



PATENT OWNER PRESENTATION

Samsung Electronics Co., Ltd. et al v. Nanoco Technologies, Ltd.

IPR2021-00183, -00184, -00185, -00186

February 23, 2022

Overview

- **Background**
- **The Banin Grounds**
- **The Obviousness Grounds**
 - Technology
 - Banin in view of Herron, Farneth, Bawendi, Treadway, and/or Braun
 - Zaban in view of Farneth, Yu, Ptatschek, Bawendi, and/or Braun
 - Lucy in view of Ahrenkiel and/or Braun

Challenged Claims

Ground	8,524,365 Claims	35 U.S.C. §	Reference(s)
1	1, 7-12, 17, 22, 23	102	Banin
2	1, 7-12, 15-17, 22, 23	103(a)	Banin
3	2-6, 18-21	103(a)	Banin, Herron
4	13, 14	103(a)	Banin, Treadway
5	1-9, 17-23	103(a)	Zaban, Farneth, Yu
6	1, 2, 4, 7-12, 17, 18, 22, 23	103(a)	Lucey, Ahrenkiel
7	13-16	103(a)	Lucey, Ahrenkiel, Treadway

Ground	7,803,423 Claims	35 U.S.C. §	Reference(s)
1	1-3, 10, 11, 13, 22-24	102	Banin
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Ground	7,588,828 Claims	35 U.S.C. §	Reference(s)
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4	1, 4, 16, 17	103(a)	Lucey, Ahrenkiel, Braun



Background

The Quantum Dot Patents

8,524,365



US8524365B2

(12) **United States Patent**
O'Brien et al.

(38) **Patent No.:** **US 8,524,365 B2**
(45) **Date of Patent:** ***Sep. 3, 2013**

(54) **PREPARATION OF NANOPARTICLE MATERIALS**

(57) **Field of Classification Search**
None
See application file for complete search history.

(75) **Inventors:** **Paul O'Brien, (High Tech. (G)); Nigel Pickett, (Mechanics (G))**

(58) **References Cited**

(73) **Assignee:** **Nanoco Technologies Ltd. (G)(U)**

(51) **U.S. PATENT DOCUMENTS**
2,700,018 A 11/17/90 Mawardi et al.
2,704,771 A 8/17/91 Olson
4,209,000 A 8/19/90 Nakawo et al.
6,134,618 A 8/20/99 Carter et al.
(COINVENTOR)

(*) **Notice:** Subject to any disclaimer, the term of this patent is extended or adjusted under 35 U.S.C. 154(b) by 0 days.
This patent is subject to a terminal disclaimer.

(52) **Applicant:** **US200702532**

(59) **FOREIGN PATENT DOCUMENTS**
CN 1704910 2/2003
JP 0776640 5/2002
(COINVENTOR)

(21) **Filed:** **Oct. 6, 2011**

(60) **OTHER PUBLICATIONS**
"Semicon. "Hanging In a Nanoparticle Processor: Global Semiconductor Consortium (GSC) of (S-C) companies", *Solid State Electronics*, vol. 45, (2003), pp. 543-549.*
(COINVENTOR)

(22) **Pub. No.:** **US 20120025155 A1** Feb. 2, 2012

(65) **Related U.S. Application Data**
Continuation of application No. 12/814,611, filed on Aug. 11, 2010, now Pat. No. 8,062,303, which is a continuation of application No. 11/277,600, filed as application No. PCT/US2005/001411, on Apr. 27, 2005, now Pat. No. 7,301,423.

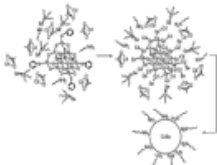
(70) **Primary Examiner:** **Nathan Empey**
Assistant Examiner: **Linda Jiang**
(74) **Attorney, Agent, or Firm:** **Wong, Cahillo, Lomach, Rutherford & Associates LLP**

(30) **Foreign Application Priority Data**

(57) **ABSTRACT**
A method of producing nanoparticles comprises effecting coagulation of a nanoparticle precursor composition to the material of the nanoparticles. The precursor composition comprises a first precursor species containing a first core to be incorporated into the growing nanoparticles and a separate second precursor species containing a second core to be incorporated into the growing nanoparticles. The coagulation is effected in the presence of a molecular cluster compound under conditions permitting settling and growth of the nanoparticles.

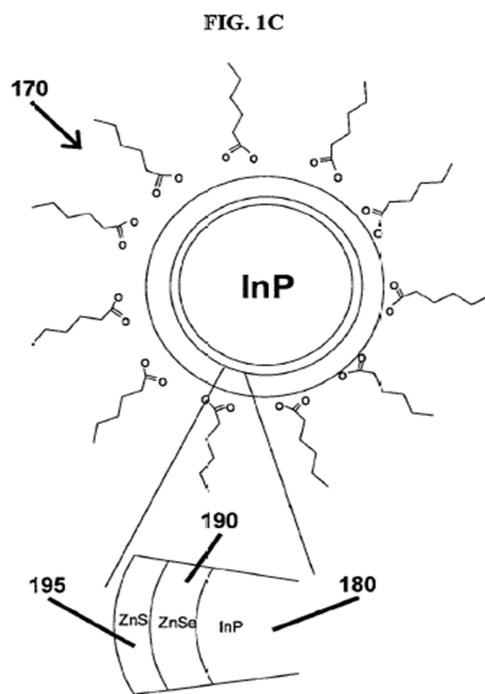
(25) **Claims, 18 Drawing Sheets**

Samsung Ex. 1001
1 of 33



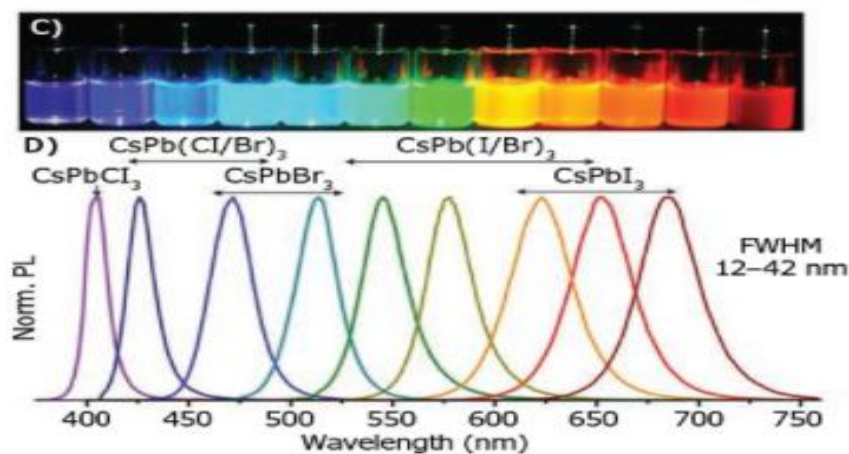
Quantum Dots

- Man-made semiconductors nanoparticles



Quantum Dots – Size Precision Is Important

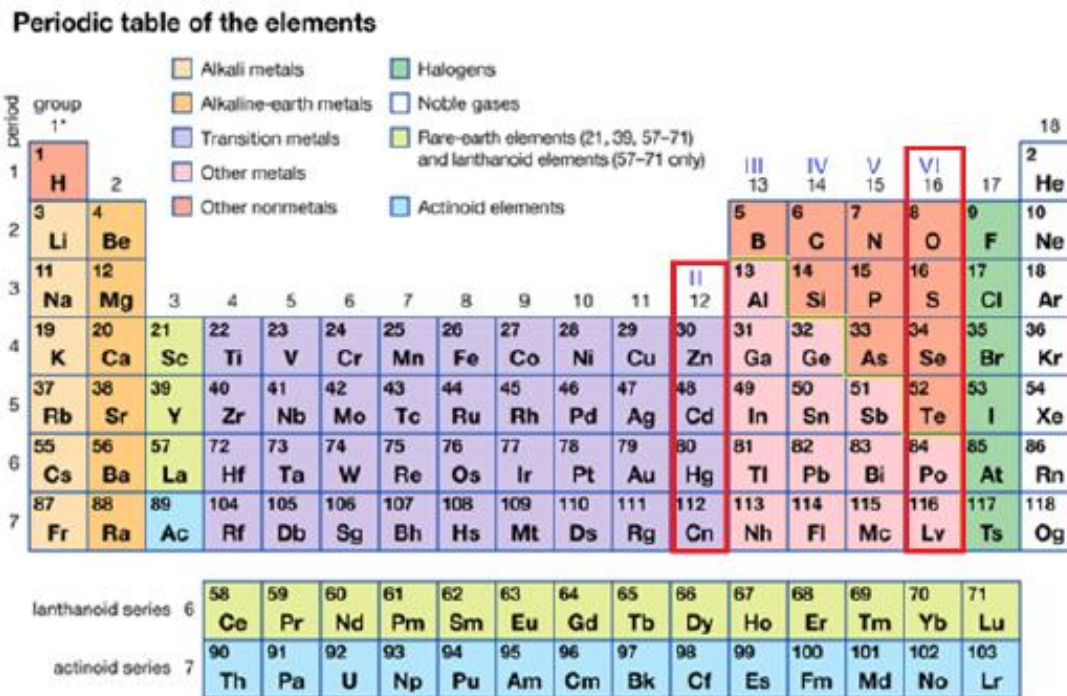
- Size of the QD dictates the wavelength, and thus the color of light emitted
- The more uniform in size, the better color precision.
- Uniformly sized QDs are called “monodisperse.”
- It is difficult to make monodisperse populations of quantum dots.



Ex. 1001, 1:20-27; POR (IPR2021-00186) at 4; Ex. 2030 (IPR2021-00186) ¶¶ 33-35.

Quantum Dots – Needed Alternative to Toxic Cadmium Selenide

- Cadmium selenide (CdSe) can be relatively easily size-controlled, but extremely toxic.
- CdSe are group 12-16 (II-IV) elements.



*Numbering system adopted by the International Union of Pure and Applied Chemistry (IUPAC). © Encyclopædia Britannica, Inc.

Ex. 2030 (IPR2021-00186) ¶¶ 36-37; Ex. 1001, 1:40-43; POR (IPR2021-00186) at 24.

Quantum Dots – Nanoco’s Quantum Dot Invention

- To achieve uniformly sized QDs, Nanoco invented a new method
 - Using uniform molecular cluster compounds in the reaction
 - Reaction typically initiated by combining a known concentration of a small well-defined molecular cluster compound with a coordinating solvent in the presence of multiple semiconductor precursors before slowly increasing the temperature

Ex. 2030 (IPR2021-00186) ¶¶ 40, 65-66; Ex, 2012 at 4; POR (IPR2021-00186) at 21-22.

Molecular Cluster Compounds

The Court hereby construes the term “**molecular cluster compound,**” for the ‘828, ‘557, ‘423, and ‘365 patents, to mean “**clusters of three or more metal atoms and their associated ligands of sufficiently well-defined chemical structure such that all molecules of the cluster compound possess the same relative molecular formula.**”

Ex. 1091 at 18; POR (IPR2021-00186) at 29.



The **Banin** Grounds

Grounds Relying on Banin Alone – Banin Lacks a MCC

Ground	8,524,365 Claims	35 U.S.C. §	Reference(s)
1	1, 7-12, 17, 22, 23	102	Banin
2	1, 7-12, 15-17, 22, 23	103(a)	Banin
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No MCC - Banin Does Not Disclose a Molecular Cluster Compound

- **If Banin does not disclose a molecular cluster compound, Petitioner's Grounds fail**
- **The Institution Decision cast doubt on whether Banin discloses a molecular cluster compound, and asked the parties to address three issues:**
 1. Do Hutchison's differently sized gold particles, with only an estimated average formula, have "sufficiently well-defined chemical structure such that all molecules have the same relative molecular formula?"
 2. Are Hutchison's "narrowly disperse" gold particles "monodisperse" gold particles?
 3. Are Banin's gold particles "ensembles of small nanoparticles" distinguished by the specifications?

InstDec (IPR2021-00186) at 17-22 ; POSR (IPR2021-00186) at 2-4.

No MCC - Banin Does Not Disclose a Molecular Cluster Compound

- Hutchison does not disclose a MCC – **No Dispute**
- **Petitioner's Concessions:**
 - Banin makes its gold particles using the Hutchison method.
 - Hutchison averages out different sized gold particles to estimate the average number of gold atoms.
 - Narrowly disperse particles are not considered monodisperse.
 - Banin's gold particles aggregate.
 - Aggregates that span a distribution of masses are not MCCs.

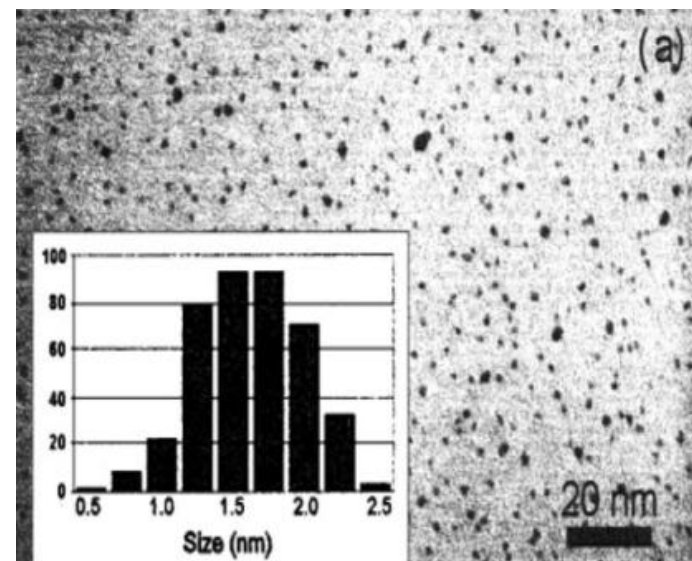
Petitioner's Reply at 4 (IPR2021-00186); POSR (IPR2021-00186) at 11; Ex. 2034, 120:3-13; InstDec (IPR2021-00186) at 19; Ex. 2030 (IPR2021-00186) ¶¶ 105-109.

No MCC - Hutchison's Suggested Formula Is Just an Average

Question 1: Does Hutchison average out the gold clusters of different sizes to estimate the average number of gold atoms?

Answer: YES.

- Petitioner concedes that Hutchison's process produce distribution of gold cluster sizes that are averaged to create a "suggested" formula
 - The "[Au101] shown in Hutchison *are consistent with a size distribution of clusters*"
 - "25% size distribution of Hutchison's gold particles"
- A distribution of cluster sizes that are averaged into a "suggested" formula are not "sufficiently well-defined chemical structure such at all molecules have the same relative molecular formula."
 - Dr. Bawendi - "aggregates ... [that] span a distribution of masses" do not meet the Court's construction of 'molecular cluster compound'"
 - Dr. Cossairt - Hutchison's does not produce particles that "possess the same relative molecular formula."



Petitioner's Reply at 3, 5 (IPR2021-00186); POSR (IPR2021-00186) at 3; Ex. 2030, ¶¶105-109 (IPR2021-00186); Ex. 2017, Fig.1(a) .

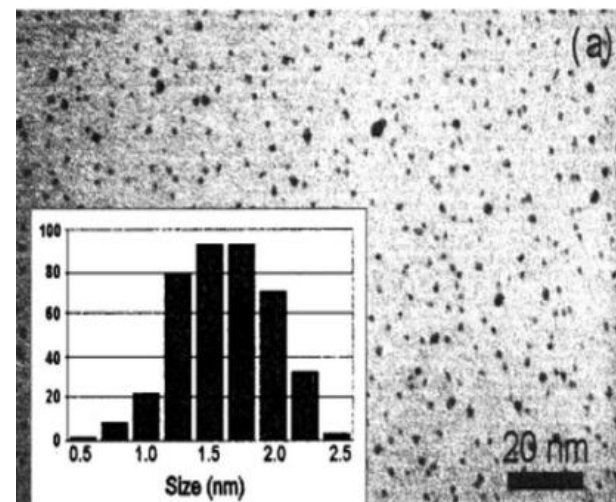
No MCC - Hutchison's Narrowly Disperse Gold Particles Are Not Monodisperse

Question 2: Are Hutchison's "narrowly disperse" gold particles "monodisperse" gold particles?

Answer: NO.

- Petitioner admits Hutchison results in "*polydisperse* clusters."
- Polydisperse particles are not molecular cluster compounds
 - Both sides' experts agree that Hutchison does not disclose a MCC

Hutchison – Polydisperse Clusters



Petitioner's Reply at 3 (IPR2021-00186); POSR (IPR2021-00186) at 3-4; Ex. 1093 ¶¶18-21 (IPR2021-00186).

No MCC - Banin's Gold Catalysts are Ensembles of Small Nanoparticles

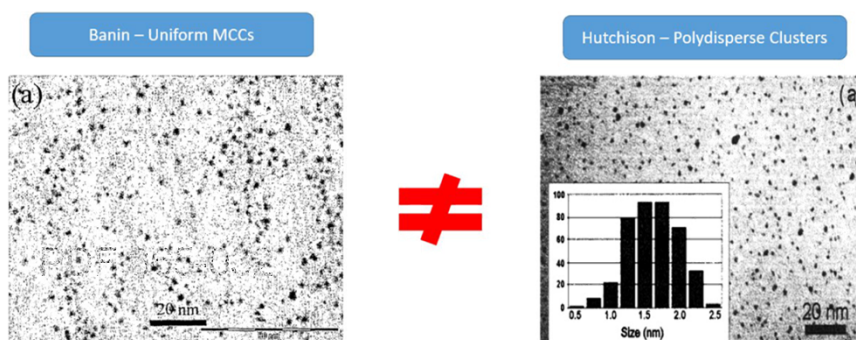
Question 3: Are Banin's gold clusters akin to ensembles of small nanoparticles expressly distinguished by the patents?

Answer: YES.

- Dr. Cossairt - "Banin's gold particles are also ensembles of small nanoparticles; Ex. 1005 at 23:23-24:2 (discussing the gold particle aggregating to form larger particles)."
- Petitioner admits that Banin's gold particles are aggregates
 - "Banin does not state (and a POSITA would not have understood Banin to suggest) that its MCCs undergo any chemical change **when they aggregate**."
- Petitioner's expert admits that aggregates are not MCCs.
 - "aggregates ... [that] span a distribution of masses" do not meet the Court's construction of 'molecular cluster compound'"

Petitioner's Reply at 7 (IPR2021-00186); POSR (IPR2021-00186) at 3-4, 14; Ex. 2034, 120:3-13; Ex. 2017, Fig.1(a); Ex. 2030 (IPR2021-00186) ¶ 114.

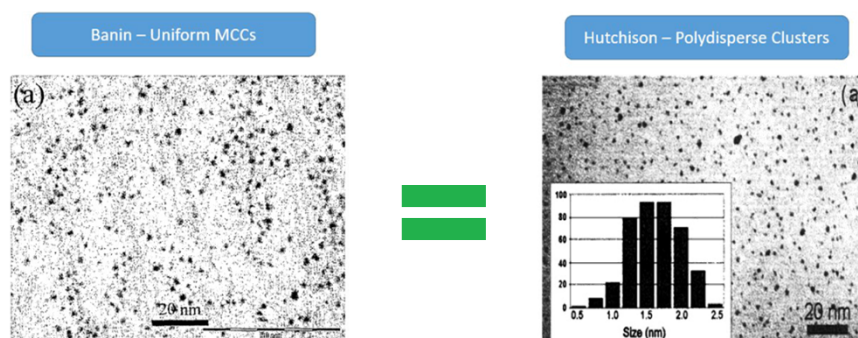
Hutchison Does Not Produce MCC, So Petitioner Brings New and Unsupported Arguments



As Petitioner’s expert Dr. Green explains, Banin’s TEM is consistent with uniform $\text{Au}_{101}(\text{PPh}_3)_{21}\text{Cl}_5$ MCCs, while those shown in Hutchison are consistent with a size distribution of clusters. Ex. 1093 ¶¶18-21. A POSITA would recognize from these images that Banin’s MCCs represent purified, size-selected MCCs, even if they were made starting with Hutchison’s method. See *id.* ¶18. This is common

Petitioner’s Reply at 3; POSR (IPR2021-00186) at 8, 11; Ex. 2034, 120:3-13; Ex. 1005, 7, 24:15-23.

No MCC - Banin Does Not Further Size-Select its Gold Catalysts



- Images do not show size selection; they are indistinguishable
- Nothing written in Banin even intimates size selection of gold particles
- Rather, Banin recognizes and accepts size distribution
 - Banin discloses “small Au clusters (1.4 nm in mean diameter)”
 - The Banin catalysts are, as recognized in Hutchinson, just an average size.

Petitioner’s Reply at 3 (IPR2021-00186);
POSR (IPR2021-00186) at 6; Ex. 1005, 20:12-16.

No MCC - Banin Does Not Further Size-Select its Gold Catalysts

- Additionally:
 - Banin only describes size-selecting the nanorod end-product, not the gold catalyst/proposed MCC
 - Nothing in Banin suggests that it performs any further processing of the gold catalysts
 - Only size-selection Petitioner identifies is centrifugation (Reply, fn.3)
 - Centrifugation is of resulting nanorods, not the gold cluster
 - Selection is for rod length, not for diameter
 - After centrifugation, nanorod population still polydispersed
 - “diameter distribution [of] 20-25% ... length distribution is 15-20%”
 - No enablement of size-selected gold particles with the precision required to produce MCCs
 - Assertions that are not enabled cannot anticipate any challenged claim

Petitioner’s Reply at 4, fn.3 (IPR2021-00186); POSR (IPR2021-00186) at 8, 11; Ex. 2034, 120:3-13; Ex. 1005, 7, 24:15-23.

No MCC - Petitioner's New Arguments Regarding the Yu Reference Fail

- Inadmissible as new matter first raised in the reply
- Even so, does not cure Banin failure to disclose an MCC
 - Yu does not disclose a molecular cluster compound
 - Yu uses the Hutchison method to make gold particles that Petitioner concede are not MCCs
 - Same failure as Banin to clarify failure of Hutchison to disclose an MCC
 - Petitioner does not allege that Yu further size-selects its gold clusters
 - Yu is not relevant for other reasons
 - Yu's seeding process is incompatible with SLS reactions
 - Yu does not make semiconductor nanoparticles
 - Yu does not use multiple precursors
 - Yu does not use a MCC

Petitioner's Reply at 9 (IPR2021-00186);
POSR (IPR2021-00186) at 6; Ex. 1010 at 9198.

Banin lacks a MCC

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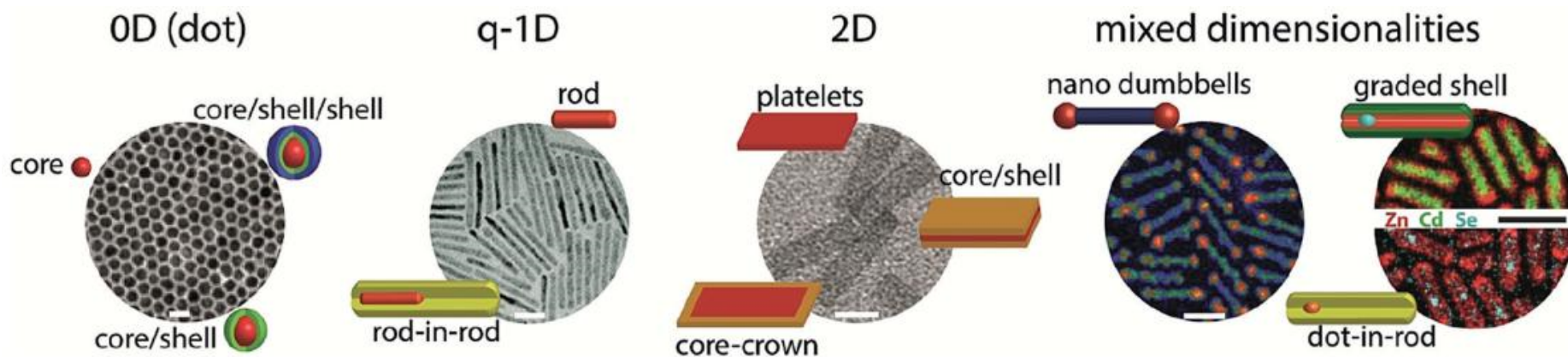
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The Obviousness Grounds - Technology

Different Types of Nanoparticles and Different Methods of Manufacture

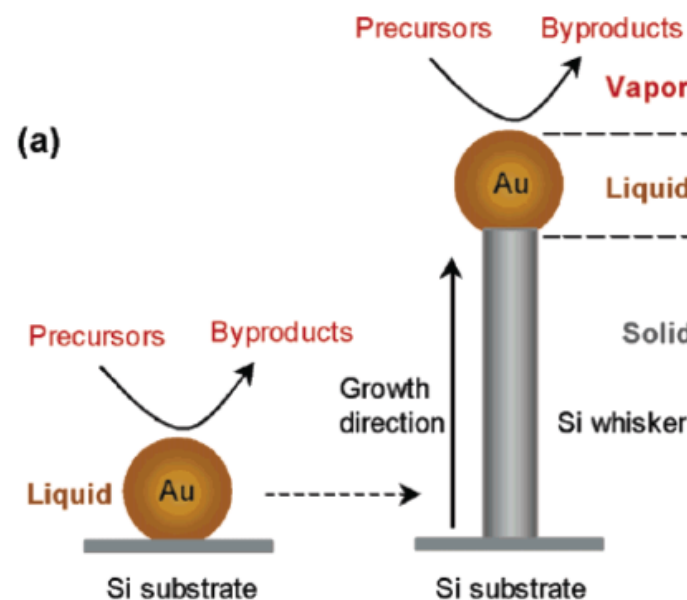
There are many types of nanoparticles, and many ways to make them.



Ex. 2036 at 4276, Ex. 2030 at ¶ 42, POR at 8.

Nanorods – Overview of VLS Reactions

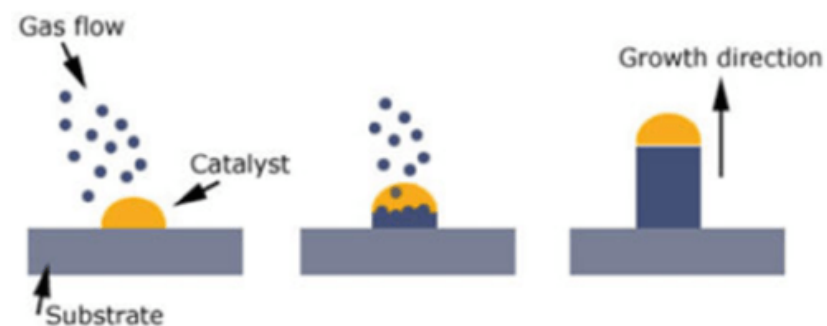
- Vapor-Liquid-Solid (VLS)
 - VLS uses vaporized precursors to deposit atoms directly on a substrate
 - At extremely high temperatures a metal melts into liquid droplets
 - These liquid metal droplets dissolve the vaporized precursor ions
 - The liquid metal becomes supersaturated and precipitates creating a long one-dimensional nanorod or whisker
 - **Vapor**-phase (silicon precursors), **Liquid** phase (melted gold droplets, called the “catalyst”), and **Solid** phase (crystalline semiconductor whiskers)



POR (IPR2021-00186); at 9-12; Ex. 2030
(IPR2021-00186) at ¶¶ 46-50; Ex. 2008 at 7514.

Nanorods – Overview of VLS Reactions

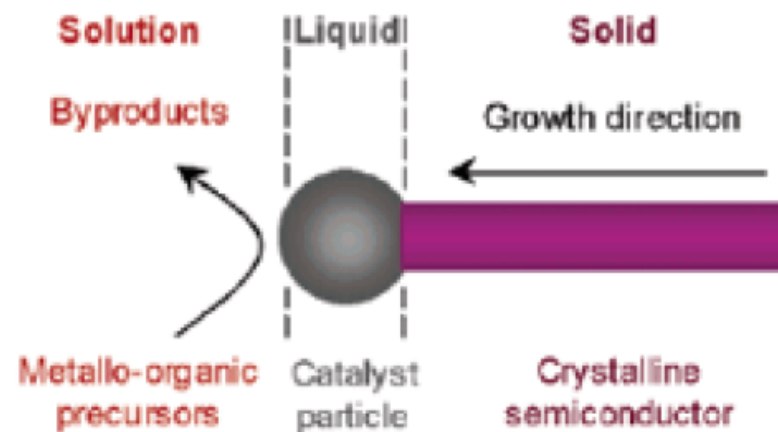
- Requirements for a catalyst:
 - (1) **A liquid metal catalyst** (e.g., gold);
 - (2) Catalyst must be able to **dissolve the substrate**;
 - (3) Solubility of **precursors**: sufficient for supersaturation;
 - (4) **Vapor pressure**: catalyst cannot vaporize, shrink in volume or decrease in radius
 - (5) **A catalyst that won't react**: the catalyst must be inert to the reaction precursors and products;
 - (6) **Compatibility of the components** of the reaction: the vapor–solid, vapor–liquid, and liquid–solid interfacial energies must be examined for compatibility before choosing a suitable catalyst



POR (IPR2021-00186); at 9-12; Ex. 2030
(IPR2021-00186) at ¶¶ 46-50; Ex. 2008 at 7514.

Nanorods – Overview of SLS Reactions

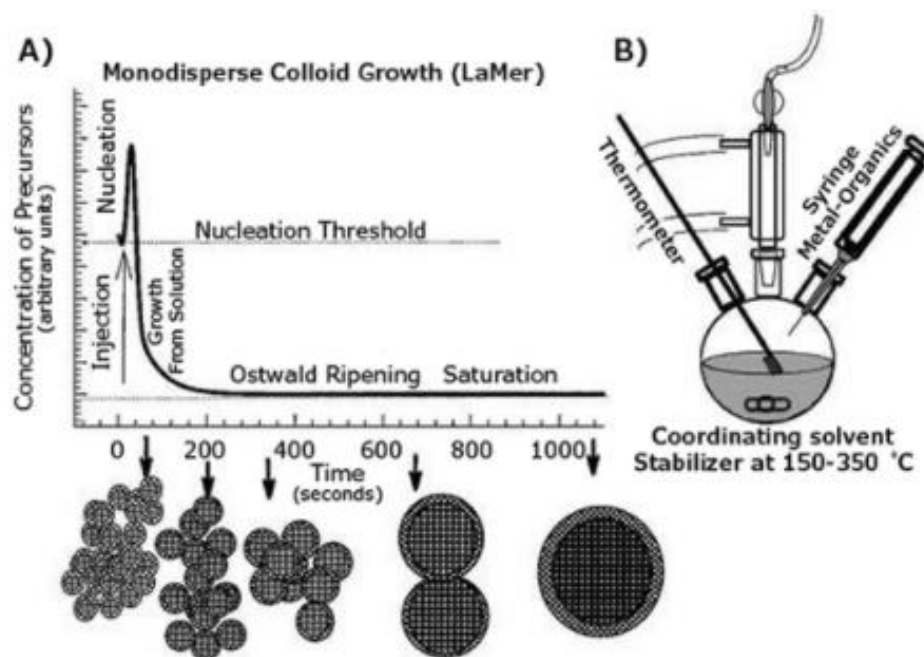
- SLS uses molten metal catalysts to dissolve precursors and create one dimensional crystal growth.
- Requirements for a catalyst include the metal's:
 - (1) **melting point**;
 - (2) **solvating abilities**; and
 - (3) **reactivity**.
- Catalyst must dissolve at least one semiconductor component and allow supersaturation.
- Metal catalyst should not react with the target semiconductor unless the catalyst is one of the constituent elements.
- Melting points typically between ~150–350 °C.
- Metal nanoparticles with low melting points are the most useful
- Gold is a common catalyst along with indium (In), bismuth (Bi), and tin (Sn).



POR (IPR2021-00186) at 12-15.

Overview of the Hot-Injection Method

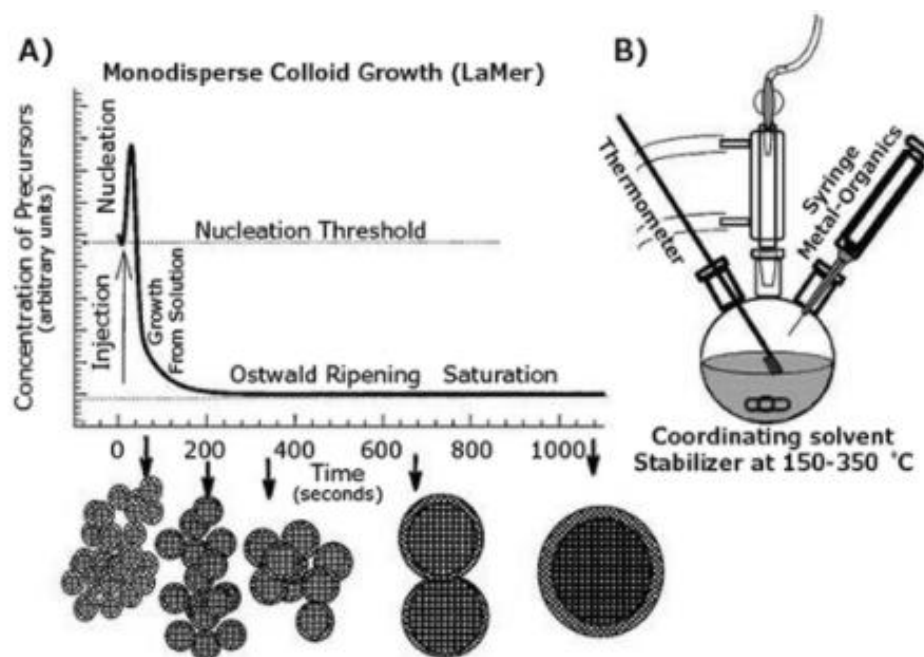
- Rapid injection of a room temperature solution of precursors into an extremely hot reaction medium in the presence of carefully chosen surfactant molecules
- Induces a sudden supersaturation of the solution, resulting in a short burst of nucleation
- Further nucleation is halted by a drop in reaction temperature
- Quantum dots then grow at a lower temperature than that of the nucleation process



Ex. 2030 (IPR2021-00186); ¶¶ 57-61;
Ex. 1037, 82; POR (IPR2021-00186); at 15-18.

Overview of the Dual Hot-Injection Method

- Different variations: single-source precursors and dual-injection
- Dual-injection is restrictive because the precursors usually cannot be pre-mixed.
 - This can be a major drawback because localized variation of precursor concentration can result in broadened size distribution of the initial nucleated crystals
 - Single-source precursors are therefore often preferred.

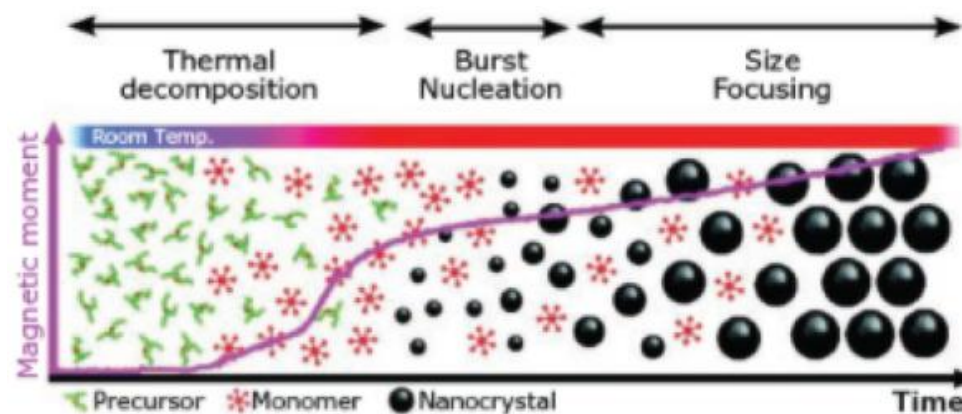


Ex. 2030 (IPR2021-00186); ¶¶ 57-61;
Ex. 1037 at 82; POR (IPR2021-00186) at 15-18.

Overview of the Heat-Up Method

- More efficient than hot-injection
 - Lower temperatures
 - Single pot reaction

- Requires very specific precursors
 - Must be highly reactive only when they reach a certain temperature
 - Should exhibit no reactivity below target temperature



Ex. 2030 (IPR2021-00186) ¶¶ 62-64; Ex. 1006 at 3154; POR (IPR2021-00186) at 54-55; POR (IPR2021-00186) at 18-19.

Different Methods of Making Different Nanoparticles

- **Semiconductor Nanorods**

- **Solution-Liquid-Solid (SLS)** ----- Banin
Ahrenkiel

- **Semiconductor Quantum Dots**

- **Hot-Injection Method** ----- Lucey
Bawendi
- **Bubbling Hydrogen Sulfide
Gas Through Aqueous
Solution of Cd Ions** ----- Braun
- **Heat-Up Method** ----- Zaban
Treadway

- **Semiconductor Cluster Compounds**

- **Recrystallization** ----- Herron
- **Chemical-Vapor Reactions** ----- Farneth
- **Chalcogenolysis and Condensation** ---- Ptatschek

- **Metal Nanoparticles**

- **Nucleation** ----- Yu

Different Methods Not Compatible

- The Board correctly noted, these methods are not all compatible.
 - “Patent Owner argues, and we agree based on the current record, that those disparate methods involve markedly different experimental conditions, reactants, and synthetic goals.” InstDec, 27.
- Petitioner provides no scientifically justified response to Board’s finding
 - Claim scope has no applicability to scientific justification for compatibility of processes
 - Patent owner’s facts are unrebutted

III. PO’S BACKGROUND SECTIONS ARE IRRELEVANT

While Petitioner disputes the “Overview of Quantum Dot Nanoparticles,” POR 3-7, Ex. 2030 ¶¶34-40, that section is irrelevant, unsubstantiated, and inconsistent with the Challenged Claims.

Additionally, PO’s discussion of different nanoparticle synthesis methods ignores the broad scope of the Challenged Claims. POR 8-23. The claims do not require nanoparticles with a specific shape, purity, quantity, or made by any specific method (so long as a MCC and seeded growth are present). *See* Ex. 1099, 99:18-101:22. Moreover, Petitioner did not argue that the synthesis method from one reference should be imported into another reference, rendering PO’s discussion irrelevant. Finally, this discussion is predicated on articles post-dating the alleged invention.

Petitioner Reply, 26.



The Obviousness Grounds - **Banin**

Banin in view of **Herron**, **Treadway**, **Bawendi**, **Farneth**, and/or **Braun**

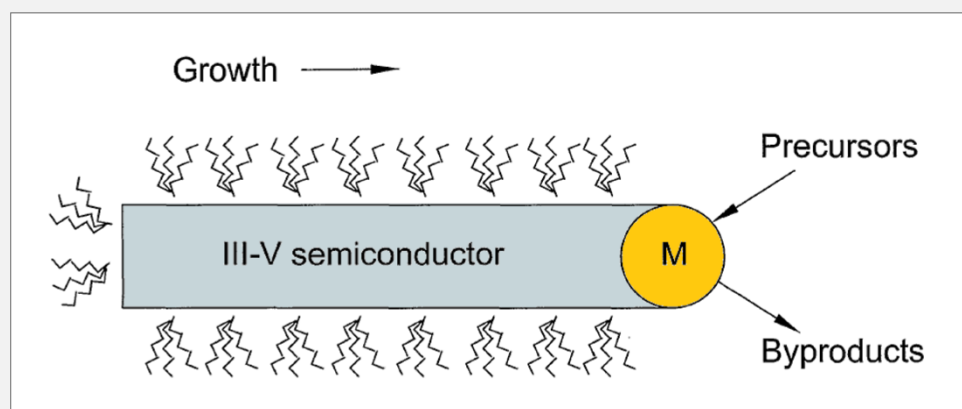
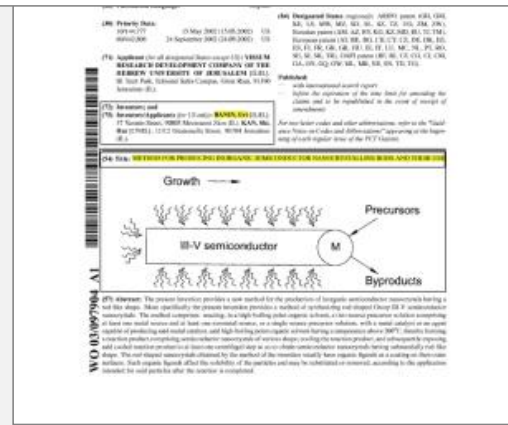
Banin – SLS Growth of Nanorods

Uses Solution-Liquid-Solid (“SLS”) method to grow nanorods and nanowires

(72) Inventors; and
 (75) Inventors/Applicants (for US only): **BANIN, Uri** [IL/IL];
 37 Yasmin Street, 90805 Mevasseret Zion (IL). **KAN, Shi,**
Hai [CN/IL]; 11/12 Guatemala Street, 96704 Jerusalem
 (IL).



(54) Title: METHOD FOR PRODUCING INORGANIC SEMICONDUCTOR NANOCRYSTALLINE RODS AND THEIR USE



Ex. 1005.

Banin – SLS Growth of Nanorods

- Banin discloses using the SLS method to create nanorods.
 - “The inset of **Fig. 10B** shows rods with gold particles on the end, demonstrating the SLS growth mechanism.”
- Banin’s SLS method uses “a metal nanoparticle as a *catalyst* for rod growth.”
 - The metal catalyst acts as a solvent and melts into droplets so that selected precursors can dissolve into the molten droplets and form a supersaturated solution that precipitates out as a quantum rod.
- Banin disclosed solvents are either TOP or TOPO at 200-360 °C.

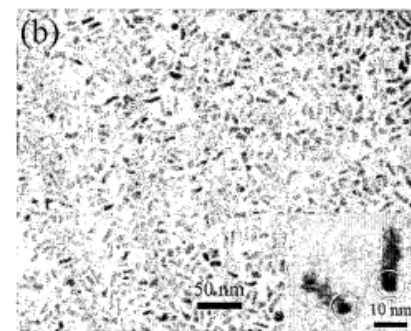
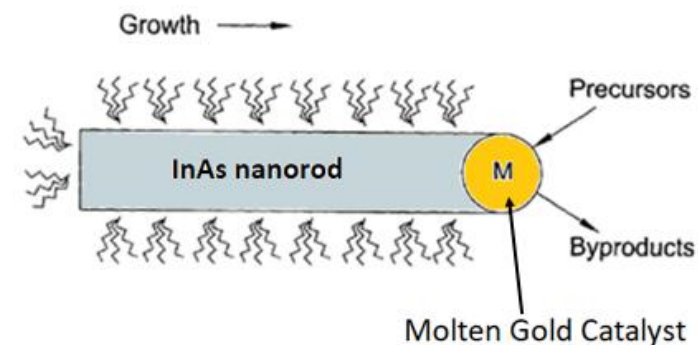


Fig. 10B



Ex. 1005, 22:25-30; POR (IPR2021-00186) at 12-15.

Grounds Relying on **Banin** in View of **Herron** – The Two Are Incompatible

Banin does not disclose use of 12-16 elements within an MCC; Petitioner attempts to use Herron to disclose a 12-16-based MCC in a subset of claims

Ground	8,524,365 Claims	35 U.S.C. §	Reference(s)
1	1, 7-12, 17, 22, 23	102	Banin
2	1, 7-12, 15-17, 22, 23	103(a)	Banin
3	2-6, 18-21	103(a)	Banin, Herron
4	13, 14	103(a)	Banin, Treadway
5	1-9, 17-23	103(a)	Zaban, Farneth, Yu
6	1, 2, 4, 7-12, 17, 18, 22, 23	103(a)	Lucey, Ahrenkiel
7	13-16	103(a)	Lucey, Ahrenkiel, Treadway

Ground	7,803,423 Claims	35 U.S.C. §	Reference(s)
1	1-3, 10, 11, 13, 22-24	102	Banin
2	1-6, 10-14, 21-25	103(a)	Banin
3	7-9	103(a)	Banin, Bawendi
4	1, 10-16, 21-24	103(a)	Zaban, Ptatschek
5	4-6, 25	103(a)	Zaban, Ptatschek, Yu
6	7-9	103(a)	Zaban, Ptatschek, Bawendi
7	1, 4, 11-16, 21, 25	103(a)	Lucey, Ahrenkiel

Ground	7,588,828 Claims	35 U.S.C. §	Reference(s)
1	1, 3-6, 8-14	103(a)	Banin, Herron
2	1, 3	103(a)	Banin, Farneth
3	1, 3-6, 14	103(a)	Zaban, Farneth, Yu
4	8-13	103(a)	Zaban, Farneth, Yu, Braun

Ground	7,867,557 Claims	35 U.S.C. §	Reference(s)
1	1-6, 16, 17	103(a)	Banin, Braun
2	1, 16, 17	103(a)	Zaban, Ptatschek, Braun
3	4-6	103(a)	Zaban, Ptatschek, Braun, Yu
4	1, 4, 16, 17	103(a)	Lucey, Ahrenkiel, Braun

Herron

Characterization of a crystalline fragment of bulk cubic CdS

– Using the solvent DMF the cluster $Cd_{32}(SC_6H_5)_{36} \cdot DMF_4$ precipitated out and was analyzed



REPORTS

Crystal Structure and Optical Properties of $Cd_{32}S_{14}(SC_6H_5)_{36} \cdot DMF_4$, a Cluster with a 15 Angstrom CdS Core

N. Herron,* J. C. Calabrese, W. E. Farneth, Y. Wang

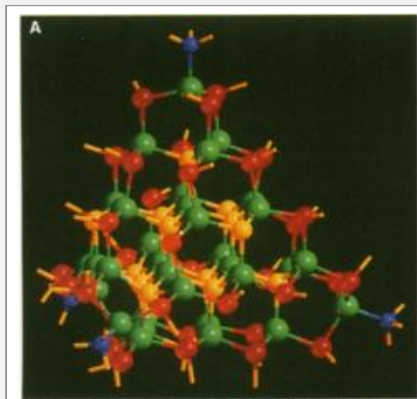


Fig. 1. (A) Crystal structure of $Cd_{32}S_{14}(SC_6H_5)_{36} \cdot DMF_4$ core.

Finally, the solubility of this new crystalline cluster may be used in the production of polymer films, such as polyvinylcarbazole, doped with the CdS cluster simply by spin-coating from a pyridine solution. Such films have been found to exhibit good photoconductive properties (10), which points to a real utility for such soluble semiconductor clusters in the preparation of materials that go into electronic devices made for this and other thin-film applications.

Ex. 1016.

No Motivation to Combine **Banin** and **Herron** and No Expectation of Success

Banin and Herron Are Incompatible – Key Points

- Petitioner fails to address key considerations for choosing a metal catalyst for SLS:
 - (1) melting point,
 - (2) solvating ability, and
 - (3) reactivity
- **Banin’s SLS process requires a metal catalyst – Herron discloses a semiconductor**
 - Petitioner never address question of why replace metal with semiconductor
- **No evidence exists that anyone has ever attempted to use semiconductor material to replace metal in an SLS reaction**

Pet. (IPR2021-00186) at 28; Petitioner’s Reply (IPR2021-00186) at 11-12; POSR (IPR2021-00186) at 15-16.

Petitioner Does Not Consider Requirements of **Banin's** Metal Catalyst

- Banin repeatedly emphasizes demand for use of a metal catalyst which
 - Acts as a solvent
 - Dissolves precursors into supersaturated solution
 - Does not react with precursors
- Petitioner proposes Herron's cadmium sulfide (CdS) semiconductor as a replacement for the Banin gold catalyst
- Petitioner gives no explanation at all for how a semiconductor would possible work in SLS
 - Provides no support for whether melting point for semiconductor material would be incompatible with the SLS process used by Banin
 - Provides no explanation for why semiconductor material would act as a solvent
 - Provides no support to guarantee that the snon-reactivity

Petitioner's Reply (IPR2021-00186) at 9; POR (IPR2021-00183) at 47; Ex. 1016 (Herron) at 1426.

Banin in view of Herron – Banin’s Metal Catalyst Must Melt

No motivation to replace Banin’s metal catalyst with a semiconductor cluster

- Petitioner contends that a “POSITA would have been motivated to replace Banin’s Au-based MCC with Herron’s Cd-based MCC.”
- Petition does not consider the melting point of Herron’s semiconductor cluster.
- Petitioner’s expert did not consider the melting point of any metal or semiconductor.
 - “Q. And in your opinion, you didn’t consider the melting temperatures of those molecular cluster compounds. Right? A. ***It wasn’t something that I particularly paid that much attention to, no.***”
 - Admitted to having no opinion related to melting points of indium, cadmium, bismuth, cadmium sulfide, cadmium selenide, or gold.

Pet. (IPR2021-00183) at 34, 36, 38; POR (IPR2021-00183) at 47-48; POSR (IPR2021-00183) at 16-17; Ex. 1005, 6:9-16, 14:3-9; Ex. 2031, 106: 7-12; 100:19–101:5.

Banin in view of Herron – Petitioner Ignores Key Considerations

No motivation to replace Banin's metal catalyst with a semiconductor cluster

- Petitioner also fails to address key considerations for choosing a metal catalyst for SLS, including
 - (1) melting point, (2) solvating ability, and (3) reactivity
- Petitioner's expert, Dr. Green, offers no opinion but Dr. Cossairt testified that a POSITA would have recognized that metals with lower melting temperatures were preferable for SLS.
- **Ahrenkiel** reviewed Banin and expressly noted that lower melting temperatures were desirable:

After this paper was submitted for publication, a paper by Banin et al. appeared on the formation of InAs quantum rods ... using gold particles as the catalyst. ... Although this method can also be applied to the synthesis of InP quantum rods, *it requires extremely small gold particles* that are about 20 Å in diameter *in order to lower the melting point of the gold particles to about 360 °C and initiate catalytic action*. Indium has a lower melting point than gold, and the method described in this paper has the potential for greater flexibility for the synthesis of quantum rods with different diameters.

POR (IPR2021-00183) at 48-49; POSR (IPR2021-00183) at 17-18; Ex. 2030 (IPR2021-00186) ¶¶ 132-43; Ex. 1012, 834.

Banin in view of Herron – Banin Requires a Metal Catalyst

No motivation to replace Banin's metal catalyst with a semiconductor cluster

- Petitioner contends that a “POSITA would have been motivated to replace Banin's Au-based MCC with Herron's Cd-based MCC.”
- As Banin acknowledges, in SLS the catalysts ***must be metal***.
 - “The SLS mechanism ***requires*** the addition or in-situ formation of a ***metal*** nanoparticle as a catalyst for rod growth.”
 - The ***metal*** catalyst can be made, for example, of a transition ***metal***, ... alloys of such ***metals...*** or core/shell layered ***metal*** particle
- At her deposition, Dr. Cossairt testified “A. ... I spoke with Dr. Buhro, who is the primary author on the Yu paper and who is the primary author on the Wang reviews that I cite in my expert declaration, and he confirmed that suspicion; that he doesn't know of any examples where semiconductor particles have been used as catalysts in SLS growth.”

Pet. at 34, 36, 38 (IPR2021-00183); POR (IPR2021-00183) at 47-48; POSR (IPR2021-00183) at 16-18; Ex. 1005, 6:9-16, 14:3-9; Ex. 2031, 106: 7-12; 100:19–101:5; Ex. 1099, 150:15-151:8; Ex. 2030 (IPR2021-00186) ¶¶ 139-46.

Banin in view of Herron – Banin Requires a Metal Catalyst

No reasonable expectation of success

- Not “a matter of routine optimization to substitute” Banin’s metal catalyst with Herron’s semiconductor.
 - No support for that vague theory.
 - Petitioner failed to meet its burden to show a reasonable expectation of success.
- The proposal to use a semiconductor cluster in SLS has never been accomplished!
- Notwithstanding its relevancy positions, Petitioner offered a new argument in its Reply
 - Petitioner contends a semiconductor could be used as a catalyst in VLS (the Pan article published after the priority date)
 - No mention of “solution-liquid-solid” or “vapor-liquid-solid” in the Petitioner or accompanying expert declaration.
- Pan supports Patent Owner’s position.
 - Pan notes that “...***this is the first example of a non-metal catalyzing nanowire growth.***”
 - Pan demonstrates the high temperatures required for VLS are incompatible with Banin’s SLS.
 - Pan: synthesis temperatures up to 650 °C.
 - Banin: synthesis below 360 °C due to solvent boiling point.
- Reliance on Pan’s VLS demonstrates Petitioner found no relevant SLS teachings!

Pet. (IPR2021-00186) at 34-35; Pet. Reply at 34; POSR (IPR2021-00183) at 18-20;
Ex. 1098, 275; Ex. 1005, 16:12-13, 20:20; Ex. 2030 (IPR2021-00186) ¶¶ 139-46.

Banin in view of Herron – Petitioner’s Arguments Fail

No motivation to replace Banin’s metal catalyst with a semiconductor cluster

- Yu and Wang both counsel using low-melting point metals
 - Wang: “The low melting metals best meeting these [three] criteria seemed to be In, Bi, and Sn”
 - Yu: “[W]e...have therefore sought monodisperse catalyst nanoparticles of low-melting metals such as Bi, Sn, and In having similar, selected sizes.”
- Petitioner contends the “clusters” disclosed by Banin and Herron are interchangeable due to Banin’s mention of possibly using Cd-based metal alloys.
 - Metal alloys are not semiconductors.
- Petitioner also cites purported cost savings due to cost of gold in Banin.
 - No consideration of the cost of Herron’s process.
- **Both uncorroborated and entitled to no weight!**
- Improper hindsight is the real reason behind the disparate combination of Banin and Herron.

Pet., 34; POR (IPR2021-00183) at 50; POSR (IPR2021-00183) at 19-20; Ex. 2030 (IPR2021-00186) ¶¶ 139-48; Ex. 2008, 7515; Ex. 1010 at 9198.

Petitioner's **Banin** in view of **Herron** Grounds Fail

Ground	8,524,365 Claims	35 U.S.C. §	Reference(s)
1	1, 7-12, 17, 22, 23	102	Banin
2	1, 7-12, 15-17, 22, 23	103(a)	Banin
3	2-6, 18-21	103(a)	Banin, Herron
4	13, 14	103(a)	Banin, Treadway
5	1-9, 17-23	103(a)	Zaban, Farneth, Yu
6	1, 2, 4, 7-12, 17, 18, 22, 23	103(a)	Lucey, Ahrenkiel
7	13-16	103(a)	Lucey, Ahrenkiel, Treadway

Ground	7,803,423 Claims	35 U.S.C. §	Reference(s)
1	1-3, 10, 11, 13, 22-24	102	Banin
2	1-6, 10-14, 21-25	103(a)	Banin
3	7-9	103(a)	Banin, Bawendi
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5	4-6, 25	103(a)	Zaban, Ptatschek, Yu
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Ground	7,588,828 Claims	35 U.S.C. §	Reference(s)
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3	4-6	103(a)	Zaban, Ptatschek, Braun, Yu
4	1, 4, 16, 17	103(a)	Lucey, Ahrenkiel, Braun

Pet. (IPR2021-00186) at 28; Petitioner's Reply at 11-12 (IPR2021-00186); POSR (IPR2021-00186) at 15-16.

Banin and Farneth Are Incompatible

Banin does not disclose a group 12-16 MCC, and Herron does not disclose a MCC with zinc ions, so Petitioner attempts to combine Farneth.

Ground	8,524,365 Claims	35 U.S.C. §	Reference(s)
1	1, 7-12, 17, 22, 23	102	Banin
2	1, 7-12, 15-17, 22, 23	103(a)	Banin
3	2-6, 18-21	103(a)	Banin, Herron
4	13, 14	103(a)	Banin, Treadway
5	1-9, 17-23	103(a)	Zaban, Farneth, Yu
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7	13-16	103(a)	Lucey, Ahrenkiel, Treadway

Ground	7,803,423 Claims	35 U.S.C. §	Reference(s)
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2	1-6, 10-14, 21-25	103(a)	Banin
3	7-9	103(a)	Banin, Bawendi
4	1, 10-16, 21-24	103(a)	Zaban, Ptatschek
5	4-6, 25	103(a)	Zaban, Ptatschek, Yu
6	7-9	103(a)	Zaban, Ptatschek, Bawendi
7	1, 4, 11-16, 21, 25	103(a)	Lucey, Ahrenkiel

Ground	7,588,828 Claims	35 U.S.C. §	Reference(s)
1	1, 3-6, 8-14	103(a)	Banin, Herron
2	1, 3	103(a)	Banin, Farneth
3	1, 3-6, 14	103(a)	Zaban, Farneth, Yu
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Ground	7,867,557 Claims	35 U.S.C. §	Reference(s)
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2	1, 16, 17	103(a)	Zaban, Ptatschek, Braun
3	4-6	103(a)	Zaban, Ptatschek, Braun, Yu
4	1, 4, 16, 17	103(a)	Lucey, Ahrenkiel, Braun

Farneth

Solid-state conversion of a series of group II/VI precursors to bulk metal sulfide

– Characterized an intermediate material $Cd_{10}S_{16}P_{12}$ with structural integrity and optical properties

Bulk Semiconductors from Molecular Solids: A Mechanistic Investigation

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Bulk Semiconductors from Molecular Solids: A Mechanistic Investigation

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Received December 12, 1991. Revised Manuscript Received April 27, 1992

We report on the mechanism of the solid-state conversion of a series of II/VI precursors of general formula $(R_4N^+)_2[S_2M_2(SPh)_2]^{2-}$ ($R = Me, Et; M = Cd, Zn$) to the bulk metal sulfide structure. On heating in vacuum or inert atmosphere, we find that the solid-state conversion proceeds in two discrete reaction steps. The first step, $\sim 200^\circ C$, can be characterized as a nucleophilic substitution or elimination initiated by the attack of a fragment of the anion cluster on the tetraalkylammonium counterion. The intermediate solid produced in this step consists of charge neutral $M_2S_2Ph_{12}$ clusters that retain the primary cluster size present in the starting material but appear to aggregate to varying degrees in the solid or solution. The intermediate cluster has very different chemical and physical properties from the starting material and, therefore, suggests some new possible routes to the preparation of bulk II/VI semiconductor films and powders from molecular precursors.

Introduction

The preparation of crystalline solids via the consolidation of molecular precursors is an increasingly important branch of solid-state chemistry.¹ II/VI compound semiconductors have been a favorite subject for such studies because of interest in the evolution of their electronic properties as the consolidation process proceeds. Consequently, a wide variety of molecular precursor approaches to these materials has been described.^{2,3} Nevertheless,

whether the goal is crystalline thin films via MOCVD or microcrystalline powders via solution- or solid-state reactions, detailed information on the mechanisms of the transformation from molecules to bulk II/VI solids is limited. In this paper we report on the mechanism of the solid-state conversion of a particular series of II/VI precursors of general formula $(R_4N^+)_2[S_2M_2(SPh)_2]^{2-}$ to the bulk metal sulfide structure. On heating, in vacuum or

(3). Characterization of the Intermediate $Cd_{10}S_{16}Ph_{12}$. We have isolated and characterized the intermediate $Cd_{10}S_{16}Ph_{12}$ material (II).

Although the structural integrity of the clusters appears to be maintained in the solid, new electronic interactions between the clusters do exist as revealed by their optical properties. Figure 8 shows the luminescence and excitation spectra of powders of II. The luminescence spectrum is

(1) Rao, C. N. R.; Gopalakrishnan, J. *New Directions in Solid State Chemistry*; Cambridge University Press: Cambridge, UK, 1986; Chapter 3.
(2) Spachel, L.; Adelman, M. J. *Acc. Chem. Soc.* 1990, 11, 209 and references therein.

(3) (a) Brennan, J. G.; Siegrist, T.; Carroll, P. J.; Szymanski, S. M.; Heydem, P.; Brus, L. E.; Stagerwald, M. L. *Chem. Mater.* 1990, 2, 402 and references therein. (b) Brennan, M. G.; Karas, A. R.; Stagerwald, M. L.; Brus, L. E. *J. Chem. Phys.* 1988, 81, 7285.
(4) Oshida, K.; Yamamoto, T. *J. Chem. Soc., Chem. Commun.* 1987, 1117.
(5) Oshida, K.; Yamamoto, T. *J. Chem. Soc.* 1991, 30, 3325.

Farneth Fails for the Same Reasons as Herron

- **Petitioner fails to address key considerations for choosing a metal catalyst for SLS:**
 - (1) melting point,
 - (2) solvating ability, and
 - (3) reactivity
- **Banin's SLS process requires a metal catalyst – Farneth discloses a semiconductor**
 - Farneth teaches a solid-state method to make its $\text{Cd}_{10}\text{S}_6\text{Ph}_{12}$ and $\text{Zn}_{10}\text{S}_6\text{Ph}_{12}$ semiconductors.
 - Petitioner never address question of why replace metal with semiconductor
 - No one has every used a semiconductor in an SLS reaction apparently

Pet. (IPR2021-00186) at 28; Petitioner's Reply at 11-12 (IPR2021-00186);
POSR (IPR2021-00186) at 15-16; Ex. 2030 (IPR2021-00183) ¶¶ 141-53.

Banin in View of Farneth – Fails for Same Reason as Herron

No motivation to combine or reasonable expectation of success

- Dr. Green failed to explain why a POSITA would have considered substituting Farneth’s **solid** semiconductor clusters for Banin’s **liquid** metal catalyst
 - Farneth suggests that the disclosed semiconductor clusters already possess nanoparticle characteristics.
 - No reason to use as a catalyst for SLS.
 - The disclosures of Banin and Farneth push them apart rather than together.
- Farneth’s cluster no more likely to succeed as a metal catalyst in Banin’s SLS method than Herron.
 - Dr. Green’s testifies that Farneth’s cluster (like Herron’s) would maintain its structural integrity => won’t melt!
 - Green did not consider the melting points of Farneth’s semiconductors.
 - No evidence Farneth discloses a “slow-melting metallic nanoparticle” than can easily exist in a “molten, liquid form” and catalyze the decomposition of metallo-organic precursors at the solution–liquid interface, as Banin’s requires.
- **Claims 1, 3 of the ’828 patent are not rendered obvious by Banin + Farneth.**

POR (IPR2021-00183) at 54-58; Ex. 1016, 1426; Ex. 1009, 916, 921-922; Ex. 2030 (IPR2021-00183) ¶¶ 141-53; Ex. 1002 ¶153; Ex. 2031, 106:7-12.

Banin and Farneth are incompatible

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3	4-6	103(a)	Zaban, Ptatschek, Braun, Yu
4	1, 4, 16, 17	103(a)	Lucey, Ahrenkiel, Braun

Bawendi is Added to Get the “Monitoring” Missing from Banin

- Banin does not disclose a “a) monitoring an average size of the nanoparticles being grown; and b) terminating nanoparticle growth when the average nanoparticle size reaches a predetermined value”
 - Claims 7-9 of the '423 patent

Pet. (IPR2021-00186) at 28; Petitioner’s Reply at 11-12 (IPR2021-00186); POSR (IPR2021-00186) at 15-16.

No Motivation to Combine **Banin** and **Bawendi** and No Expectation of Success

- **Banin's SLS** method and **Bawendi's hot-injection** method are not compatible,
 - As the Board noted in the IPR2021-00186 institution decision, “[t]hese disparate methods involve markedly different experimental conditions, reactants, and synthetic goals” (referring to Ahrenkiel’s SLS method and the Lucey’s hot-injection method)
- **Bawendi monitors the growing diameter of a quantum dot, but Banin’s SLS produce quantum rods having a predetermined diameter.**

Petitioner’s Reply at 9 (IPR2021-00184); POSR at 17-18; POR (IPR2021-00184) at 46-48; Ex. 1014, 1:26-29, 4:59-64; Ex. 2030 (IPR2021-00184) ¶¶ 132-135; InstDec (IPR2021-00186) at 17, 27.

Bawendi

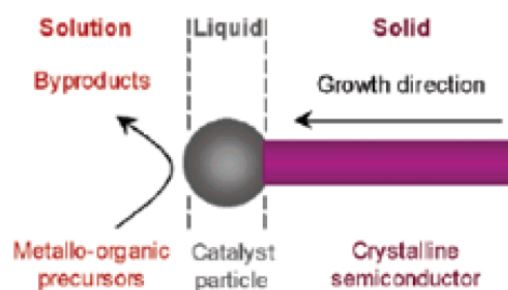
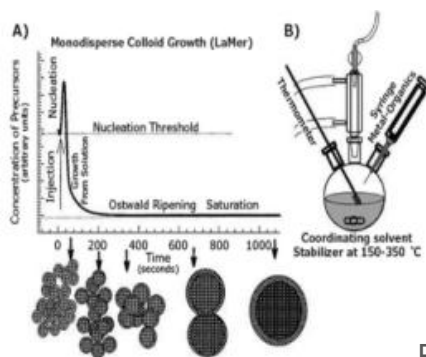
Preparation of nanocrystallites (quantum dots) using hot injection

- Injection produces a nucleus that can be grown in a controlled manner



Claims 7-9 of '423 patent Not Obvious by **Banin** in view of **Bawendi**

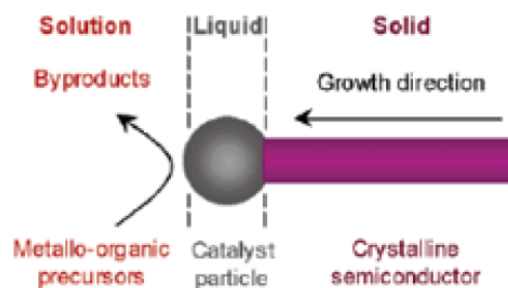
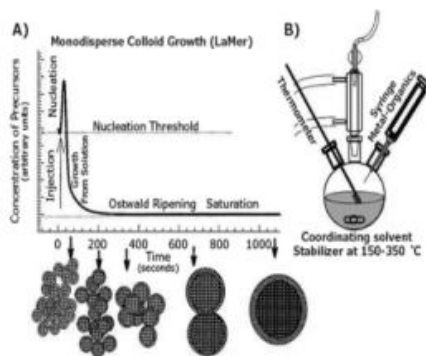
- **Claims 7-9 of '423 patent require monitoring the average size of the nanoparticles being grown.**
- **Bawendi** cold-quenches but employs a hot-injection method.
 - “organometallic reagents, such as dimethyl cadmium, injected into a hot, coordinating solvent.”
 - Has nothing to do with SLS
- As the Board noted in the IPR2021-00186 institution decision, “[t]hese disparate methods involve markedly different experimental conditions, reactants, and synthetic goals.”



Petitioner’s Reply at 9 (IPR2021-00186); POSR at 17-18 POR (IPR2021-00183) at 46-48; Ex. 1014, 1:26-29, 4:59-64; Ex. 2030 (IPR2021-00184) ¶¶132-135; InstDec (IPR2021-00186) at 17, 27.

Claims 7-9 of '423 patent Not Obvious by **Banin** in view of **Bawendi**

- **Bawendi** monitors the changing *diameters* of the nanocrystallites by “monitoring the absorption line widths of the particles [and] stopping growth at a particular nanocrystallite average diameter...”
- Banin’s particle diameters never change and are dictated by the metal catalysts, not the reaction duration!
 - no need to monitor the nanoparticle’s diameter
 - no motivation to combine
- **Banin/Bawendi cannot render claims 7-9 of '423 patent obvious**



Petitioner’s Reply at 9 (IPR2021-00186); POSR at 17-18 POR (IPR2021-00183) at 46-48; Ex. 1014, 1:26-29, 4:59-64; Ex. 2030 ¶¶ 132-135; InstDec (IPR2021-00186) at 17, 27.



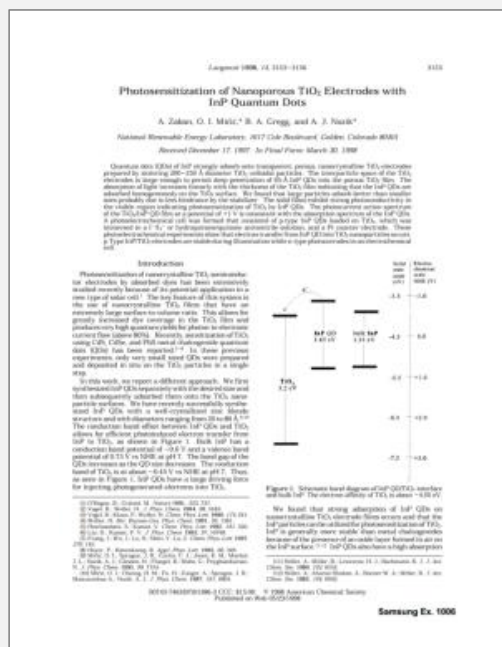
The Obviousness Grounds - Zaban

Zaban in view of Farneth, Yu, Ptatschek, Bawendi, and/or Braun

Zaban

Growth of colloidal InP quantum dots

- Three-day heat-up method according to an established preparation procedure (Ref 14)



Photosensitization of Nanoporous TiO₂ Electrodes with InP Quantum Dots

A. Zaban, O. I. Mičić,* B. A. Gregg, and A. J. Nozik*

Experimental Section

Synthesis of Colloidal InP QDs. Colloidal InP QDs were synthesized by colloidal chemistry methods using indium oxalate and tris(trimethylsilyl)phosphine (P(SiMe₃)₃) as starting reactants; the reactants are heated for 3 days at 260–280 °C in the presence of trioctylphosphine oxide (TOPO) and trioctylphosphine (TOP). Details of the preparation are given in ref 14. During

Ex. 1006.

Zaban – Role of the Zinc Acetate as a Dopant

- A dopant is a small impurity intentionally added to change the core semiconductor's electrical properties.
- Chemistry is specific; reagents, precursors, catalysts and other materials must be precisely chosen to obtain the specific result sought. Especially true with dopants
- Changing the amount of dopant changes the properties of the resultant quantum dot.

Ex. 2030 ¶¶ 160-164; Ex. 1049, 42-44; Ex. 1070; POR at 54-55; POSR at 22.

Zaban – Role of the Zinc Acetate as a Dopant

- “II-VI semiconductor nanocrystals doped with transition metals has proved to be *particularly difficult*”
- “specially designed precursors ... appear to be *necessary* to successfully incorporate low levels of [the transition metal]”).
- Zaban requires one zinc atom per quantum dot to “achieve the optimum zinc concentration and resulting QD size”

Ex. 2030 ¶¶ 160-164; Ex. 1049, 42-44; Ex. 1070; POR at 54-55; POSR at 22-23.

Zaban – Does Not Disclose Many Claim Elements

- Petitioner does not allege that Zaban discloses “a molecular cluster compound,” so adds **Farneth** or **Ptatschek**
- Petitioner does not allege that Zaban discloses “conditions permitting seeding an growth,” so adds **Yu**.
- Petitioner does not allege Zaban discloses a first or second layer provided on the nanoparticle core, so adds **Braun**.
- Petitioner does not allege that Zaban discloses “monitoring the average size of the nanoparticles being grown,” so adds **Bawendi**

Ex. 2030 ¶¶ 160-164; Ex. 1049, 42-44; Ex. 1070; POR at 54-55; POSR at 22.

Zaban, Farneth, Yu, Braun, Bawendi = 4 different methods

Ground	8,524,365 Claims	35 U.S.C. §	Reference(s)
1	1, 7-12, 17, 22, 23	102	Banin
2	1, 7-12, 15-17, 22, 23	103(a)	Banin
3	2-6, 18-21	103(a)	Banin, Herron
4	13, 14	103(a)	Banin, Treadway
5	1-9, 17-23	103(a)	Zaban, Farneth, Yu
6	1, 2, 4, 7-12, 17, 18, 22, 23	103(a)	Lucey, Ahrenkiel
7	13-16	103(a)	Lucey, Ahrenkiel, Treadway

Ground	7,803,423 Claims	35 U.S.C. §	Reference(s)
1	1-3, 10, 11, 13, 22-24	102	Banin
2	1-6, 10-14, 21-25	103(a)	Banin
3	7-9	103(a)	Banin, Bawendi
4	1, 10-16, 21-24	103(a)	Zaban, Ptatschek
5	4-6, 25	103(a)	Zaban, Ptatschek, Yu
6	7-9	103(a)	Zaban, Ptatschek, Bawendi
7	1, 4, 11-16, 21, 25	103(a)	Lucey, Ahrenkiel

Ground	7,588,828 Claims	35 U.S.C. §	Reference(s)
1	1, 3-6, 8-14	103(a)	Banin, Herron
2	1, 3	103(a)	Banin, Farneth
3	1, 3-6, 14	103(a)	Zaban, Farneth, Yu
4	8-13	103(a)	Zaban, Farneth, Yu, Braun

Ground	7,867,557 Claims	35 U.S.C. §	Reference(s)
1	1-6, 16, 17	103(a)	Banin, Braun
2	1, 16, 17	103(a)	Zaban, Ptatschek, Braun
3	4-6	103(a)	Zaban, Ptatschek, Braun, Yu
4	1, 4, 16, 17	103(a)	Lucey, Ahrenkiel, Braun

Zaban in View of Farneth (plus Yu, Braun, and/or Bawendi)

- Petitioner contends motivation to replace zinc acetate dopant with either ($\text{Cd}_{10}\text{S}_6\text{Ph}_{12}$ or $\text{Zn}_{10}\text{S}_6\text{Ph}_{12}$)
- Board already correctly rejected this combination
 - “Petitioner does not show sufficiently that either of Farneth’s cluster compounds ($\text{Cd}_{10}\text{S}_6\text{Ph}_{12}$ or $\text{Zn}_{10}\text{S}_6\text{Ph}_{12}$) would have been recognized as a known interchangeable alternative serving any function in common with the zinc acetate in Zaban’s process.”
 - “Petitioner articulates no clear reason why an ordinary skilled artisan would have viewed Farneth’s zinc-based cluster (which contributes many times the amount of zinc compared to zinc acetate) as interchangeable with the zinc acetate dopant in Zaban’s process. Petitioner’s arguments pertaining to Farneth’s non-zinc-based cluster (that is, the cadmium-based cluster) are even less clear.”

InstDec. (IPR2021-00186) at 25-26.

Zaban in View of Farneth (plus Yu, Braun, and/or Bawendi)

- Petitioner fails to address issues identified by the Board, but instead attacks the POR
- Petitioner suggested replacing Zn acetate with a Cd based cluster shows they did not comprehend the role of zinc.
- Dilution does nothing – still end up with 10 zincs in whichever QD incorporates the cluster.

POSR at 22-23.

Braun

Creates CdS semiconductor cores by bubbling H₂S gas through an aqueous solution of cadmium ions.

- Braun uses an aqueous synthesis method

Variation of the Thickness and Number of Wells in the CdS/HgS/CdS Quantum Dot Quantum Well System†

Markus Braun, Clemens Burda, and Mostafa A. El-Sayed*

In step A, CdS nanoparticles are prepared under argon atmosphere by injecting 0.6 mL H₂S gas into a 250 mL flask containing 100 mL 2 × 10⁻⁴ M aqueous solution of Cd²⁺ ions and hexametaphosphate as stabilizer while stirring at an initial pH of 7.9. The high pH value leads to a fast ionization of the H₂S gas into H⁺ and S⁻ ions and thereby initiates a fast nucleation process of many small CdS crystallites. As S⁻

Ex. 1013.

Zaban in View of Farneth (Plus Yu, and/or Braun)

Ground	8,524,365 Claims	35 U.S.C. §	Reference(s)
1	1, 7-12, 17, 22, 23	102	Banin
2	1, 7-12, 15-17, 22, 23	103(a)	Banin
3	2-6, 18-21	103(a)	Banin, Herron
4	13, 14	103(a)	Banin, Treadway
5	1-9, 17-23	103(a)	Zaban, Farneth, Yu
6	1, 2, 4, 7-12, 17, 18, 22, 23	103(a)	Lucey, Ahrenkiel
7	13-16	103(a)	Lucey, Ahrenkiel, Treadway

Ground	7,803,423 Claims	35 U.S.C. §	Reference(s)
1	1-3, 10, 11, 13, 22-24	102	Banin
2	1-6, 10-14, 21-25	103(a)	Banin
3	7-9	103(a)	Banin, Bawendi
4	1, 10-16, 21-24	103(a)	Zaban, Ptatschek
5	4-6, 25	103(a)	Zaban, Ptatschek, Yu
6	7-9	103(a)	Zaban, Ptatschek, Bawendi
7	1, 4, 11-16, 21, 25	103(a)	Lucey, Ahrenkiel

Ground	7,588,828 Claims	35 U.S.C. §	Reference(s)
1	1, 3-6, 8-14	103(a)	Banin, Herron
2	1, 3	103(a)	Banin, Farneth
3	1, 3-6, 14	103(a)	Zaban, Farneth, Yu
4	8-13	103(a)	Zaban, Farneth, Yu, Braun

Ground	7,867,557 Claims	35 U.S.C. §	Reference(s)
1	1-6, 16, 17	103(a)	Banin, Braun
2	1, 16, 17	103(a)	Zaban, Ptatschek, Braun
3	4-6	103(a)	Zaban, Ptatschek, Braun, Yu
4	1, 4, 16, 17	103(a)	Lucey, Ahrenkiel, Braun

Zaban in View of Ptatschek (plus Yu, Bawendi, and/or Braun)

Ground	8,524,365 Claims	35 U.S.C. §	Reference(s)
1	1, 7-12, 17, 22, 23	102	Banin
2	1, 7-12, 15-17, 22, 23	103(a)	Banin
3	2-6, 18-21	103(a)	Banin, Herron
4	13, 14	103(a)	Banin, Treadway
5	1-9, 17-23	103(a)	Zaban, Farneth, Yu
6	1, 2, 4, 7-12, 17, 18, 22, 23	103(a)	Lucey, Ahrenkiel
7	13-16	103(a)	Lucey, Ahrenkiel, Treadway

Ground	7,803,423 Claims	35 U.S.C. §	Reference(s)
1	1-3, 10, 11, 13, 22-24	102	Banin
2	1-6, 10-14, 21-25	103(a)	Banin
3	7-9	103(a)	Banin, Bawendi
4	1, 10-16, 21-24	103(a)	Zaban, Ptatschek
5	4-6, 25	103(a)	Zaban, Ptatschek, Yu
6	7-9	103(a)	Zaban, Ptatschek, Bawendi
7	1, 4, 11-16, 21, 25	103(a)	Lucey, Ahrenkiel

Ground	7,588,828 Claims	35 U.S.C. §	Reference(s)
1	1, 3-6, 8-14	103(a)	Banin, Herron
2	1, 3	103(a)	Banin, Farneth
3	1, 3-6, 14	103(a)	Zaban, Farneth, Yu
4	8-13	103(a)	Zaban, Farneth, Yu, Braun

Ground	7,867,557 Claims	35 U.S.C. §	Reference(s)
1	1-6, 16, 17	103(a)	Banin, Braun
2	1, 16, 17	103(a)	Zaban, Ptatschek, Braun
3	4-6	103(a)	Zaban, Ptatschek, Braun, Yu
4	1, 4, 16, 17	103(a)	Lucey, Ahrenkiel, Braun

Zaban in View of Ptatschek, Yu, Bawendi, and Braun

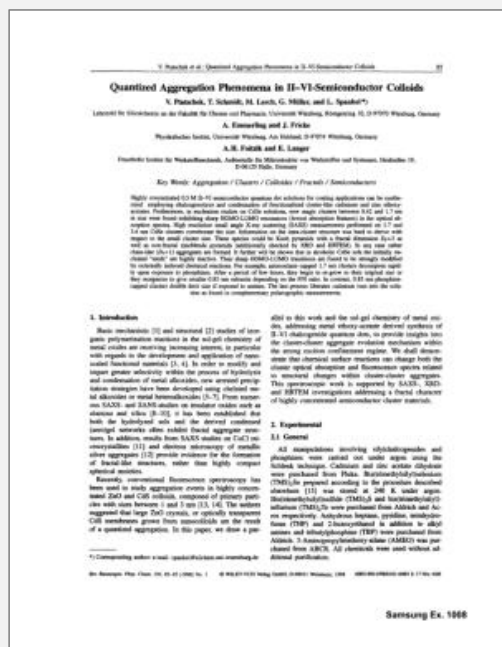
- In Nos. 2021-00184 and -00185 Petitioner contends there would be a motivation to replace Zaban's Zn acetate dopant with Ptatschek's $\text{Zn}_{10}\text{O}_4(\text{OAc})_{12}$ precursor.
- Board already rejected this combination
 - “Petitioner does not address adequately the disparate conditions, reagents, and reaction products that distinguish Zaban's method from Ptatschek's. Pet. 38–40, 47–48. On this record, we agree with Patent Owner that, given Zaban is synthesizing indium phosphide nanoparticles, there would have been little need to look for zinc-based precursors to improve Zaban's method.”

InstDec. (IPR2021-00185) at 26 (citations removed).

Ptatschek

Teaches ways to make **group II-VI** quantum dots (e.g., CdS)

- Uses a precursor with 10 zinc atoms to make group II-VI (zinc selenide and zinc sulfide) quantum dots



Quantized Aggregation Phenomena in II-VI-Semiconductor Colloids

V. Ptatschek, T. Schmidt, M. Lerch, G. Müller, and L. Spanhel*

2.2 Synthesis of Cd- and Zn-Precursors

16 g (60 mmol) cadmium acetate dihydrate (or zinc acetate dihydrate) was dehydrated using acetic anhydride. This starting procedure resulted in the production of crystalline powders composed of well known pyramidal $[Zn_4O(OAc)_6]$ -clusters (OAc=acetate). These powders were suspended in 600 ml ethanol and refluxed for 3 h whereupon the solvent was removed under reduced pressure at ambient temperature. The FTIR spectra of the resulting crystalline powders exhibit stretching vibrations of strongly associated OH-groups (at 3200 cm^{-1}), asymmetric and symmetric stretching vibrations of acetate bidentate ligands (at 1554 and 1456 cm^{-1}) as well as metal-oxygen vibrations between 500 and 400 cm^{-1} . The chemical elemental analysis MS-, XRD- and NMR-investigations revealed the presence of $[Zn_{10}O_4(OAc)_{12}]$ -clusters associated with 7 ethanol ligands and 1 water molecule.

Ex. 1008.

Zaban in View of Ptatschek, Yu, Bawendi, and Braun

- In Nos. 2021-00184 and -00185 Petitioner contends there would be a motivation to replace Zaban's Zn acetate dopant with Ptatschek's $\text{Zn}_{10}\text{O}_4(\text{OAc})_{12}$ precursor.
- Board already rejected this combination
 - Petitioner ignores the “orders of magnitude” increase in zinc content that would attend the proposed substitution. *Id.*
 - “Nor does Petitioner address how the proposed substitution would affect the final nanoparticle product grown according to the modified process of Zaban, much less show adequately that Ptatschek's $\text{Zn}_{10}\text{O}_4(\text{OAc})_{12}$ -clusters would maintain “the quality of” Zaban's final nanoparticle product.” *Id.* at 27 (citations removed).
 - “Petitioner fails to show sufficiently that an ordinarily skilled artisan would have been led to replace the zinc acetate in Zaban's process with the $\text{Zn}_{10}\text{O}_4(\text{OAc})_{12}$ -clusters disclosed in Ptatschek.”

InstDec. (IPR2021-00185) at 27.

Zaban in View of Ptatschek, Yu, Bawendi, and Braun

- The Petition completely failed to address the role of zinc acetate in Zaban. This failure is dispositive of the Zaban-based grounds. See *Intelligent Bio-Sys., Inc. v. Illumina Cambridge Ltd.*, 821 F.3d 1359, 1369 (Fed. Cir. 2016) (“**the initial petition** [must] identify ‘with particularity’ the ‘evidence that supports the grounds for the challenge to each claim.’” (quoting 35 U.S.C. § 312(a)(3))).
- *Samsung Elecs. Co. v. Elm 3DS Innovations, LLC* (“*Elm 3DS*”), 925 F.3d 1373, 1382-1383 (Fed. Cir. 2019) (“Petitioners specifically argued in its Petition that [an element from one reference] could have been used in place of the [element from another reference]. The Board ultimately determined that Petitioners’ evidence in support of that combination was insufficient. We will not fault the Board for analyzing Petitioners’ obviousness grounds in the way presented in the Petition”)

Zaban and Ptatschek (plus Yu, Bawendi, and/or Braun)

Ground	8,524,365 Claims	35 U.S.C. §	Reference(s)
1	1, 7-12, 17, 22, 23	102	Banin
2	1, 7-12, 15-17, 22, 23	103(a)	Banin
3	2-6, 18-21	103(a)	Banin, Herron
4	13, 14	103(a)	Banin, Treadway
5	1-9, 17-23	103(a)	Zaban, Farneth, Yu
6	1, 2, 4, 7-12, 17, 18, 22, 23	103(a)	Lucey, Ahrenkiel
7	13-16	103(a)	Lucey, Ahrenkiel, Treadway

Ground	7,803,423 Claims	35 U.S.C. §	Reference(s)
1	1-3, 10, 11, 13, 22-24	102	Banin
2	1-6, 10-14, 21-25	103(a)	Banin
3	7-9	103(a)	Banin, Bawendi
4	1, 10-16, 21-24	103(a)	Zaban, Ptatschek
5	4-6, 25	103(a)	Zaban, Ptatschek, Yu
6	7-9	103(a)	Zaban, Ptatschek, Bawendi
7	1, 4, 11-16, 21, 25	103(a)	Lucey, Ahrenkiel

Ground	7,588,828 Claims	35 U.S.C. §	Reference(s)
1	1, 3-6, 8-14	103(a)	Banin, Herron
2	1, 3	103(a)	Banin, Farneth
3	1, 3-6, 14	103(a)	Zaban, Farneth, Yu
4	8-13	103(a)	Zaban, Farneth, Yu, Braun

Ground	7,867,557 Claims	35 U.S.C. §	Reference(s)
1	1-6, 16, 17	103(a)	Banin, Braun
2	1, 16, 17	103(a)	Zaban, Ptatschek, Braun
3	4-6	103(a)	Zaban, Ptatschek, Braun, Yu
4	1, 4, 16, 17	103(a)	Lucey, Ahrenkiel, Braun



The Obviousness Grounds - Lucey

Lucey in view of Ahrenkiel, Treadway, and/or Braun

Lucey

Report on methods for making quantum dots using a single-source precursor

– relies on the hot injection method

United States Patent
Lucey et al.

Patent No.: US 7,193,098 B1
Date of Patent: Mar. 26, 2007

PROCESS FOR PRODUCING SEMICONDUCTOR NANOCRYSTAL CORES, CORE-SHELL, CORE-BUFFER-SHELL, AND MULTIPLE LAYER SYSTEMS IN A NON-COORDINATING SOLVENT UTILIZING IN SITU SURFACTANT GENERATION

Inventors: Derrick W. Lucey, Amherst, NY (US); David J. MacRae, Pendleton, NY (US); Paras N. Prasad, Buffalo, NY (US); Orville T. Beachley, Jr., Buffalo, NY (US)

Assignee: The Research Foundation of State University of New York, Amherst, NY (US)

PROCESS FOR PRODUCING SEMICONDUCTOR NANOCRYSTAL CORES, CORE-SHELL, CORE-BUFFER-SHELL, AND MULTIPLE LAYER SYSTEMS IN A NON-COORDINATING SOLVENT UTILIZING IN SITU SURFACTANT GENERATION

Inventors: **Derrick W. Lucey**, Amherst, NY (US); **David J. MacRae**, Pendleton, NY (US); **Paras N. Prasad**, Buffalo, NY (US); **Orville T. Beachley, Jr.**, Buffalo, NY (US)

Summary Ex. 1011

PROCESS FOR PRODUCING SEMICONDUCTOR NANOCRYSTAL CORES, CORE-SHELL, CORE-BUFFER-SHELL, AND MULTIPLE LAYER SYSTEMS IN A NON-COORDINATING SOLVENT UTILIZING IN SITU SURFACTANT GENERATION

Inventors: **Derrick W. Lucey**, Amherst, NY (US); **David J. MacRae**, Pendleton, NY (US); **Paras N. Prasad**, Buffalo, NY (US); **Orville T. Beachley, Jr.**, Buffalo, NY (US)

The initiation of the reactions occurs when the ER_n source is rapidly injected into a heated solution of ML_n source in the non-coordinating solvent. Upon injection the ER_n and M sources react yielding a seed ME nanocrystal and the elimination product of the reaction (nLR). As shown in FIG.

Another aspect of the present invention is a process for producing compounds useful as Group III precursors for producing III-V nanocrystals in accordance with processes of the present invention. The process involves reacting a

Ex. 1011.

Lucey

- Uses the hot injection method to make quantum dots from a single-source precursor.
 - See Ex. 2030 ¶¶ 61, 179; Ex. 1011, 12:38-41 (“The initiation of the reactions occurs when the ERn source is rapidly injected into a heated solution of MLn source in the non-coordinating solvent.”).

Lucey – Does Not Disclose Many Claim Elements

- Lucey does not disclose “wherein the semiconductor material comprises one or more elements not comprised within the molecular cluster compound,” so adds **Ahrenkiel**.
- Lucey does not disclose “a first precursor species containing a first ion to be incorporated into the core semiconductor material and a separate second precursor species containing a second ion to be incorporated,” so adds **Ahrenkiel**
- Lucey does not disclose “a core/shell/shell nanoparticle, where the core and second shell material are selected to form a quantum well in the first shell, and the first shell is of a different material, so adds **Braun**.

Ex. 2030 (IPR2021-00186) ¶¶ 160-164; Ex. 1049, 42-44;
Ex. 1070; POR at 54-55; POSR at 22.

Lucey and Ahrenkiel (plus Yu, Bawendi, and/or Braun)

Ground	8,524,365 Claims	35 U.S.C. §	Reference(s)
1	1, 7-12, 17, 22, 23	102	Banin
2	1, 7-12, 15-17, 22, 23	103(a)	Banin
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4	13, 14	103(a)	Banin, Treadway
5	1-9, 17-23	103(a)	Zaban, Farneth, Yu
6	1, 2, 4, 7-12, 17, 18, 22, 23	103(a)	Lucey, Ahrenkiel
7	13-16	103(a)	Lucey, Ahrenkiel, Treadway

Ground	7,803,423 Claims	35 U.S.C. §	Reference(s)
1	1-3, 10, 11, 13, 22-24	102	Banin
2	1-6, 10-14, 21-25	103(a)	Banin
3	7-9	103(a)	Banin, Bawendi
4	1, 10-16, 21-24	103(a)	Zaban, Ptatschek
5	4-6, 25	103(a)	Zaban, Ptatschek, Yu
6	7-9	103(a)	Zaban, Ptatschek, Bawendi
7	1, 4, 11-16, 21, 25	103(a)	Lucey, Ahrenkiel

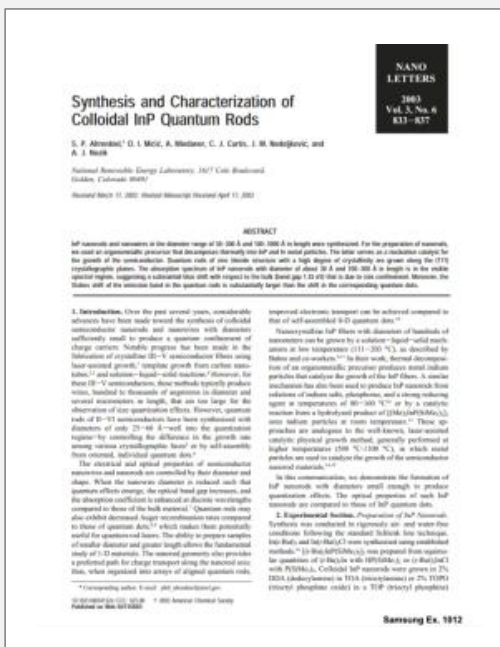
Ground	7,588,828 Claims	35 U.S.C. §	Reference(s)
1	1, 3-6, 8-14	103(a)	Banin, Herron
2	1, 3	103(a)	Banin, Farneth
3	1, 3-6, 14	103(a)	Zaban, Farneth, Yu
4	8-13	103(a)	Zaban, Farneth, Yu, Braun

Ground	7,867,557 Claims	35 U.S.C. §	Reference(s)
1	1-6, 16, 17	103(a)	Banin, Braun
2	1, 16, 17	103(a)	Zaban, Ptatschek, Braun
3	4-6	103(a)	Zaban, Ptatschek, Braun, Yu
4	1, 4, 16, 17	103(a)	Lucey, Ahrenkiel, Braun

Ahrenkiel

Synthesis of InP nanorods with small diameters

- Uses the SLS method with precursors with both indium and phosphorus

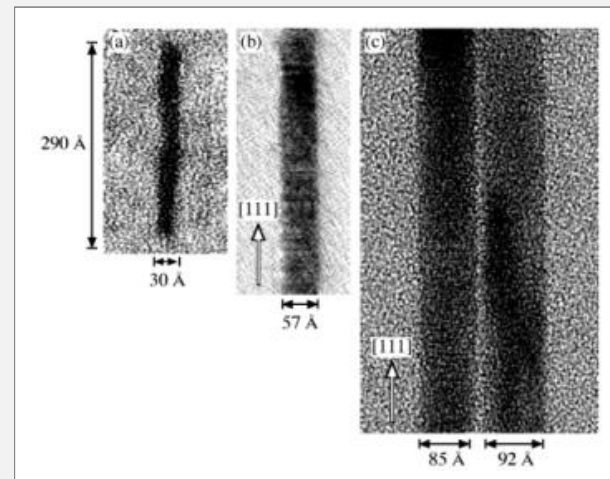


Synthesis and Characterization of Colloidal InP Quantum Rods

S. P. Ahrenkiel,* O. I. Micić, A. Miedaner, C. J. Curtis, J. M. Nedeljković, and A. J. Nozik

3. Results and Discussion. The growth of InP quantum rods and quantum wires by the solution-liquid-solid mechanism requires the presence of extremely small metal indium particles that serve as a catalyst for the growth of InP. The diameter of the indium particles determines the diameter of the nanorods. Indium particles can be introduced

molecular precursor by thermal decomposition. For the preparation of InP quantum rods, we used a mixture of $[(t\text{-Bu})_2\text{InP}(\text{SiMe}_3)_2]_2$ and $[\text{Cl}_2\text{InP}(\text{SiMe}_3)_2]_2$ precursors. When only the $[(t\text{-Bu})_2\text{InP}(\text{SiMe}_3)_2]_2$ precursor was present in the system, a large number of indium droplets and short InP fibers were produced. The $[\text{Cl}_2\text{InP}(\text{SiMe}_3)_2]_2$ precursor



Ex. 1012.

Lucey in view of Ahrenkiel

Incompatible Methods - Ahrenkiel uses the SLS method while Lucey uses hot-injection

The Board already rejected the combination of Lucey and Ahrenkiel.

- “This ground appears to us to be based on impermissible hindsight reconstruction, rather than any suggestion, identified adequately by Petitioner, in the asserted prior art references.”
- “[Hot injection and SLS are] disparate methods involve markedly different experimental conditions, reactants, and synthetic goals.”
- “Petitioner does not address adequately, if at all, the disparate features of the two methods, much less show sufficiently how or why an ordinarily skilled artisan would have reconciled the differences in a modified process of Lucey.”

Ex. 2030 (IPR2021-00186), ¶¶ 54, 179-184; Ex. 1012, 834; POR at 14-15.

Lucey in view of Ahrenkiel

Lucey Teaches Away from Ahrenkiel's Chlorine-Based Precursors

Moreover, current technologies for III-V semiconductor nanocrystals synthesis are dedicated strictly to using commercially available precursors. The most common precursors are the $M^{III}(\text{halides})_3$. When utilizing these precursors, the resultant semiconductor nanocrystals have the propensity to have halide impurities. These impurities hinder the desired electronic or optical properties of the nanocrystals.

The Group III precursors prepared in accordance with the present invention do not contain halide impurities, and do not require coordinating solvents or surfactants that upon extended exposure to high temperatures may obscure the electronic and optical properties of the resultant III-V nanocrystals.

Ex. 1011, 3:53-57, 5:14-19; 2030 (IPR2021-00186), ¶¶ 185-86; POR at 62-63.

Lucey in view of Ahrenkiel

Lucey Teaches Away from Ahrenkiel's Chlorine-Based Precursors

molecular precursor by thermal decomposition. For the preparation of InP quantum rods, we used a mixture of $[(t\text{-Bu})_2\text{InP}(\text{SiMe}_3)_2]_2$ and $[\text{Cl}_2\text{InP}(\text{SiMe}_3)_2]_2$ precursors. When only the $[(t\text{-Bu})_2\text{InP}(\text{SiMe}_3)_2]_2$ precursor was present in the system, a large number of indium droplets and short InP fibers were produced. The $[\text{Cl}_2\text{InP}(\text{SiMe}_3)_2]_2$ precursor decomposes to InP without the formation of metallic indium and can produce InP fibers in the presence of metal indium particles.¹⁸ Because of that, we added $[\text{Cl}_2\text{InP}(\text{SiMe}_3)_2]_2$ to $[(t\text{-Bu})_2\text{InP}(\text{SiMe}_3)_2]_2$ to increase the total InP precursor concentration and, consequently, the length of the InP nanorods. Thermal decomposition of the $[(t\text{-Bu})_2\text{InP}$ -

Ex. 1012, 824; 2030 (IPR2021-00186), ¶¶ 185-87; POR at 62-63.

Lucey in view of Ahrenkiel

Ahrenkiel's Precursor Decomposes to InP, not Indium alone.

- Petitioner fails to show adequately that Ahrenkiel discloses or suggests this limitation, because Ahrenkiel does not provide separate ions as required by those claims, but rather, employs a precursor composition comprising two precursor species that both provide indium phosphide (InP) itself.”

fibers were produced. The $[\text{Cl}_2\text{InP}(\text{SiMe}_3)_2]_2$ precursor decomposes to InP without the formation of metallic indium and can produce InP fibers in the presence of metal indium particles.¹⁸ Because of that, we added $[\text{Cl}_2\text{InP}(\text{SiMe}_3)_2]_2$ to

InstDec. (IPR2021-00186) at 27, Ex. 1012, 824; 2030 (IPR2021-00186), ¶¶ 185-87; POR at 62-63.

Lucey in view of Ahrenkiel, and Treadway

The Board Already Rejected this Combination

- “Treadway does nothing to cure the insufficiency of Petitioner’s showing, discussed above, with respect to the proposed combination of the disparate methods of Lucey and Ahrenkiel.”
- In this ground, moreover, Petitioner plucks elements from each of three asserted prior references without adequately tethering a reason for the proposed combination to any path illuminated by the prior art. This suggests a classic exercise of impermissible hindsight reconstruction.

InstDec. (IPR2021-00186) at 27-28.

Lucey and Ahrenkiel (plus Yu, Bawendi, and/or Braun)

Ground	8,524,365 Claims	35 U.S.C. §	Reference(s)
1	1, 7-12, 17, 22, 23	102	Banin
2	1, 7-12, 15-17, 22, 23	103(a)	Banin
3	2-6, 18-21	103(a)	Banin, Herron
4	13, 14	103(a)	Banin, Treadway
5	1-9, 17-23	103(a)	Zaban, Farneth, Yu
6	1, 2, 4, 7-12, 17, 18, 22, 23	103(a)	Lucey, Ahrenkiel
7	13-16	103(a)	Lucey, Ahrenkiel, Treadway

Ground	7,803,423 Claims	35 U.S.C. §	Reference(s)
1	1-3, 10, 11, 13, 22-24	102	Banin
2	1-6, 10-14, 21-25	103(a)	Banin
3	7-9	103(a)	Banin, Bawendi
4	1, 10-16, 21-24	103(a)	Zaban, Ptatschek
5	4-6, 25	103(a)	Zaban, Ptatschek, Yu
6	7-9	103(a)	Zaban, Ptatschek, Bawendi
7	1, 4, 11-16, 21, 25	103(a)	Lucey, Ahrenkiel

Ground	7,588,828 Claims	35 U.S.C. §	Reference(s)
1	1, 3-6, 8-14	103(a)	Banin, Herron
2	1, 3	103(a)	Banin, Farneth
3	1, 3-6, 14	103(a)	Zaban, Farneth, Yu
4	8-13	103(a)	Zaban, Farneth, Yu, Braun

Ground	7,867,557 Claims	35 U.S.C. §	Reference(s)
1	1-6, 16, 17	103(a)	Banin, Braun
2	1, 16, 17	103(a)	Zaban, Ptatschek, Braun
3	4-6	103(a)	Zaban, Ptatschek, Braun, Yu
4	1, 4, 16, 17	103(a)	Lucey, Ahrenkiel, Braun



Thank You