

UNITED STATES PATENT AND TRADEMARK OFFICE

BEFORE THE PATENT TRIAL AND APPEAL BOARD

MICRON TECHNOLOGY, INC.,
Petitioner,

v.

PRESIDENT AND FELLOW OF HARVARD COLLEGE,
Patent Owner

Case IPR No. 2017-00664
U.S. Patent No. 8,334,016
Title: VAPOR DEPOSITION OF METAL OXIDES, SILICATES AND
PHOSPHATES, AND SILICON DIOXIDE

**REPLY TO PATENT OWNER'S RESPONSE TO
PETITION FOR *INTER PARTES* REVIEW OF
U.S. PATENT NO. 8,334,016**

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Exhibit List

Micron Exhibit #	Description
Ex.1001	U.S. Patent No. 8,334,016 (“016 Patent”)
Ex.1002	Excerpts from Prosecution File History for U.S. Patent No. 8,334,016 (“016 Patent FH”)
Ex.1003	Declaration of Dr. Sanjay Banerjee (“Banerjee Decl.”)
Ex.1004	<i>Curriculum Vitae</i> of Dr. Sanjay Banerjee
Ex.1005	U.S. Patent No. 6,537,613 (“Senzaki”)
Ex.1006	Jae-Sik Min, et al., Atomic Layer Deposition of TiN Films by Alternate Supply of Tetrakis(ethylmethylamino)-Titanium and Ammonia, Japanese Journal of Applied Physics, Vol. 37, No. 9A 1998, pp. 4999-5004 (“Min”)
Ex.1007	Korean Patent No. 0156980 (“Shin”)
Ex.1008 ¹	M. Leskelä and M. Ritala, <i>Atomic Layer Epitaxy in Deposition of Various Oxide and Nitride Thin Films</i> , Journal de Physique IV Colloque, 1995, 05 (C5), pp.C5-937-C5-951 (“Leskelä I”)
Ex.1009	U.S. Patent No. 6,159,855 (“Vaartstra”)
Ex.1010	Jae-Sik Min, et al., <i>Metal-organic atomic-layer deposition of titanium-silicon-nitride films</i> , Applied Physics Letters, Volume 75, Number 11, 13 September 1999, pp.1521-1523 (“Min 1999”).
Ex.1011	S. M. George, et al., <i>Surface Chemistry for Atomic Layer Growth</i> , J. Phys. Chem, Vol. 100, No. 31, August 1, 1996 (“George”).

¹ See Ex.1017 for further publication information.

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Ex.1012 ²	Leskelä, M. and M. Ritala, <i>ALD precursor chemistry: Evolution and future challenges</i> . Journal de Physique, 1999, Vol. 9, pp. Pr8-837-Pr8-852. ("Leskelä II")
Ex.1013	A. Bastianini et al., <i>Chemical Vapor Deposition of ZrO₂ Thin Films Using Zr(NEt₂)₄ as Precursor</i> , Journal de Physique IV Colloque, 1995, 05 (C5), Vol 1, pp.C5-525-C5-531 ("Bastianini")
Ex.1014	Declaration of Roy G. Gordon regarding U.S. Patent Application No. 11/199,032, dated Aug. 12, 2008 ("Gordon Declaration")
Ex.1015	U.S. Patent No. 7,507,848 ("848 Patent")
Ex.1016	Declaration of Sharon Wiles-Young regarding Jae-Sik Min, et al., <i>Atomic Layer Deposition of TiN Films by Alternate Supply of Tetrakis(ethylmethyldamino)-Titanium and Ammonia</i> .
Ex.1017	Declaration of Sharon Wiles-Young regarding M. Leskelä and M. Ritala, <i>Atomic Layer Epitaxy in Deposition of Various Oxide and Nitride Thin Films</i> .
Ex.1018	Declaration of Sharon Wiles-Young regarding Leskelä, M. and M. Ritala, <i>ALD precursor chemistry: Evolution and future challenges</i> .
Ex.1019 ³	Declaration of Sharon Wiles-Young regarding A. Bastianini et al., <i>Chemical Vapor Deposition of ZrO₂ Thin Films Using Zr(NEt₂)₄ as Precursor</i>
Ex.1020	U.S. Patent No. 6,969,539 ("539 Patent")
Ex.1021	Pierson, H., <i>Principles, Technology, and Applications</i> . Handbook of Chemical Vapor Deposition (CVD) 2nd ed., 1999 ("Pierson")

² See Ex.1018 for further publication information.

³ See Ex.1019 for further publication information.

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Ex.1022	Fix, R., et al., <i>Chemical Vapor Deposition of Titanium, Zirconium, and Hafnium Nitride Thin Films</i> . Chemistry of Materials, 1991, Vol. 3(6), pp. 1138-1148. ("Fix")
Ex.1023	Gates, S.M., <i>Surface Chemistry in the Chemical Vapor Deposition of Electronic Materials</i> . Chemical Reviews, 1996, Vol. 96, pp. 1519-1532. ("Gates")
Ex.1024	Hampden-Smith, M.J. and Kodas, T.T., <i>Chemical Vapor Deposition of Metals: Part 1. An Overview of CVD Processes</i> . Chemical Vapor Deposition, 1995, Vol. 1, pp. 8-23. ("Hampden-Smith")
Ex.1025	Suntola, T., <i>Atomic Layer Epitaxy</i> . International Journal on the Science and Technology of Condensed Matter Films, 1992, Vol. 216, pp. 84-89. ("Suntola II")
Ex.1026	Smith, R., <i>Chemical Vapour Deposition of the Oxides of Titanium, Zirconium and Hafnium</i> . Advanced Materials for Optics and Electronics 10, 105-114, 2000, pp.105-114 ("Smith 2000")
Ex.1027	U.S. Patent No. 5,252,518 ("518 Patent")
Ex.1028	Lee, J.G. et al., <i>Comparison of TiN films produced by TDEAT (Ti[N(C₂H₅)₂]₄), TDMAT (Ti[N(CH₃)₂]₄), and a new precursor TEMAT (Ti[N(CH₃)C₂H₅]₄)</i> . Materials Research Society Symposium Proceedings Volume 427, 1996, pp. 371-376. ("Lee")
Ex.1029	Suntola, T. and J. Hyvarinen, <i>Atomic Layer Epitaxy</i> . Annu. Rev. Mater. Sci., 1985, Vol. 15, pp. 177-195. ("Suntola I")
Ex.1030	Bradley, D.C. and Thomas, I.M., <i>Metallo-organic compounds containing metal-nitrogen bonds. Part I. Some dialkylamino-derivatives of Titanium and Zirconium</i> . J. Chem. Soc., 1960, October, pp. 3857-3861. ("Bradley I")
Ex.1031	M. Ritala et al., <i>Perfectly Conformal TiN and Al₂O₃ Films Deposited by Atomic Layer Deposition</i> . Chem. Vap. Deposition 1999, Vol. (1), pp.7-9 ("Ritala I")

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Ex. 1032	Solid State Technology, Insights for Electronics Manufacturing, <i>Executive Millennium Report: 1999 Roadmap: Solutions and caveats</i> , May 1, 2000 ("STT")
Ex.1033	Goodman, C. and Pessa, M.V., <i>Atomic layer epitaxy</i> . J. Appl. Phys., 1986, Vol. 60 (3), pp. R65-R81. ("Goodman")
Ex.1034	Ritala, M. et al., <i>Atomic Layer Deposition of Oxide Thin Films with Metal Alkoxides as Oxygen Sources</i> . Science, 2000, Vol. 288, pp. 319-321. ("Ritala II")
Ex.1035	Ritala, M., Advanced ALE processes of amorphous and polycrystalline films. Appl. Surf. Sci., 1997, Vol. 112, pp. 223-230. ("Ritala III")
Ex.1036	Niinistö, L. et al., <i>Synthesis of oxide thin films and overlayers by atomic layer epitaxy for advanced applications</i> . Mat. Sci. and Eng., 1996, Vol. B41, pp. 23-29. ("Niinistö")
Ex.1037	Hampel, C.A. and Hawley, G.G., <i>Glossary of Chemical Terms</i> . 2nd ed., 1982. ("Hampel")
Ex.1038	U.S. Patent No. 6,984,591 ("Buchanan")
Ex.1039	Ritala, M. and M. Leskelä, <i>Zirconium dioxide thin films deposited by ALE using zirconium tetrachloride as precursor</i> . Applied Surface Science, 1994, Vol. 75, pp. 333-340. ("Ritala IV")
Ex.1040	Colombo, D., <i>Anhydrous Metal Nitrates as Volatile Single Source Precursors for the CVD of Metal Oxide Films</i> . Chem. Vap. Deposition, 1998, 4, No. 6, pp. 220-222. ("Colombo")
Ex.1041	Bouman, M. and Zaera, F., <i>Reductive Eliminations from Amido Metal Complexes: Implications for Metal Film Deposition</i> . J. Electrochem. Soc., 2011, Vol. 158 (8), pp. D-524-D526. ("Bouman")
Ex.1042	Harvard Complaint, <i>President and Fellows of Harvard College, v. Micron Tech., Inc.</i> , Case No. 1:16-cv-11249 (D. Mass.) ("Harvard Complaint")

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Ex.1043	Bradley, D.C. and Gitlitz, M.H., <i>Metallo-organic Compounds Containing Metal-Nitrogen Bonds. Part VI. Infrared and Nuclear Magnetic Resonance of Dialkylamido-derivatives of Titanium, Vanadium, Zirconium, Niobium, Hafnium, Tantalum, and Thorium</i> . J. Chem. Soc., 1969, pp. 980-984. (“Bradley II”)
Ex.1044	Tsyganova, E.I. et al., <i>Thermal Stability of Zirconium Organic Derivatives with Ligands of Different Nature</i> . Russian J. of General Chem., 1999, Vol. 69 (10), pp. 1532-1535. (“Tsyganova”)
Ex.1045	Graf, R.F. <i>Modern Dictionary of Electronics</i> . 6th ed. 1997, pp. 254-257 (“Graf”)
Ex.1046	Buchanan, D.A., <i>Scaling the gate dielectric: Materials, integration, and reliability</i> . IBM J. Res. Dev., 1999, Vol. 43 (3), pp. 245-264. (“Buchanan II”)
Ex.1047	1999 Edition, International Technology Roadmap for Semiconductors. (“ITRS Roadmap”)
Ex.1048	U.S. Patent No. 6,013,553 (“553 Patent”)
Ex.1049	Burggraaf, P., <i>IEDM 1999 focused on CMOS solutions</i> . Solid State Tech., Feb, 2000. (“Burggraaf”)
Ex.1050	Hubbard, K.J. and Schlom, D.G., <i>Thermodynamic stability of binary oxides in contact with silicon</i> . J. Mater. Res., 1996, Vol. 11 (11), pp. 2757-2776. (“Hubbard”)
Ex.1051	Kukli, K. et al., <i>Tailoring the dielectric properties of HfO₂-Ta₂O₅ nanolaminates</i> . Appl. Phys. Lett., 1996, Vol. 68 (26), pp. 3737-3739. (“Kukli”)
Ex.1052	Fictorie, C.P. et al., <i>Kinetic and mechanistic study of the chemical vapor deposition of titanium oxide thin films using tetrakis-(isopropoxo)-titanium(IV)</i> . J. Vacuum Sci. & Tech., 1994, Vol. 12, pp. 1108-1113. (“Fictorie”)

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Ex.1053	Houssa, M. et al., <i>Charge trapping in very thin high-permittivity gate dielectric layers</i> . Appl. Phys. Lett., 2000, Vol. 77 (9), pp. 1381-1383. ("Houssa")
Ex.1054	Kytökivi, A. et al., <i>Controlled Formation of ZrO₂ in the Reaction of ZrCl₄ Vapor with Porous Silica and γ-Alumina Surfaces</i> . Langmuir, 1996, Vol. 12, pp. 4395-4403. ("Kytökivi")
Ex.1055	Brusasco, Raymond M., <i>High Index of Refraction Films for Dielectric Mirrors Prepared by Metal-Organic Chemical Vapor Deposition</i> . SPIE- The International Society for Optical Engineering, 1989, Vol. 1047, pp. 23-32. ("Brusasco")
Ex.1056	Roy Gordon LinkedIn Profile, available at https://www.linkedin.com/in/roy-gordon-63550413 , accessed on Jan. 6, 2017
Ex.1057	Markku Leskelä LinkedIn Profile, available at https://www.linkedin.com/in/markku-leskel%C3%A4-60a5601 , accessed on Jan. 8, 2017
Ex.1058	Douglas Buchanan LinkedIn Profile, available at https://www.linkedin.com/in/douglas-buchanan-0112658 , accessed on Jan. 8, 2017
Ex.1059	Brian Vaartstra LinkedIn Profile, available at https://www.linkedin.com/in/brian-vaartstra-58009323 , accessed on Jan. 8, 2017
Ex.1060	Declaration of Sanjay Banerjee, Ph.D. in Support of Reply to Patent Owner's Response
Ex.1061	U.S. Patent No. 5,389,401 ("401 Patent")
Ex.1062	Deposition Transcript of Wayne L. Gladfelter, taken on January 11, 2018
Ex.1063	Exhibit 4 to Deposition of Wayne L. Gladfelter, taken on January 11, 2018

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Ex.1064	Putkonen, M. and Niinisto, L., <i>Zirconia thin films by atomic layer epitaxy. A comparative study on the use of novel precursors with ozone</i> , Journal of Materials Chemistry, 2001, Vol. 11, pp. 3141-3147 (“Putkonen”)
Ex.1065	Leskelä, M. W0 00/15865(“Leskelä 865”)
Ex.1066	U.S. Patent No. 5,178,911 (“911 Patent”).)
Ex.1067	Inman et al., <i>Evaluation of tetrakis(diethylamino)hafnium Precursor in the Formation of Hafnium Oxide Films Using Atomic Layer Deposition</i> , Material Research Society Symposia Proceedings, 2003, Vol. 765, pp. D3.10.1-D3.10.6 (“Inman”)
Ex.1068	Lide, D.R. et al., <i>CRC Handbook of Chemistry and Physics</i> 9-51 – 9-62 (79th ed. 1998) (“CRC Handbook”)
Ex.1069	Juppo, M. et al., <i>In Situ Mass Spectrometry Study on Surface Reactions in Atomic Layer Deposition of Al₂O₃ Thin Films from Trimethylaluminum and Water</i> , Langmuir, 2000, Vol. 16 (8), pp. 4034-4039 (“Juppo”)
Ex.1070	Wilk, G.D. et al., <i>Hafnium and zirconium silicates for advanced gate dielectrics</i> , Journal of Applied Physics, 2000, Vol. 87 (1), pp. 484-492 (“Wilk”)
Ex.1071	U.S. Pat. No. 6,042,652 (“652 Patent”)

I. INTRODUCTION

Patent Owner's ("PO") Response ("POR") does nothing to rebut Petitioner's showing that claims 1, 2, 7, 8, and 10 are obvious.

First, PO's arguments should be rejected because they largely attack a combination that Petitioner has never advanced. The Petition demonstrates that claims 1, 2, 7, and 10 are invalid in view of Senzaki's *ALD process* that uses $\text{Zr}(\text{NET}_2)_4$ and, to the extent that Senzaki does not expressly disclose "self-limiting" deposition, adapting that *ALD process* to be self-limiting in view of Min's teachings on how to make an ALD process self-limiting ("Senzaki/Min combination"). *See, e.g.*, Petition at 29-45. In response, PO does not address this combination. Instead, the POR attacks a *different* combination -- one that modifies Senzaki's *CVD process* to use (1) ALD precursors that are "suitable" for ALD, (2) Min's ALD process, (3) Min's precursors, and (4) Min's process conditions. POR at 19-20, 24-49. Because PO does not challenge Petitioner's combination, or the grounds on which the IPR was instituted (Decision at 8-14), PO's arguments are irrelevant.

Second, PO's assertion that Senzaki requires varying the temperature to form a "compositional gradient" and that it would not have been obvious how to do this using ALD is misguided. POR at 29-35. Nothing in Senzaki teaches that a compositional gradient is required. Rather, Senzaki describes an ALD process that

changes temperature to create a compositional gradient *and* it describes an ALD process that does not change the temperature and does not create a compositional gradient. Regardless, both ALD disclosures are presumed to be enabling, and PO does nothing to overcome the presumption.

Third, with respect to claim 8, PO's attack on the Senzaki/Min/Shin combination fails as a matter of law. PO argues that it would not have been obvious to combine Shin's teachings with the Senzaki/Min combination because (1) Shin's teachings relate to CVD and CVD teachings are not necessarily applicable to ALD, and (2) experimentation would be necessary to confirm that Shin's CVD teaching would apply to ALD. POR at 50-57. Here, PO applies a legally erroneous standard that a person of ordinary skill in the art ("POSA") must have absolute certainty that a prior art combination would work without any experimentation whatsoever. Not only is PO's position contrary to law, but it ignores that binary CVD reactions were known to provide a strong indication of whether an ALD reaction would work.

II. SENZAKI IN VIEW OF MIN RENDERS CLAIMS 1, 2, 7, AND 10 OBVIOUS⁴

As an initial matter, PO repeatedly misstates Petitioner's Senzaki/Min

⁴ PO argues only that the prior art does not teach or disclose limitations in claim 1; the POR is silent about the limitations in claims 2, 7, and 10.

combination, going so far to suggest that the Petition is substituting (1) Min's precursors with Senzaki's precursors, and (2) Min's ALD process for Senzaki's *CVD process*. POR at 19-20. This is *not* Petitioner's Senzaki/Min combination. The Petition makes clear that claims 1, 2, 7, and 10 are invalid in view of Senzaki's *ALD process* that uses $\text{Zr}(\text{NEt}_2)_4$ ⁵ and, to the extent Senzaki does not expressly disclose "self-limiting" ALD, dialing-in⁶ Senzaki's *ALD process* to be self-limiting in view of Min's teaching.⁷ See, e.g., Petition at 29-45.

⁵ Senzaki's $\text{Zr}(\text{NEt}_2)_4$ ALD process also uses $\text{Si}(\text{NMe}_2)_4$ and an oxidant, but this Reply focuses on $\text{Zr}(\text{NEt}_2)_4$ because PO only disputes Senzaki/Min's disclosure of a metal alkylamide. Petition at 29-35.

⁶ Dialing-in an ALD process refers to the routine process of adjusting the process conditions, e.g., temperature, to render the process self-limiting. ALD processes have been known since the 1970s, and such dialing-in was well known and routine. See Petition at 6-7, 39-40; Ex.1003, ¶55; Ex.1060, Section III.B.

⁷ When PO made these same mischaracterizations in its Preliminary Response, the Board found that "Petitioner does not assert, however, that one of ordinary skill in the art would have modified Senzaki's disclosed CVD examples. Petitioner contends, instead, that Senzaki discloses using its precursors and reactants in both a CVD process and an ALD process." Decision at 10. Although the Board

A. Senzaki Provides An Enabling Disclosure Of An ALD Process Using $\text{Zr}(\text{NEt}_2)_4$, Which Is A Metal Alkylamide

The Petition demonstrates that Senzaki discloses an *ALD process* using $\text{Zr}(\text{NEt}_2)_4$, which is a metal alkylamide. Petition at 29-35. In response, PO argues that Senzaki does not disclose which of its precursors are suitable for ALD and that a POSA would need to test each precursor to confirm that it is suitable. POR at 24-29. This argument fundamentally misses the mark.

There is no need to test each and every Senzaki precursor because Senzaki states that the “above precursors could be used for” for ALD and $\text{Zr}(\text{NEt}_2)_4$ – a metal alkylamide – is expressly identified as one of the “above precursors.” Ex.1005 at 4:4-15. Senzaki identifies “ $\text{Zr}(\text{NEt}_2)_4$ ” in the specification when it initially describes an example process. *Id.* at 3:40-43. Then, Senzaki states just two paragraphs later that “the above precursors could be used for” “atomic layer deposition.” *Id.* at 4:4-15. A POSA certainly would have understood Senzaki to disclose an ALD process using $\text{Zr}(\text{NEt}_2)_4$. *In re Hogan*, 559 F.2d 595, 608 (C.C.P.A. 1977) (noting that statements do not “stand alone” and that “the indicated that Senzaki does not provide “an explicit example of forming a metal oxide layer using a metal alkylamide in an ALD process,” the Board still found that “Petitioner has explained sufficiently where Senzaki teaches or suggests an ALD process using a metal alkylamide to form a metal oxide layer.” *Id.*

specification disclosure as a whole must be considered"); *see also* Ex.1060, ¶¶75-77.⁸ As the Board correctly pointed out, the processes disclosed in Senzaki are presumed to be enabled, Decision at 11, and therefore Senzaki's $\text{Zr}(\text{NEt}_2)_4$ ALD process is presumed to be enabled. PO fails to rebut this presumption—PO and Dr. Gladfelter do not point to any evidence suggesting that Senzaki's $\text{Zr}(\text{NEt}_2)_4$ ALD process is not suitable for ALD.

Contrary to PO's suggestion, Senzaki still teaches a metal dialkylamide ALD process even if a POSA would look up $\text{Zr}(\text{NEt}_2)_4$ in a handbook or conduct a confirming ALD experiment. POR at 28-29. Even the law of anticipation permits experimentation as long as the experimentation is not *undue*. *See, e.g., Kennametal*, 780 F.3d at 1383; *Elan Pharms., Inc. v. Mayo Found. for Med. Educ. & Research*, 346 F.3d 1051, 1054 (Fed. Cir. 2003). Here, any testing of $\text{Zr}(\text{NEt}_2)_4$ for an ALD process would have been routine, involving little more than adjusting the knob of an ALD reactor to vary the substrate temperature. *See, e.g.,* Ex.1060, ¶¶ 77-78, Ex.1006 at 7-9; Ex.2117, 26:20-29:6; Petition at 39-

⁸ Senzaki's reference to making an "[a]ppropriate choice of precursors" (Ex.1005 at 363) is not to the contrary. Indeed, the language – which appears shortly after $\text{Zr}(\text{NEt}_2)_4$ is identified as a precursor – merely reinforces that the specifically named $\text{Zr}(\text{NEt}_2)_4$ precursor is an "appropriate choice."

40. That a POSA would confirm information about a precursor such as $\text{Zr}(\text{NEt}_2)_4$ in a reference book also does not preclude obviousness. *See, e.g., MacDermid Printing Sol'ns, LLC v. E.I. DuPont de Nemours & Co.*, 2008 U.S. Dist. LEXIS 71737, at *24 (D.N.J. Sept. 4, 2008) (citing *In re Winslow*, 365 F.2d 1017, 1020 (CCPA 1966)).

Finally, Senzaki also provides an example of using $\text{Zr}(\text{NEt}_2)_4$ in a CVD process. *Id.* at 5:10-42 (Example 1). Notably, the Federal Circuit has held that when a prior art reference discloses using a metal in multiple technologies (there, CVD and MTCVD), but generally states that the metal is suitable for other technologies as well (there, PVD), the reference discloses using the metal in the other technologies (there, PVD). *Kennametal, Inc. v. Ingersoll Cutting Tool Comp.*, 780 F.3d 1376, 1381-84 (Fed. 2015). Accordingly, PO's argument that a POSA would not read Example 1 of Senzaki to apply to ALD is incorrect as a matter of law.

B. PO Attacks A Strawman In Arguing That It Would Not Have Been Obvious How To Achieve Senzaki's "Goal" Of A Compositional Gradient Using ALD

The Petition amply demonstrates that the Senzaki/Min combination discloses an ALD process using a metal alkylamide ($\text{Zr}(\text{NEt}_2)_4$), just like the claims of the 016 Patent. Petition at 29-35. PO argues, however, that it would not have been obvious to implement Senzaki using an ALD process because Senzaki

allegedly requires varying the process temperature to create a compositional gradient. PO at 29-35. PO's argument fails because Senzaki discloses an ALD process using $\text{Zr}(\text{NEt}_2)_4$ both with *and* without changing the temperature (and does not teach away from depositing a film without a compositional gradient) and both disclosures are sufficient to render the challenged claims obvious.

As the Board noted, Senzaki provides a "disclosure of ALD without temperature modification provided in the detailed description..." Decision at 11 n.7. After specifically mentioning the $\text{Zr}(\text{NEt}_2)_4$ precursor and stating that "the above precursors" can be used for ALD, Senzaki describes an ALD process that does not vary the process temperature. Ex.1005 at 3:40-42, 4:4-15. PO simply ignores this disclosure in its POR.

Rather than address the language of Senzaki or the Board's ID, PO turns to Dr. Banerjee's deposition testimony, asserting that he "recognizes" that "Senzaki's process requires the formation of a metal and metalloid compound layer with a compositional gradient of the metal and metalloid in the layer." POR at 29-30. Quite the contrary, Dr. Banerjee testified that achieving a compositional gradient in Senzaki was an "overarching goal" of Senzaki, not a requirement. Ex.2121, Banerjee Tr. at 102:3-104:20, 115:2-17. In fact, Senzaki recognizes advantages of using multicomponent metal-containing materials, such as mixed-metal/metalloid oxides, without a compositional gradient, noting that such materials "often have

unique physical properties that each individual metal/metalloid oxide/nitride component does not possess. For example, some mixed metal oxides can be used for high dielectric constant materials” *Id.* at 1:7-12. Senzaki goes on to cite several references illustrating this point. *Id.* at 1:12-23. By way of example, as Dr. Banerjee explains, these references (such as the Wilk reference) discuss the benefits of these materials without a compositional gradient in the context of “metal silicates as gate dielectric films.” *Id.* at 1:41-46; Ex.1060, ¶¶80-81. This is consistent with Senzaki, which states that “it is desirable to have materials which have a varying compositional gradient of mixed metals or metal/metalloid composition in either oxide, oxynitride or nitride form.” Ex.1005 at 2:20-23. But nothing about this “goal” or “desire” means that Senzaki’s plain language in column 4 does not disclose “ALD without temperature modification,” particularly given Senzaki’s praise of such films that do not include a compositional gradient. Decision at 11 n.7; *see also* Ex.1060, ¶¶82-84.

PO’s actual argument appears to be that Senzaki teaches away from its own disclosure of an ALD process that uses $\text{Zr}(\text{NEt}_2)_4$ “without temperature modification.” Decision at 11 n.7. But just because a prior art reference describes “better alternatives” “does not mean that an inferior combination is inapt for obviousness purposes.” *In re Mouttet*, 686 F.3d 1322, 1334 (Fed. Cir. 2012) (citing *Gurley*, 27 F.3d at 553). Accordingly, just because Senzaki discloses that a

compositional gradient through temperature variation is “desirable” does not teach away from its express disclosure of an ALD process without temperature variation. Ex.1060, ¶¶82-84.

Even if the Board were to find that the Senzaki only discloses varying the temperature to create a compositional gradient, Senzaki/Min still renders the challenged claims obvious. Ex.1060, ¶¶85-87. Notably, Senzaki describes a process in which the temperature is varied and the precursors contact the substrate by ALD. Ex.1005 at 2:38-62.⁹ As the Board correctly pointed out, Senzaki is presumed to be enabling, ID at 11, and thus its ALD process in which temperature is varied is presumed to be enabling. Ex.1060, ¶¶85-87.

PO fails to rebut this presumption. Instead, PO and Dr. Gladfelter theorize that Senzaki's two precursors ($\text{Zr}(\text{NEt}_2)_4$ and $\text{Si}(\text{NEt}_2)_4$) *may not have* a common ALD window. POR at 31-34. Tellingly, Dr. Gladfelter provides no evidence or opinion that $\text{Zr}(\text{NEt}_2)_4$ does not have an ALD window, that $\text{Si}(\text{NMe}_2)_4$ does not

⁹ PO argues that Senzaki's disclosures are all in the context of achieving a compositional gradient by changing the temperature. *See, e.g.*, POR at 12-13. If so, then all of Senzaki's processes, including the $\text{Zr}(\text{NEt}_2)_4$ ALD process in columns 3 and 4, are presumed to achieve a compositional gradient by changing the temperature. As the below illustrates, PO does not rebut this presumption.

have an ALD window, or that both do not have a common ALD window. Ex.1005 at 2:38-62, 3:40-42, 4:4-15.

PO side-steps the issue by theorizing about other precursors and “potential problems” that may arise when precursors are injected simultaneously. POR at 34. This completely ignores Senzaki's enabling disclosure of using “atomic layer deposition” to deposit “an approximately single layer” of $\text{Zr}(\text{NEt}_2)_4$ and $\text{Si}(\text{NMe}_2)_4$ precursors “in a near atomic thickness layer.” Ex.1005 at 3:40-42, 4:4-15. Tellingly, Dr. Gladfelter does not cite any specific problem with the $\text{Zr}(\text{NEt}_2)_4$ and $\text{Si}(\text{NMe}_2)_4$ process. Ex.1060, ¶¶85-87. PO's theoretical attacks cannot overcome the presumption of enablement. *See In re Antor Media Corp.*, 689 F.3d 1282, 1287-88, 1290-92 (Fed. Cir. 2012) (placing burden on patentee to show, for example, that undue experimentation is required to make or use the claims at issue).

C. A POSA Would Have Been Motivated To Combine Senzaki And Min

As an initial matter, the Petition shows that Senzaki itself indicates that it is, or can be made, self-limiting (even if it does not use the magic words “self-limiting”). Petition at 33. It does so by describing “atomic layer deposition” to deposit “an approximately single layer” of precursor molecules “in a near atomic thickness layer.” Ex.1005 at 3:40-42, 4:4-15. PO does not squarely address this argument, nor does it address its statement in its district court complaint against

Petitioner that a “process wherein deposition of the first reactant and the second reactant component are self-limiting is part of ALD.” *See* Decision at 12-13.

Even though Senzaki does not use the magic words “self-limiting,” the claims of the 016 Patent are still obvious because Senzaki in combination with Min teaches a POSA how to make an ALD process that uses a metal alkylamide self-limiting. Petition at 37-45. None of PO's arguments, each of which is addressed below, is to the contrary.

1. Senzaki And Min Do Not Have Inconsistent Goals That Would Discourage A POSA From Combining Their Teachings

PO asserts that a POSA would not have been motivated to combine Senzaki and Min because their “goals” allegedly are inconsistent. POR at 35-40. Specifically, PO argues that Min is inconsistent with Senzaki because (1) Min teaches deposition at a particular “stable” temperature and (2) Senzaki requires changing the deposition temperature. POR at 38-40. This argument should be rejected, because it ignores the actual disclosures of Senzaki and Min, ignores their common goal, and improperly attacks the references individually.

To begin, the references are not inconsistent. Ex.1060, ¶¶88-89. Both references teach depositing a film by ALD without varying the temperature. Ex.1005 at 3:40-42, 4:4-15 (Senzaki); Ex.1006 at 5001 & Fig. 4 (e.g., self-limiting ALD at 200°C) (Min). And both references also teach depositing a film by ALD

by varying the temperature. Ex.1005 at 2:33-62 (Senzaki); Ex.1006 at 5000-01 & Fig. 3 (Min) (e.g., curve (a) shows self-limiting ALD measured at five different temperatures between 170°C and 210°C). Indeed, Min concludes “that the temperature range for TiN ALD is between 170°C and 210°C.” Ex.1006 at 5000. This 40°C temperature range in Min is fully consistent with Senzaki’s temperature-varying embodiment, which recites a change of at least 40°C. Ex.1005 at 2:50-53. Even PO, when generally describing Min, notes that it discloses “a definite temperature range.” POR at 16; Ex.1063, Gladfelter Depo. at 238:17-240:21 (confirming that film thickness does not change per cycle as temperature changes in this range).¹⁰

PO also ignores the common goal of precise layer thickness control that Senzaki and Min share. Ex. 1060, ¶89. The Petition explains that an express goal of Senzaki is controlling the precise thickness of gate dielectric layers, which

¹⁰ PO also argues that Senzaki and Min do not expressly describe changing the flow rates, which Dr. Banerjee explained could be done to achieve a compositional gradient. However, PO does not argue that changing flow rates was not within the ordinary level of skill. *See* Ex.1060, ¶86. Moreover, as demonstrated above, Senzaki discloses doing ALD without changing temperature and therefore discloses ALD without a compositional gradient. *See* Section II.B.

Senzaki describes in referring to ALD as depositing “an approximately single layer of precursor molecules” “in a near atomic thickness layer,” and which was a well-known goal in the industry for gate dielectrics at the time. Petition at 38-40 (citing Ex.1003, ¶133). The Petition further explains that Min’s teachings augment this goal by describing how to dial-in a self-limiting process. *Id.* PO ignores these teachings of Senzaki and Min, focusing only on Senzaki’s teaching that it is “desirable” to achieve a compositional gradient. Accordingly, there is nothing “inconsistent” about the references.

PO’s argument also carries no weight because it improperly reads Senzaki and Min individually. “Non-obviousness cannot be established by attacking references individually where the rejection is based upon the teachings of a combination of references.” *In re Merck*, 800 F.2d 1091, 1097 (Fed. Cir. 1986). “Rather, the test is what the combined teachings of the references would have suggested to those of ordinary skill in the art.” *In re Keller*, 642 F.2d 413, 425 (CCPA 1981). Taken together, Min bolsters Senzaki’s goal of controlling film thickness. Ex.1060, ¶89.

2. It Is Irrelevant Whether Min Teaches Modifying Senzaki’s CVD Processes With Predictable Results

PO argues that a POSA “would not have understood modifying Senzaki’s CVD process in view of Min to yield predictable results nor be routine to implement.” POR at 40-42. Here again, PO mischaracterizes Petitioner’s

combination. The Petition relies on Senzaki's *ALD process*, and merely uses Min to reinforce that it was routine to dial-in an ALD process to achieve self-limiting deposition (as Senzaki itself suggests with its description of ALD as depositing "an approximately single layer of precursor molecules" "in a near atomic thickness layer"). Petition at 29-35, 37-45. As such, PO's arguments are irrelevant.

3. It Is Irrelevant Whether A POSA Was Generally Aware That Metal Alkylamides Were "Desirable"

PO argues that a "POSA would not have known that certain precursors were 'desirable' for ALD processes." POR at 43. Once more, PO mischaracterizes Petitioner's combination. The Petition notes that Senzaki and Min both describe ALD processes with metal alkylamides, that it was well known at the time that metal alkylamides were suitable and desirable for ALD (citing several supporting references), and that making an oxide or nitride with the same metal precursor was routine (again citing several supporting references). Petition at 39-41. Min's teaching of how to dial-in a metal alkylamide ALD process into the "self-limiting" ALD window would have provided a POSA with a high expectation of success that Senzaki's metal alkylamide ALD process also could be dialed into the ALD window. *Id.* The combination does not depend on the general knowledge that metal alkylamides are desirable for ALD. Senzaki specifically discloses using a

metal alkylamide precursor ($\text{Zr}(\text{NEt}_2)_4$) in ALD. Min also specifically discloses using a metal alkylamide precursor in ALD. Thus, PO's argument is off point.¹¹

4. It Is Irrelevant Whether Min Teaches Modifying Senzaki's CVD Processes With A Reasonable Expectation Of Success

PO argues that a "POSA would have had no reasonable expectation of success modifying Senzaki's CVD process in view of Min."¹² POR at 44-48. Once again, PO's argument is irrelevant, because Petitioner's combination does not rely on modifying Senzaki's *CVD process*. The Petition relies on Senzaki's *ALD process*. To the extent PO is arguing that a POSA would look to Min for how to dial-in a self-limiting metal alkylamide ALD process solely for nitride deposition, PO is simply incorrect. Min *generally* teaches how to adjust an ALD process to deposit a film in a self-limiting fashion. Petition at 39-40 (citing Leskelä (Ex.1008) for a similar teaching); Ex. 1060, Section III.B. Min teaches how to identify the process conditions, e.g., the relevant temperatures, that result in

¹¹ Nonetheless, it was well known in the prior art that metal alkylamides were suitable and desirable for ALD. Petition at 39-41; Ex. 1060, Section III.C-D.

¹² PO's argument that the Examiner allowed the 016 Patent claims over Min because Min only discloses a nitride is thus irrelevant for the same reasons—Petitioner's combination uses Senzaki's dialkylamide precursors to form a metal oxide by ALD, not Min's precursors. POR at 43-44.

self-limiting deposition. Ex.1006 at 5000-01; Ex. 1060, Section III.B. PO does not explain why this routine process would not be applicable to Senzaki's ALD processes.¹³

III. SENZAKI IN VIEW OF MIN AND SHIN RENDERS CLAIM 8 OBVIOUS

The Petition demonstrates that a POSA would have been motivated to use Shin's tetrakis(ethylmethylamino) zirconium precursor in the Senzaki/Min combination because, for example, it has characteristics comparable to the precursors of Senzaki/Min and can neutralize potential problems presented by the precursors of Senzaki/Min. Petition at 47-50. PO's POR repeats the same arguments about claim 8 that it made in its Preliminary Response and Dr. Gladfelter provides no new opinions on the issue, thereby completely ignoring the

¹³ PO argues that all of Petitioner's support for the similarities between nitride and oxide ALD rely on CVD processes. POR at 47-48. That is incorrect. For example, in describing both ALD and CVD, Senzaki describes the well-known fact that the same metal precursor generally can be used for oxide and nitride deposition. Ex.1005 at 2:33-62, 4:32-46. Moreover, even if PO were correct, it was well known that binary CVD reactions generally predict ALD behavior, thereby providing additional support for a high expectation of success in applying Min's self-limiting deposition teaching to Senzaki. Ex.1060, Section III.A.

Board's criticisms of PO's arguments in its Institution Decision. At bottom, PO is arguing that even though Shin describes its precursor as comparable to Senzaki's precursor, that Shin describes its precursor as overcoming shortcomings with Senzaki's precursor, and that Shin's precursor and Senzaki's precursor are nearly identical, a POSA would not have been motivated to use Shin's compound because she would not have known with *absolute certainty* that it would be suitable for ALD. This argument bears no weight, because the law does not require the certainty that PO demands.

The Federal Circuit has repeatedly held that obviousness requires only a "reasonable expectation of success," not "absolute predictability" or "absolute certainty." *See, e.g., Soft Gel Techs., Inc. v. Jarrow Formulas, Inc.*, 864 F.3d 1334, 1342 (Fed. Cir. 2017); *In re O'Farrell*, 853 F.2d 894, 903 (Fed. Cir. 1988). "Indeed, for many inventions that seem quite obvious, there is no absolute predictability of success until the invention is reduced to practice." *O'Farrell*, 853 F.2d at 903 (affirming obviousness). Dr. Gladfelter testified in deposition that it is a common scenario in his laboratory work to perform experiments even though he did not have absolute certainty that the reaction would work as expected. Ex.1063, at 229:20-230:2.

PO then argues that, even though the Senzaki and Shin precursors differ by only one alkyl group, a POSA would not have been motivated to consider Shin's

precursor because this difference can have a meaningful effect, e.g., the two precursors being in different phases at the same temperature. POR at 52-53. In taking this position, PO both ignores (1) the actual precursors at issue in the Senzaki/Min/Shin combination, and (2) that Shin discloses that its precursor will “exhibit[] the intermediate characteristics of” of Senzaki’s and will “incorporate the advantages” of Senzaki’s. Petition at 48 (citing Ex.1007, Shin, pp.6-7). More troublingly, PO ignores the Board’s observation that PO failed to, and now continues to fail to, explain how these alleged differences have any effect on suitability for ALD. *Id.* at 17. Here, such differences do not defeat suitability for ALD. Ex.1060, ¶¶91.

PO further argues that a precursor’s suitability in Shin’s CVD process does not *necessarily* predict ALD behavior and that, therefore, a POSA would not have had a reasonable expectation of success in using Shin’s precursor in an ALD process. POR at 53-55. Not only is PO applying the legally erroneous “certainty” standard, but PO also is ignoring that binary CVD reactions of the type described in Shin generally predict—and at a bare minimum provide a strong indication of—ALD behavior. Ex.1060, ¶¶92-93. A POSA knew that binary CVD reactions (i.e., CVD reactions in which two different precursors react to deposit a film) generally predict the effectiveness of the same precursors in ALD reactions, and thus the successful use of metal dialkylamides in binary CVD would have given a POSA at

least a reasonable expectation of success in using the same precursor in an ALD reaction. *See, e.g.*, Ex.1011, 5; Ex.1012, 15; *see also* Ex.1060, ¶¶92-93. Indeed, Senzaki confirms this, as it describes using the same alkylamide precursors for binary CVD processes and for ALD processes. Ex.1005 at 2:33-62, 3:40-4:46.¹⁴

IV. NO OBJECTIVE INDICIA SUPPORT THE NON-OBVIOUSNESS OF THE CLAIMS

None of the purported objective indicia identified in the POR supports non-obviousness. PO cites purported “unexpected properties and results,” relying on the inventors’ own statements in the specification. POR at 21. However, “unexpected results must be established by factual evidence. *Mere argument or conclusory statements in the specification does not suffice.*” *In re Soni*, 54 F.3d 746, 750 (Fed. Cir. 1995). The specification contains no objective, factual evidence against which the inventors’ subjective claims of surprise can be evaluated, and PO has cited none. Thus, PO has not demonstrated “unexpected results.”

¹⁴ PO also suggests that contamination may deter a POSA from considering Shin. POR at 53-54. First, Dr. Gladfelter’s support relates to contamination in single-source CVD processes, not binary CVD processes, and therefore are not analogous. Ex.1060, ¶¶14-20, 92. Second, a POSA would not have expected significant contamination from carbon or nitrogen. *Id.*

PO's claims of "long-felt need" (POR at 21-22) are also unsupported. PO cites the 2000 ITRS as purportedly showing that the "problem" of depositing high- κ materials inside narrow trenches existed in the mid-1990s and was not expected to be solved until "at least 2008" (POR at 22), but this misinterprets the ITRS. Rather, the ITRS shows that the industry did not anticipate this "problem" to arise until 2005, because the dielectric material for technology nodes prior to 2005 was adequately provided by existing nitrided oxide ("NO") techniques. Ex.2108, 1; Ex.1060, ¶97. Even after the need for a new high- κ material was expected to arise around 2005, the ITRS merely indicates that a solution was needed by 2008, *not* that the solution would not be found sooner. Ex.2108, 1; Ex.1060, ¶97. Thus, there is no evidence of a long-felt need that was solved by the claimed invention.

PO alleges "great commercial success" of the claimed inventions, citing sales of metal dialkylamides. POR at 22. As discussed in the Petition, however, there is no nexus between these sales and the challenged claims, which require more than just a metal dialkylamide. Petition at 42-45. PO cites no evidence that the metal dialkylamides were used to deposit oxygen-containing insulators through ALD in a self-limiting fashion, as required by the claims. Indeed, there is no evidence that the chemicals were not used for CVD reactions. Ex. 1060, ¶98.

Finally, PO cites a paper by Samsung and Genus that thanked the named inventors for “helpful discussion on alkylamide precursors.” PO misrepresents the paper, misleadingly stating that it “thanked Dr. Gordon for his teaching of the claimed inventions.” POR at 22. Alkylamide precursors, however, are not the claimed invention. Ex.1060, ¶99. PO, by admission in the 016 Patent specification (*e.g.*, Table 1), expressly identified numerous metal alkylamide precursors that were known as early as the 1960s. *See, e.g.*, Ex.1001, 15:7-8, 15:12-13 (citing 1960 Bradley & Thomas article as reference for $\text{Zr}(\text{NEt}_2)_4$ and $\text{Zr}(\text{NMe}_2)_4$); *see also* Ex.1063, at 205:10-206:2. Thus, it is simply untrue that the Samsung/Genus paper “thanked Dr. Gordon for his teaching of the claimed inventions,” and like the metal dialkylamide sales discussed above, the paper lacks a nexus to the claims, which require using a metal dialkylamide in self-limiting ALD.

V. CONCLUSION

Petitioner requests that the Board find challenged claims 1, 2, 7, 8, and 10 unpatentable.

Reply to Patent Owner's Response to Petition for
Inter Partes Review of U.S. Patent No. 8,334,016

Respectfully submitted,

Dated: January 30, 2018

By: /Jeremy Jason Lang /

Jeremy Jason Lang
Lead Counsel for Petitioner
Registration No. 73,604
Weil, Gotshal & Manges LLP
201 Redwood Shores Parkway
Redwood Shores, CA 94065
Telephone: 650-802-3000

CERTIFICATE OF COMPLIANCE

1. The undersigned certifies that this brief complies with the type volume limitations of 37 CFR § 42.24(a)(1)(i). This brief contains 4,667 words (excluding the table of contents, the table of authorities, mandatory notices under 37 CFR § 42.8, the certificate of service, certificate of compliance, and appendix of exhibits), as calculated by the “Word Count” feature of Microsoft Word 2010, the word processing program used to create it.

2. The undersigned further certifies that this brief complies with the typeface requirements of 37 CFR § 42.6(a)(2)(ii) and typestyle requirements of 37 CFR § 42.6(a)(2)(iii). This brief has been prepared in a proportionally spaced typeface using Microsoft Word 2010 in Times New Roman 14 point font.

Respectfully submitted,

WEIL, GOTSHAL AND MANGES LLP

Dated: January 30, 2018

By: / Jeremy Jason Lang /

Jeremy Jason Lang
Lead Counsel for Petitioner
Registration No. 73,604
Weil, Gotshal & Manges LLP
201 Redwood Shores Parkway
Redwood Shores, CA 94065
Telephone: 650-802-3000

CERTIFICATE OF SERVICE

The undersigned certifies, in accordance with [37 C.F.R. § 42.105 and § 42.6(e)], that service was made on the Patent Owner as detailed below.

<i>Date of service</i>	January 30, 2018
<i>Manner of service</i>	ELECTRONIC FILING
<i>Documents served</i>	Reply to Patent Owner's Response to Petition for <i>Inter Partes</i> Review of U.S. Pat. No. 8,335,016 with Exhibit List Exhibits Ex. 1060 through Ex. 1071
<i>Persons served</i>	<u>Patent Owner's Address of Record:</u> Wilmer Hale 60 State Street Boston, MA 02109 <u>Additional Addresses Known as Likely to Effect Service:</u> William Belanger Alison McCarthy Gregory D. Len Maia H. Harris Griffin Mesmer Pepper Hamilton LLP 19th Floor, High Street Tower 125 High Street Boston, MA 02110-2736

Reply to Patent Owner's Response to Petition for
Inter Partes Review of U.S. Patent No. 8,334,016

Respectfully submitted,

A handwritten signature in blue ink, appearing to read 'J. Lang', with a horizontal line extending to the right.

Jeremy Jason Lang
Lead Counsel for Petitioner
Registration No. 73,604
Weil, Gotshal & Manges LLP
201 Redwood Shores Parkway
Redwood Shores, CA 94065-1134
Telephone: 650-802-3000