

UNITED STATES PATENT AND TRADEMARK OFFICE

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BEFORE THE PATENT TRIAL AND APPEAL BOARD

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MICRON TECHNOLOGY, INC.,  
Petitioner,

v.

PRESIDENT AND FELLOWS OF HARVARD COLLEGE,  
Patent Owner.

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Case IPR2017-00664  
Patent 8,334,016 B2

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Before CHRISTOPHER L. CRUMBLEY, JON B. TORNQUIST, and  
CHRISTOPHER M. KAISER, *Administrative Patent Judges*.

TORNQUIST, *Administrative Patent Judge*.

DECISION  
Institution of *Inter Partes* Review  
37 C.F.R. § 42.108

## I. INTRODUCTION

Micron Technology, Inc. (“Petitioner”) filed a Petition (Paper 1, “Pet.”) requesting an *inter partes* review of claims 1, 2, 7, 8, and 10 of U.S. Patent No. 8,334,016 B2 (Ex. 1001, “the ’016 patent”). The President and Fellows of Harvard College (“Patent Owner”) filed a Preliminary Response to the Petition (Paper 8, “Prelim. Resp.”).

We have authority to determine whether to institute an *inter partes* review. 35 U.S.C. § 314; 37 C.F.R. § 42.4(a). The standard for instituting an *inter partes* review is set forth in 35 U.S.C. § 314(a), which provides that an *inter partes* review may not be instituted “unless the Director determines . . . there is a reasonable likelihood that the petitioner would prevail with respect to at least 1 of the claims challenged in the petition.”

After considering the Petition and the Preliminary Response, we determine that Petitioner has demonstrated a reasonable likelihood of prevailing with respect to claims 1, 2, 7, 8, and 10 of the ’016 patent. Accordingly, we institute *inter partes* review with respect to those claims.

### A. *Related Proceedings*

The parties note that the ’016 patent is at issue in *President and Fellows of Harvard College v. Micron Tech., Inc.*, 1:16-cv-11249 (D. Mass.), *President and Fellows of Harvard College v. GlobalFoundries U.S., Inc.*, IPR2017-00663, and IPR2017-00666. Pet. 2; Paper 5, 1. The parties further note that related U.S. Patent No. 6,969,539 is at issue in the above-noted district court proceedings as well as IPR2017-00662. Pet. 2; Paper 5, 1.

*B. The '016 Patent*

The '016 patent discloses “reagents for use in thin film deposition processes such as chemical vapor deposition (CVD) and atomic layer deposition (ALD).” Ex. 1001, 1:30–32.

“In CVD processes, a reactant vapor or vapor mixture is brought into contact with a heated surface on which a thin film is deposited.” *Id.* at 1:46–48. In ALD (which is a type of CVD process) “a metered amount of a first reactant component” is introduced into a deposition chamber to deposit a thin layer of this first reactant on a substrate. *Id.* at 20:57–60. Excess vapor is removed from the chamber and “a metered amount of a second reactant component is then introduced into the deposition chamber” where it “interacts with the already deposited layer of the first reactant.” *Id.* at 20:60–21:5. The '016 patent explains that, because the surface reactions in the ALD process are “self-limiting,” the process may be used to provide a “reproducible layer of predictable composition” with “improved step coverage and thickness uniformity compared to CVD with mixed vapors.” *Id.* at 1:48–54, 20:64–67, 21:5–7.

In certain embodiments of the '016 patent, metal or metalloid amides may be used as a reactant. *Id.* at 10:4–9. Table I of the '016 patent provides a list of known amides for use in the disclosed ALD process, including tetrakis(dimethylamino)–, tetrakis(diethylamino)–, and tetrakis(ethylmethylamino)–hafnium and zirconium. *Id.* at Table I.

*C. Illustrative Claim*

Claim 1, reproduced below, is the only independent claim of the '016 patent and is illustrative of the challenged claims:

1. A process for making an insulator in a microelectronic device, the process comprising:

introducing a first reactant component into a deposition chamber;  
introducing a second reactant component into the deposition chamber; and  
alternately repeating introducing the first reactant component and the second reactant component into the deposition chamber;  
wherein deposition of the first reactant component and the second reactant component are self-limiting;  
wherein said first reactant component comprises a metal alkylamide;  
wherein said second reactant component interacts with the deposited first reactant component to form the insulator;  
and  
wherein said insulator comprises oxygen and the metal from the metal alkylamide.

Ex. 1001, 30:9–26.

*D. The Asserted Grounds of Unpatentability*

Petitioner contends that claims 1, 2, 7, 8, and 10 of the '016 patent are unpatentable based on the following grounds (Pet. 29–50):<sup>1</sup>

References	Basis	Claim(s) Challenged
Senzaki <sup>2</sup> and Min <sup>3</sup>	§ 103	1, 2, 7, and 10
Senzaki, Min, and Shin <sup>4</sup>	§ 103	8

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<sup>1</sup> Petitioner also relies on a declaration from Dr. Sanjay Banerjee (Ex. 1003).

<sup>2</sup> U.S. Patent No. 6,537,613 B1, filed Apr. 10, 2000 and issued Mar. 25, 2003 (Ex. 1005).

<sup>3</sup> Jae–Sik Min, et al., *Atomic Layer Deposition of TiN Films by Alternate Supply of Tetrakis (ethylmethyldamino)–Titanium and Ammonia*, 37 JAPANESE JOURNAL OF APPLIED PHYSICS, No. 9A, 1998, pp. 4999–5004 (Ex. 1006).

<sup>4</sup> Korean Patent No. 0156980, published Jan. 28, 1997 (Ex. 1007).

Petitioner presents evidence that Min and Shin are prior art under 35 U.S.C. § 102(b) and Senzaki is prior art under §102(e). Pet. 22 n.10, 25 n.13, 27 n.14 (noting that Shin was published on January 28, 1997). Patent Owner does not, at this stage of the proceeding, challenge the prior art status of any reference.

## II. ANALYSIS

### A. *Claim Construction*

In an *inter partes* review, “[a] claim in an unexpired patent shall be given its broadest reasonable construction in light of the specification of the patent in which it appears.” 37 C.F.R. § 42.100(b); *Cuozzo Speed Techs., LLC v. Lee*, 136 S. Ct. 2131, 2142 (2016) (upholding the use of the broadest reasonable interpretation standard). In determining the broadest reasonable construction, we presume that claim terms carry their ordinary and customary meaning. *See In re Translogic Tech., Inc.*, 504 F.3d 1249, 1257 (Fed. Cir. 2007). A patentee may define a claim term in a manner that differs from its ordinary meaning; however, any special definitions must be set forth in the specification with reasonable clarity, deliberateness, and precision. *See In re Paulsen*, 30 F.3d 1475, 1480 (Fed. Cir. 1994).

Petitioner and Patent Owner do not propose a construction for any claim terms of the ’016 patent. Pet. 21; Prelim. Resp. 22. And, upon review of the parties’ arguments and supporting evidence, we determine that no claim terms require construction for purposes of this Decision. *See Vivid Techs., Inc. v. Am. Sci. & Eng’g, Inc.*, 200 F.3d 795, 803 (Fed. Cir. 1999) (“[O]nly those terms need be construed that are in controversy, and only to the extent necessary to resolve the controversy.”).

*B. Claims 1, 2, 7, and 10 over Senzaki and Min*

Petitioner contends that the subject matter of claims 1, 2, 7, and 10 would have been obvious over Senzaki and Min. Pet. 29–45.

*1. Senzaki*

Senzaki discloses a process for the deposition of “a multiple metal and metalloid compound layer with a compositional gradient of the metal and metalloid.” Ex. 1005, 2:33–36.

In the process of Senzaki, two or more metal–ligand and metalloid–ligand precursors are provided, wherein the ligands are selected from the group consisting of “alkyls, alkoxides, . . . [and] amides.” *Id.* at 2:36–43. These precursors are delivered to a deposition zone to contact a substrate under “deposition conditions.” *Id.* at 2:43–50. The temperature of the deposition is then varied by at least 40° C to deposit a metal/metalloid compound layer having a compositional gradient. *Id.* at 2:50–57.

In one exemplary embodiment, precursors  $\text{Zr}(\text{NEt}_2)_4$  and  $\text{Si}(\text{NMe}_2)_4$  are used to deposit a  $\text{Zr}_x\text{--Si}_y\text{--O}_z$  film. *Id.* at 3:40–42, 5:11–42. Senzaki explains that the metal applied may be selected from a broad range of additional metals, including titanium and hafnium. *Id.* at 4:63–5:3. Senzaki also discloses that “[a]n oxygen source can be added” as a reactant “to result in a metal–metalloid oxide” or “a nitrogen source can be added to result in a metal–metalloid nitride” layer. *Id.* at 2:57–61, 3:63–66.

Although the process is generally described in terms of thermal low pressure CVD, Senzaki explains that the disclosed precursors could also be deposited by atomic layer deposition. *Id.* at 2:50, 4:4–9. Senzaki explains:

In atomic layer deposition, an approximately single layer of precursor molecules are adsorbed on a surface. A second reactant is dosed onto the first precursor layer followed by a

reaction between the second reactant and the first reactant already on the surface. This alternating procedure is repeated to provide the desired thickness of element or compound in a near atomic thickness layer.

*Id.* at 4:9–15.

## 2. *Min*

Min discloses the use of tetrakis(ethylmethylamino)-titanium, or TEMAT, and ammonia in an ALD process to form a TiN film. Ex. 1006, 4999–5000. According to Min, the use of these components in an ALD process results in TiN films “with improved conformality,” which is “crucial for the interconnection of metals in ultralarge-scale integrated circuits.” *Id.* at 4999.

In Min, the disclosed layers are deposited on a SiO<sub>2</sub> substrate via alternate supply of TEMAT and NH<sub>3</sub> in the reactor, with an inert Argon (Ar) pulse used to purge the reactor between each step. *Id.* Figure 2 of Min shows this gas switching method:

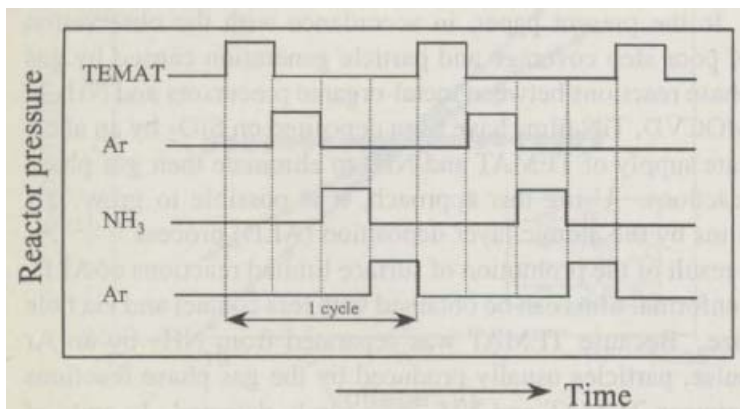


Figure 2 shows the sequential injection of gas reactants in Min. As shown in Figure 2, the reactants of Min are injected into the reactor in the following order: “TEMAT vapor pulse, Ar purge gas pulse, NH<sub>3</sub> gas pulse and Ar purge gas pulse.” *Id.* These four pulses define one cycle and may be

repeated multiple times to achieve a desired film thickness. *Id.* at 4999, 5001, Abstract.

Min explains that at temperatures of 175° C and 200° C, film thickness per cycle is saturated<sup>5</sup> at around 0.5 nm/cycle. *Id.* at 5001. “After saturation level is reached, film thickness per cycle remains constant, and saturation level is independent of both the TEMAT pulse time and the substrate temperature.” *Id.* According to Min, these results indicate that the process is “self-limiting,” which is a “distinct characteristic” of ALD processes. *Id.*

Min notes that “the carbon impurity in films produced by ALD was below” 4%, which “was much lower than the [25%] in films produced at 300°C using TEMAT without NH<sub>3</sub> by MOCVD,” and “slightly lower than the [5%] in film produced at 200°C using TEMAT with NH<sub>3</sub> by MOCVD.” *Id.* at 5002. Min postulates that this reduction in carbon atom concentration in the disclosed ALD process “can be attributed to the post-purge process<sup>6</sup> followed by the introduction of reactant gas.” *Id.*

### 3. Claim 1

Petitioner contends that Senzaki teaches or suggests every limitation of claim 1 of the '016 patent. Pet. 31–35. In particular, Petitioner contends that Senzaki teaches using both a metal dialkylamide (Zr(NEt<sub>2</sub>)<sub>4</sub>) and an oxidant in an ALD process to form an insulating oxide layer on a

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<sup>5</sup> The '016 patent explains that “[n]ormally, in an ALD process, the dose of precursor” delivered “is chosen to be large enough to cause the surface reactions to go to completion (also called ‘saturation’).” Ex. 1001, 22:22–26.

<sup>6</sup> Min describes a “post-TEMAT Ar purge” and a “post-NH<sub>3</sub> Ar purge.” Ex. 1006, 5000. We understand the term “post-purge process” to refer to these Ar purge steps.

microelectronic device. *Id.* Petitioner concedes that Senzaki does not explicitly state that the depositions of the first precursor and the second reactant are self-limiting, but contends Senzaki's disclosure of applying "an approximately single layer of precursor molecules" on the surface of the substrate would indicate to one of ordinary skill in the art that the deposition processes in Senzaki "are or can be made to be self-limiting." *Id.* at 33 (citing Ex. 1003 ¶ 121).

To the extent Senzaki does not expressly disclose that the ALD deposition processes are self-limiting, Petitioner contends one of ordinary skill in the art would have found it obvious to make the disclosed processes self-limiting in view of both Senzaki and Min. *Id.* at 37–42. Petitioner reasons that one of the express goals of Senzaki's ALD process was to provide "a near atomic thickness layer" of the desired element or compound and Min discloses that its ALD method provides a film with a "desired thickness" through the use of a self-limiting process. *Id.* at 38–39.

Patent Owner contends Senzaki and Min would not have rendered the subject matter of claim 1 obvious because Senzaki does not disclose a self-limiting ALD process using a metal alkylamide to form a metal oxide layer and one of ordinary skill in the art would not have sought to modify Senzaki in view of Min to yield such a process. Prelim. Resp. 30–31 (citing Ex. 2101 ¶¶ 179–180). First, Patent Owner contends that Petitioner's asserted combination would "change the cited CVD process described in the examples of Senzaki to an ALD process to control film thickness." Prelim. Resp. 28, 35 (asserting that "a person of ordinary skill in the art would not have modified the cited Senzaki CVD process in view of Min"), 38 ("A person of ordinary skill in the art would have had no reasonable expectation

of success modifying Senzaki's CVD process in view of Min"). 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. Pet. 32–33 (citing Ex. 1005, 4:4–15).

Second, Patent Owner contends that "Senzaki does not disclose any ALD process using a metal alkylamide to form a metal oxide layer." Prelim. Resp. 30–31 (citing Ex. 2101 ¶¶ 179–180). We agree that Senzaki does not disclose an explicit example of forming a metal oxide layer using a metal alkylamide in an ALD process. Senzaki does disclose, however, the use of a  $\text{Zr}(\text{NEt}_2)_4$  *precursor* and an oxidant *reactant* in a CVD process to form a mixed metal/metalloid oxide layer. Ex. 1005, 3:40–43, 3:54–66; Pet. 31–35. Senzaki goes on to explain that, "[i]n addition to thermal low pressure CVD, the above precursors could be used for . . . atomic layer deposition" and that in this ALD process a *precursor* is added to the reactor and then a second *reactant* is dosed onto the first precursor layer to form the desired compositional layer having a "near atomic thickness." Ex. 1005, 4:4–15 (emphases added). Finally, Senzaki indicates that the disclosed metal–metalloid materials are useful as "gate oxides." Ex. 1005, 2:18–30, 3:40–66, 5:15–23. In view of these disclosures, as well as the disclosures of Senzaki as a whole, Petitioner has explained sufficiently where Senzaki teaches or suggests an ALD process using a metal alkylamide to form a metal oxide layer.

Patent Owner also argues that the ALD disclosure of Senzaki is limited to a single paragraph and fails to provide any specifics regarding how an ALD process could be used to form a compositional gradient.

Prelim. Resp. 36, 41–44. And, according to Patent Owner, modifying the deposition temperature in an ALD process to affect the composition of the resulting layer is contrary to Min’s teaching that the growth rate of the deposited film is independent of deposition temperature (at least within the ALD window). *Id.* at 31–33, 44. Patent Owner does not explain, however, why the temperature would necessarily be modified in the ALD process of Senzaki, as opposed to the CVD processes that are the primary focus of the reference.<sup>7</sup> Moreover, a prior art patent is presumed enabled and Patent Owner’s general questions regarding how Senzaki’s ALD process could produce a compositional gradient are not, on this record, sufficient to overcome this presumption. *See In re Antor Media Corp.*, 689 F.3d 1282, 1287–88 (Fed. Cir. 2012) (explaining that “both claimed and unclaimed materials disclosed in a patent are presumptively enabling” and that it is the patentee’s burden to demonstrate otherwise).

Patent Owner also contends that Petitioner has conceded that the deposition processes of Senzaki are not self-limiting. Prelim. Resp. 30–31; Ex. 2101 ¶ 179 (“Petitioner indicates that Senzaki does not disclose wherein deposition of the first reactant component and the second reactant component are self-limiting.”). We do not agree. The Petition states that a person of ordinary skill in the art would have understood that “Senzaki’s

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<sup>7</sup> Patent Owner’s arguments are not without support in Senzaki. For example, in the summary of the invention section the disclosed process includes depositing a precursor using, *inter alia*, “atomic layer deposition,” and then varying the temperature of the second deposition step. *See* Ex. 1005, 2:33–62. Whether this disclosure is separate from the more specific disclosure of ALD without temperature modification provided in the detailed description of the invention section of Senzaki is a question of fact that is better resolved at trial. *Compare id.* at 2:44–57 with *id.* at 4:4–15.

deposition processes are . . . self-limiting.” Pet. 33; Ex. 1003 ¶ 121. It is only “[t]o the extent that [it is determined] that Senzaki does not expressly disclose ‘self-limiting’ depositions,” that Petitioner contends it would have been obvious to make the process of Senzaki self-limiting in view of Min. Pet. 33.

Substantively, the only evidence in support of Patent Owner’s argument that Senzaki’s ALD process is not self-limiting is presented by Dr. Gladfelter. Ex. 2101 ¶ 179. Dr. Gladfelter provides no support or explanation, however, for his conclusion that one of ordinary skill in the art would not have understood Senzaki to disclose a self-limiting ALD process. *Id.* Moreover, Senzaki indicates that the disclosed ALD process results in “a near atomic thickness layer” and Min discloses that “a self-limiting process” is a “distinct characteristic of [an] ALD process.” Ex. 1005, 4:15; Ex. 1006, 5001. Likewise, numerous prior art references indicate that self-limiting growth is a fundamental characteristic of ALD. *See* Ex. 1008, 937 (noting that Atomic Layer Epitaxy<sup>8</sup> (ALE) is characterized by its “self-limiting growth process”), Abstract (“As the film is growing in a self-limiting manner ALE is a promising method to deposit thin, high-quality films for micro- and optoelectronics.”); Ex. 1011, 13121 (“Whatever the name, the basic ALE strategy involves self-limiting surface reactions that provide the atomic layer control of growth.”); Ex. 1012, 837 (“The striking feature of ALD is the saturation of all the reaction and purging steps which makes growth self-limiting.”). And, in its complaint before the District Court, Patent Owner asserted that a “process wherein deposition of the first reactant

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<sup>8</sup> Dr. Banerjee explains that when ALD was originally developed in the 1970s it was referred to as ALE. Ex. 1003 ¶¶ 42–44.

and the second reactant component are self-limiting is part of ALD.” Ex. 1042 ¶ 50. Thus, on this record, Petitioner has demonstrated sufficiently that Senzaki’s ALD process is “self-limiting.”

As noted above, to the extent it is ultimately determined that Senzaki does not teach or suggest a self-limiting process, Petitioner presents arguments and supporting evidence to explain why one of ordinary skill in the art would have found it obvious in view of Min to modify the process of Senzaki to make it self-limiting. Pet. 37–42 (asserting that Min discloses a method for providing strict control over film thickness, which is an express goal of Senzaki). Patent Owner vigorously disputes the factual predicates upon which Petitioner bases its arguments. Prelim. Resp. 31–33 (asserting that Senzaki and Min have directly contrary goals), 33–36 (asserting that one of ordinary skill in the art would not have understood that Senzaki’s CVD process could be modified in a routine way to produce predictable results), 36–38 (asserting that it was not possible to ascertain whether a precursor could be used in an ALD process without extensive experimentation), 38–42 (asserting that a person of ordinary skill in the art would not have had a reasonable expectation of success in modifying the CVD process of Senzaki in view of Min), 42–44 (asserting that it would not have been obvious to modify Senzaki’s CVD process as precursor chemistry is unpredictable and CVD precursors cannot be assumed to work in an ALD process).

The factual dispute presented by the parties, which is supported by the competing testimony of Dr. Banerjee and Dr. Gladfelter, creates a material issue of fact. In such a situation, we must view the competing testimonial evidence “in the light most favorable to the petitioner” when deciding whether to institute an *inter partes* review.

*See* 37 C.F.R. § 42.108(c). And, when the testimonial evidence is viewed in this fashion, we determine that Petitioner has demonstrated sufficiently that one of ordinary skill in the art would have sought to combine Senzaki and Min to use a metal dialkylamide in a self-limiting ALD process to form an insulating oxide layer.

In view of the foregoing, Petitioner has demonstrated a reasonable likelihood that claim 1 of the '016 patent would have been obvious over Senzaki and Min.

*4. Dependent Claims 2, 7, and 10*

Claim 2 depends from claim 1 and further requires “wherein the insulator insulates a gate or a capacitor.” Ex. 1001, 30:27–28. Claim 7 depends from claim 2 and further requires “wherein the metal alkylamide is a zirconium dialkylamide,” and claim 10 depends from claim 7 and further requires “wherein the zirconium dialkylamide is tetrakis(diethylamido)zirconium.” *Id.* at 30:37–38, 30:43–44.

Petitioner contends Senzaki expressly discloses applying the disclosed films as a “gate dielectric” in “IC fabrication,” which would insulate a gate. Pet. 36 (citing Ex. 1005, 3:40–53, 4:4–15, 5:11–42; Ex. 1003 ¶ 127). Petitioner further contends that  $\text{Zr}(\text{NEt}_2)_4$  is tetrakis(diethylamido) zirconium, a zirconium dialkylamide, as recited in claims 10 and 7, respectively. *Id.*

Patent Owner does not directly address Petitioner’s arguments with respect to claims 2, 7, and 10, but generally contends that these claims would not have been obvious over Senzaki and Min because claim 1, from which claims 2, 7, and 10 depend, would not have been obvious over these references. Prelim. Resp. 27–30.

For the reasons set forth above, Petitioner has demonstrated sufficiently that the subject matter of claim 1 is taught or suggested in Senzaki and Min. Petitioner has also identified sufficiently where Senzaki discloses the subject matter of claims 2, 7, and 10. Thus, on this record, Petitioner demonstrates a reasonable likelihood that claims 2, 7, and 10 would have been obvious over Senzaki and Min.<sup>9</sup>

*C. Claim 8 over Senzaki, Min, and Shin*

Claim 8 depends from claim 7 and recites, “wherein the zirconium dialkylamide is tetrakis(ethylmethyamido)zirconium.” Ex. 1001, 30:39–40. Petitioner contends that the subject matter of claim 8 would have been obvious in view of Senzaki, Min, and Shin. Pet. 45–50.

*1. Shin*

Shin discloses precursor compounds used to deposit a metal nitride film over a silicon substrate. Ex. 1007, 1.<sup>10</sup> In particular, Shin disclose the use of an organometallic precursor compound defined by the formula  $M[N(CH_3)C_2H_5]X$ , wherein M is selected from the metallic elements

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<sup>9</sup> Petitioner also contends that it would have been obvious to use a hafnium dialkylamide precursor, as recited in claim 3, in Senzaki’s ALD process. Pet. 37. Petitioner does not assert a ground with respect to claim 3. Nor does Petitioner address claim 3 in its claim analysis or identify a prior art reference that teaches or suggests the use of hafnium dialkylamide precursors in an ALD process. Indeed, in another portion of the Petition Petitioner concedes that “Senzaki does not disclose hafnium-based precursors.” *Id.* at 31. Accordingly, to the extent Petitioner intended to include claim 3 within its ground based on Senzaki and Min, Petitioner has not demonstrated sufficiently that claim 3 would have been obvious over these references.

<sup>10</sup> Citations to Shin are to page numbers provided in the lower right corner of the document.

belonging to group 3, group 4, and group 5 on the periodic table, and X is an integer between 3–5. *Id.* at 1–2, 7.

Shin discloses that any metallic element within groups 3–5 on the periodic table is “suitable,” but focuses its discussion primarily on the deposition of titanium films. *Id.* at 7. Shin notes that tetrakis diethyl amino amino titanium and tetrakis dimethyl amino amino titanium were widely used as precursors for the deposition of titanium nitride thin films. *Id.* Use of the dimethyl form, however, was found to result in carbon impurities “of about 27–30% within the film,” and use of the diethyl form, although it “showed a decrease of 15–25% in the content of carbon impurity and an increase in the speed of deposition,” was found to be inferior to the use of the dimethyl form with respect to depositing a thin film with an even thickness. *Id.* at 5–6.

Shin discloses that tetrakis(ethylmethyamido) titanium “exhibits the intermediate characteristics” of tetrakis dimethyl amino amino titanium and tetrakis diethyl amino amino titanium, and can “neutralize more or less” the problems caused by those two compounds. *Id.* at 7. Shin also notes that the disclosed compounds “can be made easily,” and provides an example process for forming both tetrakis(ethylmethyamino) titanium and tetrakis(ethylmethyamino) zirconium. *Id.* at 7–9.

## 2. *Analysis*

Petitioner asserts that Shin expressly discloses tetrakis(ethylmethyamino) zirconium,<sup>11</sup> as recited in claim 8. Pet. 27–28,

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<sup>11</sup> Shin discloses tetrakis(ethylmethyamido) zirconium, whereas the claims recite tetrakis(ethylmethyamino) zirconium (tetrakis vs. tetrakis). Neither party asserts that there is a difference between these two compounds.

46–47. Petitioner further asserts that a person of ordinary skill in the art would have sought to use this compound in the ALD process of Senzaki and Min. In support, Dr. Banerjee testifies that Shin’s disclosure that the –ethylmethylamino form of a transition metal dialkylamide “exhibits the intermediate characteristics of” the –diethylamino and –dimethylamino forms of the same transition metal dialkylamides, while neutralizing the shortcomings of these compounds, would have suggested to one of ordinary skill in the art to try this intermediate form in Senzaki’s ALD process. Ex. 1003 ¶¶ 142–143; Pet. 48–49.

Patent Owner responds that changing even a single alkyl group can result in unpredictable changes in the properties of a compound and “could affect a precursor’s applicability” in an ALD process. Prelim. Resp. 46–47 (citing Ex. 2101 ¶ 220). In support, Patent Owner notes that Table 1 of the ’016 patent shows that  $\text{Hf}(\text{NEt}_2)_4$ , which differs from  $\text{Hf}(\text{NMe}_2)_4$  by only one alkyl group, is liquid at room temperature whereas  $\text{Hf}(\text{NMe}_2)_4$  is a solid. *Id.* at 47 (citing Ex. 2116, Table 1; Ex. 2101 ¶ 220).

As noted by Patent Owner, Table 1 of the ’016 patent indicates that  $\text{Hf}(\text{NEt}_2)_4$  is liquid at room temperature and that  $\text{Hf}(\text{NMe}_2)_4$  does not become a liquid until it reaches 30° C. Ex. 1001, Table 1. Neither Patent Owner nor Dr. Gladfelter explains sufficiently, however, why this result is unpredictable. Nor do they explain why the phase of a precursor at room temperature is relevant to whether that precursor will be useful in an ALD process; indeed, the ’016 patent indicates that  $\text{Hf}(\text{NMe}_2)_4$  may be used successfully in an ALD process to form  $\text{HfO}_2$ . *Id.* at 26:45–27:43.

Patent Owner also argues that Shin is directed to CVD and not ALD and “[j]ust because one CVD precursor described in Shin was found to be

successful in achieving self-limiting ALD, it does not mean that the rest of the CVD precursors in Shin will work in ALD.” Prelim. Resp. 47–50. Here, Shin discloses the use of both tetrakis(ethylmethyl)titanium and zirconium and discloses that the ethylmethyl– form provides advantages over the diethyl and dimethyl forms in a CVD process. Ex. 1007, 7, Abstract.

Whether these disclosures, when combined with the disclosures of Senzaki and Min, would have given an ordinary artisan a reasonable expectation of success in using tetrakis(ethylmethyl)zirconium in the ALD process of Senzaki to produce an oxide insulating layer, as asserted by Petitioner and Dr. Banerjee, is a question of material fact that is best resolved at trial.

As noted above, Petitioner provides an explanation, supported by both documentary and testimonial evidence, as to why one of ordinary skill in the art would have sought to combine Senzaki, Min, and Shin to result in the process of claim 8. And, although Patent Owner vigorously contests the factual predicates underlying this explanation, on this record, and given the requirement that we view the testimonial evidence in the light most favorable to Petitioner at this stage of the proceeding, *see* 37 C.F.R. § 42.108(c), we determine that Petitioner has demonstrated a reasonable likelihood that claim 8 of the ’016 patent would have been obvious over Senzaki, Min, and Shin.

### III. CONCLUSION

Upon consideration of the Petition and the Preliminary Response, we conclude that Petitioner has demonstrated a reasonable likelihood that claims 1, 2, 7, 8, and 10 would have been obvious over the recited prior art.

Accordingly, we institute *inter partes* review with respect to these claims as set forth in the Order.

#### IV. ORDER

It is hereby

ORDERED that, pursuant to 35 U.S.C. § 314, an *inter partes* review is instituted on the following grounds:

(1) Whether claims 1, 2, 7, and 10 are unpatentable under 35 U.S.C. § 103 as having been obvious over Senzaki and Min; and

(2) Whether claim 8 is unpatentable under 35 U.S.C. § 103 as having been obvious over Senzaki, Min, and Shin;

FURTHER ORDERED that no ground other than those specifically granted above are authorized for *inter partes* review as to the claims of the '016 patent.

IPR2017-00664  
Patent 8,334,016 B2

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