , , , , , , , , , , , , , , , , , , </u> | |
| <u>`</u> | 09/458,919 | 12/10/99 BF | 30DKIN | | D | JPP-1222NP |
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| | ANN M KNAB | | 0242/0 | 1119 | NOT AS | SIGNED |
| | 53 NORTH PL WALLINGFORD | AINS INDUSTRIAL | - ROAD | | | |
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| is given avoid ab 37 CFR for a sm | TWO MONTHS FROM TI andonment: Extensions 136(a): If any of items 1 | Date have been assigned to the DATE OF THIS NOTICE of time may be obtained by or 3 through 5 are indicate with 37 CFR 1.27, or □\$ | i within which filing a petition filing a petition ad as missing | . The items indicated to to file all required item accompanied by the , the SURCHARGE se | extension fee under extension fee under of forth in 37 CFR 1.1 | required below to the provision's of (6) of |
| If all red | quired items on this fo | rm are filed within the pe I) | riod set abo | ve, the total amount | owed by applican | tas a |
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| Ľ | missing.insufficient. | | | haala (iliaa faa amalia | , file a secold continue | 4-4 |
| 6 | Applicant must submit \$_ laiming such status (37 | CFR 1.27). | complete the | basic filing fee and/oi | r file a small entity s | tatement |
| 2. 1 | The following additional o | | 00 | | | |
| | \$forforfor | total claim | s over 20. ent claims ove | ər 3 | • • • • • | |
| ے ا ا ا ا ا ا ا | Applicant-must either su The cath or declaration: is missing or unsigned does not cover the no An oath or declaration in the above Application Nu The signature(s) to the o 1.43 or 1.47. A properly signed oath o Application Number and | ewly submitted items. compliance with 37 CFR 1 umber and Filing Date is re ath or declaration is/are by r declaration in compliance | ees or cancel 1. 63, includir quired. • a person oth • with 37 CFF | g residence informat ler than inventor or p R 1.63, identifying the | ion and identifying t erson qualified unde | he application by er 37 CFR 1.42, |
| | An oath or declaration in inventor(s), identifying th \$50.00 processing fea 'our filing receipt was ma 'he application was filed Applicant must file a ver | compliance with 37 CFR is application by the above a Is required since your c ailed in error because your in a language other than E <i>fied English translation of</i> and a statement that the tran | 1.63 listing th Application heck was re check was re inglish. the applicatio | e names of all invent Number and Filing D. turned without payr oturned without paym n, the \$130.00 set fo | ate, is required. nent (37 CFR 1.21(ent. rth in 37 CFR 1.17(I | m)). |
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| Custor Initial f | ner Service Center Patent Examination Divis | ion (703) 308-1202 | | | | |
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<u>PATENT</u>

IN THE UNITED STATES PATENT AND TRADEMARK OFFICE

Attorney Docket No.: JPP-1222NP Group Art Unit: 1754

| : | Dmitri Brodkin, Carlino Panzera, Paul Panzera |
|---|---|
| : | 09/458,919 |
| : | 12/10/99 |
| : | PRESSABLE LITHIUM DISILICATE GLASS CERAMICS |
| | • |

Honorable Commissioner of Patents and Trademarks Washington, D.G. 20231 Attention: BOX MISSING PARTS

Sir:

* In response to the Notice to File Missing Parts of Application Filing Date Granted, mailed January 19, 2000, submitted herewith are the following:

- 1. Declaration/Power of Attorney
- 2. Recordation Form Cover Sheet (in duplicate)
- 3. Assignment
- 4. Notice to File Missing Parts of Application, Filing Date Granted.
- 5. Return Date Stamped Post Card

The Patent Office is authorized to charge my Deposit Account No. <u>50-0718</u> the total of \$65.00 for the Declaration/Power of Attorney along with any additional fees necessary for the advancement of the application. Two copies of this sheet are enclosed for that purpose.

Respectfully submitted,

A#WX_Knab Attorney for Applicant Registration No. 33,331 Telephone: 203-265-7397, Ext. 508

Certificate of Mailing I hereby certify that this correspondence is being deposited with the United States Postal Service with sufficient postage as Express Mail in an envelope with Label No. <u>EK217582243US</u> Addressed to: Commissioner of Patents and Trademarks

Washington, D. C. 20231 on <u>February 14, 2000</u> <u>Augural MC With</u> Suzano McHugh Date: <u>February 14, 2000</u> Jeneric/Pentron Incorporated 53 North Plains Industrial Road Wallingford, Connecticut 06492 Date: February 14, 2000



DECLARATION AND POWER OF ATTORNEY

As below named inventor, I hereby declare that:

My residence, post office address and citizenship are as stated below next to my/our name,

I believe I am the original, first and sole inventor (if only one name is listed below) or an original, first and joint inventor (if plural names are listed below) of the subject matter which is claimed and for which a patent is sought on the invention entitled <u>PRESSABLE</u> <u>LITHIUM DISILICATE GLASS CERAMICS</u> the specification of which

(check one)

_____ is attached hereto. <u>X</u> was filed on <u>December 10, 1999</u> as Application Serial No. <u>09/458,919</u> and was amended on ______

(if applicable)

I hereby state that I have reviewed and understand the contents of the above-identified specification, including the claims, as amended by any amendment referred to above.

I acknowledge the duty to disclose information which is material to the examination of this application in accordance with Title 37, Code of Federal Regulations, 1.56(a).

I hereby claim foreign priority benefits under title 35, United States Code 119 of any foreign application(s) for patent or inventor's certificate listed below and have also identified below any foreign application for patent or inventor's certificate having a filing date before that of the application on which priority is claimed:

Prior Foreign Application(s).

| | | · | Prio Clair | - |
|----------|-----------|------------------------|---------------|----|
| (Number) | (Country) | (Day/Month/Year Filed) | Yes | No |
| · | | (Day/Month/Year Filed) | Yes | No |
| (Number) | (Country) | | Yes | No |
| (Number) | (Country) | (Day/Month/Year Filed) | | |

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I hereby claim the benefit under Title 35, United States Code, 120 of any United States application(s) listed below and, insofar as the subject matter of each of the claims of this application is not disclosed in the prior United States application in the manner provided by the first paragraph of Title 35, United States Code, 112, I acknowledge the duty to disclose material information as defined in Title 37, Code of Federal Regulations, 1.56(a) which occurred between the filing date of the prior application and the national or PCT international filing date of this application:

(Application Serial No.)

(Filing Date)

(Status) (patented, pending, abandoned)





(Application Serial No.)

(Filing Date)

(Status) (patented, pending, abandoned)

I hereby claim the benefit under Title 35, United States Code, § 119(e) of any United States provisional applications(s) listed below:

| PROVISIONAL APPLICATION NUMBER | FILING DATE |
|--------------------------------|-------------|
| 60/153,916 | 09/14/99 |
| 60/122,558 | 03/02/99 |
| 60/111,872 | 12/11/98 |

POWER OF ATTORNEY: As a named inventor, I hereby appoint the following attorney(s) and/or agent(s) to prosecute this application and transact all business in the Patent and Trademark Office connected therewith.

Ann M. Knab

Send Correspondence to: JENERIC/PENTRON INCORPORATED 53 North Plains Industrial Road Wallingford, Connecticut 06492 Registration No. 33,331

Direct Telephone Calls To: Ann M. Knab (203) 265-7397, Extension 508



I hereby declare that all statements made herein of my own knowledge are true and that all statements made on information and belief are believed to be true; and further that these statements were made with the knowledge that willful false statements and the like so made are punishable by fine or imprisonment, or both, under Section 1001 of Title 18 of the United States Code and that such willful false statements may jeopardize the validity of the application or any patent issued thereon.

| Full name of sole | | |
|-----------------------|---|--------|
| or first inventor: | Dmitri Brodkin | |
| Inventor's signature: | Diriti Russlin | 2/8/00 |
| Residence: | 75 Herbert Terrace, West Orange, New Jersey 07052 | Date |
| Citizenship: | Russia | |

Post Office Address: 75 Herbert Terrace, West Orange, New Jersey 07052

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| | | |
| Full name of second | | |
| joint inventor, if any: | Carlino Panzera | |
| Inventor's signature: | Edgen | Ear 8 2000 |
| | | Date |
| Residence: | 7 Huntsman Lane, Belle Mead, New Jersey 08502 | |
| Citizenship: | U.S.A. | |
| Post Office Address: | 7 Huntsman Lane, Belle Mead, New Jersey 08502 | , |
| | | |
| | | |
| Full name of third | | |
| joint inventor, if any: | | |
| | | f I |
| Inventor's signature: | Tal | 2/8/00 |
| Residence; | 1 Middleton Drive, Mt. Holly, New Jersey 08060 | Date |
| Citizenship: | U.S.A. | |
| Post Office Address: | 1 Middleton Drive, Mt. Holly, New Jersey 08060 | |
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GC Corporation, et al. Exhibit 1006, p.52

| APPLICATION NUMBER PLANABECEPT DATE INST NUMED APPLICANT ATTOMENT DOCAT HOTALE 0974658,919 12/10/99 DRODKIN DP-12.22010 0974658,919 12/10/99 DRODKIN DP-12.22010 CANN M KHA9 SSJ NORTH PLALING INDUGTRIAL ROAD DIT ASSIGNED MAELL INGERORD CT 06492 1754 DOTATION DIT ASSIGNED DIT ASSIGNED MOTICE TO FILE MISSING PARTS OF APPLICATION Filing Date Granted Polication Number and Filing Date have been assigned to this application. The items indicated below, however, are missing. Applicant was TWO MONTHS FROM THE DATE OF THIS NOTEC within which the BeloRHARGE set forth in 37 CFR 1.16(e) of 2465.00 Signal and the set of the association is a set of the set | | | A CHARGE OF COMMENT OF COMMENT | UNITED STATES Patent and Trade Address: COMMISSIC Washington | mar, otting | D TRADEMARKS |
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| <page-header>DIA DATA DATA DATA DATA DATA DATA DATA D</page-header> | 53 NORTH PL | | | PATER | ARKOTIS A | SSIGNED |
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| Pullag Date Granted polication Number and Filing Date have been assigned to this application. The items indicated below, however, are missing. Applicant wern TWO MONTHS FROM THE DATE OF THIS NOTICE within which to file all required items and pay any release required below to d abandonment. Fight 136(a). If any of items to r3 through 5 are indicated as missing, the SURCHARGE set forth in 37 CFR 1.6(a) of 0.465.00 a small entity in compliance with 37 CFR 1.27, or I \$31000 for a non-small entity, must also be timely submitted in reply inf NOTCE to avoid abandonment. The guired items on this form are filed within the period set above, the total amount owed by applicant as a mail entity (statement filed) In non-small entity is \$ The institution must submit \$\$ The form multiple dependent claims over 20. The following additional claims fees are due: The following additional claims fees are due: The opticant must submit the additional claim sees or cancel additional claims for which fees are due. The statutory obsic filing fees the additional claim fees or cancel additional claims for which fees are due. The opticant must submit the additional claim fees or cancel additional claims for which fees are due. The opticant must submit the additional claim fees or cancel additional claims for which fees are due. The opticant must submit the additional claim fees or cancel additional claims for which fees are due. The opticant must submit the additional claim fees or cancel additional claims for which fees are due. The optication Number and Filing Date is required. An oth or declaration in compliance with 37 CFR 1.63, including residence information and identifying the application by the above Application Number and Filing Date is required. An oth or declaration in compliance with 37 CFR 1.63 identifying the application by the above Application Number and Filing Date is required. An oth or declaration in compliance with 37 CFR 1.63 identifying the application by the above Application Number and Filing Date i | | | | DATEMP | | 01/19/00 |
| In the NOTICE to avoid abandonment. Irrequired items on this form are filed within the period set above, the total amount owed by applicant as a mail entity is simal entity is simal entity is simal submits. In the statutory basic filing fee is: Insting. Institution: Applicant must submit \$ | ven TWO MONTHS FROM TI d abandonment. Extensions CFR 1:136(a). If any of items 1 | Fi Date have been assigned HE DATE OF THIS NOT of time may be obtained or 3 through 5 are indi | Ing Date Gran to this application FICE within which by filing a petition cated as missing | ted . The items indicated b to file all required item accompanied by the , the SURCHARG se | below, however, are ns and pay any fees extension fee unde et forth in 37 CFR 1 | s required below to er the provision's of .16(e) of \$65.00 |
| mail entity (statement filed) non-small entity is \$ | his NOTICE to avoid abando | onment. | | | | i i |
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| Insufficient. Applicant must submit \$ | | fee is: | $t \circ t$ | | 4 | |
| claiming such status (37 CFR 1.27). 2. The following additional claims fees are due: for | insufficient. | | | | | ÷. |
| <pre> for</pre> | claiming such status (37 | CFR 1.27). | to complete the | basic filing fee and/o | r file a small entity | statement |
| for | 2. The following additional c | laims fees are due: | | | | |
| for multiple dependent claim surcharge. Applicant-must either submit the additional claim fees or cancel additional claims for which fees are due. 3. The defit or declaration: (design of unsigned. (does pot cover the newly submitted items. An oath or declaration in compliance with 37 CFR 1. 63, including residence information and identifying the application by the above Application Number and Filing Date is required. 4. The signature(s) to the oath or declaration is/are by a person other than inventor or person qualified under 37 CFR 1.42, 1.43 or 1.47. A properly signed oath or declaration in compliance with 37 CFR 1.63, identifying the application by the above Application Number and Filing Date, is required. 5. The signature of the following joint inventor(s) is missing from the oath or declaration: An oath or declaration in compliance with 37 CFR 1.63 listing the names of all inventors and signed by the omitted inventor(s), identifying this application by the above Application Number and Filing Date, is required. 6. A \$50.00 processing fee is required since your check was returned without payment. 8. The application was filed in a language other than English. Application must file a verified English translation of the application is accurate (37 CFR 1.52(d)). 9. OTHER: A copy of this notice to "Attention: Box Missing Parts." A copy of this notice to "Attention: Box Missing Parts." A copy of this notice to "Attention: Box Missing Parts." A copy of this notice to "Attention: Box Missing Parts." A copy of this notice to "Attention: Box Missing Parts." A copy of this notice to "Attention: Box Missing Parts." A copy of this notice to "Attention: Box Missing Parts." A copy of this notice to "Attention: Box Missing Parts." A copy of this notice to "Attention: Box Missing Parts." A copy of this | \$for | | | | 3 | |
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| An Dath or declaration in compliance with 37 CFR 1. 63, including residence information and identifying the application by the above Application Number and Filing Date is required. 4. The signature(s) to the oath or declaration is/are by a person other than inventor or person qualified under 37 CFR 1.42, 1.43 or 1.47. A properly signed oath or declaration in compliance with 37 CFR 1.63, identifying the application by the above Application Number and Filing Date, is required. 5. The signature of the following joint inventor(s) is missing from the oath or declaration: An oath or declaration in compliance with 37 CFR 1.63 listing the names of all inventors and signed by the omitted inventor(s), identifying this application by the above Application Number and Filing Date, is required. 6. A \$50.00 processing fee is required since your check was returned without payment (37 CFR 1.21(m)). 7. Your filing receipt was mailed in error because your check was returned without payment. 8. The application was filed in a language other than English. Application was filed in a language other than English. Application was filed and a statement that the translation is accurate (37 CFR 1.52(d)). 9. OTHER: ect the reply and any questions about this notice to "Attention: Box Missing Parts." A copy of this notice MUST be returned with the reply. stomer Service Center lial Patent Examination Division (703) 308-1202 | Applicant-must either su 3. The cath or declaration: | <i>ibmit the additional clai</i> d. | | additional claims for | which fees are du | 6. |
| A properly signed oath or declaration in compliance with 37 CFR 1.63, identifying the application by the above Application Number and Filing Date, is required. 5. The signature of the following joint inventor(s) is missing from the oath or declaration: An oath or declaration in compliance with 37 CFR 1.63 listing the names of all inventors and signed by the omitted Inventor(s), identifying this application by the above Application Number and Filing Date, is required. 6. A \$50.00 processing fee is required since your check was returned without payment (37 CFR 1.21(m)). 7. Your filing receipt was mailed in error because your check was returned without payment. 8. The application was filed in a language other than English. Applicant must file a verified English translation of the application, the \$130.00 set forth in 37 CFR 1.17(k), unless previously submitted, and a statement that the translation is accurate (37 CFR 1.52(d)). 9. OTHER: ect the reply and any questions about this notice to "Attention: Box Missing Parts." A copy of this notice MUST be returned with the reply. stomer Service Center tial Patent Examination Division (703) 308-1202 U.S. GPO 198 U.S. | An oath or declaration in the above Application Nu 4. The signature(s) to the o | compliance with 37 CF Imber and Filing Date i | s required. | | | |
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| inventor(s), identifying this application by the above Application Number and Filing Date, is required. 6. A \$50.00 processing fee is required since your check was returned without payment (37 CFR 1.21(m)). 7. Your filing receipt was mailed in error because your check was returned without payment. 8. The application was filed in a language other than English. Applicant must file a verified English translation of the application, the \$130.00 set forth in 37 CFR 1.17(k), unless previously submitted, and a statement that the translation is accurate (37 CFR 1.52(d)). 9. OTHER: ect the reply and any questions about this notice to "Attention: Box Missing Parts." A copy of this notice <u>MUST</u> be returned with the reply. stomer Service Center tial Patent Examination Division (703) 308-1202 U.S. GPO 1990; Store and a statement is a store and a statement is a store and a statement is a store and a store | | | | oath or declaration: | | |
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| APPLICATION NO. | FILING DATE | FIRST NAME | DINVENTOR | | ATTORNEY DOCKET NO. |
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| 09/458.919 | 12/10/99 | BRODKIN | | D | JPP-1222NP |
| r | | | | · · | EXAMINER |
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| ANN M KNAB | ITRON INCORP | ORATED | | ART UNIT | PAPER NUMBER |
| | AINS INDUST | | | 1755 DATE MAILED: | 4 |
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Please find below and/or attached an Office communication concerning this application or proceeding.

Commissioner of Patents and Trademarks

| | () | Application No. | Applicant(s) |
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| ¥ | office Action Summer | 09/458,919 | BRODKIN ET AL. |
| | Office Action Summary | Examiner | Art Unit |
| | | C. Melissa Koslow | 1755 |
| eriod fo | The MAILING DATE of this communication a rReply | ppears on the cover sheet wit | h the correspondence address |
| THE M - Exten after - If the - If NO - Failur - Any n | DRTENED STATUTORY PERIOD FOR RE MAILING DATE OF THIS COMMUNICATIO isions of time may be available under the provisions of 37 CFR SIX (6) MONTHS from the mailing date of this communication. period for reply is specified above is less than thirty (30) days, a period for reply is specified above the maximum statutory per re to reply within the set or extended period for reply will, by sta eply received by the Office later than three months after the main d patent term adjustment. See 37 CFR 1.704(b). | N. 1.136 (a). In no event, however, may a reply wilhin the statutory minimum of thir iod will apply and will expire SIX (6) MON tute, cause the application to become Al | reply be timely filed ty (30) days will be considered timely. ITHS from the mailing date of this communication. BANDONED (35 U.S.C. § 133). |
| 1)[]] | Responsive to communication(s) filed on _ | · | |
| 2a) | | This action is non-final. | |
| 3) | Since this application is in condition for all closed in accordance with the practice und | | |
| Dispositi | on of Claims | | |
| 4)🛛 | Claim(s) 1-77 is/are pending in the applica | tion. | |
| | 4a) Of the above claim(s) is/are with | drawn from consideration. | л |
| 5)🖂 | Claim(s) 5-7.10-15.18.19.25.26.47.52 and | 57-62 is/are allowed. | · · · · · · |
| 6)🛛 | Claim(s) 1-4. 8.16.17.20-24.27.28.35.36.39 | <u>)-41,43-46,48-51,53-55, 68, (</u> | 69, 72-74 and 77 is/are rejected. |
| 7)🛛 | Claim(s) <u>9, 56, 63-67, 70, 71, 75 and 76</u> is | /are objected to. | |
| 8) | Claims are subject to restriction an | d/or election requirement. | |
| Applicati | ion Papers | | |
| _ | The specification is objected to by the Exar | miner. | |
| 10) | The drawing(s) filed on is/are object | ed to by the Examiner. | |
| 11) | The proposed drawing correction filed on _ | is: a) approved b) |] disapproved. |
| 12) | | | |
| Priority (| under 35 U.S.C. § 119 | | |
| 13) | Acknowledgment is made of a claim for for | eign priority under 35 U.S.C. | § 119(a)-(d). |
| a) | □ All b)□ Some * c)□ None of: | | |
| | 1. Certified copies of the priority docum | ents have been received. | |
| | 2. Certified copies of the priority docum | ents have been received in a | Application No |
| * (| 3. Copies of the certified copies of the application from the Internationa See the attached detailed Office action for a | Bureau (PCT Rule 17.2(a)) | |
| 14) | Acknowledgement is made of a claim for d | | |
| Attachmer | (10) | | |
| | n(s) ilce of References Cited (PTO-892) | | |
| 16) 🛄 Nol 17) 🔲 Info | lice of Draftsperson's Patent Drawing Review (PTO-94 ormation Disclosure Statement(s) (PTO-1449) Paper N | 8) 19) Notice | w Summary (PTO-413) Paper No(s) of Informal Patent Application (PTO-152) |
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| GC | Corpor | ation, | et al. |
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| | Exhibit | 1006, | p.55 |

The listing of references in the specification is not a proper information disclosure statement. 37 CFR 1.98(b) requires a list of all patents, publications, or other information submitted for consideration by the Office, and MPEP § 609 A(1) states, "the list may not be incorporated into the specification but must be submitted in a separate paper." Therefore, unless the references have been cited by the examiner on form PTO-892, they have not been considered.

This application has been filed with informal drawings which are acceptable for examination purposes only. Formal drawings will be required when the application is allowed.

The disclosure is objected to because of the following informalities: In table 1, in the Range 1 column, "toabout" should be "to about". The meaning of "about 0" in table 1 is unclear. If the lower limit of the amount is zero, then "about" should be deleted. If the lower limit is greater than zero, then the table should state "greater than 0" instead of "about 0". Appropriate correction is required.

Claims 9 and 28 are objected to under 37 CFR 1.75(c), as being of improper dependent form for failing to further limit the subject matter of a previous claim. Applicant is required to cancel the claims.

Claim 9 depends from claim 6. The third step in claim 6 is a heat treatment step which converts the glass to a glass-ceramic. This step occurs after the quenching step. One of ordinary skill in the art would know that the lithium disilicate of claim 6 refers to a lithium disilicate glass-ceramic containing lithium disilicate crystals, from the teachings in the specification and the cited prior art which teaches lithium disilicate dental products. Therefore, it is clear to one of ordinary skill in the art that the heat treatment step which converts the glass to a glass-ceramic in

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Page 3

Application/Control Number: 09/458,919 Art Unit: 1755

claim 6 produces lithium disilicate crystals. Thus, the heat treatment step in claim 6 effects crystallization of lithium disilicate. Accordingly, claim 9 does not further limit claim 6. Claim 28 does not further limit claim 27 since the listed function of sintering is an inherent function of sintering.

Claims 3, 63 and 67 objected to because of the following informalities: "Na2O" in claim 3 should be "Na₂O". The leading zeros in claims 63 and 67 are missing. Appropriate correction is required.

Claims 8, 16, 17, 20-22, 27-42, 68, 69, 72-74 and 77 are rejected under 35 U.S.C. 112, first paragraph, as containing subject matter which was not described in the specification in such a way as to reasonably convey to one skilled in the relevant art that the inventors, at the time the application was filed, had possession of the claimed invention.

The subject of these claims is not taught by the specification. Page 5, line 18 teaches the pellet is partially or fully sintered. The specification is silent as to the density of the sintered blank or pellet. An article can be fully sintered and not have full or 100% density. Thus, the specification does not support claim 8. The specification is silent as to the sintering temperatures of the pellets or blanks. The specification does not teach the lithium disilicate dental product is a core and that the core has one of more coatings. Page 3, lines 22-26 and page 7, lines 1-5 both teach producing a lithium disilicate blank or pellet by melting the starting glass at temperatures in the range of about 1200-1600°C and quenching the melt to form a glass or cooling the glass melt to the crystallizing temperature to form a glass-ceramic. The specification does not teach quenching the formed glass-ceramic of the second cooling process. The specification never states the sintering step imparts sufficient strength for further handing of the blank.

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Page 4

Example 11 in table 2 provides the basis for the glass composition of claims 68, 69 and 72. Example 13 in table 2 provides the basis for the glass composition of claims 73, 74 and 77. The compositional values in these examples in table 2 are exact values. Thus, the taught values do not support the claimed compositions where the values are modified y the term "about". It is suggested to deleted "about" from the values in claims 68, 72, 73 and 77. The taught flexural strengths are 420 ± 60 MPa and 440 ± 60 MPa. Thus, the examples teach the flexural strengths in the ranges of 360-280 MPa and 380-500 MPa. These ranges do not support the claimed ranges of 440 MPa and greater and 420 MPa and greater. The claimed flexural ranges should be limited to the taught ranges.

The following is a quotation of the appropriate paragraphs of 35 U.S.C. 102 that form the basis for the rejections under this section made in this Office action:

A person shall be entitled to a patent unless -

(b) the invention was patented or described in a printed publication in this or a foreign country or in public use or on sale in this country, more than one year prior to the date of application for patent in the United States.

(e) the invention was described in a patent granted on an application for patent by another filed in the United States before the invention thereof by the applicant for patent, or on an international application by another who has fulfilled the requirements of paragraphs (1), (2), and (4) of section 371(c) of this title before the invention thereof by the applicant for patent.

The following is a quotation of 35 U.S.C. 103(a) which forms the basis for all

obviousness rejections set forth in this Office action:

(a) A patent may not be obtained though the invention is not identically disclosed or described as set forth in section 102 of this title, if the differences between the subject matter sought to be patented and the prior art are such that the subject matter as a whole would have been obvious at the time the invention was made to a person having ordinary skill in the art to which said subject matter pertains. Patentability shall not be negatived by the manner in which the invention was made.

Claims 1-3 and 48-50 are rejected under 35 U.S.C. 102(b) as being anticipated by Goto et

al.

This reference teaches a lithium disilicate glass-ceramic. Examples 1, 3, 6, 8 and the comparison example teach compositions which fall within the claimed ranges. The presence of PbO is not excludes due to the use of applicants' use of "comprising". Since the composition falls within the claimed composition, the taught glasses must inherently be pressable, absent any showing to the contrary. The claimed glass-ceramic reads upon that taught.

Claims 1-3, 43-45, 48-50 and 53-55 are rejected under 35 U.S.C. 103(a) as being unpatentable over Goto et al.

This reference teaches a lithium disilicate glass-ceramic comprising 65-83 wt% silica, 8-13 wt% Li₂O, 0-7 wt% K₂O, 0-7 wt% alumina, 1-4 wt% P₂O₅, 0.5-5.5 wt% MgO, 0-5 wt% ZnO, 0-5 wt% PbO, 0.5-5.5 wt% MgO + PbO + ZnO and 0-2 wt% As₂O₃ and/or Sb₂O₃. Column 4, lines 13-18 teach the presence of alumina is desirable to improve the chemical durability of the glass-ceramic. Therefore, one of ordinary skill in the art would have it obvious to include alumina in the taught glass-ceramic. The taught glass-ceramic composition overlaps the claimed glass-ceramic compositions. Product claims with numerical ranges which overlap prior art ranges were held to have been obvious under 35 USC 103. *In re Wertheim* 191 USPQ 90 (CCPA 1976); *In re Malagari* 182 USPQ 549 (CCPA 1974); *In re Fields* 134 USPQ 242 (CCPA 1962); *In re Nehrenberg* 126 USPQ 383 (CCPA 1960). Since the composition overlaps the claimed composition, the taught glasses must inherently be pressable, absent any showing to the contrary, when it has a composition within the overlapping compositions. The reference suggests the claimed glass-ceramics.

Claims 48-51 are rejected under 35 U.S.C. 103(a) as being unpatentable over Petticrew.

Page 5

Page 6

This reference teaches lithium disilicate glass-ceramic dental restorations produced from pressable lithium disilicate glass-ceramic. The taught glass-ceramic compositions are taught in column 9 and in table II. The taught glass-ceramic comprises 75-85 wt% silica, 2-5.0 wt% alumina, 8-12 wt% Li₂O, 1.5-5 wt% P₂O₅, 0-2 wt% of Na₂O, 0-2 wt% K₂O, 0-2 titania, 0-3 wt% zirconia and 0-5 wt% coloring agents. The taught glass-ceramic composition overlaps the claimed glass-ceramic compositions. The value 5.0 falls within the range of "about 5.1". Product claims with numerical ranges which overlap prior art ranges were held to have been obvious under 35 USC 103. *In re Wertheim* 191 USPQ 90 (CCPA 1976); *In re Malagari* 182 USPQ 549 (CCPA 1974); *In re Fields* 134 USPQ 242 (CCPA 1962); *In re Nehrenberg* 126 USPQ 383 (CCPA 1960). The reference suggests the claimed glass-ceramics.

Claims 1, 2, 4, 23, 24, 43, 44 and 46 are rejected under 35 U.S.C. 102(e) as being anticipated by Schweiger et al.

This reference teaches lithium disilicate glass-ceramic dental restorations, such as inlays, onlays and the other products taught in column 8, lines 55-60. Examples 1 and 14 teach glass compositions which fall within the clamed ranges. The reference teaches and the examples show the taught glass-ceramics are pressable. The claimed glass-ceramics read upon those taught.

Claims 1-4, 23, 24, 43-46, 58-51 are rejected under 35 U.S.C. 103(a) as being unpatentable over Schweiger et al.

This reference teaches lithium disilicate glass-ceramic dental restorations, such as inlays, onlays and the other products taught in column 8, lines 55-60. The taught glass-ceramic is composed of 57-80 wt% silica, 0-5.0 wt% alumina, 0.1-6 wt% La₂O₃, 0.1-7 wt% Al₂O₃ + La₂O₃, 11-19 wt% Li₂O, 0-5 wt% MgO, 0-8 wt% ZnO, 0.5-13.5 wt% K₂O, 0.5-11 wt% P₂O₅, 0-8 wt%

Page 7

colorant and 0-6 wt% of at least one of B₂O₃, F, Na₂O, zirconia, BaO and SrO. The taught glassceramic composition overlaps the claimed glass-ceramic compositions. The value 5.0 falls within the range of "about 5.1". Column 7, lines 17-30 and column 8, lines 12-15 teach the presence of alumina is desirable to improve the chemical durability of the glass-ceramic and that it improves the pressability of the glass-ceramic. Therefore, one of ordinary skill in the art would have it obvious to include alumina in the taught glass-ceramic. Product claims with numerical ranges which overlap prior art ranges were held to have been obvious under 35 USC 103. *In re Wertheim* 191 USPQ 90 (CCPA 1976); *In re Malagari* 182 USPQ 549 (CCPA 1974); *In re Fields* 134 USPQ 242 (CCPA 1962); *In re Nehrenberg* 126 USPQ 383 (CCPA 1960). The reference teaches the glass-ceramic is pressable. The reference teaches producing glass-ceramic blanks of this glass-ceramic (col. 4,lines 65-67). The reference suggests the claimed glassceramics.

Claims 5-7, 10-15, 18, 19, 25, 26, 47, 52 and 57-62 are allowable over the cited art of record.

Claims 56, 64-66, 70, 71, 75 and 76 are objected to as being dependent upon a rejected base claim, but would be allowable if rewritten in independent form including all of the limitations of the base claim and any intervening claims.

Claims 63 and 67 would be allowable if rewritten or amended to overcome the objection, set forth in this Office action.

Claims 16, 17, 68, 69, 72-74 and 77 would be allowable if rewritten or amended to overcome the rejections under 35 U.S.C. 112, set forth in this Office action.

The claimed process is not taught or suggested by the cited art of record. There is no suggestion or teaching in the art to form a sintered blank from lithium disilicate glass-ceramic particles. The cited art of record all form sintered glass blanks or form blanks from the crystallized quenched glass and then utilize these blanks to form dental products by machining or by injection molding. The claimed glass-ceramic compositions of claims 58, 63, 68 and 73 are not taught or suggested by the cited art of record. The cited art of record does not teach or suggest a dental restoration composed of the glass-ceramic of claim 53. There is no teaching or suggestion in the art to use the glass-ceramic of Goto et al to form a dental restoration.

Any inquiry concerning this communication or earlier communications from the examiner should be directed to Melissa Koslow whose telephone number is (703) 308-3817. The examiner can normally be reached on Monday-Thursday from 7:30 AM to 4:00 PM. The examiner can also be reached on alternate Fridays.

If attempts to reach the examiner by telephone are unsuccessful, the examiner's supervisor, Mark Bell, can be reached at (703) 308-3823.

The fax number for Amendments filed under 37 CFR 1.116 or After Final communications is (703) 872-9311. The fax number for all other official communications is (703) 872-9310.

Any inquiry of a general nature or relating to the status of this application or proceeding should be directed to the Group receptionist whose telephone number is (703) 308-0661 or (703) 308-0662.

cmk January 29, 2001

C. Melissa Koslow

C. Melissa Koslow – Primary Examiner Tech. Center 1700

GC Corporation, et al. Exhibit 1006, p.62

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| | A | 5,968,856 | Oct. 1999 | Schweige | r et al | 106 | 35 | | | |
| | В | 5,507,981 | Apr. 1996 | Petticrew | | 264 | 16 | | | |
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0-892 (Rev. 03-98)

Part of Paper No. 4

| | NFORMATION DISCLO der 37 CFR 1.97(b) or 1.97 | | Docket No. JPP-1222NP |
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| Re Application Of: DM | ATRI BRODKIN, CARLINO | PANZERA, PAUL PANZERA | 775. 5710 |
| Serial No. 09/458,919 | Filing Date 12/10/99 | Examiner KOSLOW, C. | Group Art Unit |
| OTPE | HUM DISILICATE GLASS C | ERAMICS | |
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| | | FR 1.97(b) | |
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| INFORMATION DISCLOSURE | Filing Date | 12/10/99 |
| INFORMATION DISCLOSURE STATEMENT BY APPLICANT | First Named Inventor | BRODKIN |
| | Group Art Unit | 1755 |
| (use as many sheets as necessary) | Examiner Name | KOSLOW, C. |
| TRADEMAN Sheet 1 of 3 | Attorney Docket Number | JPP-1222NP |
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| aminer | Cite | U.S. Patent Docu | iment d Code² | Name of Patentee or Applicant | Date of Publication of Cited Document | Pages, Columns, Lines, Where Relevant |
|--------|------|------------------|------------------|-------------------------------|--|--|
| tials' | No.1 | Number (if I | known) | of Cited Document | MM-DD-YYYY | Passages or Relevant Figures Appear |
| 2 | | 2,920,971 | | STOOKEY | 01-12-1960 | |
| | 1 | 3,006,775 | | CHEN | 10-31-1961 | |
| | | 3,157,522 | 1 | STOOKEY | 11-17-1964 | |
| | | 3,238,085 | | HAYAMI | 03-01-1966 | ······ |
| | | 3,460,987 | | MCMILLAN | 08-12-1969 | |
| | | 3,650,817 | | ΜΟΤΟΥUKI | 03-21-1972 | |
| ent | AL | 3,804,608 | | HEDLEY | 08-16-1974 4 | 974 |
| | V | 3,809,543 | | GASKELL | 05-07-1974 | |
| 4 | | 3,816,704 | | BOROM | 06-11-1974 | |
| 5 . | | 3,907,577 | 100 | KIEFER | 09-23-1975 | |
| | | 3,931,438 | | BEALL | 01-06-1976 | |
| | | 3,977,857 | | ΜΑΤΤΟΧ | 08-31-1976 | RECEIVED |
| | | 4,189,325 | | BARRETT | 02-19-1980 | |
| | | 4,414,282 | | MCCOLLISTER | 11-08-1983 | |
| | | 4,480,044 | | MCALINN | 10-30-1984 | MAY 0 9 2001 |
| | | 4,515,634 | | WU | 05-07-1985 | TOATA |
| | | 4,672,152 | | SHINOHARA | 06-09-1987 | TC 1700 |
| | | 5,219,799 | | BEALL | 06-15-1993 | |
| | | 5,466,285 | | КАМІҮА | 11-14-1995 | |
| | 1 | 5,702,514 | | PETTICREW | 12-30-1997 | |

| | | | 100 | | FOR | EIGN PATENT DOCUMEN | ΓS | |
|-----------------------|---|--------------------------|------------------------------|-----------------|--|--|--|--|
| Examiner Initials* | | Cite No. ¹ | Cite Foreign Patent Document | | Name of Patentee or Applicant of Cited Document | Date of Publication of Cited Document MM-DD-YYYY | Pages, Columns, Lines, Where Relevant Passages or Relevant Figures Appear | |
| <u> </u> |) | | | UK 924,996 | | MCMILLAN | 05-01-1963 | |
| | | | | UK 943,599 | | MCMILLAN | 12-04-1963 | |
| | | | | UK 1312700 | 1.0 | DAVIES | 04-04-1973 | |
| 1 | | | | UK 1467459 | 1 1 | DAVIES | 03-16-1977 | 41 |
| . <u>9</u> | | | 121.4 | JP 01145348 | | NAOMI | 07-06-1989 | |
| | | 1.1 | | EP 0 536 572 A1 | | CROOKER | 09-17-1992 | a de la competencia d |
| | | | 1.1.1.12 | JP 62072547 | | TOSHIMICHI | 03-04-1987 | and a state of the |
| | | | | EP 0781731 A1 | | ΤΟΜΟΥUKI | 07-02-1997 | |
| | 1 | | | JP 10188260 | | MASAHIRO | 07-21-1998 | |
| | | | | JP 50094016 | e ji se | SOJIRO | 07-26-1975 | |

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¹ Unique citation designation number. Bee attached Kinds of U.S. Patent Documents. ³ Enter Office that issued the document, by the two-letter code (WIPO Standard ST.3). ⁴ For Japanese patent documents, the indication of the year of the reign of the Emperor must precede the serial number of the patent document. ⁵ Kind of document by the appropriate symbols as indicated on the document under WIPO Standard ST. 16 if possible. ⁶ Applicant is to place a check mark here if English language Translation is attached.

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| Application Number | 09/458,919 |
| Filing Date | 12/10/99 |
| First Named Inventor | BRODKIN |
| Group Art Unit | 1755 |
| Examiner Name | KOSLOW, C. |
| Attorney Docket Number | JPP-1222NP |

Sheet 2 of 3

| | | U.S. Patent Docur | ment | U.S. PATENT DOCU | | |
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| Examiner Initials* | Cite No. ¹ | Number Kind | i Code² nown) | Name of Patentee or Applicant of Cited Document | Date of Publication of Cited Document MM-DD-YYYY | Pages, Columns, Lines, Where Relevant Passages or Relevant Figures Appear |
| 9 | <u> </u> | 5,744,208 | | BEALL | 04-28-1998 | Figures Appear |
| | <u> </u> | 5,820,989 | | REED | 10-13-1998 | |
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¹ Unique citation designation number. See attached Kinds of U.S. Patent Documents. ³ Enter Office that issued the document, by the two-letter code (WIPO Standard ST.3). ⁴ For Japanese patent documents, the indication of the year of the reign of the Emperor must precede the serial number of the patent document. ⁵ Kind of document by the appropriate symbols as indicated on the document under WIPO Standard ST. 16 if possible. ⁶ Applicant is to place a check mark here if English language Translation is attached.

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| | Initials | No.1 | item (book, magazi | ne, journal, serial, syr publisher, ci | nposium, catalog, etc.), date, pa ity and/or country where publish | age(s), volume-issue number(s), ed. | Т | | | | | |
| | ~ | | FABER, K.T., EVANS, | A.G., "Crack De | flection Processees -II. Ex | periment," Acta metall. 31, 4, pp. | | | | | | |
| | | | 577-584, 1983. | | | | | | | | | |
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| | 0 | | Journal of the American | Ceramic Society. | gin and Microstructure in I Vol. 58, No. 9-10 pp. 385- | Lithium Disilicate Glass-Ceramics," 391, 1975. | | | | | | |
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| In Re Application Of: DI Serial No. | MITRI BRODKIN, CARLINO I | PANZERA, PAUL PANZERA | |
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| Serial No. | | | |
| 09/458,919 | Filing Date 12/10/99 | Examiner KOSLOW, C | Group Art Unit 1755 |
| Title: PRESSABLE LIT | HIUM DISILICATE GLASS CI | ERAMICS | |
| as described belov Charge the Credit any Charge an | (Only complete if Applicant elects bunt of is atta | tent of Fee to pay the fee set forth in 37 CFR 1.17() inched. to charge and credit Deposit Acc it is enclosed. Certificate of Mailing b | ount No. 50-0718 |
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CERTIFICATE OF MAILING BY "EXPRESS MAIL" (37 CFR 1.10) Docket No. Applicant(s): DMITRI BRODKIN, CARLINO PANZERA, PAUL PANZERA JPP-1222NP Serial No. Filing Date Examiner Group Art Unit 09/458,919 12/10/99 KOSLOW, C 1755 Invertine P PRESSABLE LITHIUM DISILICATE GLASS CERAMICS APR 2 4 2001 I hereby certify that the following correspondence: STATEMENT UNDER 37 CFR 1.97(e) ACCOMPANYING INFORMATION DISCLOSURE STATEMENT & INFORMATION DISCLOSURE STATEMENT (Identify type of correspondence) is being deposited with the United States Postal Service "Express Mail Post Office to Addressee" service under 37 CFR 1.10 in an envelope addressed to: The Assistant Commissioner for Patents, Washington, D.C. 20231 on APRIL 23, 2001 (Date) ANNMARIE TENERO (Typetfor Printed Name of Person Mailing Correspondence) Person Mailing Cor EK904749519US ("Express Mail" Mailing Label Number) Note: Each paper must have its own certificate of mailing. RECEIVED MAY 0 9 2001 TC 1700 P06A/REV02

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4/23/01 Date:

In Re Application of:

Annmarie Tenero

PATENT

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IN THE UNITED STATES PATENT AND TRADEMARK OFFICE

Inventor: Brodkin et al. Serial No.: 09/458,919 Filed: December 10, 1999

Group Art Unit: 1755

) Examiner: C. M. Koslow

PRESSABLE LITHIUM For: DISILICATE GALSS CERAMICS

AMENDMENT A

)

Assistant Commissioner of Patents Washington, D.C. 20231

Sir:

Please enter the following amendments to the above-identified application in

response to the Official Action mailed January 31, 2001.

In the specification:

Please amend Table 1 at page 6 as follows:

Table 1

| Oxide, | Range 1 | Range 2 | Range 3 | Range 4 | Range 5 | Range 6 |
|--------------------------------|-----------------------|------------------------|--------------------------|-------------------------|--------------------------|--------------------------|
| wt% SiO ₂ | about 62 to about | about 64 to about | about 62 to about 85 | about 64 to about 70 | about 64 to about 70 | about 62 to about 76 |
| B ₂ O ₃ | 0 to about 4.9 | 0 - about 2.7 | 0- about 4.9 | 0- about 2.7 | about 0.5 - about 3.0 | 0 - about 5 |
| Al ₂ O ₃ | about 1.5 to about 10 | about 1.5 to about 6.0 | about 5.1-to about 10 | about 5.2 to about 9.0 | about 1.5 to about 6.0 | about 1.5 to about 10 |



| F 0 to about 1.5 | | 0 to about 1.5 | about 0 to about | 0 to about 1.5 | 0 to about 1.5 | 0 to about 1.5 |
|--------------------------------|----------------------|----------------------|-----------------------|--------------------|-------------------------|------------------------|
| ZnO | 0 to about 5 | 0 to about 2 | 0 to about 5 | 0 to about 2 | | 0 to about 5 |
| CaO | 0 to about 7 | 0 to about 0.9 | 0 to about 7 | 0 to about 0.9 | 0 to about 0.9 | 0 to about 7 |
| MgO | 0 to about 2 | 0 to about 2 | 0 to about 2 | about 0 to about 2 | | 0 to about 2 |
| BaO | 0 to about 7 | 0 to about 7 | 0 to about 7 | about 0 to about 7 | 0 to about 7 | 0 to about 7 |
| SrO | 0 to about 1 | 0 to about 1 | 0 to about 1 | about 0 to about 1 | 0 to about 1 | 0 to about 1 |
| Cs ₂ O | 0 to about 5 | 0 to about 5 | 0 to about 5 | about 0 to about 5 | 0 to about 5 | 0 to about 5 |
| Li2O | about 8 to about | about 10 to about | about 8 to about | about 10 to about | about 10 to about | about 8 to about |
| | 19 | 15 | 19 | 15 | 15 | 19 |
| K ₂ O | about 2.5 to about 7 | about 2.5 to about 5 | 0 to about 7 | 0 to about 5 | about 2.2 to about 5 | 0 to about 7 |
| Na ₂ O | 0 to about 5 | 0 to about 3 | 0 to about 5 | 0 to about 3 | about 0.5 to about 3 | 0 to about 5 |
| TiO ₂ | 0 to about 2 | 0 to about 2 | 0 to about 2 | 0 to about 2 | 0 to about 2 | 0 to about 2 |
| ZrO ₂ | 0 to about 3 | 0 to about 3 | 0 to about 3 | 0 to about 3 | 0 to about 3 | 0 to about 3 |
| P ₂ O ₅ | 0.5 to about 12 | about 2 to about 7 | about 0.5 to about 12 | about 2 to about 7 | about 2 to about 7 | 0.3 to about 7.0 |
| SnO ₂ | 0 to about 1 | 0 to about 1 | 0 to about 1 | 0 to about 1 | 0 to about 1 | 0 to about 1 |
| Sb ₂ O ₁ | 0 to about 1 | 0 to about 1 | 0 to about 1 | 0 to about 1 | 0 to about 1 | 0 to about 1 |
| Y ₂ O ₃ | 0 to about 3 | 0 to about 3 | 0 to about 3 | 0 to about 3 | 0 to about 3 | 0 to about 3 |
| CeO ₂ | 0 to about 1 | 0 to about 1 | 0 to about 1 | 0 to about 1 | 0 to about 1 | 0 to about 1 |
| Eu ₂ O ₃ | 0 to about 1 | 0 to about 1 | 0 to about 1 | 0 to about 1 | 0 to about 1 | 0 to about 1 |
| Tb ₄ O ₇ | 0 to about 1 | 0 to about 1 | 0 to about 1 | 0 to about 1 | 0 to about 1 | 0 to about 1 |
| Nb ₂ O ₅ | 0 to about 2 | 0 to about 2 | 0 to about 2 | 0 to about 2 | 0 to about 2 | 0 to about 2 |
| Ta ₂ O ₅ | 0 to about 2 | 0 to about 2 | 0 to about 2 | 0 to about 2 | 0 to about 2 | about 0.5 to about 8.0 |

Please replace the paragraph beginning at page 7, line 25 and continuing to line 7 on page 8, with the following paragraph:

The powders may be used to form and fuse (sinter) pressable pellets and/or blanks of desired shapes, sizes and structures. Sintering of the pellets or blanks is carried out at temperatures in the range of about 800° to about 1000°C, and preferably in the range of about 850° to about 950°C. Sintering imparts sufficient strength for handling of the pellets or blanks. These pellets and blanks may be used for pressing cores or other frameworks or shapes for dental restorations. The cores may be provided with one or more coatings. The coatings may be selected from a ceramic, a sintered ceramic, a glass ceramic, a porcelain, a glass, a glaze, a composite and mixtures thereof. The coatings preferably have a firing temperature in the range of about 700° to about 900°C and a coefficient of thermal expansion (measured from room temperature to its transition temperature) of within about $\pm 2.0 \times 10^{-6/\circ}$ C of the dental core (measured at the same temperature in the range of about 800° to about 1200°C, and more preferably in the range of about 850° to about 950°C, and most preferably at less than about 930°C, under vacuum and with the application of pressure of between about 2 to about 8 bar (0.2 - 0.8

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MPa) and preferably no greater than about 6 bar (0.6 MPa) to obtain a dental restoration. Moreover, it is possible that the blanks may be machined to a dental restoration of desired geometry using commercially available milling equipment such as the Maho HGF 500 5 Axis CNC Milling Machine available from Fraunhofer Institut Produktionstechnologie, Germany.

/ Please amend Table 2 at pages 10 and 11 as follows:

Table 2

| | | | | | T | En (| Table Ex. 7 | Ex. 8 | Ex. | Ex. | Ex. | Ex. | Ex. | Ex. | Ex. |
|--------------------------------|-------|----------|-------|-------|----------|-------|----------------|-------|---------|----------|-------|-----------------|--------|------------|-------|
| Oxide, wt% | Ex. 1 | Ex. 2 | Ex. 3 | Ex. 4 | Ex. 5 | Ex. 6 | <u>,</u> | | 9 | 10 | 11 | <u>12</u> 65 | 13 | 14 68.9 | 15 |
| SiO ₂ | 68.8 | 70.0 | 70.1 | 65.4 | 70.7 | 70.9 | 68.1 | 69.5 | 72 | 69.0 | about | 65 | about | 08.9 | 12 |
| | | 1.1 | 1 | | | | 1.1 | | | | 68.8 | | 67.2 | | |
| · . | | | | | | | | 1.3 | 2.9 | | about | 1.0 | about | 0.9 | 2.0 |
| B ₂ O ₃ | 1.2 | 1.0 | | | | | | 1.5 | 2.7 | | | | | | |
| | | | | | | | ļ | | | | 1.3 | | 1.2 | | |
| Al ₂ O ₃ | 4.8 | 4.9 | 5.2 | 8.1 | 5.3 | 5.3 | 4.7 | 4.8 | 5.1 | 6.0 | about | 5 | about | 4.7 | 4.5 |
| 711203 | | | | | | | | | | | 4.8 | | 4.7 | l | |
| | | | | | | | | | | <u> </u> | · | | | | |
| F | | | | 1.4 | 0.2 | | | | | | | | | | |
| ZnO | | | | | | | | | | | | | | | |
| | | 0.5 | 2.2 | | | | 2.0 | 2.0 | | | about | 1 | about | 0.5 | 0 |
| CaO | 0.9 | 0,5 | 2.2 | | | | | | | | 1.0 | | 1 | | · · · |
| | | | | 100 | 1.1 | | | | | | 1.0 | | | | ļ |
| MgO | | | | | | | | | | | | | | | |
| BaO | 2.8 | 1.4 | + | 3.8 | 5.4 | 5.4 | | | 2.0 | 2.7 | about | 2.7 | about | 1.4 | 2.0 |
| BaO | 2.0 | | | | | | | | | | 2.8 | | 2.7 | | |
| | 1.0 | | | | | | | | 1.1.1.2 | | | | | | |
| SrO | | | | | | | | | | | | | | | |
| Cs ₂ O | | | | 4.1 | 4.9 | 4.9 | | | | | | | | | |
| | | <u> </u> | | | 11.0 | 10.8 | 14.3 | 14.6 | 13.0 | 15.3 | about | 15 | about | 14.3 | 13.0 |
| Li ₂ O | 14.4 | 14.7 | 14.7 | 13.4 | 11.0 | 10.0 | 14.5 | 14.0 | 15.0 | 10.0 | | | 1 14 1 | | · |
| | | | | | | ľ | | | | | 14.4 | | 14.1 | | |
| K ₂ O | 2.5 | 2.5 | 4.6 | 1.7 | | | 4.4 | 4.5 | 2.0 | 2.1 | about | 2.2 | about | 2.0 | 2.0 |
| K ₂ O | 2.5 | | | 1 | | | | | | | 2.2 | | 2.2 | | |
| | | | | | - - | | _ | | | | | | about | 1.3 | |
| Na ₂ O | 1.4 | 1.4 | | | | | - | • | | - | | t 1.4 | | 1.5 | |
| | | | | | | ľ | | | | | 1.5 | | 1.4 | | |
| - | | | | | | | | | | | | | | | |
| TiO ₂ | | | | | | | | _ | | | | | | | |
| ZrO ₂ | | | • | - | - | | | | | | | | | | |
| P ₂ O ₅ | 3.3 | 3.6 | 3.4 | 2.7 | 2.7 | 2.7 | 3.3 | 3.4 | 3.0 | 4.0 | abou | ıt 3.5 | abou | 1 3.5 | 3.0 |
| | | | | | | - I- | | 1 | | 1 | 3.3 | 1 | 3.2 | 1 | 1 |

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BLO
| $\begin{array}{c ccccccccccccccccccccccccccccccccccc$ | about 0.4 | 0.3 | 0.5 |
|--|--------------|------|--------------|
| $\begin{array}{c ccccccccccccccccccccccccccccccccccc$ | about 0.4 | 0.3 | 0.6 |
| $ \begin{array}{c ccccccccccccccccccccccccccccccccccc$ | 0.4 | | |
| Eu ₂ O ₃ 0.5 Tb ₄ O ₇ 0.8 0.9 | | | |
| Tb ₄ O ₇ 0.8 0.9 | | | 0.6 |
| Tb ₄ O ₇ 0.8 0.9 | | | |
| | | 0.3 | |
| K Nb ₂ O ₅ | | | |
| • Ta ₂ O ₅ | about | 1.8 | |
| $\begin{array}{c ccccccccccccccccccccccccccccccccccc$ | 2.0 | | |
| | | | |
| Three- Point Flexural 430± 420± | 440+ | | 400 <u>+</u> |
| Strength per ISO 70 60 | 60 | | 50 |
| 6872, MPa | | - | |
| As- pressed | 25 | | 34 |
| Opacity (relativeo | | | |
| pacity units) | | | |
| ansy 8.7 10.1 CTE 10.4 10.5 10.5 | 10.5 | 10.0 | 9.9 |
| (25°C- 500°C), 10 ⁶ °C ¹ | | | |

In the claims:

Please cancel claims 3, 9, 28 and 48 through 51.

Please amend claims 1, 8, 27, 43, 53, 56, 63, 64, 65, 66, 67, 69 and 70 as follows:

1. (Amended) A glass-ceramic composition consisting essentially of in weight percent:

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about 62 to about 85% SiO2; about 1.5 to about 10% Al₂O₃; about 8 to about 19% Li2O; about 2.5 to about 7% K2O; about 0.5 to about 12% P2O5; up to about 4.6% B₂Q₃; up to about 1.5% F; up to about 7% CaO;

up to about 7% BaO; up to about 1% SrO; up to about 5% Cs_2O ; up to about 5% Na₂O; up to about 2% TiO₂; up to about $\ CrO_2$; up to about 1% SnO₂; up to about 1%Sb₂O₃; up to about 3% χ_2O_3 ; up to about 1% C up to about 1% Eu2 up to about 1% Th up to about 2% Nb and C up to about 2% Ta₂O₅ 8. (Amended) The method of claim wherein the blank is fully sintered. (21)多 Amended) A method of making a lithium disilicate dental product comprising: melting a starting glass at temperatures within the range of about 1200 to about 1600°C; quenching the glass; cooling the quenched glass to the crystallization temperature to form a glass-ceramic; comminuting the glass ceramic to a powder; compacting the powder to a starting blank; and sintering the blank. (Amended) A glass-ceramic composition consisting essentially of in weight percent: about 64 to about 70% SiO_2 ; 28 5



about 1.5 to about 6 Al_2O_3 ; about 10 to about 15% Li_2O ;

about 2.5 to about 5% K₂O; about 2 to about 7 % P_2O_5 up to about 1.5% F; up to about 7% BaO; up to about 1% SrO; up to about 5% Cs₂O; up to about 2.7% B₂O₃; up to about 0.9% CaO; up to about 3% Na₂O; up to about 2% TiO₂; up to about 3% ZrO₂; up to about 1% SnO₂; up to about 1% Sb₂O₃; up to about $3\% Y_2O_3$; up to about 1% CeO₂; up to about 1% Eu₂O₃; up to about 1% Tb₄O₇; up to about 2% Nb₂O₅; and up to about 2% Ta₂O₅.

3 53. (Amended) A glass-ceramic composition consisting essentially of in weight percent:

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about 64 to about 70% SiO_2 ; about 5.2 to about 9 Al_2O_3 ; about 10 to about 15% Li_2O ; about 2 to about 7 % P_2O_5 ; up to about 1.5% F; up to about 7% BaO; up to about 1% SrO;

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up to about 5% Cs_2O ; up to about 5% K_2O ; up to about 2.7% B_2O_3 ; up to about 0.9% CaO; up to about 3% Na₂O; up to about 3% Na₂O; up to about 2% TiO₂; up to about 3% ZrO₂; up to about 1% SnO₂; up to about 1% Sb₂O₃; up to about 1% CeO₂; up to about 1% CeO₂; up to about 1% Eu₂O₃; up to about 1% Tb₄O₇; up to about 2% Nb₂O₅; and up to about 2% Ta₂O₅.

(Amended) A dental restoration comprising the glass-ceramic composition comprising :
about 64 to about 70% SiO₂;
about 5.2 to about 9 Al₂O₃;
about10 to about 15% Li₂O; and
about 2 to about 7 % P₂O₅.

(Amended) A glass-ceramic composition comprising in weight percent:

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about 62 to about 76% SiO_2 ; about 1.5 to about 10 Al_2O_3 ; about 8 to about 19% Li_2O ; about 0.3 to about 7% P_2O_5 ; and about 0.5 to about 8% Ta_2O_5 .

A. (Amended) A glass-ceramic composition comprising:
about 62 to about 76% SiO₂;
about 1.5 to about 10 Al₂O₃;
about 8 to about 19% Li₂O;
about 0.3 to about 7% P₂O₅;
about 0.5 to about 8% Ta₂O₅: and
whereby the glass-ceramic is pressable.

(Amended) A glass-ceramic composition comprising in weight percent:

about 62 to about 76% SiO₂ about 1.5 to about 10 Al₂O about 8 to about 19% $\text{Li}_2\phi$; about .3 to about 7% $P_2 \phi_5$; about .5 to about 8% T/a₂O₅; up to about 5% B2O3# up to about 5% $Na_2\phi$; up to about $7\% K_2$, up to about 1.5%/F; up to about 5% ZnO; up to about 2%/MgO; up to about 7% BaO; up to about 1/% SrO; up to about/5% Cs₂O; up to about 7% CaO; up to about 2% TiO2; up to about 3% ZrO₂; up to about 1% SnO2; up to about 1% Sb₂O₃; up/to about 3% Y₂O₃; up to about 1% CeO2;

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up to about $1\% \text{ Eu}_2^2\text{O}_3$; up to about $1\% \text{ Tb}_4\text{O}_7$; and up to about $2\% \text{ Nb}_2\text{O}_5$.

A dental restoration comprising the glass-ceramic comprising:

about 62 to about 76% SiO₂; about 1.5 to about 10 Al₂O₃; about 8 to about 19% Li₂O; about 0.3 to about 7% P2O5; and about 0.5 to about 8% Ta₂O₅.

(Amended) A method of making a dental restoration comprising:

melting a starting glass composition comprising in weight percent

about 62 to about 76% SiO₂;

about 1.5 to about 10 Al₂O₃;

about 8 to about 19% Li₂O;

about 0.3 to about 7% P_2O_5 ; and

about 0.5 to about 8% Ta₂O₅; at temperatures within the range of about

1200° to about 1600°C;

quenching the glass melt;

subjecting the quenched glass to one or more heat treatments in the

temperature range of from about 400° to about 1100°C to convert the glass into a

glass-ceramic;

comminuting the glass ceramic to a powder;

compacting the powder to a starting blank;

sintering the blank; and

pressing the blank into the dental restoration.

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 \mathcal{F} \mathcal{F} (Amended) The glass-ceramic of claim \mathcal{F} having a flexural strength in the range of 360 to 480 MPa.

in the range of 380 to 500 MPa.

REMARKS

Claims 1, 8, 27, 43, 53, 56, 63, 64, 65, 66, 67, 69 and 70 have been amended to more particularly define and distinctly claim the instant invention. Claims 3, 9, 28 and 48 through 51 have been cancelled. Attached hereto is a marked-up version of the changes made to the claims by the current amendment. The attached page is captioned **VERSION WITH MARKINGS TO SHOW CHANGES MADE**. The specification has been amended to clarify any inconsistencies therein. No new matter has been added. In regard to the objections to the drawings, Applicants respectfully submit that the drawings will be amended upon receipt of the Notice of Allowance. The allowabilty of claims 5 - 7, 10 - 15, 18, 19, 25, 26, 47, 52 and 57 - 62 is hereby acknowledged. In view of the foregoing amendments and following remarks, reconsideration of the application is respectfully requested.

Table 1 was amended to remove the word "about" before the number 0. Moreover, the word "about" was inserted before those numbers from which it was inadvertently left out. Support for such additions is found in the originally filed claims. The subject matter in originally filed claims 16, 17, 20 - 22, 68, 72, 73 and 77 has been incorporated into the specification at pages 7 and 8, and in Table 2, examples 11 and 13. No new matter was added. "It must be kept in mind that an original claim is part of the disclosure and might adequately set forth subject matter which is completely absent from the specification." *Ex parte Gould*, 6 USPQ2d 1680, 1681 (bd. Pat. App. & Int'f 1987); See also *In re Benno*, 768 F.2d 1340, 1346, 226 USPQ 683, 686-687 (Fed. Cir. 1985); *Twin Disc, Inv. v. United States*, 10 Cl. Ct. 713, 231 USPQ 417, 435 (Cl. Ct. 1986). Accordingly, the originally filed claims 16, 17, 20 - 22, 68, 72, 73 and 77 provide support for the amendments to the specification.

Claims 1 - 3 and 48 - 50 were rejected under 35 U.S.C. 102(b) as being anticipated by Goto et al., U.S. Patent No. 5,391,522. The Examiner states that the presence of PbO is not excluded due to the use of applicants' use of "comprising"

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language. Claims 1-3, 43-45, 48-50 and 53-55 were rejected under 35 U.S.C. 103(a) as being unpatentable over Goto '522. The Examiner contends that the composition in Goto '522 overlaps with the claimed glass-ceramic compositions. The Examiner concludes that since the composition overlaps the claimed composition, the taught glasses must inherently be pressable. For the reasons set forth below, the rejections are respectfully traversed.

The claimed invention is now directed to a glass-ceramic composition consisting essentially of SiO₂; Al₂O₃; Li₂O; K₂O; and P₂O as main ingredients in the composition. The following components may also be present: B₂O₃; F; CaO; BaO; SrO; Cs₂O; Na₂O; TiO₂; ZrO₂; SnO₂; Sb₂O₃; Y₂O₃; CeO₂; Eu₂O₃; Tb₄O₇; Nb₂O₅; and Ta₂O₅. There is mention of MgO, or ZnO in the claimed composition. Goto '522 requires the presence of MgO and ZnO in an amount of at least 0.5 %. The claimed invention is not anticipated or rendered obvious over Goto '522.

Claims 48 - 51 were rejected under 35 U.S.C. 103 as being unpatentable over Petticrew, U.S. Patent No. 5,507,981. Claims 48 - 51 were cancelled, therefore the rejection is hereby moot.

Claims 1, 2, 4, 23, 24, 43, 44 and 46 were rejected under 35 U.S.C. 102(e) as being anticipated by Schweiger et al., U.S. Patent No. 5,968,856. The Examiner states that the reference teaches lithium disilicate glass-ceramic dental restorations, and glass compositions which fall within the claimed ranges. Claims 1 - 4, 23, 24, 43 - 46, 58 - 51were rejected under 35 U.S.C. 103(a) as being unpatentable over Schweiger '856. The Examiner contends that it would have been obvious for one of ordinary skill in the art to include alumina in the taught glass-ceramic. The Examiner concludes that the reference suggests the claimed glass-ceramics. For the reasons set forth below, the rejections are respectfully traversed.

As stated above, the claimed invention is now directed to a glass-ceramic composition consisting essentially of SiO₂; Al₂O₃; Li₂O; K₂O; and P₂O as main ingredients in the composition. The following components may also be present: B₂O₃; F; CaO; BaO; SrO; Cs₂O; Na₂O; TiO₂; ZrO₂; SnO₂; Sb₂O₃; Y₂O₃; CeO₂; Eu₂O₃; Tb₄O₇; Nb₂O₅; and Ta₂O₅. Schweiger '856 requires the presence of La₂O₃ in an amount of at least 0.1 percent. As set forth in Schweiger '856, La₂O₃ and Al₂O₃ are required in the

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composition to provide a free-flowing glass ceramic that can be pressed in the plastic state. The claimed invention excludes the presence of La₂O₃. Moreover, the present invention excludes the presence of MgO and ZnO which may be present in the Schweiger '856 composition. The claims are not anticipated or rendered obvious by the Schweiger '856 reference.

The allowability of claims 5 - 7, 10 - 15, 18, 19, 25, 26, 47, 52 and 57 - 62 is hereby acknowledged. Claims 56, 64, 65, 66, have been amended to include the limitations of the base claim and any intervening claim. Claims 63 and 67 have been rewritten to overcome the objections in the Office Action.

Accordingly, it is believed that claims 1 - 47 and 52 - 77 specify patentable subject matter and are now in condition for allowance. Applicants therefore respectfully request favorable reconsideration and allowance of this application. The Examiner is requested to telephone Applicants' attorney at the number listed below if it will advance the prosecution of this case. If necessary, the Examiner is authorized to charge further fees necessary to advance the prosecution in this case from Deposit Account No. <u>500718</u>.

Respectfully submitted,

Date: April 3 2001

Ann M. K 'nah

Attorney for Applicants Registration No. 33,331

Jeneric/Pentron Inc. 53 North Plains Industrial Road Wallingford, CT 06492 Telephone (203) 265-7397 x508

Attorney Docket: JPP-1222NP





VERSION WITH MARKINGS TO SHOW CHANGES MADE

In the specification:

Table 1 has been amended as follows:

| Oxide, | Range 1 | Range 2 | Range 3 | Range 4 | Range 5 | Range 6 |
|--------------------------------|-------------------------|-------------------------|----------------------------------|-----------------------------------|--------------------------|-------------------------|
| wt% | | | | 1 (1) 1 | at and CA to all and | about 62 to about |
| SiO ₂ | about 62 to about 85 | about 64 to about 70 | about 62 <u>- to about</u> 85 | about 64 <u>to about</u> - 70 | about 64 to about 70 | 76 |
| B ₂ O ₃ | about 0 to_about 4.9 | about 0 - about 2.7 | about 0-about 4.9 | about 0- <u>about 2</u> .7 | about 0.5 - about 3.0 | about 0 - about 5 |
| Al ₂ O ₃ | about 1.5 to about 10 | about 1.5 to about 6.0 | about 5.1- <u>to about</u> 10 | about 5.2 <u>to about</u> -9.0 | about 1.5 to about 6.0 | about 1.5 to about 10 |
| F | about 0 to about | about 0 to about | about 0 to about 1.5 | about 0 to about 1.5 | about 0 to about 1.5 | about 0 to about 1.5 |
| ZnO | about 0 to about 5 | about 0 to about 2 | about 0- to about 5 | about 0- to about 2 | | about 0 to about 5 |
| CaO | about 0 to about 7 | about 0 to about | about 0- <u>to about 7</u> | about 0- <u>to about</u> 0.9 | about 0 to about 0.9 | about 0 to about 7 |
| MgO | about 0 to about 2 | about 0 to about 2 | about-0 to about 2 | about 0 to about 2 | | about 0 to about 2 |
| BaO | about 0 to about 7 | about 0 to about 7 | about 0 to about 7 | about 0 to about 7 | about 0 to about 7 | about 0 to about 7 |
| SrO | about 0 to about 1 | about 0 to about 1 | about 0 to about 1 | about 0 to about 1 | about 0 to about 1 | about 0 to about 1 |
| Cs ₂ O | about 0 to about 5 | about 0 to about 5 | about-0 to about 5 | about 0 to about 5 | about 0 to about 5 | about 0 to about 5 |
| Li ₂ O | about 8 to about | about 10 to about | about 8- <u>to</u> about19 | about 10- <u>to about</u> 15 | about 10 to about 15 | about 8 to about 19 |
| K₂O | about 2.5 to about | about 2.5 to about 5 | about 0- <u>to about 7</u> | about 0- to about 5 | about 2.2 to about 5 | about 0 to about 7 |
| Na ₂ O | about 0 to about 5 | about 0 to about 3 | about 0- <u>to about 5</u> | about 0- to about 3 | about 0.5 to about 3 | about 0 to about 5 |
| TiO ₂ | about 0 to about 2 | about 0 to about 2 | about 0 to about 2 | about 0 to about 2 | about 0 to about 2 | about 0 to about 2 |
| ZrO ₂ | about 0 to about 3 | about 0 to about 3 | about 0 to about 3 | about 0 to about 3 | about 0 to about 3 | about 0 to about 3 |
| P ₂ O ₅ | about 0.5 to about 12 | about 2 to about 7 | about 0.5- <u>to about</u> 12 | about 2- <u>to about 7</u> | about 2 to about 7 | about 0.3 to about 7.0 |
| SnO ₂ | about 0 to about 1 | about 0 to about 1 | about-0 to about 1 | about 0 to about 1 | about 0 to about 1 | about 0 to about 1 |
| Sb ₂ O ₃ | about 0 to about 1 | about 0 to about 1 | about 0 to about 1 | about-0 to about 1 | about 0 to about 1 | about 0 to about 1 |
| Y ₂ O ₃ | about 0 to about 3 | about 0 to about 3 | about 0 to about 3 | about 0 to about 3 | about-0 to about 3 | about 0 to about 3 |
| CeO ₂ | about 0 to about 1 | about 0 to about 1 | about 0 to about 1 | about 0 to about 1 | about-0 to about 1 | about 0 to about 1 |
| Eu ₂ O ₃ | about 0 to about 1 | about 0 to about 1 | about 0 to about 1 | about 0 to about 1 | about-0 to about 1 | about 0 to about 1 |
| Tb ₄ O ₇ | about 0 to about 1 | about 0 to about 1 | about 0 to about 1 | about-0 to about 1 | about-0 to about 1 | about 0 to about 1 |
| Nb ₂ O ₅ | about 0 to about 2 | about 0 to about 2 | about 0 to about 2 | about-0 to about 2 | about 0 to about 2 | about 0 to about 2 |
| Ta ₂ O ₅ | about 0 to about 2 | about 0 to about 2 | about 0 to about 2 | about-0 to about 2 | about-0 to about 2 | about 0.5 to about 8.0 |

Paragraph beginning at line 25 of page 7 and continuing to line 7 of page 8 has been amended as follows:

The powders may be used to form and fuse <u>(sinter)</u> pressable pellets and/or blanks of desired shapes, sizes and structures. <u>Sintering of the pellets or blanks is carried out at</u> <u>temperatures in the range of about 800° to about 1000°C, and preferably in the range of</u> <u>about 850° to about 950°C. Sintering imparts sufficient strength for handling of the</u> <u>pellets or blanks.</u> These pellets and blanks may be used for pressing cores or other frameworks or shapes for dental restorations. <u>The cores may be provided with one or</u> <u>more coatings.</u> The coatings may be selected from a ceramic, a sintered ceramic, a glass <u>ceramic, a porcelain, a glass, a glaze, a composite and mixtures thereof.</u> The coatings



preferably have a firing temperature in the range of about 700° to about 900°C and a coefficient of thermal expansion (measured from room temperature to its transition temperature) of within about $\pm 2.0 \times 10^{-6/\circ}$ C of the dental core (measured at the same temperature range). The blank or pellet may be subjected to viscous deformation at a temperature in the range of about 800° to about 1200°C, and more preferably in the range of about 850° to about 950°C, and most preferably at less than about 930°C, under vacuum and with the application of pressure of between about 2 to about 8 bar (0.2 - 0.8 MPa) and preferably no greater than about 6 bar (0.6 MPa) to obtain a dental restoration. Moreover, it is possible that the blanks may be machined to a dental restoration of desired geometry using commercially available milling equipment such as the Maho HGF 500 5 Axis CNC Milling Machine available from Fraunhofer Institut Produktionstechnologie, Germany.

Table 2 has been amended as follows:

| | | | | | | ta a second | Table | 2 | | 1.1 | | | | 1. | |
|--------------------------------|-------|-------|------------|-------|-------|-------------|-------|-------|----------|-----------|--------------|-----------|--------------|-----------|-----------|
| Oxide, wt% | Ex. 1 | Ex. 2 | Ex. 3 | Ex. 4 | Ex. 5 | Ex.6 | Ex. 7 | Ex. 8 | Ex. 9 | Ex. 10 | Ex. 11 | Ex. 12 | Ex. 13 | Ex. 14 | Ex. 15 |
| SiO ₂ | 68.8 | 70.0 | 70.1 | 65.4 | 70.7 | 70.9 | 68.1 | 69.5 | 72 | 69.0 | <u>about</u> | 65 | <u>about</u> | 68.9 | 72 |
| | | | | | | | | | | | 68.8 | - | 67.2 | | |
| B ₂ O ₃ | 1.2 | 1.0 | | | | | | 1.3 | 2.9 | | <u>about</u> | 1.0 | about | 0.9 | 2.0 |
| | | | | | | | | | | | 1.3 | | 1.2 | | |
| Al ₂ O ₃ | 4.8 | 4.9 | 5.2 | 8.1 | 5.3 | 5.3 | 4.7 | 4.8 | 5.1 | 6.0 | <u>about</u> | 5 | <u>about</u> | 4.7 | 4.5 |
| | | | 5-1 1-1 | | | | | | | | 4.8 | | 4.7 | | |
| F | | | | 1.4 | 0.2 | | | | | | | | | | |
| ZnO | | | | | | | | | | | | | | | |
| CaO | 0.9 | 0.5 | 2.2 | | | | 2.0 | 2.0 | | | <u>about</u> | 1 | about | 0.5 | 0 |
| | | ļ | | | | | | - | | | 1.0 | | 1 | | |
| MgO | | | | | | | | | | | ==== | | | | |
| BaO | 2.8 | 1.4 | | 3.8 | 5.4 | 5.4 | | | 2.0 | 2.7 | about | 2.7 | <u>about</u> | 1.4 | 2.0 |
| | | | | | | 1.1 | | | | | 2.8 | | 2.7 | | |
| SrO | | | | | [| | | | | | | | | | |
| Cs ₂ O | | | | 4.1 | 4.9 | 4.9 | | | | | | | | | |
| Li ₂ O | 14.4 | 14.7 | 14.7 | 13.4 | 11.0 | 10.8 | 14.3 | 14.6 | 13.0 | 15.3 | about | 15 | about | 14.3 | 13.0 |
| | | | | | | | 1 | | | | 14.4 | | 14.1 | | |

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| K20 | 2.5 | 2.5 | 4.6 | 1.7 | | | 4.4 | 4.5 | 2.0 | 2.1 | about | 2.2 | about | 2.0 | 2.0 |
|-----------------------------------|-----|------|---------|---------|------------|----------|----------|-----|--------|----------------------------|--------------|-----|--------------|----------|----------|
| .120 | | | | | | | | | | | | | | | |
| | | | | | | ļ | | | | | 2.2 | | 2.2 | | |
| Na ₂ O | 1.4 | 1.4 | | | | | | | | | <u>about</u> | 1.4 | about | 1.3 | |
| | | | | | | | | | | | 1.5 | | 1.4 | | |
| | | | | | | | | | | | 1.5 | | 1.4 | | |
| TiO2 | | | | | | | | | ****** | | | | | | |
| ZrO ₂ | | | | | | | | | | | | | | | , |
| P ₂ O ₅ | 3.3 | 3.6 | 3.4 | 2.7 | 2.7 | 2.7 | 3.3 | 3,4 | 3.0 | 4.0 | about | 3.5 | about | 3.5 | 3.(|
| 205 | 5.5 | 5.0 | 5.4 | 2.7 | 2.7 | 2.7 | 5.5 | | 5.0 | 1.0 | | 5.5 | | 5.5 | 5.0 |
| | | | | | | 1 | | | | | 3.3 | 1 | 3.2 | | |
| SnO ₂ | | | | | | | * | | | | | | | | |
| Sb ₂ O ₃ | | | | | | | | | | | | | | | |
| | | | | | | | | | | | | | - | | |
| Y ₂ O ₃ | | | | | | | | | 0.5 | 0.8 | | | | | 0.5 |
| CeO ₂ | | | | | | | 0.4 | | 0.5 | | | 0.4 | about | 0.3 | 0. |
| | | | | | | | | × . | | 1999 - 1999 1994 - 1999 | | | 0.4 | | • |
| | | - | | | - 4 - 4 | | | | | | | | 0.4 | | |
| Eu ₂ O ₃ | | | | | | | | | 0.5 | | | | | | 0. |
| Tb ₄ O ₇ | | | | | | | 0.8 | | | | | 0.9 | | 0.3 | |
| Nb ₂ O ₅ | | | | | | | | | | | | | ****** | | |
| | | ļ | | - · · · | <u> </u> | | | · | | | ļ | | | | |
| Ta ₂ O ₅ | | | | | | | 2.0 | | | | | 1.9 | about | 1.8 | |
| | | | | 19 | | 19 | 1 | | | | | | 2.0 | 1.1 | ľ |
| Three- | | | <u></u> | | <u> </u> | + | <u> </u> | | . | | - | | · · · · | <u> </u> | |
| Point | | | | | 1 | | 1.1 | | | | 1 | | | | 1 |
| Flexural Strength | | | | | | | | | | 430 <u>+</u> | 420 <u>+</u> | | 440 <u>+</u> | | 40 |
| per ISO | | | | | | | | | | 70 | 60 | | 60 | | 5 |
| 6872, | | | | | | | | | | | | | | | |
| MPa As- | | | | | | | 1 | | | 50 | 42 | | 25 | | 3 |
| pressed | | | | 1 | | | | 1 | | | | 1 | | | |
| Opacity (relativeo | | | | | | | | | | 1 | | | | | |
| pacity | | | | | | | | | | | | | İ | | 1 |
| units) CTE | | 10.4 | | | | | | + | + | 8.7 | 10.1- | | 10.5 | 10.0 | 9 |
| (25°C- | | 10.4 | | | | | | | | | | | 1 10.0 | | |
| 500°C), | ł | | | | | | | | | | 10.6 | | | | |
| 10 ⁻⁶ °C ⁻¹ | | | | | | <u> </u> | 1 | | _l | 1 | | | | 1 | <u>.</u> |

In the claims:

Claims 3, 9, 28 and 48 through 51 have been cancelled.

Claims 1, 8, 27, 43, 53, 56, 63, 64, 65, 66, 67, 69 and 70 have been amended as follows:

GC Corporation, et al. Exhibit 1006, p.84

1. (Amended) A glass-ceramic composition comprising consisting essentially of in weight percent: about 62 to about 85% SiO₂; about 1.5 to about 10% Al₂O₃; about 8 to about 19% Li₂O; about 2.5 to about 7% K₂O; and about 0.5 to about 12 % $P_2O_{5:}$ up to about 4.9% B₂O₃; up to about 1.5% F; up to about 7% CaO; up to about 7% BaO; up to about 1% SrO; up to about 5% Cs₂O; up to about 5% Na2O; up to about 2% TiO2; up to about 3% ZrO₂; up to about 1% SnO2; up to about 1% Sb₂O₃; up to about 3% Y₂O₃; up to about 1% CeO2; up to about 1% Eu2O3; up to about 1% Tb₄O₇; up to about 2% Nb2O5; and up to about 2% Ta₂O₅.

8. (Amended) The method of claim 6 wherein the blank is <u>fully</u> sintered. to full density.

27. (Amended) A method of making a lithium disilicate dental product comprising:

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melting a starting glass comprising a composition at temperatures within the range of about 1200 to about 1600°C;

quenching the glass;

cooling the <u>quenched glass melt</u> to the crystallization temperature to form a glass-ceramic;

quenching the glass ceramic;

comminuting the glass<u>-</u>ceramic to a powder; compacting the powder to a starting blank; and sintering the blank.

43. (Amended) A glass-ceramic composition comprising consisting essentially of in weight percent:

about 64 to about 70% SiO_2 ; about 1.5 to about 6 Al₂O₃; about 10 to about 15% Li₂O; about 2.5 to about 5% K₂O; and about 2 to about 7 % P_2O_{57} up to about 1.5% F; up to about 7% BaO; up to about 1% SrO; up to about 5% Cs₂O; up to about 2.7% B₂O₃; up to about 0.9% CaO; up to about 3% Na₂O; up to about 2% TiO2; up to about 3% ZrO₂; up to about 1% SnO2; up to about 1% Sb₂O₃; up to about 3% Y₂O₃: up to about 1% CeO2; up to about 1% Eu₂O₃;

GC Corporation, et al. Exhibit 1006, p.86



up to about 1% Tb₄O₇; up to about 2% Nb₂O₅; and up to about 2% Ta₂O₅.

53. A glass-ceramic composition comprising<u>consisting</u> essentially of in weight percent:

about 64 to about 70% SiO₂; about 5.2 to about 9 Al₂O₃; about10 to about 15% Li2O; and about 2 to about 7 % P₂O_{5:} up to about 1.5% F; up to about 7% BaO; up to about 1% SrO; up to about 5% Cs₂O; up to about 5% K₂O; up to about 2.7% B2O3; up to about 0.9% CaO; up to about 3% Na₂O; up to about 2% TiO2; up to about 3% ZrO₂; up to about 1% SnO₂; up to about 1% Sb₂O₃; up to about 3% Y₂O₃; up to about 1% CeO2; up to about 1% Eu₂O₃; up to about 1% Tb₄O₇; up to about 2% Nb₂O₅; and up to about 2% Ta2O5.

56. A dental restoration comprising the glass-ceramic composition comprising : of claim 53

6





about 64 to about 70% SiO_2 ; about 5.2 to about 9 Al_2O_3 ; about 10 to about 15% Li_2O ; and about 2 to about 7 % P_2O_5 .

63. (Amended) A glass-ceramic composition comprising in weight

percent:

about 62 to about 76% SiO₂; about 1.5 to about 10 Al₂O₃; about 8 to about 19% Li₂O; about $\underline{0}$.3 to about 7% P₂O₅; and about $\underline{0}$.5 to about 8% Ta₂O₅.

64. (Amended) <u>AThe glass-ceramic composition of claim 63comprising:</u> about 62 to about 76% SiO₂;
about 1.5 to about 10 Al₂O₃;
about 8 to about 19% Li₂O;
about 0.3 to about 7% P₂O₅;
about 0.5 to about 8% Ta₂O₅; and
whereby the glass-ceramic is pressable.

65. (Amended) <u>A The</u> glass-ceramic composition of claim 63 further comprising in weight percent:

7

about 62 to about 76% SiO₂; about 1.5 to about 10 Al₂O₃; about 8 to about 19% Li₂O; about .3 to about 7% P₂O₅; about .5 to about 8% Ta₂O₅; up to about 5% B₂O₃-; up to about 5% Na₂O; up to about 7% K₂O;





up to about 1.5% F; up to about 5% ZnO; up to about 2% MgO; up to about 7% BaO; up to about 1% SrO; up to about 5% Cs_2O ; up to about 7% CaO; up to about 2% TiO₂; up to about 3% ZrO₂; up to about 1% SnO₂; up to about 1% Sb₂O₃; up to about 3% Y₂O₃; up to about 1% CeO₂; up to about 1% Eu₂O₃; up to about 1% Tb₄O₇; and up to about 2% Nb₂O₅.

66. (Amended) A dental restoration comprising the glass-ceramic of elaim 63 comprising:

about 62 to about 76% SiO_2 ; about 1.5 to about 10 Al_2O_3 ; about 8 to about 19% Li_2O_3 ; about 0.3 to about 7% P_2O_5 ; and about 0.5 to about 8% Ta_2O_5 .

67. (Amended) A method of making a dental restoration comprising: melting a starting glass composition comprising in weight percent about 62 to about 76% SiO₂; about 1.5 to about 10 Al₂O₃; about 8 to about 19% Li₂O; about <u>0</u>.3 to about 7% P₂O₅; and

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about $\underline{0}.5$ to about 8% Ta₂O₅; at temperatures within the range of about 1200° to about 1600°C;

quenching the glass melt;

subjecting the quenched glass to one or more heat treatments in the temperature range of from about 400° to about 1100°C to convert the glass into a

glass-ceramic;

comminuting the glass ceramic to a powder;

compacting the powder to a starting blank;

sintering the blank; and

pressing the blank into the dental restoration.

69. (Amended) The glass-ceramic of claim 68 having a flexural strength of equal to or greater than about 420in the range of 360 to 480 MPa.

74. (Amended) The glass-ceramic of claim 73 having a flexural strength of equal to or greater than about 440in the range of 380 to 500 MPa.

9



UNITED STAT United States Patent and Trademark Office Address: COMMISSIONER OF PATENTS AND TRADEMARKS Washington, D.C. 20231 ATTORNEY DOCKET NO. FIRST NAMED INVENTOR BRUDKIN JPP-1222NP T:

ANN M KNAR JENERIC PENTRON INCORPORATED 53 NORTH PLAINS INDUSTRIAL ROAD WALLINGFORD CT 06492

FILING DATE

12/10/99

APPLICATION NO.

09/458,9

| E | XAMINER |
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| Koslov | V, C |
| ART UNIT | PAPER NUMBER |
| 1755 | |

Please find below and/or attached an Office communication concerning this application or proceeding.

IM22/0531

Commissioner of Patents and Trademarks

PTO-90C (Rev.11/00)

GC Corporation, et al. Exhibit 1006, p.91

1- File Copy

| Notice of Allowability | Examiner Melissa K | oslow | Art Unit | |
|--|---|---|---|---|
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| · · · · · | pears on the cover sh | eet with th | e correspondence | address |
| Laims being allowable, PROSECUTION ON THE MERITS previously mailed), a Notice of Allowance and Issue Fee S NOTICE OF ALLOWABILITY IS NOT A GRANT OF PA initiative of the Office or upon petition by the applicant | S IS (OR REMAINS) CLC e Due or other appropria TENT RIGHTS. This ap t. See 37 CFR 1.313 a | SED in this ite commun plication is nd MPEP 13 | application. If not ication will be mail subject to withdray 08. | included herewith ed in due course. wal from issue at |
| This communication is responsive to <i>the amendra</i> | ment of 23 April 2001 | | | P |
| The allowed claim(s) is/are <u>1, 2, 4-8, 10-27, 29</u> | | | | |
| The drawings filed on are | e acceptable as formal | drawings. | | |
| Acknowledgement is made of a claim for foreigr | | | (a)-(d). | |
| a_{a} \Box All b) \Box Some* c) \Box None of the: | | | | |
| 1. Certified copies of the priority documents | have been received. | | | |
| 2. Certified copies of the priority documents | | Applicatio | n No | , |
| 3. Copies of the certified copies of the priori application from the International Bure | ty documents have be au (PCT Rule 17.2(a)) | en receive | | |
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| blicant has THREE MONTHS FROM THE "MAILING DAT ed below. Failure to timely comply will result in ABAN TENDABLE FOR SUBMITTING NEW FORMAL DRAWING complying with the REQUIREMENT FOR THE DEPOSIT | | | FCI ARATION. Th | is three-month period |
| Note the attached EXAMINER'S AMENDMENT reason(s) why the oath or declaration is defici | or NOTICE OF INFOR | MAL APPLI DATH OR I | CATION (PTO-15 DECLARATION IS | 2) which gives REQUIRED. |
| X Applicant MUST submit NEW FORMAL DRAW | NGS | | | |
| (a) \Box including changes required by the Notice of | Draftsperson's Patent | Drawing F | Review (PTO-948) | attached |
| 1) 🗌 hereto or 2) 🗌 to Paper No | | | | |
| (b) including changes required by the proposed approved by the examiner. | | | | |
| (c) including changes required by the attached Paper No | | | | |
| Identifying indicia such as the application number drawings should be filed as a separate paper with | (see 37 CFR 1.84(c)) a transmittal letter ad | should be v dressed to | vritten on the drav the Official Drafts | wings. The person. |
| \Box Note the attached Examiner's comment regard | ing REQUIREMENT FO | R THE DEF | POSIT OF BIOLOG | ICAL MATERIAL. |
| ny reply to this letter should include, in the upper rig JMBER). If applicant has received a Notice of Allow e NOTICE OF ALLOWANCE should also be included | vance and Issue Fee D | PPLICATIC ue, the ISS | on Number (Ser Sue Batch Numi | IES CODE/SERIAL 3ER and DATE of |
| tachment(s) | | | | P |
| Notice of References Cited (PTO-892) | _ | | of Informal Patent Ap | |
| Notice of Draftsperson's Patent Drawing Review (PTO-5 | | _ | w Summary (P10-41 er's Amendment/Con | 3), Paper No |
| Information Disclosure Statement(s) (PTO-1449), Paper ☐ Examiner's Comment Regarding Requirement for Depos | | | er's Statement of Rea | |
| Material | | writing | | A Ant |
| Other | | | F | MELISSA KOSLOW PRIMARY EXAMINER ART UNIT 1755 |



NOTICE OF ALLOWANCE AND ISSUE FEE DUE

IM2270531

ANN M KNAB JEMERIC PENTRON INCORPORATED 53 NORTH PLAINS INDUSTRIAL ROAD WALLINGFORD CT 06492

| APPL | ICATION NO. | FILING DATE | TOTAL CLAIMS | EXAMINER AND GROUP ART | UNIT | DATE MAILED |
|------------|--------------|--------------|--------------|------------------------|-------|-------------|
| | 09/458,919 | 12/10/99 | 020 I | COSLOW, C | 1755 | 05/31/01 |
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| E OF F | PRESSABLE LT | THIUM DISILI | CATE GLASS | CERAMICS | | |

| ATTY'S | DOCKET NO. | CLASS-SUBCLASS | BATCH NO. | APPLN. TYPE | SMALL ENTITY | FEE DUE | DATE DUE |
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| 0 | JPR-1222NP | 801-005 | .000 6 | 399 UTIL | LITY YES | \$620.00 | 08/31/01 |

THE APPLICATION IDENTIFIED ABOVE HAS BEEN EXAMINED AND IS ALLOWED FOR ISSUANCE AS A PATENT. <u>PROSECUTION ON THE MERITS IS CLOSED.</u>

THE ISSUE FEE MUST BE PAID WITHIN <u>THREE MONTHS</u> FROM THE MAILING DATE OF THIS NOTICE OR THIS APPLICATION SHALL BE REGARDED AS ABANDONED. <u>THIS STATUTORY PERIOD CANNOT BE EXTENDED.</u>

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| I. Review the SMALL ENTITY status shown above. If the SMALL ENTITY is shown as YES, verify your current SMALL ENTITY status: | If the SMALL ENTITY is shown as NO: |
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| A. If the status is changed, pay twice the amount of the FEE DUE shown above and notify the Patent and Trademark Office of the change in status, or | A. Pay FEE DUE shown above, or |
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ANN M KNAB JENERIC PENTRON INCORPORATED 53 NORTH PLAINS INDUSTRIAL ROAD WALLINGFORD CT 06492

| APPLICATION NO. | FILING DATE | TOTAL CLAIMS | EX. | AMINER AND GROUP | ART UNIT | DATE MAILED |
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| 09/458,919 | 12/10/99 | 070 | KOSLOW, | C | 17! | 55 06/04/0 |
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08-24-01 GAU /75 Docket No. CERTIFICATE OF MAILING BY "EXPRESS MAIL" (37 CFR 1.10) JPP-1222NP CPA Applicant(s): DMITRIER ODALA, CARLINO PANZERA, PAUL PANZERA 16 2 3 2001 Filing Date Examiner Group Art Unit Serial No. 12/10/99 KOSLOW, C 09/458,919 1755 DISILICATE GLASS CERAMICS ERAN. Mallon & MRE TRADE PRESSAB Invention: AUG 2 8 2001 I hereby certify that the following correspondence: TC 1700 TRANSMITTAL OF INFORMATION DISCLOSURE STATEMENT'; POST CARD (Identify type of correspondence) is being deposited with the United States Postal Service "Express Mail Post Office to Addressee" service under 37 CFR 1.10 in an envelope addressed to: The Assistant Commissioner for Patents, Washington, D.C. 20231 on 8/23/01 (Date) ANNMARIE TENERO Correspondence) (Type EK904747305US ("Express Mail" Mailing Label Number) Note: Each paper must have its own certificate of mailing. ١ Match & net P06A/REV02



Exhibit 1006, p.96

| TRANSMITTAL OF INFORMATION DISCLOS (Under 37 CFR 1.97(b) or 1.97(c | | Docket No. JPP-1222NP CPA |
|---|--|--|
| Re Application Of: DMITRI BRODKIN, CARLINO PAR | NZERA, PAUL PANZERA | |
| Serial No. 09/458,919 Alife 2 3 2001 Serial No. | Examiner KOSLOW, C. | Group Art Unit 1755 |
| Title: PRESSABLE LITERATE GLASS CERA | MICS | |
| | | |
| Assistant Comm | ess to: issioner for Patents n, D.C. 20231 | |
| 37 CF | R 1.97(b) | |
| The Information Disclosure Statement submitted h of a national application other than a continued p three months of the date of entry of the national s application; before the mailing of a first Office Action Action after the filing of a request for continued exa | rosecution application under tage as set forth in 37 CFR 1 on on the merits, or before the amination under 37 CFR 1.114 | 37 CFR 1.53(d); within 1.491 in an international e mailing of a first Office |
| | R 1.97(c) | a manifed an addition of the OT |
| The Information Disclosure Statement submitted h CFR 1.97(b), provided that the Information Disclo Final Action under 37 CFR 1.113, a Notice of otherwise closes prosecution in the application, and | osure Statement is filed befor Allowance under 37 CFR 1 | re the mailing date of a |
| the statement specified in 37 CFR 1.97(e); | | |
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| | Substitute for form 1449A/PTC AUG 2 3 2001 & | | | | Co | omplete if Known |
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| | | | | U.S. PATENT DOC | UMENTS | |
|---------------------------------------|--------------------------|--|--|-------------------------------|--|--|
| Examiner Initials* | Cite No. ¹ | U.S. Patent Number | t Document Kind Code ² (if known) | Name of Patentee or Applicant | Date of Publication of Cited Document MM-DD-YYYY | Pages, Columns, Lines, Where Relevant Passages or Relevant Figures Appear |
| | | 4,189,325 | | BARRETT | 02-19-1980 | |
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¹ Unique citation designation number. ⁻See attached Kinds of U.S. Patent Documents. ³ Enter Office that issued the document, by the two-letter code (WIPO Standard ST.3). ⁴ For Japanese patent documents, the indication of the year of the reign of the Emperor must precede the serial number of the patent document. ⁵ Kind of document by the appropriate symbols as indicated on the document under WIPO Standard ST. 16 if possible. ⁶ Applicant is to place a check mark here if English language Translation is attached.

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| | | OTHER PRIOR ART NON PATENT LITERATURE DOCUMENTS | |
|-----------------------|--------------|--|----|
| Examiner Initials* | Cite No.1 | 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, page(s), volume-issue number(s), publisher, city and/or country where published. | T2 |
| Q | | DILMORE, M.F., CLARK, D.E., HENCH. L.L, Corrosion Behavior of Lithia Disilicate Glass in Aqueous Solutions of Aluminum Compounds. Ceramic Bullletin, pg. 1111-1124. Vol. 58, No. 11 (1979). | |
| Q | | FREIMAN, S.W., HENCH, L.L., Effect of Crystallization on the Mechanical Properties of Li2O-SiO2 Glass-Ceramics. Journal of the American Ceramic Society. pg. 86-90 Vol, 55, No.2. | |
| 9 | | HENCH, L.L., FRIEMAN, S.W., KINSER, D.L., The early stages of crystallisation of a Li2O-2SiO2 glass. Physics and Chemistry of Glasses. pg. 58-63. Vol. 12, No. 2 April 1971. | |
| 9 | | MCCRACKEN, W.J., CLARK, D.E., HENCH, .L.L, Aqueous Durability of Lithium Disilicate Glass-Ceramics. Ceramic Bulletin. pg. 1218-1223. Vol61, No. 11 (1982). | |
| 9 | | MCCRACKEN, W.J., PERSON, W.B., HENCH, L.L., Polarized infrared reflection spectroscopy of single crystal lithia-silicates and quartz. Journal of Materials Science. pg. 3853-3864. 20, 1985. | |
| 9 | | OHUCHI, F., CLARK, D.E., HENCH, L.L., Effect of Crystallization on the Auger Election Signal Decay in an Li2O 2SiO2 Glass and Glass-Ceramic. Journal of American Ceramic Society. pg. 500-503. Sept-Oct. 1979. | |
| 0 | | PALMER, R.A., LINDBERG, W.R., HENCH, L.L., Fatigue Properties of Li2O 2SiO2 Glass and Glass Ceramic. Journal of The American Ceramic Society - Discussions and Notes. pg. 319-320. Vol. 62, No.5-6. May-June 1979 | |
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| RANSMITTAL OF INFORM (Under 37 CF | ATION DISCLOS R 1.97(b) or 1.97(| SURE STATEMENT (c)) | Docket No. JPP-1222NP CPA |
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| | | | | U.S. PATENT DOC | UMENTS | |
|----------------------|--------------------------|-------------------------|--|--|--|---|
| Examiner Initials | Cite No. ¹ | U.S. Patent D Number | Document Kind Code ² (if known) | Name of Patentee or Applicant of Cited Document | Date of Publication of Cited Document MM-DD-YYYY | Pages, Columns, Lines, Where Relevant Passages or Relevant. Figures Appear |
| 0 | | 2,106,744 | | HOOD | 02-01-1938 | 0 |
| 0 | | 2,515,940 | | STOOKEY | 07-18-1950 | ALCEN/ |
| 0 | | 2,628,160 | | STOOKEY | 02-10-1953 | -IVED |
| 9 | | 2,684,911 | | STOOKEY | 07-27-1954 | AUG 2 0 |
| 0 | | 2,779,13 | 6 | STOOKEY | 01-29-1957 | - ~ U 2001 |
| 0 | | 2,971,853 | | STOOKEY | 02-14-1961 | Tra |
| G | | 3,013,362 | | CALKINS | 12-19-1961 | 101700 |
| 0 | | 3,032,429 | | ARTHUR | 05-01-1962 | |
| 0 | • | 3,114,646 | | CURRIE | 12-17-1963 | |
| 9 | | 3,130,061 | | MCMAHON | 04-21-1964 | |
| 0 | | 3,170,805 | | MCMILLAN | 02-23-1965 | |
| 0 | | 3,231,456 | | MCMILLAN | 01-25-1966 | |
| 0 | | 3,236,610 | | MCMILLAN | 02-22-1966 | |
| 0 | | 3,252,811 | | BEALL | 05-24-1966 | |
| a | | 3,253,975 | | OLCOTT | 05-31-1966 | |
| Ch | | 3,380,838 | | SACK | 04-30-1968 | |
| \mathcal{A} | | 3,397,076 | | LITTLE | 08-13-1968 | |
| Q_{j} | | 3,436,109 | | LOOSE | 04-01-1969 | |
| 0 | | 3,458,330 | | TRYGGVE | 07-29-1969 | |
| C | | 3,463,646 | | LITTLE | 08-26-1969 | |

| | | | | FOR | EIGN PATENT DOCUMEN | TS | | | |
|--------------|------|---------------------|---------------------|-------------------|----------------------------------|--|--|--|--|
| Examiner Cit | Cite | Cite | | Foreign Patent Do | cument Kind Code ⁵ | Name of Patentee or Applicant of Cited Document | Date of Publication of Cited Document | Pages, Columns, Lines, Where Relevant Passages or Relevant | |
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| | | 1 | | U.S. PATENT DOCUMENTS | | | | | |
|----------------------|--------------|---|--|--|--|--|--|--|--|
| Examiner Initials | Cite No.1 | U.S. Patent Document Kind Code ² (<i>if known</i>) | | Name of Patentee or Applicant of Cited Document | Date of Publication of Cited Document MM-DD-YYYY | Pages, Columns, Lines, Where Relevant Passages or Relevant Figures Appear | | | |
| Q | | 3,511,681 | | HUEY | 05-12-1970 | | | | |
| Ō | | 3,499,787 | | INOUE | 03-10-1970 | | | | |
| Q | | 3,537,868 | | KOSAKA | 11-03-1970 | | | | |
| Q. | | 3,561,984 | | EPPLER | 02-09-1971 | | | | |
| Q | | 3,564,587 | | ELLIS | 02-16-1971 | × | | | |
| \overline{Q} | | 3,600,204 | | BEALL | 08-17-1971 | | | | |
| 0 | | 3,647,489 | | MCMILLAN | 03-07-1972 | | | | |
| Q | | 3,663,193 | | WILSON | 05-16-1972 | | | | |
| C) | | 3,929,494 | | VERES | 12-30-1975 | | | | |
| \mathcal{O} | | 3,939,295 | | ROBERTSON | 02-17-1976 | | | | |
| S | | 4,097,295 | | CHYUNG | 06-27-1978 | | | | |
| <u>S</u> | | 4,125,651 | | CAMPBELL | 11-14-1978 | | | | |
| O_{\perp} | | 4,186,021 | | CHYUNG | 01-29-1980 | | | | |
| 0 | | 4,196,004 | | BERRETZ | 04-01-1980 | | | | |
| 0 | | 4,222,760 | | CHYUNG | 09-16-1980 | | | | |
| 0 | | 4,289,538 | | ORSO | 09-15-1981 | | | | |
| 0 | | 4,340,645 | | O'CONOR | 07-20-1982 | | | | |
| 4 | | 4,391,914 | | BEALL | 07-05-1983 | | | | |
| 9 | , | 4,431,451 | | MABIE | 02-14-1984 | | | | |
| O | | 4,473,653 | | RUDOI | 09-25-1984 | | | | |

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|---------------|--------------------------|---------------------|--------------------|-----------------------------|--|-----------------------------|------------------------------|--|----|
| | FOREIGN PATENT DOCUMENTS | | | | | | | | |
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¹ Unique citation designation number. See attached Kinds of U.S. Patent Documents. ³ Enter Office that issued the document, by the two-letter code (WIPO Standard ST.3). ⁴ For Japanese patent documents, the indication of the year of the reign of the Emperor must precede the serial number of the patent document, ⁵ Kind of document by the appropriate symbols as indicated on the document under WIPO Standard ST. 16 if possible. ⁶ Applicant is to place a check mark here if English language Translation is attached.

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| | | · | U.S. PATENT DOCUMENTS | | | | | |
|----------------------|--------------------------|---------------|---|--|--|--|--|--|
| Examiner Initials | Cite No. ¹ | U.S. Patent D | ocument Kind Code ² (if known) | Name of Patentee or Applicant of Cited Document | Date of Publication of Cited Document MM-DD-YYYY | Pages, Columns, Lines, Where Relevant Passages or Relevant Figures Appear | | |
| | | 4,515,634 | (if known) | -wu | -05-07-1985- | | | |
| 177 | | 5,176,961 | | CROOKER | 01-05-1993 | | | |
| 18 | | 5,200,369 | | CLIFFORD | 04-06-1993 | | | |
| 10- | | 5,232,878 | | KASUGA | 08-03-1993 | | | |
| 18- | | 5,308,391 | | КОММА | 05-03-1994 | | | |
| In | | 5,308,803 | | CLIFFORD | 05-03-1994 | · · · · · · · · · · · · · · · · · · · | | |
| 175- | | 5,330,939 | | MARAZZI | 07-19-1994 | · · · · · · · · · · · · · · · · · · · | | |
| <u> </u> | | 5,507,981 | | PETTICREW | | | | |
| Hm= | | 5,552,350 | | HORNOR | 09-03-1996 | · · · · · · · · · · · · · · · · · · · | | |
| 10 | + | 5,567,217 | | GOTO | 10-22-1996 | | | |
| 1a | | 5,580,363 | | GOTO | 12-03-1996 | | | |
| 10- | + | 5,849,649 | | POOLE | 12-15-1998 | | | |
| 0 | | 5,872,069 | | ABE | 02-16-1999 | | | |
| Q. | | 5,874,376 | | TAGUCHI | 02-23-1999 | | | |
| 10 | | 5,895,719 | | FYLES | 04-20-1999 | · · · · · · · · · · · · · · · · · · · | | |
| 0 | 1 | 5,897,885 | | PETTICREW | 04-27-1999 | | | |
| a | | 5.968,856 | | SCHWEIGER | 10-19-1999 | | | |
| 107 | | 5,997,977 | | ZOU | 12-07-1999 | | | |
| 0 | 1 | 6,121,175 | | DRESCHER | 09-19-2000 | | | |
| 10 | - | 3,450,54 | +6 | STONG | 06-17-1969 | | | |

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| Examiner Initials | Cite No.1 | Office | Foreign Patent Do | Pages, Columns, Lines, Where Relevant Passages or Relevant Figures Appear | Т ⁶ | | | |
| G | | Onice | JP 03271130 | | HISAKUNI | 03-12-1991 | | |
| | | ┨──── | JP 49-128011 | | ISHIZUKA | 12-07-1974 | · · · · · | |
| a | | ╞╴╴ | JP 49-128011 | | NETSLIKAGAKU | -03-04-1987- | | _ |
| -11- | | | CA 696,415 | | MCMILLAN | 10-20-1964 | | -1-1 |
| G_ | | | CA 090,415 | | MCMILLAN | 11-19-1968 | | -1- |
| G- | | | CA 1,120,960 | | BARRETT | 03-30-1982 | | <u> </u> |
| Cr_ | ├ ──── | | CA 2,213,390 | · · · · · · | SCHWEIGER | 03-05-1998 | | <u> </u> |
| 8 | <u> </u> | + | EP 0 536 479 A1 | | BEALL | 04-14-1993 | | - ` |
| 0 | ╂──── | + | EP 695 726 A1 | | FRANK | 02-07-1996 | | + |
| 2 | | | EP 0 827 941 B1 | | SCHWEIGER | 11-03-1999 | <u> </u> | 1 |

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¹ Unique citation designation number. See attached Kinds of U.S. Patent Documents. ³ Enter Office that issued the document, by the two-letter code (WIPO Standard ST.3). ⁴ For Japanese patent documents, the indication of the year of the reign of the Emperor must precede the serial number of the patent document. ⁵ Kind of document by the appropriate symbols as indicated on the document under WIPO Standard ST. 16 if possible. ⁶ Applicant is to place a check mark here if English language Translation is attached.

| Please type a plus sign (+) inside this + + Under the Paperwork Reduction Act of 1995, no persons are required to re- | Patent and Tradema sports to a collection of informatio | PTO/SB/08A (08-00) ed for use through 10/(1)2002 OMB 0651-0031 rk Office: U.S. DEPART NEOTOC COMMERCE n unless it contains a valid OMP control number. |
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| Substitute for form 1449A/PTO | | mplete if Known |
| | Application Number | 09/458,919 |
| INFORMATION DISCLOSURE | Filing Date | 12-10-99 |
| STATEMENT BY APPLICANT | First Named Inventor | BRODKIN |
| | Group Art Unit | 1755 |
| (use as many sheets as necessary) | Examiner Name | C. KOSLOW |
| Sheet 4 of 6 | Attorney Docket Number | JPP-1222NP |

| | | | | U.S. PATENT DOCU | MENTS | |
|-----------------------|---|---------|--|------------------|------------|---------------------------------------|
| Examiner Initials* | niner Cite U.S. Patent Document s' No. ¹ Number Kind Code ² Name of Patentee or Applicant (<i>it known</i>) of Cited Document | | Date of Publication of Cited Document MM-DD-YYYY Figures Appear | | | |
| • | | 5,820,9 | 89- | REED | 10-13-1998 | |
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| C | | | EP 0916625A1 | | SCHWEIGER | 05-19-1999 | | V | | | |
| 0 | | | DE 2313347 | | SCHULER | 10-04-1973 | | V | | | |
| 9 | | | DE 2314721 | | BOROM | 10-11-1973 | | V | | | |
| 0 | | | DE 2314722 | | BOROM | 10-11-1973 | | | | | |
| 4 | | | DE 2451121 | | BOROM | 05-07-1975 | | V | | | |
| 0 | | | DE 3039930A1 | | DANIELSON | 05-14-1981 | | | | | |
| 0 | | | DE 4020893A1 | | KASUGA | 01-03-1991 | | V | | | |
| 0 | | | DE 1421886 | | HAYAMI | 04-22-1971 | | | | | |
| C | | | UK 940706 | | SIMENS-SCHUCKERTWER | 10-30-1963 | | V | | | |
| 2 | | | UK 955,437 | | KOGYOGIJUTSUIN | 04-15-1964 | | V | | | |
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*EXAMINER: Initial if reference considered, whether or not citation is in conformance with MPEP 609. Draw line through citation if not in conformance and not considered. Include copy of this form with next communication to applicant.

¹ Unique citation designation number. See attached Kinds of U.S. Patent Documents. ³ Enter Office that issued the document, by the two-letter code (WIPO Standard ST.3). ⁴ For Japanese patent documents, the indication of the year of the reign of the Emperor must precede the serial number of the patent document. ⁵ Kind of document by the appropriate symbols as indicated on the document under WIPO Standard ST. 16 if possible. ⁶ Applicant is to place a check mark here if English language Translation is attached.

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TO ALL TO WHOM THESE PRESENTS SHALL COME:

UNITED STATES DEPARTMENT OF COMMERCE United States Patent and Trademark Office

February 21, 2017

THIS IS TO CERTIFY THAT ANNEXED IS A TRUE COPY FROM THE RECORDS OF THIS OFFICE OF THE FILE WRAPPER AND CONTENTS OF:

APPLICATION NUMBER: 09/458,919 FILING DATE: December 10, 1999 PATENT NUMBER: 6,455,451 ISSUE DATE: September 24, 2002

> By Authority of the Under Secretary of Commerce for Intellectual Property and Director of the United States Patent and Trademark Office

JOHN A BURSON Certifying Officer

PART (7) OF (2) PART(S)

GC Corporation, et al. Exhibit 1006, p.107

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| | (us | se as many s | heets a | s nec | essary) | Group Art Examiner I | | 1755 C. KOSLOW | Allo |
| (use as many sheets as necessar Sheet 5 of 6 | | | | | 6 | | ocket Number | JPP-1222NP | CPA 20200 |
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| nitials* | No.1 | Number | Kind C | | of Cited Docur | nent | Cited Docume MM-DD-YYY | | Passages or Relevant Figures Appear |
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| Ċ | | tt- | UK 1,136,501 | | | WILLIAM | 12-11-1968 | | $\overline{\mathbf{V}}$ |
| 9 | | | UK 1,312,700 | | | DAVIES | 04-04-1973 | | V |
| 4 | | | FR 2 655 264 | | | DENRY | 06-07-1991 | | V |
| | | | JP62072547 | | | NETSUKAGAKU | 04-03-87 | | |
| _C_ | | | <u>JP01145348</u> | | | TOSHIBA GLASS | 06-07-1989 | | - |
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| Examiner Signature | Date Considered | 1012-1101 |
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*EXAMINER: Initial if reference considered, whether or not citation is in conformance with MPEP 609. Draw line through citation if not in conformance and not considered. Include copy of this form with next communication to applicant.

¹ Unique citation designation number. See attached Kinds of U.S. Patent Documents. ³ Enter Office that issued the document, by the two-letter code (WIPO Standard ST.3). ⁴ For Japanese patent documents, the indication of the year of the reign of the Emperor must precede the serial number of the patent document. ⁵ Kind of document by the appropriate symbols as indicated on the document under WIPO Standard ST. 16 if possible. ⁶ Applicant is to place a check mark here if English language Translation is attached.

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| | èr/ | BRODKIN | | C. KOS | - | 175 | |
| TRADEMA | 7 | | Assistant Was | Address to: Commissioner f Box CPA shington, D.C. 2 | or Patents 0231 | I.53(d), (continued pros n <u>12/10/99</u> TC 7 | · · |
| This is a application | request for filing on (CPA)) of pri | g a 🛛 continue or application n | ation or 🔲 divis umber | sional applicatio 09/458,919 | n under 37 CFR 1 filed o | 1.53(d), (continued pros n <u>12/10/99</u> | section 1 |
| and entit | led: ABLE LITHIUM | I DISILICATE (| GLASS CERAMIC | 2 | | rc ₁ | >0 ₂₀₀₇ >0 >00 |
| 1. 🗖 | Enter the unent nonprovisional | | nt previously filed | on | | under 37 CFR 1.1 | 16 in the prior |
| 2. 🗋 | A preliminary a | mendment is er | nclosed. | | | | |
| 3. 🔲 | This applicatior | n is being filed b | y fewer than all th | e inventors nan | ned in the prior ap | plication, 37 CFR 1.53 | (d)(4). |
| | a. 🗋 DEL | ETE the followi | ng inventor(s) nan | ned in the prior | nonprovisional ap | plication: | |
| | | | 0 () | | | NA 00000130 100714 - | e an 1915 - Andre F |
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| | b. 🗋 The | inventor(s) to b | e deleted are set t | forth on a separ | ate sheet attache | d hereto. | |
| 4. 🗆 | A new power of | f attorney or au | horization of ager | nt is enclosed. | | | |
| 5. 🛛 | Small Entity Sta | atus: | | | | | |
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| 6. 🗖 | The fee for this | application is c | alculated as follow | ws: | | | |
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| CONTINUED PROSECUTION APPLICATION (CPA) REQUEST TRANSMITTAL (Small Entity) (Only for Continuation or Divisional Applications Under 37 CFR 1.53(d)) |
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| 7. X The Commissioner is hereby authorized to credit overpayments or charge the following fees to Deposit Account No. 50-0718 |
| fees required under 37 C.F.R. 1.16. fees required under 37 C.F.R. 1.17. fees required under 37 C.F.R. 1.18. |
| 8. A check in the amount of is enclosed. |
| 9. 🛛 An Information Disclosure Statement (IDS) is enclosed: |
| a. 🛛 PTO-1449 |
| b. 🛛 Copies of IDS Citations |
| 10. 🛛 Also enclosed: |
| Transmittal of Formal Drawings and 1 Sheet of Formal Drawings; International Search Report, Post Card. |
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| 11. The prior application's correspondence address will carry over to this CPA UNLESS a new correspondence address is provided below: |
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[Page 2 of 3]

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| 2 - 100 V. 100 V. | (1) complete as defined by 37 C.F.R. 1.51(b), or (2) compliance with 35 U.S.C. 371. A Notice will be preissues and designs, to the effect that the patent | must be a nonprovisional application that is either:) the national stage of an international application in placed on a patent issuing from a CPA, except for issued on a CPA and is subject to the twenty-year fore, the prior application of a CPA may have been |
| | C-I-P NOT PERMITTED: A continuation-in-part ap 1.53(d), but must be filed under 37 C.F.R. 1.53(b). | plication cannot be filed as a CPA under 37 C.F.R. |
| | expressly abandon the prior application as of the | CATION: The filing of this CPA is a request to filing date of the request for a CPA. 37 C.F.R. onal or continuation-in-part of an application that is |
| (Januar 10.17) 2. 2 | confidentiality by the Applicant under 35 U.S.C. 122 entitled under the provisions of 37 C.F.R. 1.14 to a | of this CPA will be construed to include a waiver of 2 to the extent that any member of the public who is access to, copies of, or information concerning, the opies of, or similar information concerning, the other |
| | sentence of the specification and none should b application is submitted, it will not be entered. A r | rence to the prior application is needed in the first be submitted. If a sentence referencing the prior equest for a CPA is the specific reference required d the application number identified in such request, |
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| ed: t | 8/16/01 | |
| | | ANN M. KNAB |
| | | Typed or printed name |
| | | 33,331 Registration Number (if applicable) |
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| | 5 | □ Inventor(s) |
| | | Assignee of complete interest |
| | | Attorney or agent of record |

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GC Corporation, et al. Exhibit 1006, p.111

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Docket No. RANSMITTAL OF FORMAL DRAWINGS JPP-1222NP CPA tion Of: DMITRI BRODKIN, CARLINO PANZERA, PAUL PANZERA Filing Date Art Unit Serial No. Batch No. Examiner AUG 2 0 2001 TC 1700 09/458,919 12/10/99 C. KOSLOW Invention: PRESSABLE LITHIUM DISILICATE GLASS CERAMICS Address to: Assistant Commissioner for Patents Washington, D.C. 20231 Transmitted herewith are: 1 sheets of formal drawing(s) for this application. Each sheet of drawing indicates the identifying indicia suggested in 37 CFR Section 1.84(c) on the reverse side of the drawing. Dated: 8/16/01 ANN M. KNAB **REG NO. 33,331** I certify that this document and attached formal drawings are being deposited on with the U.S. Postal Service as first class mail under 37 C.F.R. 1.8 and addressed to the Assistant Commissioner for Patents, Washington, D.C. 20231. Signature of Person Mailing Correspondence Typed or Printed Name of Person Mailing Correspondence P23B/REV01



FIG. 1

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SERIAL NO.: 09/458,919 INVENTOR: DMITRI BRODKIN, CARLINO PANZERA, PAUL PANZERA DOCKET NO.: JPP-1222NP CPA CONTACT PERSON: ANN KNAB, REG NO. 33,331

(203) 265-7397 x508

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Docket No. **CERTIFICATE OF MAILING BY "EXPRESS MAIL" (37 CFR 1.10)** Applicant(s): DMITRL BRODKIN, CARLINO PANZERA, PAUL PANZERA JPP-1222NP Group Art Unit Serial No Filing Date Examiner Ξ. 09/458,91 12/10/99 KOSLOW, C 1755 **I DISILICATE GLASS CERAMICS** PRE Invention: RECEIVET I hereby certify that the following correspondence: CONTINUED PROSECUTION APPLICATION; TRANSMITTAL OF FORMAL DRAWINGS; TRANSMITTAL OF INFORMATION DISCLOSURE STATEMENT'; INTERNATIONAL SEARCH REPORT ; POST CARD (Identify type of correspondence) is being deposited with the United States Postal Service "Express Mail Post Office to Addressee" service under 37 CFR 1.10 in an envelope addressed to: The Assistant Commissioner for Patents, Washington, D.C. 20231 on 8/16/01 (Date) ANNMARIE TENERO or Printed Name of Person Mailing Correspondence) EK904747464US ("Express Mail" Mailing Label Number) Note: Each paper must have its own certificate of mailing. P06A/REV02

| TRANSMITTAL OF INFORMATION DISCLO (Under 37 CFR 1.97(b) or 1.97 | | Docket No. JPP-1222NP CPA |
|---|--|---|
| In Re Application DMITRIGRODKIN, CARLINO F | PANZERA, PAUL PANZERA | |
| Serial No. | Examiner | Group Art Unit |
| 09/458,919 375 AT & TRADUME 12/10/99 | KOSLOW, C. | 1755 |
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| *This certificate may only be used if paying by deposit account. Signature ANN M. KNAB ATTORNEY REG. NO.: 33,331 | Dated: 8/16/01 | |
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P10A/REV02

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UNITED STA1 DEPARTMENT OF COMMERCE Patent and Trademark Office

Address: COMMISSIONER OF PATENTS AND TRADEMARKS Washington, D.C. 20231

| APPLICATION NO. | FILING DATE | FIRST NAMED | DINVENTOR | A | ATTORNEY DOCKET NO. |
|--------------------------|------------------------------|-------------|-----------|----------------------|---------------------|
| 09/458,919 | 9 12/10/99 | BRODKIN | | Ĭ.> | JPP-1222NP |
| Γ | < | | | E | EXAMINER |
| | | IM52/1025 | • | | |
| ANN M KNAI | 3 | | r | <u> </u> | , (°). |
| JENERIC PE | ENTRON INCORP | ORATED | | ART UNIT | PAPER NUMBER |
| 53 NORTH F WALLINGFOF | PLAINS INDUST RD CT 06492 | RIAL ROAD | | 1755 DATE MAILED: | 13 |
| | | | | | 10/25/01 |
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Please find below and/or attached an Office communication concerning this application or proceeding.

Commissioner of Patents and Trademarks

PTO-90C (Rev. 2/95)

GC Corporation, et al. Exhibit 1006, p.117

1- File Copy

| 0 | Application No. | Applicant(s) |
|--|--|--|
| | 09/458,919 | BRODKIN ET AL. |
| Office Action Summary | Examiner | Art Unit |
| | C. Melissa Koslow | 1755 |
| The MAILING DATE of this communication ap Period for Reply | pears on the cover sheet with th | he correspondence address |
| A SHORTENED STATUTORY PERIOD FOR REPL | Y IS SET TO EXPIRE 3 MON | TH(S) FROM |
| THE MAILING DATE OF THIS COMMUNICATION. Extensions of time may be available under the provisions of 37 CFR 1. after SIX (6) MONTHS from the mailing date of this communication. If the period for reply specified above, its maximum statutory period Failure to reply within the set or extended period for reply will, by statut Any reply received by the Office later than three months after the mailin earned patent term adjustment. See 37 CFR 1.704(b). | 136(a). In no event, however, may a reply b ly within the statutory minimum of thirty (30) will apply and will expire SIX (6) MONTHS e. cause the application to become ABAND | be timely filed) days will be considered timely. from the mailing date of this communication. ONED (35 U.S.C. § 133) |
| 1) Responsive to communication(s) filed on <u>16</u> | August 2001 | |
| | his action is non-final. | |
| 3) Since this application is in condition for allow | | prosecution as to the merits is |
| closed in accordance with the practice under | Ex parte Quayle, 1935 C.D. 1 | 1, 453 O.G. 213. |
| Disposition of Claims | | |
| 4)⊠ Claim(s) <u>1,2,4-8,10-27,29-47 and 52-77</u> is/ar | | |
| 4a) Of the above claim(s) is/are withdra | wn from consideration. | |
| 5)⊠ Claim(s) <u>4-8,10-23,25-27,29-44,46,47,52-54,</u> | <u>56-64 and 66-77</u> is/are allowed | • |
| 6) Claim(s) <u>1,2 and 24</u> is/are rejected. | | |
| 7) Claim(s) $45,55$ and 65 is/are objected to. | | |
| 8) Claim(s) are subject to restriction and/ | or election requirement. | |
| Application Papers | | |
| 9) The specification is objected to by the Examine | | |
| 10) The drawing(s) filed on <u>16 August 2001</u> is/are: | | |
| Applicant may not request that any objection to the | | |
| 11) The proposed drawing correction filed on | | pproved by the Examiner. |
| If approved, corrected drawings are required in re 12) The oath or declaration is objected to by the E | | |
| | kammer. | |
| Priority under 35 U.S.C. §§ 119 and 120 | | |
| 13) Acknowledgment is made of a claim for foreig | n priority under 35 U.S.C. § 11 | 9(a)-(d) or (f). |
| a) All b) Some * c) None of: | | |
| Certified copies of the priority documen Certified copies of the priority documen | | action No. |
| 3. Copies of the certified copies of the prior | | |
| application from the International But * See the attached detailed Office action for a list | reau (PCT Rule 17.2(a)). | - |
| 14) Acknowledgment is made of a claim for domest | ic priority under 35 U.S.C. § 1 | 19(e) (to a provisional application). |
| a) | | |
| Attachment(s) | | |
| Notice of References Cited (PTO-892) Notice of Draftsperson's Patent Drawing Review (PTO-948) Information Disclosure Statement(s) (PTO-1449) Paper No(s) § | 5) 🔲 Notice of Inform | mary (PTO-413) Paper No(s) nal Patent Application (PTO-152) |
| S. Patent and Trademark Office TO-326 (Rev. 04-01) Office A | ction Summary | Part of Paper No. 13 |

The request filed on 16 August 2001 for a Continued Prosecution Application (CPA) under 37 CFR 1.53(d) based on parent Application No. 09/458,919 is acceptable and a CPA has been established. An action on the CPA follows.

The references cited on the Information Disclosure Statements of 16 August 2001 and 23 August 2001 having a line drawn through them have already been considered and were cited by either the Examiner on form PTO-892 or by applicants in an earlier Information Disclosure Statement.

The non-English references cited in the Information Disclosure Statements of 16 August 2001 and 23 August 2001 have been considered with respect to the provided English abstracts, English translations and English equivalent references.

Claim 45 is objected to under 37 CFR 1.75(c), as being of improper dependent form for failing to further limit the subject matter of a previous claim. Applicant is required to cancel the claim.

The subject matter in this claim is found in claim 43 from which claim 45 depends.

Claim 55 is objected to under 37 CFR 1.75(c), as being of improper dependent form for failing to further limit the subject matter of a previous claim. Applicant is required to cancel the claim.

The subject matter in this claim is found in claim 53 from which claim 55 depends.

Claim 65 is objected to because of the following informalities: The leading zeros are missing from the amount of phosphorus oxide and tantalum oxide. Appropriate correction is required.

Page 2

The following is a quotation of the appropriate paragraphs of 35 U.S.C. 102 that form the basis for the rejections under this section made in this Office action:

A person shall be entitled to a patent unless -

(b) the invention was patented or described in a printed publication in this or a foreign country or in public use or on sale in this country, more than one year prior to the date of application for patent in the United States.

Claims 1, 2 and 24 are rejected under 35 U.S.C. 102(b) as being clearly anticipated by GB 1,312,700: EP 536,479: DE 2,451,121; US 3,816,704 or Borom et al.

All of these references teach glass-ceramic compositions. Borom et al and Example 1 in the translation of DE 2,451,121 teach a glass-ceramic, in the shape of a rod or cane, comprising 71.8 wt% silica, 5.1 or 4.8 wt% alumina, 12.6 wt% lithia, 4.8 or 5.1 wt% potassium oxide and 2.5 wt% phosphorous pentaoxide. Rods or canes are known shapes for blanks. This composition falls within the claimed composition. Since the taught composition falls within that claimed, one of ordinary skill in the art would expect it to inherently be pressable, absent any showing to the contrary. GB 1,312,700 teaches a glass-ceramic preform or blank comprising 73 wt% silica, 5.1 wt% alumina, 13.5 wt% lithia, 5.5 wt% potassium oxide, 2.7 wt% phosphorous pentaoxide and 0.2 wt% zirconia. This composition falls within the claimed composition. Since the taught composition falls within that claimed, one of ordinary skill in the art would expect it to inherently be pressable, absent any showing to the contrary. US 3,816,704 teaches, in examples II-IV and VI, glass-ceramic rods composed of 71.1-73.41 wt% silica, 3.9-5.1 wt% alumina, 12.6-15.6 wt% lithia, 4.66-6.2 wt% potassium oxide and 1.65-3.2 wt% phosphorous pentaoxide. Example II also contains 3.2 wt% boron oxide. Rods or canes are known shapes for blanks. This composition falls within the claimed composition. Since the taught composition falls within that claimed, one of ordinary skill in the art would expect it to inherently be pressable, absent any

showing to the contrary. EP 536,479 teaches, in examples 3, a glass-ceramic slab or blank comprising 74.2 wt% silica, 3.54 wt% alumina, 15.4 wt% lithia, 3.25 wt% potassium oxide and 23.37 wt% phosphorous pentaoxide. This composition falls within the claimed composition. Since the taught composition falls within that claimed, one of ordinary skill in the art would expect it to inherently be pressable, absent any showing to the contrary. The claimed glassceramic reads upon that taught.

Claims 1 and 2 are rejected under 35 U.S.C. 102(b) as being clearly anticipated by JP 1145,348.

The abstract for this reference teaches it is addressed to glass-ceramic compositions in terms of weight percentage. The table on page 288 teaches specific glass-ceramic compositions. Composition 1 teaches a glass-ceramic consisting or 74.2 wt% silica, 4.3 wt% alumina, 12.3 wt% lithia, 4.5 wt% potassium oxide, 2.5 wt% phosphorous pentaoxide, 1 wt% boron oxide, 0.1 wt% fluorine and 0.3 wt% antimony trioxide. This composition falls within the claimed composition. Since the taught composition falls within that claimed, one of ordinary skill in the art would expect it to inherently be pressable, absent any showing to the contrary. The claimed glass-ceramic reads upon that taught.

Claims 4-8, 10-23, 25-27, 29-44, 46, 47, 52-54, 56-64 and 66-77 are allowable over the cited art of record.

Claim 65 would be allowable if rewritten or amended to overcome the objection set forth in this Office action.

The claimed glass-ceramic composition, methods and dental restoration are not taught or suggested by the cited art of record.

Page 4

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Any inquiry concerning this communication or earlier communications from the examiner should be directed to Melissa Koslow whose telephone number is (703) 308-3817. The examiner can normally be reached on Monday-Thursday from 7:30 AM to 4:00 PM. The examiner can also be reached on alternate Fridays.

If attempts to reach the examiner by telephone are unsuccessful, the examiner's supervisor, Mark Bell, can be reached at (703) 308-3823.

The fax number for Amendments filed under 37 CFR 1.116 or After Final communications is (703) 872-9311. The fax number for all other official communications is (703) 872-9310.

Any inquiry of a general nature or relating to the status of this application or proceeding should be directed to the Group receptionist whose telephone number is (703) 308-0661 or (703) 308-0662.

cmk October 25, 2001

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C. Melissa Koslow Primary Examiner Tech. Center 1700

Page 5

| | Application/Control No. 09/458,919 | Applicant(s)/Patent Under Reexamination BRODKIN ET AL. | |
|----------------------------|---------------------------------------|--|-------------|
| Notice of References Cited | Examiner | Art Unit | D 4.64 |
| | C. Melissa Koslow | 1755 | Page 1 of 1 |

U.S. PATENT DOCUMENTS

| | - | | U.S. PATENT DOCUMENTS | | |
|---|--|---|---|--|---|
| | Document Number Country Code-Number-Kind Code | Date MM-YYYY | Name | Classif | ication |
| А | US-3,816,704 | 06-1974 | Borom et al | 219 | 462 |
| в | US- | | | | • |
| С | US- | | | | |
| D | US- | | | | |
| E | US- | | | | |
| F | US- | | | | |
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| | B C D F G H I J K L | Document Number Country Code-Number-Kind CodeAUS-3,816,704BUS-CUS-DUS-EUS-FUS-GUS-HUS-IUS-JUS-KUS-LUS- | Document Number Country Code-Number-Kind CodeDate MM-YYYYAUS-3,816,70406-1974BUSCUSDUSEUSFUSGUSHUSJUSJUSLUS | Document Number Country Code-Number-Kind Code Date MM-YYYY Name A US-3,816,704 06-1974 Borom et al B US- Image: Comment Number Kind Code Image: Comment Number Kind Code C US-3,816,704 06-1974 Borom et al B US- Image: Comment Number Kind Code Image: Comment Number Kind Code C US- Image: Comment Number Kind Code Image: Comment Number Kind Code D US- Image: Comment Number Kind Code Image: Comment Number Kind Code D US- Image: Comment Number Kind Code Image: Comment Number Kind Code D US- Image: Comment Number Kind Code Image: Comment Number Kind Code F US- Image: Comment Number Kind Code Image: Comment Number Kind Code G US- Image: Comment Number Kind Code Image: Comment Number Kind Code J US- Image: Comment Number Kind Code Image: Comment Number Kind Code J US- Image: Comment Number Kind Code Image: Comment Number Kind Code K US- Image: Comment Number Kind Code <td>Document Number Country Code-Number-Kind Code Date MM-YYYY Name Classif A US-3,816,704 06-1974 Borom et al 219 B US- 1 219 219 C US-3,816,704 06-1974 Borom et al 219 B US- 1 1 1 1 C US- 1</td> | Document Number Country Code-Number-Kind Code Date MM-YYYY Name Classif A US-3,816,704 06-1974 Borom et al 219 B US- 1 219 219 C US-3,816,704 06-1974 Borom et al 219 B US- 1 1 1 1 C US- 1 |

FOREIGN PATENT DOCUMENTS

| * | | Document Number Country Code-Number-Kind Code | Date MM-YYYY | Country | Name | Classif | ication |
|---|---|--|-----------------|-----------------|------------------|---------|---------|
| * | N | DE 2,451,121 | 05-1974 | Germany | Borom et al | | |
| * | 0 | EP 536,479 | 04-1993 | Europe | Beall et al | | |
| * | Р | GB 1,312,700 | 04-1973 | UK | Davies et al | | |
| * | Q | JP 1-145,348 | 06-1989 | Japan | Toshiba Glass KK | | |
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| | | | | NON-PATENT DOCU | MENTS | | |

| * | | Include as applicable: Author, Title Date, Publisher, Edition or Volume, Pertinent Pages) |
|---|---|---|
| * | U | Borom et al, "Strength and Microstructure in Lithium Disilicate Glass-Ceramics", Jour. Amer. Chem. Soc., Vol. 58, pp. 385-91, 1975. |
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*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. 13

| PETITION FOR E | XTENSION OF TIME UND (Small Entity) | DER 37 CFR 1.136(a) | Docket No. JPP-1222NP CPA |
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| IAN 3 | MITRIBRODKIN, CARLINO I | PANZERA, PAUL PANZERA | Hiy al |
| Serial No. | ADEMINITY Filing Date 12/10/99 | Examiner KOSLOW, C. | Group Art Unit |
| Invention: PRESSABL | E LITHIUM DISILICATE GLAS | SS CERAMICS | |
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| is enclosed. has already been The fee for the extension A check in the am The Commissione overpayment, to D A duplicate copy of If an additional ext | ount of the fee is enclosed. It is hereby authorized to charge Deposit Account No. 50-0718 of this sheet is enclosed. | nd is to be paid as follows: any fees which may be required e consider this a petition therefor | · · · · · · · · · · · · · · · · · · · |
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| ANN M. KNAB, ESQ. REG. NO. 33,331 | | on first class mail under | ocument and fee is being deposite with the U.S. Postal Service 37 C.F.R. 1.8 and is addressed to th oner for Patents, Washington, D. |
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| In Re Application: DMITRIBRODKIN, CARLINO PA | NZERA, PAUL PANZERA | 775 2/15 |
| Serial No. Filing Date | Examiner | Group Art Un <u>it</u> |
| 09/458,919 12/10/99 | KOSLOW, C. | 1755 / |
| PRESSABLE LITHIUM DISILICATE GLASS CER | AMICS | |
| • | ent of Fee ts to pay the fee set forth in 37 CFR | |
| A check in the amount of is attact The Assistant Commissioner is hereby authorized to as described below. A duplicate copy of this sheet Charge the amount of \$240.00 Credit any overpayment. Charge any additional fee required. | o charge and credit Deposit Acc | count No. 50-0718 |
| Certificate of Transmission by Facsimile* | Certificate of Mailing l | oy First Class Mail |
| I certify that this document and authorization to charge deposit account is being facsimile transmitted to the United States Patent and Trademark Office (F | I certify that this document an with as first class mail under addressed to the Assistant Co Washington, D.C. 20231. | the U.S. Postal Service 37 C.F.R. 1.8 and is |
| Signature | Signature of Person M | lailing Correspondence |
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| *This certificate may only be used if paying by deposit account. Signature NN M. KNAB FTORNEY REG. NO.: 33,331 | Dated: 1/31/02 | HECEWE |
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| Re Application Of: DMITROBRODKIN, CARLIN | O PANZERA, PAUL PANZERA | | |
| Serial No. Company Filing Date 09/458,919 12/10/99 | Examiner KOSLOW, C. | Group Art Unit 1755 | |
| Title: PRESSABLE LITHIUM DISILICATE GLASS | CERAMICS | | |
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| 3 | 37 CFR 1.97(b) | | |
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| 3 | 37 CFR 1.97(c) | | |
| A The Information Disclosure Statement submit CFR 1.97(b), provided that the Information I Final Action under 37 CFR 1.113, a Notic otherwise closes prosecution in the application | Disclosure Statement is filed befor e of Allowance under 37 CFR 1. | e the mailing date of a | |
| the statement specified in 37 CFR 1.97 | 7(e); | | |
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| | NZERA, PAUL PANZERA | | | |
| Serial Notifice Transmitter Filing Date 09/458,919 12/10/99 | Examiner KOSLOW, C. | Group Art Unit 1755 | | |
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| The Assistant Commissioner is hereby authorized to as described below. A duplicate copy of this sheet Charge the amount of \$240.00 Credit any overpayment. Charge any additional fee required. Certificate of Transmission by Facsimile* | is enclosed. Certificate of Mailing by I certify that this document and f | First Class Mail ee is being deposited le U.S. Postal Service C.F.R. 1.8 and is | | |
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| Typed or Printed Name of Person Signing Certificate | Typed or Printed Name of Perso | n Mailing Certificate | | |
| *This certificate may only be used if paying by deposit account. Signature NN M. KNAB TTORNEY REG. NO.: 33,331 | Dated: 1/31/02 | | | |

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| | I hereby certification the date shown b | rtificate of Mailing elow, this correspondence is being deposited with the United to Addressee", Mailing Label No. <u>EK904747892US</u> ner for Patents, Washington, D.C. 20231. <u>Annmarie Tenero</u> <u>Annmarie Tenero</u> <u>PATENT</u> |
| | IN THE UNITED STATES P | ATENT AND TRADEMARK OFFICE |
| | In Re Application of: |) |
| | |) |
| | Inventor: Brodkin et al. |) |
| | Serial No.: 09/458,919 |) Group Art Unit: 1755 |
| | Filed: December 10, 1999 |) |
| | |) Examiner: C. M. Koslow |
| | For: PRESSABLE LITHIUM DISILICATE GLASS CERAMI |) Examiner: C. M. Koslow) CS) ENDMENT B |
| | | ENDMENT B |
| | Assistant Commissioner of Patents Washington, D.C. 20231 | |
| | Sir: | |
| | Please enter the following amen | dments to the above-identified application in |
| | response to the Official Action mailed O | Detober 25, 2001. |
| | In the claims: | |
| | Please cancel claims 1, 2, 45 and | |
| _ | Please amend claims 4, 24 and 6 | 5 as follows: |
| BI | Soft (amended). A dental nessentially of: | restoration comprising a glass-ceramic consisting |

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about 62 to about 85% SiO₂; about 1.5 to about 10% Al₂O₃; about 8 to about 19% Li2O; about 2.5 to about 7% K₂O; about 0.5 to about 12 % P2O5; up to about 4.9% B₂O₃; up to about 1.5% F; up to about 7% CaO; up to about 7% BaO; up to about 1% SrO; up to about 5% Cs₂O; up to about 5% Na₂O; up to about 2% TiO₂; up to about 3% ZrO2; up to about 1% SnO₂; up to about 1% Sb₂O₃; up to about 3% Y₂O₃; up to about 1% CeO₂; up to about 1% Eu₂O₃; up to about 1% Tb₄O₇; up to about 2% Nb₂O₅; and up to about 2% Ta₂O₅

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 $5^{4}/_{24}$ (amended). A blank for machining the dental restoration of claim A.

(twice amended) A glass-ceramic composition comprising in weight percent:

about 62 to about 76% SiO₂; about 1.5 to about 10 Al₂O₃; about 8 to about 19% Li₂O; about 0.3 to about 7% P_2O_5 ;

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GC Corporation, et al. Exhibit 1006, p.130

<u>}</u>



about 0.5 to about 8% Ta₂O₅. up to about 5% B_2O_3 ; up to about 5% Na₂O; up to about 7% K₂O; up to about 1.5% F; up to about 5% ZnO; up to about 2% MgO; up to about 7% BaO; up to about 1% SrO; up to about 5% Cs₂O; up to about 7% CaO; up to about 2% TiO₂; up to about 3% ZrO₂; up to about 1% SnO₂; up to about 1% Sb₂O₃; up to about 3% Y₂O₃; up to about 1% CeO2; up to about $1\% Eu_2O_3$; up to about 1% Tb₄O₇; and up to about 2% Nb₂O₅.

REMARKS

Claims 4, 24 and 65 have been amended to more particularly define and distinctly claim the instant invention. Claims 1, 2, 45 and 55 have been cancelled. Attached hereto is a marked-up version of the changes made to the claims by the current amendment. The attached page is captioned **VERSION WITH MARKINGS TO SHOW CHANGES MADE**. The allowability of claims 4 - 8, 10 - 23, 25 - 27, 29 - 44, 46, 47, 52 - 54, 56 - 64 and 66 - 77 is hereby acknowledged. In view of the foregoing amendments and following remarks, reconsideration of the application is respectfully requested.

Claims 45, 55 and 65 were objected to. Claims 45 and 55 have been cancelled and claim 65 has been amended to overcome the informalities therein.

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Claims 1, 2 and 24 were rejected under 35 U.S.C. 102 (b) as being clearly anticipated by GB 1,312,700; EP 536,479; DE 2,451,121 and US 3,816,704 to Borom et al. Claims 1 and 2 were rejected under 35 U.S.C. 102 (b) as being clearly anticipated by JP 1,145,348.

In response thereto, claims 1 and 2 have been cancelled. Claim 24 has been amended to depend from claim 4 and to include the word machining. "Blank" is the term of art used to describe the block of material used in computer assisted design/computer assisted milling (cad/cam) operations. Support for this amendment is found on page 4 at lines 16 and 17 of the application. None of the references show or suggest a blank or block of glass-ceramic material for machining into a dental restoration. Rods or canes are not shapes of material used in machines for milling or machining into a desired shape. U.S. Patent Nos. 5,030,392, 6,174,168 and 6,190,171 are cited to show use of the term "blank" as a block of material used for machining into a desired shape. See claim 7 of U.S. Patent 5,030,392; see col. 2, line 1 and line10 - 11 of U.S. Patent No. 6,174,168; and col. 4, lines 50 - 65. The claim is not anticipated or obvious over the cited references.

Accordingly, it is believed that claims 4 - 8, 10 - 27, 29 - 44, 46, 47, 52 - 54, 56 - 77 is hereby acknowledged specify patentable subject matter and are now in condition for allowance. Applicants therefore respectfully request favorable reconsideration and allowance of this application. The Examiner is requested to telephone Applicants' attorney at the number listed below if it will advance the prosecution of this case. If necessary, the Examiner is authorized to charge further fees necessary to advance the prosecution in this case from Deposit Account No. 500718.

Respectfully submitted,

Ann M. Knab

Attorney for Applicants Registration No. 33,331

Date: January 31, 2002

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GC Corporation, et al. Exhibit 1006, p.132 \sum



Jeneric/Pentron Inc. 53 North Plains Industrial Road Wallingford, CT 06492 Telephone (203) 265-7397 x508

Attorney Docket: JPP-1222NP

GC Corporation, et al. Exhibit 1006, p.133

VERSION WITH MARKINGS TO SHOW CHANGES MADE

In the claims:

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Claims 1, 2, 45 and 55 were cancelled.

Claims 4, 24 and 65 were amended.

1. (amended) A glass ceramic composition consisting essentially of in weight percent:

about 62 to about 85% SiO₂;

about 1.5 to about 10% Al₂O₃;

about 8 to about 19% Li2O;

about 2.5 to about 7% K₂O;

about 0.5 to about 12 % P₂O₅;

up to about 4.9% B₂O₃;

up to about 1.5% F;

up to about 7% CaO;

up to about 7% BaO;

up to about 1% SrO; up to about 5% Cs₂O;

up to about 5% Na₂O;

up to about 2% TiO₂;

up to about 3% ZrO2;

up to about 1% SnO₂;

up to about 1% Sb₂O₃;

up to about 3%-Y2O3;

up to about 1% CeO2;

up to about 1% Eu₂O₃;

up to about 1% Tb₄O₇; up to about 2% Nb₂O₅; and

up to about 2% Ta2O5-

2. The glass-ceramic of claim 1 whereby the glass-ceramic is pressable.

GC Corporation, et al. Exhibit 1006, p.134 1

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4 (amended). A dental restoration comprising the a glass-ceramic of claim 1-consisting essentially of: about 62 to about 85% SiO₂; about 1.5 to about 10% Al₂O₃; about 8 to about 19% Li2O; about 2.5 to about 7% K2O; about 0.5 to about 12 % P2O5; up to about 4.9% B2O3; up to about 1.5% F; up to about 7% CaO; up to about 7% BaO; up to about 1% SrO; up to about 5% Cs₂O; up to about 5% Na₂O; up to about 2% TiO₂; up to about 3% ZrO2; up to about 1% SnO₂; up to about 1% Sb₂O₃; up to about 3% Y₂O₃; up to about 1% CeO2; up to about 1% Eu₂O₃; up to about 1% Tb₄O₇; up to about 2% Nb2O5; and up to about 2% Ta2O5.

24 (amended). A blank for the manufacture of a <u>machining the</u> dental restoration comprising the composition of claim 1 of claim 4.

45 (amended). The glass-ceramic composition of claim 43 further comprising in weight percent:

GC Corporation, et al. Exhibit 1006, p.135 Ť.



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up to about 1.5% F; up to about 7% BaO; up to about 1% SrO; up to about 5%-Cs2O; up to about 2.7% B2O3; up to about 2% ZnO; up to about 0.9% CaO; up to about 2% MgO; up to about 3% Na₂O; up to about 2% TiO2; up to about 3%-ZrO2; up to about 1% SnO2; up to about 1% Sb2O3; up to about 3% Y2O3; up to about 1% CeO2; up to about 1% Eu2O3; up to about 1% Tb4O7; up to about 2% Nb2O5; and up to about 2% Ta2O5-

55. The glass-ceramic composition of claim 53 further comprising in weight percent:

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 up to about 1.5% F;

 up to about 7% BaO;

 up to about 1% SrO;

 up to about 5% Cs2O;

 up to about 5% K2O;

 up to about 2% B2O;

 up to about 2% ZnO;

 up to about 0.9% CaO;

 up to about 2% MgO;

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up to about 3% Na₂O; up to about 2% TiO₂; up to about 3% ZrO₂; up to about 1% SnO₂; up to about 1% Sb₂O₃; up to about 3% Y₂O₃; up to about 3% Y₂O₃; up to about 1% CeO₂; up to about 1% Eu₂O₃; up to about 1% Tb₄O₇; up to about 2% Nb₂O₅; and up to about 2% Ta₂O₅.

65. (twice amended) A glass-ceramic composition comprising in weight percent:

about 62 to about 76% SiO₂; about 1.5 to about 10 Al₂O₃; about 8 to about 19% Li₂O; about 0.3 to about 7% P₂O₅; about 0.5 to about 8% Ta₂O₅. up to about 5% B₂O₃; up to about 5% Na₂O; up to about 7% K₂O; up to about 1.5% F; up to about 5% ZnO; up to about 2% MgO; up to about 7% BaO; up to about 1% SrO; up to about 5% Cs₂O; up to about 7% CaO; up to about 2% TiO₂; up to about 3% ZrO₂;

> GC Corporation, et al. Exhibit 1006, p.137





up to about 1% SnO_2 ; up to about 1% Sb_2O_3 ; up to about 3% Y_2O_3 ; up to about 1% CeO_2 ; up to about 1% Eu_2O_3 ; up to about 1% Tb_4O_7 ; and up to about 2% Nb_2O_5 .

GC Corporation, et al. Exhibit 1006, p.138

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| ITLE OF INVENTION: P | PRESSABLE LITHIUM DIS | LICATE GLASS | S CERAMICS | RODKIN | | | JPP-1222NP | | 9478 |
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II. PART B - FEE(S) TRANSMITTAL should be completed and returned to the United States Patent and Trademark Office (USPTO) with your ISSUE FEE and PUBLICATION FEE (if required). Even if the fee(s) have already been paid, Part B - Fee(s) Transmittal should be completed and returned. 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.

III. All communications regarding this application must give the application number. Please direct all communications prior to issuance to Box ISSUE FEE unless advised to the contrary.

IMPORTANT REMINDER: Utility patents issuing on applications filed on or after Dec. 12, 1980 may require payment of maintenance fees. It is patentee's responsibility to ensure timely payment of maintenance fees when due.

Page 1 of 3

PTOL-85 (REV. 07-01) Approved for use through 01/31/2004.

| oplicant(s): DM | MENT TRANSMITT | FAL LETTER (Sm Lino panzera, pau | all Entity) L PANZERA | | Docket No. JPP-1222NP CPA | | |
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| Serial No. 09/458,919 | Serial No. 09/458,919 JAN 3 1 2002 P2/10/99 KOSLOW, C. | | | | | | |
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| hereby certify that the following cor | respondence: | | |
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| Notice of Allowability | 09/458,919 Examiner | BRODKIN ET AL. | |
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| | C. Melissa Koslow | 1755 | |
| The MAILING DATE of this communication All claims being allowable, PROSECUTION ON THE ME herewith (or previously mailed), a Notice of Allowance (P NOTICE OF ALLOWABILITY IS NOT A GRANT OF PA of the Office or upon petition by the applicant. See 37 CF 1. X This communication is responsive to <u>the amendm</u> | RITS IS (OR REMAINS) CLOSED i TOL-85) or other appropriate comm TENT RIGHTS . This application is FR 1.313 and MPEP 1308. ent of 31 Jan. 2002. | n this application. If not include unication will be mailed in due | ed course. THIS |
| 2. 🛛 The allowed claim(s) is/are <u>4-8, 10-27, 29-44, 46,</u> | <u>47, 52-54 and 56-77</u> . | | |
| 3. The drawings filed on are accepted by the | | | |
| 4. ☐ Acknowledgment is made of a claim for foreign pri a) ☐ All b) ☐ Some* c) ☐ None of the: | | or (f). | |
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| (a) The translation of the foreign language prov | | | |
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| Applicant has THREE MONTHS FROM THE "MAILING E below. Failure to timely comply will result in ABANDONN | IENT of this application. THIS THI | REE-MONTH PERIOD IS NOT | EXTENDABLE |
| 7. A SUBSTITUTE OATH OR DECLARATION must NFORMAL PATENT APPLICATION (PTO-152) which gi | be submitted. Note the attached EX ves reason(s) why the oath or decla | AMINER'S AMENDMENT or N aration is deficient. | IOTICE OF |
| 3. CORRECTED DRAWINGS must be submitted. | | | |
| (a) 🔲 including changes required by the Notice of D | raftsperson's Patent Drawing Revie | ew (PTO-948) attached | |
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| Identifying indicia such as the application number (see a of each sheet. The drawings should be filed as a separate | 87 CFR 1.84(c)) should be written on the paper with a transmittal letter add | he drawings in the top margin (n ressed to the Official Draftsperso | ot the back) on. |
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| TOTAL CLAIMS 66 | APPLN. TYPE | SMALL ENTITY | | UE FEE | PUBL | ICATION FE | E | TOTAL FEE(S) DUE | | DATE DUE |
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Determination of Patent Term Adjustment under 35 U.S.C. 154 (b) (application filed on or after May 29, 2000)

The patent term adjustment to date is 0 days. If the issue fee is paid on the date that is three months after the mailing date of this notice and the patent issues on the Tuesday before the date that is 28 weeks (six and a half months) after the mailing date of this notice, the term adjustment will be 0 days.

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) system. (http://pair.uspto.gov)

PTOL-85 (REV. 07-01) Approved for use through 01/31/2004.

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Page 1 of

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PART B - FEE(S) TRANSMITTAL

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Complete and mail this form, together with applicable fee(s), to:

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(12) United States Patent

Brodkin et al.

PRESSABLE LITHIUM DISILICATE GLASS (54) CERAMICS

- Inventors: Dmitri Brodkin, West Orange, NJ (75)(US); Carlino Panzera, Belle Mead, NJ (US); Paul Panzera, Mt. Holly, NJ (US)
- Assignee: Jeneric/Pentron, Inc., Wallingford, CT (73) (US)
- This patent issued on a continued pros-Notice: (*)ecution application filed under 37 CFR 1.53(d), and is subject to the twenty year patent term provisions of 35 U.S.C. 154(a)(2).

Subject to any disclaimer, the term of this patent is extended or adjusted under 35 U.S.C. 154(b) by 0 days.

(21) Appl. No.: 09/458,919

(56)

(22) Filed: Dec. 10, 1999

Related U.S. Application Data

- Provisional application No. 60/153,916, filed on Sep. 14, 1999, provisional application No. 60/122,558, filed on Mar. 2, 1999, and provisional application No. 60/111,872, filed on Dec. 11, 1998. (60)
- (51) Int. Cl.⁷ C03B 27/012; C03B 32/00; A61C 13/00
- U.S. Cl. 501/5; 106/35; 264/16; (52)264/19; 433/201.2; 433/202.1; 433/212.1
- Field of Search 264/16, 19; 106/35; (58) 501/5; 433/201.1, 202.1, 212.1

References Cited

U.S. PATENT DOCUMENTS

2/1938 Hood 2,106,744 A

7/1950 Stookey 2,515,940 A

(List continued on next page.)

FOREIGN PATENT DOCUMENTS

| | Тоныны | |
|----|---------|---------|
| CA | 696415 | 10/1964 |
| CA | 799407 | 11/1968 |
| CA | 1120960 | 3/1982 |
| CA | 2213390 | 3/1998 |
| DF | 1421886 | 4/1971 |

C C

(List continued on next page.)

OTHER PUBLICATIONS

Borom et al, "Strength and Microstructure in Lithium Disilicate Glass-Ceramics", Jour. Amer. Chem. Soc., vol. 58, pp. 385-391, 1975.*

(List continued on next page.)

Primary Examiner-C. Melissa Koslow (74) Attorney, Agent, or Firm-Ann M. Knab

ABSTRACT (57)

This invention is directed to lithium disilicate $(\mathrm{Li}_2\mathrm{Si}_2\mathrm{O}_5)$ based glass-ceramics comprising silica, lithium oxide, alumina, potassium oxide and phosphorus pentoxide. The glass-ceramics are useful in the fabrication of single and multi-unit dental restorations (e.g. anterior bridges) made by heat pressing into refractory investment molds produced using lost wax techniques. The glass-ceramics have good pressability, i.e., the ability to be formed into dental articles by heat-pressing using commercially available equipment. In accordance with one embodiment directed to the process of making the glass-ceramics, the compositions herein are melted at about 1200° to about 1600° C., thereafter quenched (e.g., water quenched or roller quenched) or cast into steel molds, or alternately, cooled to the crystallization temperature. The resulting glass is heat-treated to form a glass-ceramic via a one or two step heat-treatment cycle preferably in the temperature range of about 400° to about 1100° C. The resulting glass ceramics are then pulverized into powder and used to form pressable pellets and/or blanks of desired shapes, sizes and structures which are later pressed into dental restorations.

66 Claims, 1 Drawing Sheet



(10) Patent No.: *Sep. 24, 2002 (45) Date of Patent:

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JP JP

U.S. PATENT DOCUMENTS

| 2 (20) (0) | • · · · · · |
|---------------|---|
| 2,628,160 A | 2/1953 Stookey |
| 2,684,911 A | 7/1954 Stookey |
| 2,779,136 A | 1/1957 Stookey |
| 2,920,971 A | 1/1960 Stookey |
| 2,971,853 A | 2/1961 Stookey |
| 3,006,775 A | |
| 3,013,362 A | |
| | 12/1961 Calkins |
| 3,032,429 A | 5/1962 Arthur |
| 3,114,646 A | 12/1963 Currie |
| 3,130,061 A | 4/1964 McMahon |
| 3,157,522 A | 11/1964 Stookey |
| 3,170,805 A | 2/1965 McMillan |
| 3,231,456 A | 1/1966 McMillan |
| 3,236,610 A | • · · · · · · · · · · · · · · · · · · · |
| | 2/1966 McMillan |
| | 3/1966 Hayami |
| 3,252,811 A | 5/1966 Beall |
| 3,253,975 A | 5/1966 Olcott |
| 3,380,838 A | 4/1968 Sack |
| 3,397,076 A | 8/1968 Little |
| 3,436,109 A | 4/1969 Loose |
| 3,450,546 A | - (+ |
| 3,458,330 A | 6/1969 Stong |
| | 7/1969 Tryggve |
| 3,460,987 A | 8/1969 McMillan |
| 3,463,646 A | 8/1969 Little |
| 3,499,787 A | 3/1970 Inoue |
| 3,511,681 A | 5/1970 Huey |
| 3,537,868 A | 11/1970 Kosaka |
| 3,561,984 A | 2/1971 Eppler |
| 3,564,587 A | |
| 3,600,204 A | |
| | 8/1971 Beall |
| 3,647,489 A | 3/1972 McMillan |
| 3,650,817 A | 3/1972 Motoyuki |
| 3,663,193 A | 5/1972 Wilson |
| 3,804,608 A | 4/1974 Hedley |
| 3,809,543 A | 5/1974 Gaskell |
| 3,816,704 A * | |
| 3,907,577 A | 219/402 |
| 3,929,494 A | |
| | * * · · · · · |
| | 1/1976 Beall |
| 3,939,295 A | 2/1976 Robertson |
| 3,977,857 A | 8/1976 Mattox |
| 4,097,295 A | 6/1978 Chyung |
| 4,125,651 A | 11/1978 Campbell |
| 4,186,021 A | 1/1980 Chyung |
| 4,189,325 A | 2/1980 Barrett |
| 4,196,004 A | |
| 4,222,760 A | |
| | 9/1980 Chyung |
| 4,289,538 A | 9/1981 Orso |
| 4,340,645 A | 7/1982 O'Conor |
| 4,391,914 A | 7/1983 Beall |
| 4,414,282 A | 11/1983 McCollister |
| 4,431,451 A | 2/1984 Mabie |
| 4,473,653 A | 9/1984 Rudoi |
| 4,480,044 A | 10/1984 McAlinn |
| 4,515,634 A | |
| 4,672,152 A | 5/1985 Wu |
| | 6/1987 Shinohara |
| 5,176,961 A | 1/1993 Crooker |
| 5,200,369 A | 4/1993 Clifford |
| 5,219,799 A | 6/1993 Beall |
| 5,232,878 A | 8/1993 Kasuga |
| 5,308,391 A | 5/1994 Komma |
| 5,308,803 A | 5/1994 Clifford |
| 5 380 85- | 7/1994 Marazzi |
| 5 80 | 2/1005 0 |
| E 166 00- 1 | - 301/4 |
| 5 505 004 · · | 1/1995 Kamiya |
| 5 550 0 50 | 4/1996 Petticrew 264/16 |
| 5,552,350 A | 9/1996 Hornor |
| 5,567,217 A 1 | 0/1996 Goto |
| 5,580,363 A 1 | 2/1996 Goto |
| 5,702,514 A 1 | 2/1997 Petticrew |
| | |
| | |

١

| 5,744,208 A 4/1998 Beall |
|---|
| 5,820,989 A 10/1998 Reed |
| 5,849,649 A 12/1998 Poole |
| 5,872,069 A 2/1999 Abe |
| 5,874,376 A 2/1999 Taguchi |
| 5.895,719 A 4/1999 Fyles |
| 5,897,885 A 4/1999 Petticrew |
| Total of the second s |
| 5,968,856 A * 10/1999 Schweiger et al 106/35 5,997,977 A 12/1999 Zou |
| 6,121,175 A 9/2000 Drescher |
| A second presenter |
| FOREIGN PATENT DOCUMENTS |
| 2313347 10/1973 |
| 2314721 10/1973 |
| 2314722 10/1973 |
| 2451121 * 5/1974 |
| 3039930 A1 5/1981 |
| 4020893 A1 1/1991 |
| 0 536 572 A1 9/1992 |
| 536479 * 4/1993 |
| 0 536 479 A1 4/1993 |
| 695 726 A1 2/1996 |
| 0781731 A1 7/1997 |
| 0916625 A1 5/1999 |
| 0 827 941 B1 11/1999 |
| 2 655 264 6/1991 |
| 924996 5/1963 |
| 940706 10/1963 |
| 943599 12/1963 |
| 955437 4/1964 |
| 1136501 12/1968 |
| 1312700 * 4/1973 |
| 1467459 3/1977 |
| 49-128011 12/1974 |
| 50094016 7/1975 |
| 62072547 3/1987 |
| 01145348 6/1989 |
| 1145348 # 6/1989 |
| 01145348 7/1989 |
| 03271130 3/1991 |
| 10188260 7/1998 |
| OTHER PUBLICATIONS |
| ore, M.F., Clark, D.E., Hench. L.L, Corrosion Behavior |

Dilmore, M.F., Clark, D.E., Hench. L.L, Corrosion Behavior of Lithia Disilicate Glass in Aqueous Solutions of Aluminium Compounds. Ceramic Bulletin, pp. 1111–1124. vol. 58, No. 11 (1979).

Freiman, S.W., Hench, L.L., Effect of Crystallization on the Mechanical Properties of Li2O–SiO2 Glass–Ceramics. Journal of the American Ceramic Society. pp. 86–90 vol. 55, No. 2.

Hench L.L., Frieman, S.W., Kinser, D.L., The early stages of crystallisation of a Li2O–2SiO2 glass. Physics and Chemistry of Glasses. pp. 58–63. vol. 12, No. 2 Apr. 1971.

McCracken, W.J., Clark, D.E., Hench, L.L, Aqueous Durability of Lithium Disilicate Glass-Ceramics. Ceramic Bulletin. pp. 1218–1223. vol. 61, No. 11 (1982).

McCracken, W.J., Person, W.B., Hench, L.L., Polarized infrared reflection spectroscopy of single crystal lithia–silicates and quartz. Journal of Materials Science. pp. 3853–3864. 20, 1985.

Ohuchi, F., Clark, D.E., Hench, L.L., Effect of Crystallization on the Auger Election Signal Decay in an Li2O 2SiO2 Glass and Glass-Ceramic. Journal of American Ceramic Society. pp. 500–503. Palmer, R.A., Lindberg, W.R., Hench, L.L., Fatigue Properties of Li2O 2SiO2 Glass and Glass Ceramic. Journal of The American Ceramic Society –Discussions and Notes. pp. 319–320. vol. 62, No. 5–6. May–Jun. 1979.

Faber, K.T., Evans, A.G., "Crack Deflection Processes –II.
Experiment," Acta metall. 31, 4, pp. 577–584, 1983.

Borom, M.P., Turkalo, A.M., "Strength and Microstructure in Lithium Disilicate Glass-Ceramics," Journal of the American Ceramic Society, vol. 58, No. 9–10 pp. 385–391, 1975.

* cited by examiner

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FIG. 1

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PRESSABLE LITHIUM DISILICATE GLASS CERAMICS

CROSS-REFERENCE TO RELATED APPLICATIONS

This application claims priority to U.S. Provisional Application No. 60/153,916, filed Sep. 14, 1999, U.S. Provisional Application No. 60/122,558, filed Mar. 2, 1999, and U.S. Provisional Application No. 60/111,872, filed Dec. 11, 1998, all which are incorporated herein by reference.

FIELD OF INVENTION

This invention relates generally to glass-ceramics comprising lithium disilicate and more specifically to glassceramics for use in the manufacture of dental restorations and methods of manufacture thereof.

BACKGROUND OF THE INVENTION

The use of lithium disilicate glass ceramics for use in ²⁰ dental restorations has been suggested in the prior art. U.S. Pat. No. 4,189,325 to Barret et al. is directed to a glass-ceramic comprising lithium disilicate for use in dental restorations. The glass ceramic requires the presence of Nb₂O₅ and Pt as nucleation agents. Barret et al. introduced ²⁵ dental restorations made from castable lithium disilicate glass-ceramics in the Li₂O—CaO—Al₂O₃—SiO₂ system nucleated by Pt and Nb₂O₅. According to Barret et al., dental restorations are made by casting a melt into an investment mold, and devitrifying thereafter. ³⁰

U.S. Pat. No. 4,515,634 to Wu et al. is directed to a castable glass-ceramic composition wherein the glass is melted and cast into a shape and is crystallized after it has been shaped. Therefore, the crystallization process is performed by the technician making the restoration, not the manufacturer of the dental material. Wu set al. suggests one way to improve properties of castable lithium disilicate dental restorations within the same Li2O-CaO-Al2O3-SiO₂ system as described by Barrett et al. is by utilization of P2O5 as a nucleating agent. Both Barrett et al. and Wu et al. describe castable compositions having CaO as an essential ingredient believed to improve chemical durability of the resulting glass-ceramics. Chemical durability is one of the major issues that the Wu and Barrett inventions fail to address. For example, total alkali leaching rates for materials presented in Wu's examples were four to five times higher than those for commercial dental porcelain.

Castable dental ceramics as described in Barret et al. and Wu et al. employ melting glass ingots supplied by a manufacturer and casting dental articles into a refractory investment mold. Following the casting process, the cast articles are devitrified (crystallized) by the required heat-treatment steps. This process is very similar to casting metals whereby a heat-treatment step follows the casting process to increase hardness and strength.

U.S. Pat. Nos. 5,507,981 and 5,702,514 to Petticrew teach lithium disilicate compositions for use in dental restorations, but the method described therein implies forming glass into the shape of a dental restoration at temperatures much higher ₆₀ than the melting temperature of lithium disilicate and heat-treating the dental restoration after forming to convert the glass into a glass-ceramic.

German Patent Application No. DE19647739 to Schweiger et al. is directed to lithium disilicate compositions for 65 use in dental restorations. The glass-ceramic bodies or blanks used to press dental restorations are defined as

"sinterable glass ceramics" which are produced from the starting amorphous glass powder by simultaneous sintering and powder crystallizing, which process is also known as surface crystallization. The glass must be in powder form to be crystallized. Additionally, the lithium disilicate compositions therein require the presence of $La_{2}O_{3}$, MgO and

Many of the lithium disilicate compositions in the prior art require casting of the glass into the desired shape and ¹⁰ crystallizing thereafter. The glass must be formed into the finally desired shape and thereafter heat treated to crystallize into a lithium disilicate phase. This may result in structural and other problems, since the microstructure is not formed by the dental materials manufacturer, but by the technician ¹⁵ fabricating the dental restoration. Overprocessing by a technician may change the microstructure of the material to something not preferred or desired by the dental materials manufacturer. Moreover, some of the prior art compositions require the forming of the glass ceramics by surface ²⁰ crystallization, limiting the forming and compositional possibilities of the material.

It is desirable to provide a lithium disilicate glass-ceramic which is pressable after the lithium disilicate is formed. It is beneficial to provide a lithium disilicate glass ceramic for use in the fabrication of dental restorations wherein crystallization is carried out by the dental materials manufacturer in the most controlled manner. It is beneficial to provide translucent lithium disilicate glass ceramics having high strength and good pressability.

SUMMARY OF THE INVENTION

This invention is directed to lithium disilicate (Li₂Si₂O₅) based glass-ceramics comprising silica, lithium oxide, alumina, potassium oxide and phosphorus pentoxide in addition to other components listed below. The glassceramics are useful in the fabrication of single and multiunit dental restorations including but not limited to orthodontic appliances, bridges, space maintainers, tooth replacement appliances, splints, crowns, partial crowns, dentures, posts, teeth, jackets, inlays, onlays, facing, veneers, facets, implants, abutments, cylinders, and connectors made by heat pressing into refractory investment molds produced using lost wax techniques. The glass-ceramics have good pressability, i.e., the ability to be formed into dental articles by heat pressing, also known as hot pressing, or injection molding, using commercially available equipment.

In accordance with one embodiment directed to the process of making the glass-ceramics, the compositions herein are melted at about 1200° to about 1600° C. and preferably in the range of about 1300° to about 1400° C. for a period of time, preferably for about 4 hours and thereafter quenched (e.g., water quenched or roller quenched) or cast into steel molds, or alternately, cooled to the crystallization temperature.

The resulting glass is heat-treated to form a glass-ceramic via a one or two step heat-treatment cycle preferably in the temperature range of about 400° to about 1100° C. This crystallization heat treatment may comprise a nucleation step and a crystal growth step. Depending on the composition, the first, nucleation step, may be carried out in the range of about 450° C. to about 700° C. and preferably in the range of about 500° C. to about 650° C. for about 0.5 to about 4 hours and the second, crystal growth step, may be carried out in the range of about 500° C. to about 800° C. to about 930° C. and preferably in the range of about 800° C. to about 930° C.

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The resulting glass ceramics are then pulverized into powder and used to form pressable pellets and/or blanks of ⁵ desired shapes, sizes and structures. Additives may be mixed with the powder prior to forming into pellets or blanks. These pellets and blanks may be used for pressing cores or other frameworks or shapes for dental restorations. The blank or pellet may be subjected to viscous deformation at ¹⁰ a temperature in the range of about 800° to about 1200° C., and more preferably in the range of about 850° to about 950° C., and most preferably at less than about 930° C., under vacuum and with the application of pressure of between about 2 to about 8 bar (0.2 to 0.8 MPa) and preferably no ¹⁵ greater than about 6 bar (0.6 MPa) to obtain a dental restoration. Moreover, it is possible that the blanks may be machined to a dental restoration of desired geometry.

BRIEF DESCRIPTION OF THE DRAWINGS

Features of the present invention are disclosed in the accompanying drawing, wherein:

FIG. 1 is perspective view of a plunger system in a pressing furnace for use in the fabrication of dental resto- 25 rations in accordance with the invention.

DESCRIPTION OF THE INVENTION

As will be appreciated, the present invention provides 30 glass-ceramic compositions comprising a glassy matrix and lithium disilicate (Li2Si2O5). The glass-ceramics are useful for the fabrication of dental restorations. The compositions of the lithium disilicate glass-ceramics comprise inter alia, silica, lithium oxide, alumina, potassium oxide and phosphorus pentoxide in the ranges given in Table 1 below. The 35 glass-ceramic compositions of the invention have a combination of properties useful for dental restorations. The glass-ceramics have good pressability, i.e., the ability to be formed into dental articles by heat pressing, also known as 40 hot pressing, or injection molding, using commercially available equipment.

Pressable ceramics employ some form of hot-pressing or injection-molding of the glass-ceramic materials, which are typically in the form of pellets. The pellets contain one or more crystalline phases and their morphology as well as volume fraction are not significantly altered in the course of pressing. One reason for this is that the pressing temperature is typically lower than the melting temperature of the crystalline phases. This is a major advantage because microstructure is formed in the controlled conditions by the manufacturer of the glass-ceramic materials, e.g., pellets. Following pressing, the resulting dental article does not require crystallization heat-treatment.

The glass-ceramic pellets can be formed by a number of processes: (1) Glass can be cast into the shape of pellet. Pellets are taken from the mold and crystallized. These pellets can not be shaded by the addition of pigments. (2) Glass can be crystallized in bulk and subsequently milled into powder. Pigments and other additives can be added to the powder. The powder is formed into a pellet and partially or fully sintered. Pigments, if added, create color centers that impart a certain color to a translucent body of the dental article pressed from the pellet. The mechanism of crystallization.

Alternatively, surface crystallization may be utilized to crystallize a portion of the glass into one or more crystal phases. This involves milling glass into powder. Pigments and other additives can be added to the powder. This glass powder (amorphous, not crystalline) is formed into a pellet. The glass pellet is sintered and crystallized in the same firing cycle. Not all glass-ceramic materials can be crystallized and sintered simultaneously. Only certain materials and compositions prone to surface crystallization can be processed this way. The glass-ceramics processed from glass powder via simultaneous sintering and crystallization are sometimes called "sintered" glass-ceramics. Another term that can be used is "sinterable" glass-ceramics.

The compositions herein are prepared by mixing, in the desired proportions, the oxides and/or compounds that decompose to form the oxides, followed by fusing the ingredients to obtain the compositions in Table 1. Convenient raw material include lithium carbonate, silica, alumina, carbonates of K, Na, Ca, animonium phosphate, tricalcium aluminate, aluminum phosphate or aluminum metaphosphate and if necessary, Ta_2O_5 , CeO_2 , Tb_4O_7 , titanium dioxide, and zirconium dioxide.

TABLE 1

| Oxide wt % | Range 1 | Range 2 | Range 3 | Range 4 | Range 5 | Range 6 |
|--------------------------------|--------------------------|-------------------------|----------------------------|-------------------------|-------------------------|-------------------------|
| SiO ₂ | about 62 to about 85 | about 64 to about 70 | about 62 to about 85 | about 64 to about 70 | about 64 to about 70 | about 62 to about 76 |
| B ₂ O ₃ | 0 to about 4.9 | 0 - about 2.7 | 0 - about 49 | 0 - about 27 | about 0.5 - about 3.0 | 0 - about 5 |
| Al ₂ O ₃ | about 1.5 to about 10 | about 1.5 to about 6.0 | about 5.1 - to about 10 | about 5.2 to about 9.0 | about 1.5 to about 6.0 | about 1.5 to about 10 |
| F | 0 to about 1.5 | 0 to about 1.5 | about 0 to about 1.5 | 0 to about 1.5 | 0 to about 1.5 | 0 to about 1.5 |
| ZnO | 0 to about 5 | 0 to about 2 | 0 to about 5 | 0 to about 2 | _ | 0 to about 5 |
| CaO | 0 to about 7 | 0 to about 0.9 | 0 to about 7 | 0 to about 0.9 | 0 to about 0.9 | 0 to about 7 |
| MgO | 0 to about 2 | 0 to about 2 | 0 to about 2 | about 0 to about 2 | | 0 to about 2 |
| BaO | 0 to about 7 | 0 to about 7 | 0 to about 7 | about 0 to about 7 | | 0 to about 2 |
| SrO | 0 to about 1 | 0 to about 1 | 0 to about 1 | about 0 to about 1 | | 0 to about 1 |
| Cs ₂ O | 0 to about 5 | 0 to about 5 | 0 to about 5 | about 0 to about 5 | | 0 to about 5 |
| Li ₂ O | about 8 to about | about 10 to about | about 8 to about | | about 10 to about | about 5 to about |
| | 19 | 15 | 19 | 15 | 15 | 19 |
| K₂O | about 2.5 to about | about 2.5 to about | - | 0 to about 5 | about 2.2 to about | |

| TABLE 1-continued | | | | | | | | | | |
|--------------------------------|-----------------|--------------------|-----------------------|--------------------|--------------------|------------------------|--|--|--|--|
| Oxide wt % | Range 1 | Range 2 | Range 3 | Range 4 | Range 5 | Range 6 | | | | |
| Na ₂ O | 0 to about 5 | 0 to about 3 | 0 to about 5 | 0 to about 3 | about 0.5 to about | 0 to about 5 | | | | |
| TiO ₂ | 0 to about 2 | 0 to about 2 | 0 to about 2 | 0 to about 2 | 0 to about 2 | 0 to about 2 | | | | |
| ZrO ₂ | 0 to about 3 | 0 to about 3 | 0 to about 3 | 0 to about 3 | 0 to about 3 | 0 to about 3 | | | | |
| $P_{2}O_{5}^{-}$ | 0.5 to about 12 | about 2 to about 7 | about 0.5 to about 12 | about 2 to about 7 | about 2 to about 7 | 0.3 to about 7.0 | | | | |
| SnO ₂ | 0 to about 1 | 0 to about 1 | 0 to about 1 | 0 to about 1 | 0 to about 1 | 0 to about 1 | | | | |
| Sb ₂ O ₃ | 0 to about 1 | 0 to about 1 | 0 to about 1 | 0 to about 1 | 0 to about 1 | 0 to about 1 | | | | |
| Y_2O_3 | 0 to about 3 | 0 to about 3 | 0 to about 3 | 0 to about 3 | 0 to about 3 | 0 to about 3 | | | | |
| CeO ₂ | 0 to about 1 | 0 to about 1 | 0 to about 1 | 0 to about 1 | 0 to about 1 | 0 to about 1 | | | | |
| Eu ₂ O ₃ | 0 to about 1 | 0 to about 1 | 0 to about 1 | 0 to about 1 | 0 to about 1 | 0 to about 1 | | | | |
| Tb ₄ O ₇ | 0 to about 1 | 0 to about 1 | 0 to about 1 | 0 to about 1 | 0 to about 1 | 0 to about 1 | | | | |
| Nb ₂ O ₃ | 0 to about 2 | 0 to about 2 | 0 to about 2 | 0 to about 2 | 0 to about 2 | 0 to about 2 | | | | |
| Ta ₂ O ₅ | 0 to about 2 | 0 to about 2 | 0 to about 2 | 0 to about 2 | 0 to about 2 | about 0.5 to about 8.0 | | | | |

The compositions in Table 1 are melted at about 1200° to about 1600° C. and preferably in the range of about 1300° to about 1400° C. for a period of time, preferably for about 4 hours and thereafter quenched (e.g., water quenched or roller quenched), cast into steel molds, or alternately, cooled to the crystallization temperature. If the melt is cooled to the crystallization temperature, it may remain in the same furnace.

The resulting glass is heat-treated to form glass-ceramics using a one or a two step heat-treatment cycle preferably in the temperature range of about 400° to about 1100° C. This 30 crystallization heat-treatment may comprise a nucleation step and a crystal growth step. Depending on the composition, the first, nucleation step, may be carried out in the range of about 450° C. to about 700° C. and preferably in the range of about 500° C. to about 650° C. for about 0.5 $_{35}$ to about 4 hours and the second, crystal growth step, may be carried out in the range of about 800° C. to about 1000° C. and preferably in the range of about 4800° C. to about 930° C. for about 930° C. for about 0.5 to about 48 hours. The most preferable heat treatment comprises about a one hour soak at about 645° C. $_{40}$ and a subsequent four hour soak at about 850° C.

The glass ceramics comprise lithium disilicate. The resulting glass ceramics are then pulverized into powder sieved to -200 mesh to provide powder with average particle sizes of about 30 to about 40 microns. Pigments, 45 fluorescing agents, opacifying agents, and the like may be added to the powder in a wide range in an amount between about 0 and about 6 wt % and preferably in the amount of between about 0 and about 5 wt % and most preferably in the amount of about 0% to about 3 wt %. Moreover, reinforcing signates may be added to the powder in an amount of from about 0 to about 30 vol % and more preferably in a amount of from about 0 to about 20 vol %. The reinforcing agents may include fibers, whiskers, and particulate fillers and may be fabricated of any known material, preferably a glass or 55 ceramic material.

The powders may be used to form and fuse (sinter) pressable pellets and/or blanks of desired shapes, sizes and structures. Sintering of the pellets or blanks is carried out at temperatures in the range of about 800° to about 1000° C., 60 and preferably in the range of about 850° to about 950° C. Sintering imparts sufficient strength for handling of the pellets or blanks. These pellets and blanks may be used for pressing cores or other frameworks or shapes for dental restorations. The cores may be provided with one or more 65 coatings. The coatings may be selected from a ceramic, a sintered ceramic, a glass ceramic, a porcelain, a glass, a

glaze, a composite and mixtures thereof. The coatings preferably have a firing temperature in the range of about 700° to about 900° C. and a coefficient of thermal expansion (measured from room temperature to its transition temperature) of within about $\pm 2.0 \times 10^{-6}$ /°C. of the dental core (measured at the same temperature range). The blank or pellet may be subjected to viscous deformation at a temperature in the range of about 800° to about 1200° C., and more preferably in the range of about 850° to about 930° C., under vacuum and with the application of pressure of between about 2 to about 8 bar (0.2–0.8 MPa) and preferably no greater than about 6 bar (0.6 MPa) to obtain a dental restoration. Moreover, it is possible that the blanks may be machined to a dental restoration of desired geometry using commercially available milling equipment such as the Maho HGF 500 5 Axis CNC Milling Machine available from Fraunhofer Institut Produktionstechnologie, Germany.

In an alternative method herein, the compositions in Table 1 are melted at about 1200° to about 1600° C. and preferably in the range of about 1300° to about 1400° C. for a period of time, preferably for about 4 hours and thereafter water quenched or cast into steel molds.

The quenched glass is comminuted to a powder. Pigments, fluorescing agents, opacifying agents, and the like may be added to the powder in a wide range in an amount between about 0 and about 6 wt % and preferably in the amount of between about 0 and about 5 wt % and most preferably in the amount of about 0% to about 3 wt %. Moreover, reinforcing agents may be added to the powder in an amount of from about 0 to about 30 vol % and more preferably in an amount of from about 0 to about 20 vol %. The reinforcing agents may include fibers, whiskers, and particulate fillers and may be fabricated of any known material, preferably a ceramic material.

The powder is compacted into a pellet or starting blank. The blank is thereafter simultaneously sintered and crystallized. Heat treatment may be one or more cycles in the temperature range of about 400° to about 1100° C. The crystallization may comprise a nucleation step and a crystal growth step. Depending on the composition, the first, nucleation step, may be carried out in the range of about 450° C. to about 700° C. and preferably in the range of about 500° C. to about 650° C. for about 0.5 to about 4 hours and the second, crystal growth step, may be carried out in the range of about 800° C. to about 1000° C. and preferably in the range of about 830° C. to about 930° C. for about 0.5 to about 48 hours. The most preferable heat treatment comprises about a one hour soak at about 645° C.

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The blank or pellet may be subjected to viscous deformation at a temperature in the range of about 800° to about 1200° C., and more preferably in the range of about 850° to about 950° C., and most preferably at less than about 930° C., under vacuum and with the application of pressure of 5 about between about 2 to about 8 bar (0.2-0.8 MPa) and preferably no greater than 6 bar (0.6 MPa) to obtain a dental restoration. Moreover, it is possible that the blanks may be machined to a dental restoration of desired geometry.

To achieve the required combination of properties, 10 namely sufficient strength, formability below 950° C., by heat-pressing using commercially available dental presses such as the Autopress® Plus available from Jeneric/Pentron, Wallingford, Conn., translucency and chemical durability, the optimal chemical combinations and crystallization treatment of the present invention are necessary. The best properties are obtained when the lithium metasilicate (Li2SiO3) and silica phases are nearly absent, the volume fraction of Li_3PO_4 is less than about 5% and the volume fraction of lithium disilicate ($Li_2Si_2O_5$) is between about 35% and ²⁰ about 60%. High aspect ratio morphology of the lithium disilicate phase is important and is believed to enhance mechanical properties, i.e., strength and fracture toughness of the glass-ceramic.

 Li_2O and SiO_2 are instrumental in crystallizing the ²⁵ required amount of the lithium dislicate phase in the com8

positions of the present invention. Additionally, BaO and Cs₂O stabilize the residual glass and boost the refractive index of the residual glass to match that of lithium disilicate. Al₂O₃ and to a lesser extent, B₂O₃, if less than 3%, yield chemically durable glass-ceramics that exhibit a sufficiently low solubility. Alkali (Na, K, Cs) and alkaline earth metal (Ca, Ba) oxides are required to lower processing temperatures of the glass-ceramic. However some of them affect chemical durability more than others. With respect to the following group of alkali metals and alkaline earth metals of potassium, calcium, sodium, and barium; potassium is associated with the smallest decrease in chemical durability of lithium containing glasses, with calcium being next, and sodium and barium affecting chemical durability the most. However, the best combination of properties is achieved when those oxides are used in combination to achieve the so-called "mixed alkali effect." F lowers the viscosity of the glass-matrix and enhances formability at temperatures below about 950° C. Y₂O₃ in combination with Ce₂O₃, Eu_2O_3 and Tb_4O_7 modify refractive index as well as impart fluorescence. Nb₂O₅ and Ta₂O₅ modify the refractive index as well as aid nucleation and chemical durability of the resulting glass ceramics.

The following Table 2 illustrates examples of the compositions of the invention.

| - T A | Dľ | T | \mathbf{a} |
|-------|----|----------|--------------|
| - I A | 61 | · P. | |

| Oxide, wt % | Ex. I | Ex. 2 | Ex. 3 | Ex. 4 | Ex. 5 | Ex. 6 | Ex. 7 | Ex. 8 | Ex. 9 | Ex. 10 | Ex. 11 | Ex. 12 | Ex. 13 | Ex. 14 | Ex. 15 |
|--|-------|---------|-------|-------------|-------------|-------------|-------|-------|----------|-----------|---------------|-----------|-------------------|-----------|-----------|
| SiO ₂ | 68.8 | 70.0 | 70.1 | 65.4 | 70.7 | 70.9 | 68.1 | 69.5 | 72 | 69.0 | about 68.8 | 65 | about 67.2 | 68.9 | 72 |
| B ₂ O ₃ | 1,2 | 1.0 | | — | — | | — | 1.3 | 2.9 | | about 1.3 | 1.0 | about 1.2 | 0.9 | 2.0 |
| Al_2O_3 | 48 | 4.9 | 5.2 | 8.1 | 5.3 | 5.3 | 4.7 | 4.8 | 5.1 | 6.0 | about 4.8 | 5 | about 4.7 | 4.7 | 4.5 |
| F ZnO | — | | — | 1.4 | 0.2 | | — | | | _ | _ | | _ | | — |
| CaO | 0.9 | 0.5 | 2.2 | | _ | _ | 2.0 | 2.0 | _ | | about 1.0 | 1 | about 1 | 0.5 | 0 |
| MgO | | · | — | | _ | _ | | | | | | | — | | — |
| BaO | 2.8 | 1.4 | | 3.8 | 5.4 | 5.4 | — | — | 2.0 | 2.7 | about 2.8 | 2.7 | about 2.7 | 1.4 | 2.0 |
| SrO | | | — | _ | | | | | — | _ | | | — | | _ |
| Cs ₂ O Li ₂ O | 14.4 | 14.7 | 14.7 | 4.1 13.4 | 4.9 11.0 | 4.9 10.8 | 14.3 | 14.6 | 13.0 | 15.3 | about 14.4 | 15 | about 14.1 | 14.3 | 13.0 |
| K_2O | 2.5 | 2.5 | 4.6 | 1.7 | — | — | 4,4 | 4.5 | 2.0 | 2.1 | about | 2.2 | about | 2.0 | 2.0 |
| Na_2O | 1.4 | 1.4 | | | — | _ | | — | _ | | about 1.5 | 1.4 | about 1.4 | 1.3 | — |
| TiO_2 | | | | _ | _ | _ | | _ | _ | | _ | — | | | |
| ZrO ₂ P ₂ O ₅ | 3.3 | 3.6 | 3.4 | 2.7 | 2.7 | 2.7 | 3.3 | 3.4 | 3.0 | 4.0 | about | 3.5 | about | 3.5 | 3.0 |
| | | | | | | | | | | | 3.3 | | 3.2 | | |
| SnO2 Sb2O3 | _ | | | | | | | | | | | | | | _ |
| Y_2O_3 | _ | _ | | | _ | _ | | | 0.5 | 0.8 | | | _ | | 0.5 |
| CeO ₂ | _ | _ | | - | - | - | 0.4 | _ | 0.5 | _ | - | 0.4 | about 0,4 | 0.3 | 0.6 |
| Eu ₂ O ₃ | | — | | _ | _ | | | | 0.5 | | - | _ | _ | | 0.6 |
| Tb ₄ O ₇ | _ | | | | | | 0.8 | — | | — | | 0.9 | — | 0.3 | — |
| Nb ₂ O ₅ Ta ₂ O ₅ | _ | | | | | _ | 2.0 | | _ | | | 1.9 | about 2.0 | 1.8 | _ |
| Three- | | | | | | | | | | 430 : | ± 420 ± | | 440 ± | : | 400 ± |
| Point Flexural Strength per ISO 6872, | | | | | | | | | | 70 | 60 | | 60 | | 50 |

MPa

| TABLE 2-continued | | | | | | | | | | | | | | | |
|---|-------|-------|-------|-------|-------|-------|-------|-------|----------|-----------|--------------|-----------|-----------|-----------|-----------|
| Oxide, wt % | Ex. 1 | Ex. 2 | Ex. 3 | Ex. 4 | Ex. 5 | Ex. 6 | Ex. 7 | Ex. 8 | Ex. 9 | Ex, 10 | Ex. 11 | Ex. 12 | Ex. 13 | Ex. 14 | Ex. 15 |
| As- pressed Opacity (relative opacity units) | | | | | | | | | | 50 | 42 | | 25 | | 34 |
| CTE (25° C.– 500° C.), 10 ⁻⁶ ° C. ⁻¹ | ı | 10.4 | | | | | | | | 8.7 | 10.1 10.6 | | 10.5 | 10.0 | 9.9 |

The following examples illustrate the invention.

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EXAMPLES

Glass-ceramic compositions of the present invention were ²⁰ utilized to make glass-ceramic pellets. These pellets were used to make rectangular bars and rods for measuring the flexural strength by heat-pressing these pellets into the cavity of the refractory investment mold. The process was the same as that used to make dental restorations. The three-point flexure strength was greater or equal to 30° MPa. This strength is sufficient for multi-unit restorations such as anterior bridges. The compositions have a significant amount of glass phase, i.e., approximately 50% glass phase which enhances the pressability thereof. ²⁰

Moreover, the closeness of refractive indices of the matrix glass (~1.5) and that of the crystallized phase—lithium disilicate—(~1.55) allows for the possibility of translucent glass-ceramics. Specifically in the present invention, the $_{35}$ refractive index of the glass matrix is increased to match that of the lithium disilicate phase by adding small amounts of

heavy ions such as, but not limited to, Sr, Y, Nb, Cs, Ba, Ta, Ce, Eu and Tb.

The following examples in Table 3 illustrate the effect of different additions on translucency, strength and reactivity with investment of the resulting glass ceramics. The composition of Example 11 (Table 2) was selected as a control composition for the purposes of this study. This composition was modified by adding 0.13 mole % of CeO₂, or 0.06 mole % of Tb_4O_7 , or 0.26 mole % of Ta_2O_5 , or La_2O_3 , or Y_2O_3 ; and combinations of the latter with CeO₂. Opacity was measured on the pressed disk using an optical densitometer. Reactivity with investment was evaluated qualitatively by visual inspection of disks and copings prior to and after sand-blasting of the reaction layer. Surfaces of the disks were inspected for pittings under low-magnification (8×) stereomicroscope. Compositions comprising combinations of Ta₂O₅, and CeO₂ were found to have the best combination of high translucency (low opacity) and low reactivity with the investment.

| Oxide, wt % | Ex. 11* | Ex. 16 | Ex. 17 | Ex. 18 | Ex. 19 | Ex. 20 | Ex. 21 | Ex. 22 | Ex. 13* | Ex. 23 |
|---|------------|-----------|-----------|-----------|-----------|-----------|-----------|-----------|------------|---------------|
| SiO ₇ | 68.8 | 68.12 | 68.5 | 68.2 | 67.5 | 67.8 | 67.9 | 68.0 | 67.2 | 67.5 |
| B ₂ O ₃ | 1.3 | 1.2 | 1.2 | 1.2 | 1.2 | 1.2 | 1.2 | 1.2 | 1.2 | 1.2 |
| Al ₂ O ₃ | 4.8 | 4.7 | 4.8 | 4.7 | 4.7 | 4.7 | 4.7 | 4.7 | 4.7 | 4.7 |
| CaO | 1.0 | 1.0 | 1.0 | 1.0 | 1.0 | 1.0 | 1.0 | 1.0 | 1.0 | 1.0 |
| BaO | 2.8 | 2.7 | 2.8 | 2.7 | 2.7 | 2.7 | 2.7 | 2.7 | 2.7 | 2.7 |
| Li ₂ O | 14.4 | 14.3 | 14.3 | 14.3 | 14.1 | 14.2 | 14.2 | 14.2 | 14.1 | 14.1 |
| K ₂ O | 2.2 | 2.2 | 2.2 | 2.2 | 2.2 | 2.2 | 2.2 | 2.2 | 2.2 | 2.2 |
| Na ₂ O | 1.5 | 1.4 | 1.5 | 1.4 | 1.4 | 1.4 | 1.4 | 1.4 | 1.4 | 1.4 |
| P_2O_5 | 3.3 | 3.3 | 3.3 | 3.3 | 3.3 | 3.3 | 3.3 | 3.3 | 3.2 | 3.3 |
| Y ₂ O ₃ | | 1.04 | | | | | 1.04 | | | |
| CeO ₂ | | | 0.39 | | | | 0.39 | 0.39 | 0.39 | 0.39 |
| Tb ₄ O ₇ | | | | 0.85 | | — | | 0.85 | — | |
| Ta ₂ O ₃ | | — | | | 2.02 | | _ | | 2.01 | |
| La2O3 | | _ | | | _ | 1.5 | | _ | | 1.49 |
| 3-pt Flexure | 420 ± | | | | | | | | 440 ± | |
| Strength per ISO 6872, MPa | 60 | | | | | | | | 60 | |
| As-Pressed Rod 3- | 370 ± | | | | | | | | 370 ± | |
| pt Flexure Strength, MPa | 40 | | | | | | | | 30 | |
| As-pressed Opacity (relative opacity units) | 42 | 35 | 39 | 37 | 27 | 25 | 31 | 32 | 25 | 24 |
| Reactivity with investment material | - | Medium | Lower | Higher | Medium | Higher | Lower | Medium | Lower | The Highes |

TABLE 3

*Correspond to compositions in Table 2.

Pellets of the compositions of Examples 2, 11, 13 and 14 were used to press a variety of dental articles in the Auto-Press® dental press (Jeneric/Pentron, Wallingford, Conn.) at pressing cycles carried out under vacuum and involving heating from 700° C. to 920° C. and holding the temperature 5 for 20 minutes prior to initiation of the pressing cycle. Pressure of 0.5 MPa was applied for 7 minutes through a mold-plunger assembly schematically shown in FIG. 1. The plunger assembly used to press the pellets into dental restorations may be the system set forth in copending 10 commonly assigned application Ser. No. 09/431,659, filed Nov. 1, 1999, which is hereby incorporated by reference. Disks of compositions of examples 11 and 13 were pressed as described above and chemical solubility was measured according to ISO 6872 and found to be significantly lower 15 than the acceptable limit of 100 ug/cm².

The glass-ceramics of the present invention have the capability to be pressed into a single or multi-unit dental restorations at temperatures below about 950° C. using already existing, commercially available equipment such as the Autopress® available from Jeneric/Pentron, Wallingford, Conn. Pressability or ability to flow and be pressed into complex shapes of dental restorations at these temperatures is achieved due to the presence of a sufficient amount of the residual glass in the resulting glass-ceramic, in the range of about 15%-60% by volume. The glassceramics of the present invention have the capability to be shaded by admixing pigments to the glass-ceramic powder by methods commonly used for dental porcelains.

As will be appreciated, the present invention provides a ³⁰ simple and effective method for producing lithium disilicate glass-ceramic compositions and dental restorations therefrom. While various descriptions of the present invention are described above, it should be understood that the various 35 features can be used singly or in any combination thereof. Therefore, this invention is not to be limited to only the specifically preferred embodiments depicted herein.

Further, it should be understood that variations and modifications within the spirit and scope of the invention may occur to those skilled in the art to which the invention pertains. Accordingly, all expedient modifications readily attainable by one versed in the art from the disclosure set forth herein that are within the scope and spirit of the present invention are to be included as further embodiments of the 45 present invention. The scope of the present invention is accordingly defined as set forth in the appended claims.

What is claimed is:

1. A glass-ceramic composition consisting essentially of in weight percent: 50

about 64 to about 70% SiO₂; about 1.5 to about 6 Al₂O₃; about 10 to about 15% Li₂O; about 2.5 to about 5% K₂O: about 2 to about 7% P2O5 up to about 1.5% F; up to about 7% BaO; up to about 1% SrO; up to about 5% Cs₂O; up to about 2.7% B₂O₃; up to about 0.9% CaO; up to about 3% Na₂O; up to about 2% TiO2; up to about 3% ZrO₂; up to about 1% SnO2;

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- up to about 1% Sb₂O₃;
- up to about 3% Y₂O₃; up to about 1% CeO₂;
- up to about 1% Eu₂O₃;
- up to about 1% Tb₄O₇;
- up to about 2% Nb₂O₅; and up to about 2% Ta₂O₅.
- 2. The glass-ceramic composition of claim 1 whereby the glass-ceramic is pressable.
- 3. A glass-ceramic composition consisting essentially of in weight percent:
 - about 64 to about 70% SiO₂;
- about 5.2 to about 9 Al₂O₃;
- about 10 to about 15% Li₂O:
- about 2 to about 7% P2O5;
- up to about 1.5% F;
- up to about 7% BaO;
- up to about 1% SrO;
- up to about 5% Cs2O;
- up to about 5% K₂O;
- up to about 2.7% B₂O₃;
- up to about 0.9% CaO;
- up to about 3% Na₂O;
- up to about 2% TiO₂;
- up to about 3% ZrO2;
- up to about 1% SnO2;
- up to about 1% Sb2O3;
- up to about 3% Y2O3;
- up to about 1% CeO₂:
- up to about 1% Eu₂O₃;
- up to about 1% Tb₄O₇;
- up to about 2% Nb₂O₅; and
- up to about 2% Ta₂O₅.
- 4. The glass-ceramic composition of claim 3 whereby the glass-ceramic is pressable.
- 5. A glass-ceramic composition comprising in weight percent:

about 64 to about 70% SiO₂;

- about 1.5 to about 6 Al₂O₃;
- about 10 to about 15% Li₂O;
- about 2 to about 7% P2O5;
- about 2.2 to about 5% K2O;
- about 0.5 to about 3% Na₂O; and
- about 0.5 to about 3% B₂O₃.

6. The glass-ceramic composition of claim 5 whereby the glass-ceramic is pressable.

7. The glass-ceramic composition of claim 5 further 55 comprising in weight percent:

- up to about 1.5% F:
- up to about 7% BaO;
- up to about 1% SrO;
- up to about 5% Cs₂O; 60
- up to about 0.9% CaO;
- up to about 2% TiO₂;
- up to about 3% ZrO₂;
- up to about 1% SnO₂; up to about 1% Sb₂O₃;
 - up to about 3% Y₂O₃;

up to about 1% CeO₂; up to about 1% Eu₂O₃; up to about 1% Tb₄O₇; up to about 2% Nb₂O₅; and 5 up to about 2% Ta₂O₅. 8. A glass-ceramic composition comprising in weight percent: about 62 to about 76% SiO2; about 1.5 to about 10 Al₂O₃; about 8 to about 19% Li₂O; about 0.3 to about 7% P2O5; and about 0.5 to about 8% Ta₂O₅. 9. A glass-ceramic composition comprising: about 62 to about 76% SiO2; about 1.5 to about 10 Al₂O₃; about 8 to about 19% Li₂O; about 0.3 to about 7% P₂O₆; about 0.5 to about 8% Ta₂O₅: and whereby the glass-ceramic is pressable. 10. A glass-ceramic composition comprising in weight percent: about 62 to about 76% SiO₂; about 1.5 to about 10 Al₂O₃; about 8 to about 19% Li₂O; about 0.3 to about 7% P2O5; about 0.5 to about 8% Ta₂O₅; up to about 5% B₂O₃; up to about 5% Na₂O; up to about 7% K₂O; up to about 1.5% F; up to about 5% ZnO; up to about 2% MgO; up to about 7% BaO; up to about 1% SrO; up to about 5% Cs2O; up to about 7% CaO; up to about 2% TiO₂; up to about 3% ZrO2; up to about 1% SnO₂; up to about 1% Sb₂O₃; up to about 3% Y₂O₃; up to about 1% CeO2; up to about 1% Eu₂O₃; up to about 1% Tb_4O_7 ; and up to about 2% Nb₂O₅. 11. A glass-ceramic composition comprising in weight percent: about 68.8% SiO2; about 1.3% B2O3; about 4.8% Al₂O₃; about 1.0% CaO; about 2.8% BaO; about 14.4% Li2O; about 2.2% K2O; about 1.5% Na₂O; and about 3.3% P2O5. 12. The glass-ceramic of claim 11 having a flexural strength in the range of 360 to 480 MPa.

13. The glass-ceramic composition of claim 11 whereby the glass-ceramic is pressable. 14. A glass-ceramic composition comprising in weight percent: about 67.2% SiO₂; about 1.2% B2O3; about 4.7% Al₂O₂; about 1.0% CaO; about 2.7% BaO; 10 about 14.1% Li2O; about 2.2% K₂O; about 1.4% Na₂O; about 3.2% P2O5; 15 about 0.4% CeO2; and about 2% Ta₂O₅. 15. The glass-ceramic of claim 14 having a flexural strength in the range of 380 to 500 MPa. 16. The glass-ceramic composition of claim 14 whereby 20 the glass-ceramic is pressable. 17. A method of making a lithium disilicate dental restoration comprising: melting a starting glass composition at temperatures within the range of about 1200 to about 1600° C.; 25 quenching the glass melt; subjecting the quenched glass to one or more heat treatments in the temperature range of from about 400° to about 1100° C. to convert the glass into a glassceramic: 30 comminuting the glass ceramic to a powder; compacting the powder to a starting blank; sintering the blank; and pressing the blank into the dental restoration. 35 18. A method of making a lithium disilicate dental product comprising: melting a starting glass composition at temperatures within the range of about 1200 to about 1600° C.; quenching the glass melt; 40 subjecting the quenched glass to one or more heat treatments in the temperature range of from about 400° to about 1100° C. to convert the glass into a glassceramic; comminuting the glass ceramic to a powder; 45 compacting the powder to a starting blank; and sintering the blank. 19. The method of claim 18 wherein the blank is manufactured to a desired shape and structure. 20. The method of claim 18 wherein the blank is fully sintered. 21. The method of claim 18 wherein the powder is sieved to the required mesh size prior to the compacting step. 22. The method of claim 18 further including adding 55 additives to the powder prior to the compacting step. 23. The method of claim 22 wherein the additives comprise pigments, fluorescing agents, opacifying agents. 24. The method of claim 23 wherein the additives comprise reinforcing agents. 25. The method of claim 24 wherein the reinforcing agents comprise one of fibers, whiskers, particles and mixtures thereof. 26. The method of claim 24 wherein the reinforcing agents are fabricated of ceramic material. 27. The method of claim 18 wherein the sintering of the 65 starting blank comprises heating to a temperature in the

range of about 800° to about 1000° C.

28. The method of claim 18 wherein the sintering of the starting blank comprises heating to a temperature in the range of about 850° to about 950° C.

29. The method of claim 18 wherein the sintered starting blank is subjected to viscous deformation at a temperature in the range of from about 850° to about 950° C. under vacuum and at a pressure not exceeding about 6 bar to obtained the desired shape.

30. The method of claim 18 wherein the sintered blank is machined to a dental restoration of desired shape.

31. The method of claim 18 wherein the dental product is a dental core and is provided with one or more coatings.

32. The method of claim 31 wherein the one or more coatings is selected from a ceramic, a sintered ceramic, a glass ceramic, a porcelain, a glass, a glaze, a composite and 15 mixtures thereof.

33. The method of claim 31 wherein the one or more coatings has a firing temperature in the range of about 700° to about 900° C. and a coefficient of thermal expansion (measured from room temperature to its transition 20 temperature) of within about $\pm 2.0 \times 10^{-6}$ /°C. of the dental product (measured at the same temperature range).

34. The method of claim 18 wherein the glass-ceramic comprises in weight percent:

about 62 to about 85% SiO2;

about 1.5 to about 10% Al₂O₃;

about 8 to about 19% Li₂O;

about 2.5 to about 7% K2O; and

about 0.5 to about 12% P2O5.

35. The method of claim 34 wherein the glass-ceramic further comprises in weight percent:

up to about 4.9% B₂O₃;

up to about 1.5% F:

up to about 5% ZnO;

up to about 7% CaO;

- up to about 2% MgO;
- up to about 7% BaO;
- up to about 1% SrO;
- up to about 5% Cs₂O;
- up to about 5% Na₂O;
- up to about 2% TiO₂;
- up to about 3% ZrO2;
- up to about 1% SnO₂:
- up to about 1% Sb₂O₃;
- up to about 1% Sb₂O₃;
- up to about 3% Y₂O₃;
- up to about 1% CeO₂;
- up to about 1% Eu₂O₃;
- up to about 1% Tb₂O₇;
- up to about 2% Nb2O5; and
- up to about 2% Ta₂O₅.

36. A method of making a lithium disilicate dental product comprising:

melting a starting glass at temperatures within the range of about 1200 to about 1600° C.; 60

quenching the glass;

- cooling the quenched glass to the crystallization temperature to form a glass-ceramic;
- comminuting the glass ceramic to a powder;

compacting the powder to a starting blank; and sintering the blank.

37. The method of claim 36 wherein the powder is sieved to the required mesh size prior to the compacting step.

38. The method of claim 36 further including adding additives to the powder prior to the compacting step.

39. The method of claim 38 wherein the additives comprise pigments, fluorescing agents, opacifying agents.

40. The method of claim 38 wherein the additives comprise reinforcing agents.

41. The method of claim 40 wherein the reinforcing 10 agents comprise one of fibers, whiskers, particles and mixtures thereof.

42. The method of claim 40 wherein the reinforcing agents are fabricated of ceramic material.

43. The method of claim 36 wherein the sintering of the starting blank comprises heating to a temperature in the range of about 800 to about 1000° C.

44. The method of claim 36 wherein the sintering of the starting blank comprises heating to a temperature in the range of about 850 to about 950° C.

45. The method of claim 36 wherein the sintered starting blank is subjected to viscous deformation at a temperature in the range of from about 850 to about 950° C, under vacuum and at a pressure not exceeding about 7 bar to obtained the desired shape.

46. The method of claim 36 wherein the sintered blank is 25 machined to a dental restoration of desired shape.

47. The method of claim 36 wherein the dental product is a core and is provided with one or more coatings.

48. The method of claim 47 wherein the one or more

30 coatings is selected from a ceramic, a sintered ceramic, a glass ceramic, a porcelain, a glass, a glaze, a composite and mixtures thereof.

49. The method of claim 47 wherein the one or more coatings has a firing temperature in the range of about 700

to about 900° C. and a coefficient of thermal expansion (measured from room temperature to its transition temperature) of within about $\pm 2.0 \times 10^{-6}$ /°C. of the dental product (measured at the same temperature range).

50. The method of claim 47 wherein the glass-ceramic 40 comprises in weight percent:

about 62 to about 85% SiO₂;

about 1.5 to about 10% Al₂O₃;

about 8 to about 19% Li2O;

- about 2.5 to about 7% K2O; and
- 45 about 0.5 to about 12% P2O5.

51. A method of making a lithium disilicate dental restoration comprising:

melting a starting glass composition comprising in weight percent about 64 to about 70% SiO₂;

about 1.5 to about 6 Al₂O₃;

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about 10 to about 15% Li2O;

- about 2.5 to about 5% K2O; and
- about 2 to about 7% P2O5; at temperatures within the range of about 1200° to about 1600° C.;

quenching the glass melt;

subjecting the quenched glass to one or more heat treatments in the temperature range of from about 400° to about 1100° C. to convert the glass into a glassceramic:

comminuting the glass ceramic to a powder; compacting the powder to a starting blank;

sintering the blank; and

pressing the blank into the dental restoration. 65 52. A method of making a lithium disilicate dental restoration comprising:

about 14.4% Li₂O; about 2.2% K₂O;

| 1/ | |
|---|-----|
| melting a starting glass composition comprising in weight percent about 62 to about 85% SiO ₂ ; | |
| about 5.1 to about 10 Al_2O_3 ; | |
| about 8 to about 19% Li ₂ O; and | 5 |
| about 0.5 to about 12% P_2O_5 ; at temperatures within the range of about 1200° to about 1600° C.; | ., |
| quenching the glass melt; | |
| subjecting the quenched glass to one or more heat treat- ments in the temperature range of from about 400° to about 1100° C. to convert the glass into a glass- ceramic; | 10 |
| comminuting the glass ceramic to a powder; | |
| compacting the powder to a starting blank; | 15 |
| sintering the blank; and | 1.5 |
| pressing the blank into the dental restoration. 53. A method of making a lithium disilicate dental resto- ration comprising: | |
| melting a starting glass composition comprising in weight | 20 |
| percent about 64 to about 70% SiO_2 ; about 5.2 to about 9 Al ₂ O ₃ ; | |
| about 5.2 to about 9 A_2O_3 , about 10 to about 15% Li ₂ O; and | |
| about 10 to about 15% P_2O_5 ; at temperatures within the range of about 1200° to about 1600° C.; | 25 |
| quenching the glass melt; | |
| subjecting the quenched glass to one or more heat treat- ments in the temperature range of from about 400° to about 1100° C. to convert the glass into a glass- ceramic; | 30 |
| comminuting the glass ceramic to a powder; | |
| compacting the powder to a starting blank; | |
| sintering the blank; and | 35 |
| pressing the blank into the dental restoration. | 55 |
| 54. A method of making a lithium disilicate dental resto- | |
| ration comprising: | |
| melting a starting glass composition comprising in weight percent | 40 |
| about 64 to about 70% SiO_2 ; | |
| about 1.5 to about 6 Al_2O_3 ; | |
| about 10 to about 15% Li_2O ; | |
| about 2 to about 7% P_2O_5 ; | 45 |
| about 2.2 to about 5% K_2O ; | |
| about 0.5 to about 3% Na_2O ; and | |
| about 0.5 to about 3% B ₂ O ₃ ; at temperatures within the | |
| range of about 1200° to about 1600° C.; | 50 |
| quenching the glass melt; | |
| subjecting the quenched glass to one or more heat treat- ments in the temperature range of from about 400° to about 1100° C. to convert the glass into a glass- | |
| ceramic; | 55 |
| comminuting the glass ceramic to a powder; | ~ |
| compacting the powder to a starting blank; | |
| sintering the blank; and | |
| pressing the blank into the dental restoration. 55. A method of making a dental restoration comprising: | 60 |
| melting a starting glass composition comprising in weight percent about 68.8% SiO ₂ ; | |
| about $1.3\%B_2O_3$; | |
| about 4.8% Al ₂ O ₃ ; | 65 |
| about 1.0% CaO; | |
| about 2.8% BaO; | |
| | |

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about 1.5% Na₂O; and about 3.3% P2O5; at temperatures within the range of about 1200° to about 1600° C.; quenching the glass melt; subjecting the quenched glass to one or more heat treatments in the temperature range of from about 400° to about 1100° C. to convert the glass into a glassceramic; comminuting the glass ceramic to a powder; compacting the powder to a starting blank; sintering the blank; and pressing the blank into the dental restoration. 56. A method of making a dental restoration comprising: melting a starting glass composition comprising in weight percent about 68.8% SiO2; about 1.3% B2O3; about 4.8% Al₂O₃; about 1.0% CaO; about 2.8% BaO; about 14.4% Li₂O: about 2.2% K₂O;

about 1.5% Na2O; and

- about 3.3% $P_2O_5;$ at temperatures within the range of about 1200° to about 1600° C.;
- quenching the glass melt;
- subjecting the quenched glass to one or more heat treatments in the temperature range of from about 400 to about 1100° C. to convert the glass into a glassceramic;
- comminuting the glass ceramic to a powder;

compacting the powder to a starting blank;

sintering the blank; and

- pressing the blank into the dental restoration.
- 57. A dental restoration comprising a glass-ceramic consisting essentially of:

about 62 to about 85% SiO₂;

about 1.5 to about 10% Al₂O₃;

- about 8 to about 19% Li₂O;
- about 2.5 to about 7% K₂O;
- about 0.5 to about 12% P2O5;
- up to about 4.9% B₂O₃;
- up to about 1.5% F;
- up to about 7% CaO;
- up to about 7% BaO;
- up to about 1% SrO;
- up to about 5% Cs₂O;
- up to about 5% Na₂O;

up to about 2% TiO_2 ;

- up to about 3% ZrO₂;
- up to about 1% SnO₂;
- up to about 1% Sb₂O₃;
- up to about $3\% Y_2O_3$;
- up to about 1% CeO₂;
- up to about 1% Eu₂O₃;
- up to about 1% Tb_4O_7 ; up to about 2% Nb_2O_5 ; and
- up to about 2% Ta_2O_5 .

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58. The dental restoration of claim **57** formed into a component selected from orthodontic appliances, bridges, space maintainers, tooth replacement appliances, splints, crowns, partial crowns, dentures, posts, teeth, jackets, inlays, onlays, facing, veneers, facets, implants, abutments, 5 cylinders, and connector.

59. A blank for machining the dental restoration of claim **57**.

60. A dental restoration comprising the glass-ceramic of claim 1.

61. A dental restoration comprising the glass-ceramic composition comprising:

about 64 to about 70% SiO₂;

about 5.2 to about 9 AI_2O_3 ;

about 10 to about 15% Li2O; and

about 2 to about 7% P₂O₅.

62. A dental restoration comprising the glass-ceramic of claim 5.

63. A dental restoration comprising the glass-ceramic 20 comprising:

about 62 to about 76% SiO₂;

about 1.5 to about 10 Al₂O₃;

about 8 to about 19% Li_2O ;

about 0.3 to about 7% P_2O_5 ; and

about 0.5 to about 8% Ta₂O₅.

64. A dental restoration comprising the glass-ceramic of claim 14.

65. A dental restoration comprising the glass-ceramic of claim 11.

66. A method of making a dental restoration comprising: melting a starting glass composition comprising in weight percent about 62 to about 76% SiO₂;

about 1.5 to about 10 Al₂O₃;

about 8 to about 19% Li2O;

about 0.3 to about 7% P_2O_5 ; and

about 0.5 to about 8% Ta_2O_5 ; at temperatures within the range of about 1200° to about 1600° C.;

¹⁵ quenching the glass melt;

subjecting the quenched glass to one or more heat treatments in the temperature range of from about 400° to about 1100° C. to convert the glass into a glassceramic;

comminuting the glass ceramic to a powder; compacting the powder to a starting blank; sintering the blank; and

pressing the blank into the dental restoration.

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