

UNITED STATES PATENT AND TRADEMARK OFFICE

BEFORE THE PATENT TRIAL AND APPEAL BOARD

BASF CORPORATION,
Petitioner

v.

ENTHONE, INC.
Patent Owner

Case IPR2016-00696
Patent 7,303,992 B2

Before KEVIN F. TURNER, SHERIDAN K. SNEDDEN, and
KEVIN W. CHERRY, *Administrative Patent Judges*.

TURNER, *Administrative Patent Judge*

DECISION
Final Written Decision
35 U.S.C. § 318(a) and 37 C.F.R. § 42.73

I. INTRODUCTION

This *inter partes* review, instituted pursuant to 35 U.S.C. § 314, challenges the patentability of claims 1–15, 17–22, and 26–28 of U.S. Patent No. 7,303,992 B2 (Exhibit 1001, “the ’992 Patent”), owned by Patent Owner Enthone, Inc. (“Enthone”). We have jurisdiction under 35 U.S.C. § 6. This Final Written Decision is entered pursuant to 35 U.S.C. § 318(a) and 37 C.F.R. § 42.73.

For the reasons discussed below, Petitioner has not shown by a preponderance of the evidence that claims 1–15, 17–22, and 26–28 of the ’992 Patent are unpatentable.

A. Procedural History

BASF Corporation (hereinafter, “BASF”) filed a corrected petition (Paper 4, “Pet.”) for *inter partes* review of claims 1–15, 17–22, and 26–28 of the challenged patent. Enthone filed a Preliminary Response. Paper 8 (“Prelim. Resp.”).

We instituted an *inter partes* review of claims 1–15, 17–22, and 26–28 (“the instituted claims”) of the ’992 Patent as unpatentable under 35 U.S.C. § 103(a)¹ over the following references. Paper 10 (“Institution Decision” or “Dec.”).

¹ The Leahy-Smith America Invents Act (“AIA”), Pub. L. No. 112-29, 125 Stat. 284, 287–88 (2011), revised 35 U.S.C. § 103, effective March 16, 2013. Because the challenged patent was filed before March 16, 2013, we refer to the pre-AIA version of § 103 in this Decision.

References	Claims Challenged
Barstad ² , Alling ³ , and BASF Catalog ⁴	1–15, 17–22, and 26–28
Morrissey ⁵ , Alling, and BASF Catalog	1–15, 17–22, and 26–28

Dec. 24.

Subsequent to institution, Enthone filed a Patent Owner Response to the Petition (Paper 17, “PO Resp.”) and BASF filed a Reply to Patent Owner’s Response (Paper 20, “Reply”). Enthone also filed objections to BASF’s exhibits (Paper 21) but did not file a motion to exclude any exhibits; given the outcome of this Final Written Decision, we determine Enthone’s objections to be moot.

An oral hearing was held on May 10, 2017. A transcript of the hearing has been entered into the record. Paper 30 (“Tr.”).

B. Related Matters

BASF indicates that the ’992 Patent was asserted in *Enthone, Inc. v. BASF Corp.*, Case No. 1:15-cv-233-TJM/RFT, in the U.S. District Court for the Northern District of New York. Pet. 1; *see also* Prelim. Resp. 21. U.S. Patent No. 7,815,786 B2 (“the ’786 Patent”), which issued from a divisional application based on the patent application that issued as the ’992 Patent,

² US Patent No. 6,444,110 B2, filed May 17, 1999, issued September 3, 2002 (Ex. 1003, “Barstad”).

³ US Patent Publication No. 2002/0127847 A1, filed November 3, 2001, published September 12, 2002 (Ex. 1006, “Alling”).

⁴ BASF, Pluronic and Tetronic Block Copolymer Surfactants (1989) (Ex. 1005, “BASF Catalog”).

⁵ US Patent Publication No. 2002/0043467 A1, filed October 12, 2001, published April 18, 2002 (Ex. 1004, “Morrissey”).

was also asserted in that civil action. Pet. 1. The '786 Patent is also the subject of an *inter partes* review in Case IPR2016-00697, instituted concurrently with the instant proceeding.

Previously, the '992 Patent and the '786 Patent were subject to requests for *inter partes* review, namely Cases IPR2014-00243 and IPR2014-00246, respectively, by Moses Lake Industries, Inc., the petitioner therein. We declined to institute review based on the petitions filed. See IPR2014-00243, Paper 6 ("243 Dec."). Subsequently, Moses Lake Industries, Inc. filed additional petitions directed to the same patents, Cases IPR2014-01394 and IPR2014-01395, but no institution decision was made because the parties settled and the requests were terminated.

C. The '992 Patent

The '992 Patent relates to a method for electrolytically plating copper onto a substrate having submicron-sized interconnect features using a source of copper ions and suppressor compound having polyether groups. Ex. 1001, Abs. The '992 Patent discusses known systems that rely on so-called "superfilling" or "bottom-up growth" to deposit copper into high aspect ratio features, where the superfilling involves filling a feature from the bottom up, rather than at an equal rate on all its surfaces, to avoid seams and pinching off that can result in voiding. *Id.* at 2:6–11. The '992 Patent discloses a suppressor compound formed from a combination of propylene oxide (PO) repeat units and ethylene oxide (EO) repeat units present in a PO:EO ratio between about 1:9 and about 9:1 and bonded to a nitrogen-

containing species, wherein the molecular weight of the suppressor compound is between about 1000 and about 30,000. *Id.* at 3:25–30.

D. Illustrative Claims

Claims 1, 2, and 17 of the challenged claims of the '992 Patent are independent. Claims 1 and 17 are illustrative and are reproduced below, with emphases added:

1. A method for electroplating a copper deposit onto a semiconductor integrated circuit device substrate with electrical interconnect features including submicron-sized features having bottoms, sidewalls, and top openings, the method comprising:

immersing the semiconductor integrated circuit device substrate including submicron-sized features having bottoms, sidewalls, and top openings wherein said submicron-sized features include high aspect ratio features having dimensions such that the high aspect ratio features have aspect ratios of at least about 3:1 into an electrolytic plating composition comprising a source of Cu ions in an amount sufficient to electrolytically deposit Cu onto the substrate and into the electrical interconnect features and *a polyether suppressor compound comprising a combination of propylene oxide (PO) repeat units and ethylene oxide (EO) repeat units present in a PO:EO ratio between about 1:9 and about 9:1 and bonded to a nitrogen-containing species, wherein the molecular weight of the suppressor compound is between about 1000 and about 30,000;* and

supplying electrical current to the electrolytic composition to deposit Cu onto the substrate and *superfill the submicron-sized features by rapid bottom-up deposition at a rate of growth in the vertical direction which is greater than a rate of growth in the horizontal direction.*

17. A method for electroplating a copper deposit onto a semiconductor integrated circuit device substrate with electrical interconnect features including submicron-sized features having bottoms, sidewalls, and top openings, the method comprising:

immersing the semiconductor integrated circuit device substrate into the electrolytic plating composition comprising a source of Cu ions in an amount sufficient to electrolytically deposit Cu onto the substrate and into the electrical interconnect features, an accelerator, and a suppressor; and

supplying electrical current to the electrolytic composition to deposit Cu onto the substrate and *superfill the submicron-sized features by rapid bottom-up deposition at a vertical Cu deposition growth rate in features from the bottoms of the features to the top openings of the features which is greater than 15 times faster than a field deposition growth rate on substrate surfaces outside the features.*

Ex. 1001, 18:19–46, 20:40–59.

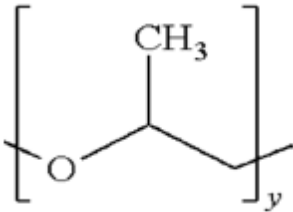
II. DISCUSSION

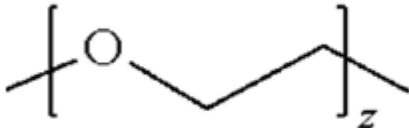
A. Claim Construction

In an *inter partes* review, the Board interprets claims in an unexpired patent using the “broadest reasonable construction in light of the specification of the patent in which [they] appear[.]” 37 C.F.R. § 42.100(b); see *Cuozzo Speed Techs., LLC v. Lee*, 136 S. Ct. 2131, 2144–46 (2016) (upholding the use of broadest reasonable construction standard). Consistent with the broadest reasonable construction standard, claim terms are presumed to have their ordinary and customary meaning as understood by one of ordinary skill in the art in the context of the entire patent disclosure at the time of the invention. *In re Translogic Tech., Inc.*, 504 F.3d 1249, 1257

(Fed. Cir. 2007). An inventor may provide a meaning for a term that is different from its ordinary meaning by defining the term in the specification with “reasonable clarity, deliberateness, and precision.” *In re Paulsen*, 30 F.3d 1475, 1480 (Fed. Cir. 1994). Moreover, limitations are not to be read from the specification into the claims. *In re Van Geuns*, 988 F.2d 1181, 1184 (Fed. Cir. 1993). In addition, the Board may not “construe claims during [an *inter partes* review] so broadly that its constructions are *unreasonable* under general claim construction principles.” *Microsoft Corp. v. Proxyconn, Inc.*, 789 F.3d 1292, 1298 (Fed. Cir. 2015). We construe the challenged claims according to these principles.

In the Institution Decision, we construed the following terms and adopted the following constructions:

Claim term	Adopted Construction
“suppressor”	compound that inhibits electrolytic plating and facilitates superfilling of features in a system having an accelerator
“superfill”	filling a feature from the bottom up, rather than at an equal rate on all its surfaces, to avoid seams and pinching off that can result in voiding
“propylene oxide (PO) repeat units”	the chemical structure represented by the formula C ₃ H ₆ O with the following structure reproduced below:  The diagram shows the chemical structure of a propylene oxide repeat unit. It consists of a three-membered ring containing an oxygen atom (O) and two carbon atoms. One carbon atom is bonded to a methyl group (CH ₃). The entire ring is enclosed in large square brackets with a subscript 'y' at the bottom right. Single bonds extend from the two carbon atoms in the ring through the brackets, indicating the polymer chain continues.

“ethylene oxide (EO) repeat units”	the chemical structure represented by the formula C_2H_4O with the following structure reproduced below:  The diagram shows a chemical structure of an ethylene oxide repeat unit. It consists of an oxygen atom (represented by a circle) connected to two carbon atoms (represented by vertices in a zigzag line). The entire unit is enclosed in brackets with a subscript 'z' to indicate it is a polymer repeat unit.
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Dec. 7–8. In Patent Owner’s Response, Enthone asserts that we “properly” construed the subject claim limitations, adding that “[i]n the context of superfilling, those skilled in the art use the term ‘suppressor’ to identify a class of compounds with shared functional attributes, rather than specific structural features.” PO Resp. 25 (citations omitted). BASF’s Reply does not explicitly address the construction of any claim terms.

We continue to adopt the above-noted constructions for purposes of this Decision. To the extent it is necessary for us to construe other claim terms in this Decision, we do so below in the context of analyzing whether the prior art renders the claims unpatentable.

B. Principles of Law

To prevail in challenging Patent Owner’s claims, Petitioner must demonstrate by a preponderance of the evidence that the claims are unpatentable. 35 U.S.C. § 316(e); 37 C.F.R. § 42.1(d). “In an [*inter partes* review], the petitioner has the burden from the onset to show with particularity why the patent it challenges is unpatentable.” *Harmonic Inc. v. Avid Tech., Inc.*, 815 F.3d 1356, 1363 (Fed. Cir. 2016) (citing 35 U.S.C. § 312(a)(3) (requiring *inter partes* review petitions to identify “with

particularity . . . the evidence that supports the grounds for the challenge to each claim”)). This burden never shifts to Patent Owner. *See Dynamic Drinkware, LLC v. Nat’l Graphics, Inc.*, 800 F.3d 1375, 1378 (Fed. Cir. 2015) (citing *Tech. Licensing Corp. v. Videotek, Inc.*, 545 F.3d 1316, 1326–27 (Fed. Cir. 2008)) (discussing the burden of proof in *inter partes* review). Furthermore, Petitioner cannot satisfy its burden of proving obviousness by employing “mere conclusory statements.” *In re Magnum Oil Tools Int’l, Ltd.*, 829 F.3d 1364, 1380 (Fed. Cir. 2016).

Petitioner has asserted each of the challenged claims of the ’992 Patent is unpatentable under 35 U.S.C. § 103(a) over various references. A claim is unpatentable under 35 U.S.C. § 103(a) if the differences between the claimed subject matter and the prior art are such that the subject matter, as a whole, would have been obvious at the time of the invention to a person having ordinary skill in the art. *KSR Int’l Co. v. Teleflex, Inc.*, 550 U.S. 398, 406 (2007). The question of obviousness is resolved based on underlying factual determinations, including: (1) the scope and content of the prior art; (2) any differences between the claimed subject matter and the prior art; (3) the level of ordinary skill in the art; and (4) objective evidence of nonobviousness. *Graham v. John Deere Co.*, 383 U.S. 1, 17–18 (1966). “A determination of whether a patent claim is invalid as obvious under § 103 requires consideration of all four *Graham* factors, and it is error to reach a conclusion of obviousness until all those factors are considered.” *Apple v. Samsung Elecs. Co., Ltd.*, 839 F.3d 1034, 1048 (Fed. Cir. 2016) (en banc) (citations omitted). “This requirement is in recognition of the fact that each

of the *Graham* factors helps inform the ultimate obviousness determination.”
Id.

C. Level of Ordinary Skill

In determining whether an invention would have been obvious at the time it was made, 35 U.S.C. § 103 requires us to resolve the level of ordinary skill in the pertinent art at the time of the invention. *Graham*, 383 U.S. at 17. “The importance of resolving the level of ordinary skill in the art lies in the necessity of maintaining objectivity in the obviousness inquiry.” *Ryko Mfg. Co. v. Nu-Star, Inc.*, 950 F.2d 714, 718 (Fed. Cir. 1991). The person of ordinary skill in the art is a hypothetical person who is presumed to have known the relevant art at the time of the invention. *In re GPAC, Inc.*, 57 F.3d 1573, 1579 (Fed. Cir. 1995). The level of ordinary skill in the art is reflected by the prior art of record. *Okajima v. Bourdeau*, 261 F.3d 1350, 1355 (Fed. Cir. 2001).

Factors that may be considered in determining the level of ordinary skill in the art include, but are not limited to, the types of problems encountered in the art, the sophistication of the technology, and educational level of active workers in the field. *Id.* In a given case, one or more factors may predominate. *Id.* Generally, it is easier to establish obviousness under a higher level of ordinary skill in the art. *Innovation Toys, LLC v. MGA Entm’t, Inc.*, 637 F.3d 1314, 1323 (Fed. Cir. 2011) (“A less sophisticated level of skill generally favors a determination of nonobviousness . . . while a higher level of skill favors the reverse.”).

In its Petition, BASF argued that one of ordinary skill in the art “would have had at least a bachelor’s degree in materials science, chemistry, or a related discipline, and would have had approximately two or more years of experience as a process engineer.” Pet. 18 (citing Ex. 1002 ¶ 9). BASF continued that such a person “would be capable of reading and understanding the open literature (including patents) related to electroplating processes for semiconductor manufacturing and would be able to modify an existing process based on the teachings found in the open literature.” *Id.* In Patent Owner’s Response, Enthone argues that such a definition “would not require any expertise or experience in the field relating to the ‘992 patent,” and that such a person “would have understood the fundamental differences between processes and compositions generally used in electrolytic plating and those used for superfilling submicron vias or trenches with high aspect ratios. PO Resp. 43–44 (citing Ex. 2006, 2–3). In the Institution Decision, we did not explicitly define one of ordinary skill in the art, but we characterized how such skilled artisans would have viewed the cited references in our analysis. See Dec. 13, 15–17.

In Patent Owner’s Response, Enthone asserts that BASF’s declarant, Dr. Bruce Pound, admitted that he did not have relevant experience in the field relating to the ‘992 Patent. PO Resp. 43–44 (citing Ex. 2038, 22:11–23:9). Enthone also argues that Dr. Pound, based on his testimony and background, lacks a general understanding of the superfilling mechanisms. *Id.* at 43, n.6. Enthone also argues that persons of ordinary skill in the art “would have appreciated that it was unpredictable whether a given molecule

other than the nonionic compounds used at the time (*i.e.* a polyoxyalkylene or an alkoxyated alcohol) would have served as a ‘suppressor’ effective for superfilling.” *Id.* at 44 (citing Ex. 2015, C492; Ex. 2016, D156; Ex. 2017, D632; Ex. 2031 ¶¶ 23–40, 46–47). As well, Enthone argues that its declarant, Dr. Dale Barkey, is better equipped to evaluate issues from the viewpoint of one of ordinary skill in the art because of his research and experience. *Id.* at 44, n.7.

In its Reply, BASF argues that the prior art of record reflects the level of ordinary skill in the art and that the parties generally agree on the education and experience level of such skilled artisans. Reply 6–7. BASF takes issue, however, with what it sees as Enthone’s attempting to have such experience “be in a very narrow field.” *Id.* at 7. BASF also argues that this narrowing of the relevant field is an attempt to cast Dr. Barkey “as the only potential expert,” and that both declarants have similar experience and both are “more than capable of understanding and applying the literature and scientific principles relevant to the ‘992 Patent.” *Id.* at 7–8.

Based on the arguments of the parties, we agree that one of ordinary skill in the art would have had at least a bachelor’s degree in materials science, chemistry, or a related discipline, and would have had approximately two or more years of experience as a process engineer. We are further persuaded that such skilled artisans would have had to understood the differences between conformal and superfilling electrolytic plating, and that they would have appreciated that it was unpredictable whether a given molecule would have served as a suppressor effective for superfilling. This

was conformed at Oral Hearing, where counsel for BASF acknowledged that it was not uncommon to try a plating bath component, even if the mechanisms of how it may work are not fully understood. See Tr. 25.

E. Asserted Grounds of Obviousness of Claims 1–15, 17–22, and 26–28 over Barstad, Morrissey, Alling, and BASF Catalog

BASF asserts that claims 1–15, 17–22, and 26–28 are unpatentable under 35 U.S.C. § 103 as obvious over the combination of Barstad, Alling, and BASF Catalog. Pet. 20–32. BASF also asserts that claims 1–15, 17–22, and 26–28 are unpatentable under 35 U.S.C. § 103 as obvious over the combination of Morrissey, Alling, and BASF Catalog. *Id.* at 32–43. We consider these grounds together as we determine the issues of whether it would have been obvious to incorporate teachings from Alling and BASF Catalog into either Barstad or Morrissey to be the same.

At Oral Hearing, counsel for BASF indicated that the independent claims of the '992 Patent would stand or fall together, so that all of the claims could be found to be unpatentable or patentable together. Tr. 11. As such, we need only consider claims 1 and 17 to determine the patentability of the challenged claims.

We begin with a brief review of the cited references, BASF's contentions and Enthone's counterarguments.

1. Summary of Asserted Prior Art

a. Summary of Barstad

Barstad discloses compositions and methods for copper electroplating that are particularly effective in difficult to plate areas having high aspect

ratios and small diameter vias. Ex. 1003, Abs. The processes are applicable to plating a wide variety of articles, including printed circuit boards and other electronic packaging devices. *Id.* at 2:45–48. The compositions disclosed are “particularly useful for filling microvias and trenches required by current and anticipated semiconductor fabrication requirements (including microvias having aspect ratios of at least 4:1 and diameters of 200 nm or less) by reliably plating copper deposits that are essentially or completely free of voids, inclusions or other plating imperfections.” *Id.* at 2:48–54.

Barstad discloses that the brightener concentration in the preferred electroplating compositions should be maintained and that a “surfactant-type suppressor agent” should be included in the electroplating compositions to achieve bottom-filling deposition. *Id.* at 3:8–25. Barstad also provides that “the suppressor enables enhanced plating rate at the bottom of a microvia, permitting copper to plate the entire aperture space in a substantially ‘bottom-fill’ manner, without premature sealing of the aperture top that can result in inclusions or voids.” *Id.* at 3:26–30. Barstad also discloses:

Particularly suitable surfactants for plating compositions of the invention are commercially available polyethylene glycol copolymers, including polyethylene glycol copolymers. Such polymers are available from e.g. BASF (sold by BASF under Tetronic and Pluronic tradenames), and copolymers from Chemax.

Id. at 6:51–56.

b. Summary of Morrissey

Morrissey discloses a process for electroplating copper onto submicron, high-aspect-ratio features of a semiconductor integrated circuit device substrate, while minimizing the concentrations of the organic additives in the plating bath. Ex. 1004 ¶¶ 11, 47. Morrissey seeks to achieve this through use of two different acids in the bath. *Id.* at Abs. Its plating compositions “are particularly useful to plate difficult work pieces, such as circuit board substrates with small diameter, high aspect ratio microvias and other apertures.” *Id.* ¶ 46. Morrissey notes that surfactants may be added to the electroplating baths, with particularly suitable surfactants containing Tetronic[®] polymers. *Id.* ¶ 42.

c. Summary of Alling

Alling discloses compositions and methods for electrolytic deposition of metal, such as copper, onto semiconductor wafers. Ex. 1006, Abs. Alling describes a process in which a conductive metal and a more resistive metal may be plated in alternating layers using the same plating bath. *Id.* Alling details that “[p]articularly suitable suppressor agents for plating compositions of the invention are commercially available polyethylene glycol copolymers, including polyethylene glycol copolymers. Such polymers are available from e.g. BASF (sold by BASF under Tetronic[®] and Pluronic[®] tradenames).” *Id.* ¶ 43. Alling further states that such suppressor agents are typically added to copper electroplating solutions in a wide range of concentrations between 1 and 10,000 ppm, preferably 5 to 10,000 ppm. *Id.* ¶ 44.

d. BASF Catalog

BASF Catalog is a brochure identifying various Pluronic[®] and Tetronic[®] surfactant products. These surfactant products are block copolymers of ethylene oxide and propylene oxide. Ex. 1005, 3. BASF Catalog notes that “BASF surfactants range from flowable liquids of varying viscosities to pastes, prills, and cast solids, with molecular weights from 1,000 to 30,000.” *Id.* It also describes a spectrum of surfactant products with varying properties: “[t]his brochure covers four block copolymer types—a total of 90 different products—that provide formulating chemists with a broad range of physical properties and surfactant functions to meet targeted performance requirements.” *Id.*

With respect to BASF Catalog, BASF contends that “[t]he last digit of the Tetronic[®] product number, multiplied by 10, is the weight percent of EO in the specific molecule.” Pet. 10 (citing Ex. 1007, 111). BASF’s expert, Dr. Pound, calculated the PO:EO ratios for each Tetronic[®] product, with his calculations provided in Table 1 of his Declaration. See Pet. 10–11. Enthone accepts the PO:EO ratios of the Tetronic[®] surfactants as computed by BASF. Prelim. Resp. 21. As such, we accept the calculated the PO:EO ratios for the listed products in BASF Catalog for purposes of this Decision.

2. BASF’s Contentions

BASF argues that Barstad discloses copper electroplating processes for filling high-aspect ratio, sub-micron features, such as vias or trenches, where the solutions employed may contain accelerators and suppressors, and that both Barstad and Alling teach that their electroplating compositions may

contain Tetronic[®] polymers. Pet. 14–15. BASF also argues that Alling provides that Tetronic[®] polymers are particularly suitable as suppressor agents and that BASF Catalog provides for compounds having PO:EO ratios that fall within the claimed ranges. *Id.* at 15. BASF also acknowledges that Barstad does not disclose the use of Tetronic[®] polymers as suppressor agents in its electroplating solution, but argues that it would have been obvious to a person of ordinary skill in the art in view of Alling and BASF Catalog. *Id.* at 15–16. BASF also argues that the genus of Tetronic[®] products is small, that all Tetronic[®] polymers are comprised of propylene oxide and ethylene oxide groups, have the same general chemical structure, and therefore would be expected to behave similarly, and Alling does not identify any Tetronic[®] polymers that would not work. *Id.* at 16.

In addition, with respect to the claimed relative growth rates of copper in certain areas with respect to other areas, BASF argues that the suppressor achieving those relative rates disclosed in the '992 Patent has the same chemical structure and molecular weight as Tetronic[®] 704, one of the Tetronic[®] products listed in the BASF Catalog. *Id.* at 17. Based on this, BASF argues that one of ordinary skill in the art using Tetronic[®] polymers as suppressors, as taught by Alling, would have arrived at the claimed vertical growth rates recited in the claims. *Id.* BASF also argues that the claimed concentrations of “polyether suppressor” lies within the range expressly taught in Alling and that the claimed suppressor concentration range would have been obvious. *Id.* at 17–18.

BASF also argues that a person of ordinary skill in the art would have been motivated to use Tetronic[®] polymers as suppressors in Barstad's compositions and that a person of ordinary skill in the art would have consulted BASF Catalog to identify the available Tetronic[®] polymers. *Id.* at 19 (citing Ex. 1002 ¶¶ 20, 29).

With respect to the second ground, BASF argues that Morrissey discloses copper electroplating processes for filling high-aspect ratio, sub-micron features, such as vias or trenches, where the solutions employed may contain accelerators and suppressors, and that both Morrissey and Alling teach that their electroplating compositions may contain Tetronic[®] polymers. Pet. 14–15. BASF also argues that Alling provides that Tetronic[®] polymers are particularly suitable as suppressor agents and that BASF Catalog provides for compounds having PO:EO ratios that fall within the claimed ranges. *Id.* at 15. BASF also acknowledges that Morrissey does not disclose the use of Tetronic[®] polymers as suppressor agents in its electroplating solution, but argues that it would have been obvious to a person of ordinary skill in the art in view of Alling and BASF Catalog. *Id.* at 15–16. BASF also argues that the genus of Tetronic[®] products is small, that all Tetronic[®] polymers are comprised of propylene oxide and ethylene oxide groups, have the same general chemical structure, and therefore would be expected to behave similarly, and Alling does not identify any Tetronic[®] polymers that would not work. *Id.* at 16.

BASF also argues that a person of ordinary skill in the art would have been motivated to use Tetronic[®] polymers as suppressors in Morrissey's

compositions and that a person of ordinary skill in the art would have consulted BASF Catalog to identify the available Tetronic® polymers. Pet. 19 (citing Ex. 1002 ¶¶ 20, 29).

3. Enthone's Contentions

Enthone contends that to combine Alling with Barstad or Morrissey, “BASF must demonstrate that a person of ordinary skill [in the art] would have had (1) a motivation or other reason to use a Tetronic surfactant (meeting the structural requirements of the claim—*e.g.*, PO:EO ratio, molecular weight) in place of the suppressor used in its primary reference (Barstad or Morrissey), and (2) a reasonable expectation that this compound would function as a ‘suppressor’ for superfilling.” PO Resp. 26 (citing *Intelligent Bio-Systems, Inc. v. Illumina Cambridge Ltd.*, 821 F.3d 1359, 1367-78 (Fed. Cir. 2016)).

4. Analysis

We are persuaded that BASF must demonstrate a specific motivation or reason to use a specific compound or series of compounds, with a reasonable expectation that the plating bath components would allow for superfilling. Independent claims 1 and 17 make clear that the methods must provide for superfilling: claim 1 provides that it must “superfill the submicron-sized features by rapid bottom-up deposition at a rate of growth in the vertical direction which is greater than a rate of growth in the horizontal direction,” and claim 17 provides that it must “superfill the submicron-sized features by rapid bottom-up deposition at a vertical Cu deposition growth rate in features from the bottoms of the features to the top

openings of the features which is greater than 15 times faster than a field deposition growth rate on substrate surfaces outside the features.” Thus, any combination must have a reasonable expectation of success in achieving the results required by the claims. Although Barstad and Morrissey both disclose superfilling, as discussed above, a person of ordinary skill in the art must have a reasonable expectation that any changes to the plating bath components, as contemplated by the instant grounds of unpatentability, will leave the ability to provide superfilling undisturbed.

Enthone further contends that the literature pre-dating the '992 Patent demonstrates that “[t]he complexity and unpredictability of the chemistry—compounded by the complexity of the superfilling mechanism—obviated any basis for predicting the effectiveness for superfilling of any suppressor whose structure or properties varied in any material respect from suppressors that had already been demonstrated.” PO Resp. 9–18. BASF agrees with this characterization, to some extent, acknowledging that there is not a known mechanism to look at the structure of a compound and know that it will work as a component of a plating bath, but counters that would not be an impediment to trying that plating bath component. Tr. 73:13–75:7. We determine that Enthone’s evidence of unpredictability is persuasive and entitled to substantial weight, and demonstrates that a person of ordinary skill in the art would not have had any basis for predicting the effectiveness for superfilling of any suppressor whose structure or properties varied in any material respect from suppressors that had already been demonstrated. *See, e.g.*, Ex. 2031 ¶¶ 22–40; Ex. 1009, 2978; Ex. 2010, C270–C271; Ex. 2011,

C702; Ex. 2015, C2492; Ex. 2016, D156; Ex. 2017, D632. Based on this unpredictability, we are persuaded that there must be some specific motivation to employ specific plating bath components, given the general unpredictability in the field and the art.

Enthone contends that Alling is not directed to a process for superfilling features of a semiconductor substrate. PO Resp. 36–42. As Enthone points out, Alling fails to specifically address submicron features, superfilling, bottom-up filling, aspect ratios, voids, or seam formation. PO Resp. 36 (citing Ex. 1006; Ex. 2031 ¶¶ 69, 92). Although it is true that Alling addresses “the need for copper electrodeposition in semiconductor chip manufacture, including ‘chip interconnects . . . at dimensions of 200 nm and less’” (Reply 15 (citing Ex. 1006 ¶¶ 6, 7)), we do not agree that this necessarily implies superfilling. The dimensions disclosed need not connote vias or features with high aspect ratios, such that we are not persuaded that the mentioning of such dimensions would necessarily suggest superfilling. BASF also points to the testimony of Dr. Barkey, providing that superfilling would be necessary for the successful electrodeposition of copper in chip interconnects of the size addressed in Alling. Reply 15 (citing Ex. 1028, 154:20–155:3). Although we accept Dr. Barkey’s testimony as being correct, we credit Dr. Barkey’s other testimony regarding Alling that the mentioning of chip interconnects at dimensions of 200 nm and less in Alling would not have informed one of ordinary skill in the art that Alling would have had specific utility to superfilling because those specific dimensions

could effectively receive electrodeposition without superfilling. *See* Ex. 2031 ¶ 69.

BASF also points to the prosecution history of Alling as “mak[ing] clear that Alling’s reference to chip interconnects would have indicated to a person of ordinary skill in the art that the disclosure was applicable to superfilling.” *Id.* We do not find this to be dispositive as the prosecution history of Alling has not been asserted as prior art in this proceeding. The prosecution history may be used to show what topics the examiner and attorneys were considering in the prosecution of Alling, but it does not necessarily demonstrate that one of ordinary skill in the art, in viewing Alling as patented, would have considered it directly applicable to superfilling.

BASF also points out that we indicated that we did not agree with Enthone’s assertions that the field and the art are unpredictable. Reply 20 (citing Dec. 18; Prelim. Resp. 52–53). Based on the record developed during the trial, however, we are now persuaded that the field and the art are, indeed, unpredictable, as discussed above.

BASF also argues that Barstad implies that Tetronic[®] copolymers may be used as suppressors, and that Alling resolves any ambiguity by explicitly stating that “[p]articularly suitable suppressor agents” include copolymers “sold by BASF under Tetronic and Pluronic trade-names.” Reply 4 (citing Ex. 1003, 3:19–30, 6:5–62; Ex. 1006 ¶ 43). We do not agree. As Enthone points out, the compositions described in Barstad and Morrissey include a Tetronic compound as a surfactant and not as a suppressor, as claimed,

where each reference describes “suppressors” and “surfactants” as separate components of the electrodeposition bath. PO Resp. 35 (citing Ex. 1003, 1:19–26; Ex. 1004 ¶¶ 2, 26). Although BASF argues that “Barstad does not contain the clear delineation between suppressors and surfactants” (Reply 9–12), we are not persuaded that Barstad does not disclose each distinctly. Nonetheless, BASF argues that any ambiguity in Barstad and Morrissey is resolved by Alling (*id.* at 12), such that it is apparent that neither ground rests on the distinctiveness of descriptions of “suppressors” and “surfactants.” With respect to Alling, although Alling makes clear that Tetronic copolymers are particularly suitable suppressor agents, it does not, however, specify that they would have particular or any applicability to methods requiring superfilling, as discussed above.

With respect to whether the proffered combination would have been obvious under an obvious to try rationale, BASF appears to be uncertain whether such a rationale has been asserted. Tr. 74:17–75:12. Nonetheless, BASF argues that “there is no requirement that the success of new additives be predicted based on their structure or mechanism.” Reply 20–21 (citing *Par Pharm., Inc. v. TWI Pharms., Inc.*, 773 F.3d 1186, 1198 (Fed. Cir. 2014); *In re O’Farrell*, 853 F.2d 894, 903 (Fed. Cir. 1988)). We agree, but still note that given the explicit requirements of claims 1 and 17 toward superfilling, there would need to be some motivation to try the specific class of compounds from the many detailed in Alling. Although BASF attempts to distinguish the combinations detailed in the instant grounds of unpatentability from those where the prior art merely provides general

guidance or a general approach (Reply 22 (citing *Pfizer, Inc. v. Apotex, Inc.*, 480 F.3d 1348, 1367 (Fed. Cir. 2007))), we are persuaded that Alling only provides general guidance when viewed in the context of superfilling. In addition, we find persuasive Enthone’s arguments regarding faults with any obvious to try rationale for combining Alling with Barstad and Morrissey. See PO Resp. 50–55.

In addition, we find the testimony of Dr. Pound to be less persuasive than the testimony of Dr. Barkey, with respect to how one of ordinary skill in the art would have viewed Alling. Dr. Pound testifies that in view of Alling, “a person of ordinary skill in the art would likely have used Tetronic[®] polymers as suppressors in copper electroplating compositions” (Ex. 1002 ¶ 31), but that testimony omits considerations of superfilling. Dr. Pound also testifies that

In view of the knowledge provided by IBM’s achievements and the teachings by Alling, Barstad, and Morrissey, a person of skill in the art would have used Tetronic polymers as suppressor agents in copper electroplating solutions and would have optimized the concentration of the suppressors and other agents to maximize the rate of superfilling and ensure that void formation is avoided.

Id. at ¶ 29. This testimony does not indicate what in Alling would have suggested the use of Tetronic polymers as suppressor agents in the context of superfilling. Dr. Pound also contends that “[t]he broader disclosure in Alling can be shown to relate to superfilling” (Ex. 1029 ¶ 21), but Alling does not tie that broad applicability to the specific use of Tetronic polymers as suppressor agents. Dr. Pound also testifies as to the similarities between

Alling's descriptions of suppressor agents and Barstad's descriptions of Surfactant-type suppressor (*id.* at ¶ 29), but this is less remarkable when it is clear that both patent references were drafted by the same law firm for the same client. *Compare* Ex. 1003 at [73], [74], *with* Ex. 1006 at [73], [75].

In contrast, we credit Dr. Barkey's testimony that "[o]ne skilled in the art could have seen how [Alling's disclosed processes] might operate in conformally depositing traces on a printed circuit board, but not how it could apply to filling submicron features of a semiconductor chip." Ex. 2031

¶ 64. Dr. Barkey also testifies that in view of Alling, one of ordinary skill in the art would:

not encounter, acknowledge, or deal with the much more complex phenomenon of superfilling which requires that the suppressor have balanced properties that allow it to adsorb to the cathodic copper surface but be subject to removal, displacement, or exclusion by an accelerator that is also present in the electrodeposition solution, and to a degree that varies along a vertical gradient within the submicron feature so as to promote superfilling.

Id. at ¶ 69. Similarly, Dr. Barkey testifies that "Alling never describes any process or composition for either superfilling or bottom-up filling of any hole, trench or via. Nor does Alling contain any mention of aspect ratio." *Id.* at ¶ 94. Lastly, we concur with Dr. Barkey that "[n]othing in the Alling reference would have suggested the possible use of a Tetronic surfactant as a suppressor for superfilling of submicron features, much less provided any basis for expectation of success. *Id.* at ¶ 91.

As such, we agree with Enthone that BASF has not demonstrated through the Petition that the combinations of Barstad, Morrissey, Alling, and BASF Catalog would have conveyed to one of ordinary skill in the art the limitations recited in claims 1 and 17. Given the dependence of claims 2–15, 18–22, and 26–28 on claims 1 and 17, Petitioner has also failed to demonstrate, by preponderance of the evidence, that the subject matter of those claims would have been obvious to one of ordinary skill in the art in view of the teachings of Barstad, Morrissey, Alling, and BASF Catalog, as combined in the manner proposed by BASF in the Petition.

III. CONCLUSION

For the foregoing reasons, we determine that BASF has not shown by a preponderance of the evidence that claims 1–15, 17–22, and 26–28 of the '992 Patent are unpatentable under 35 U.S.C. § 103 over Barstad, Alling, and BASF Catalog, or over Morrissey, Alling, and BASF Catalog.

IV. ORDER

Accordingly, it is:

ORDERED that claims 1–15, 17–22, and 26–28 of the '992 Patent have not been shown to be unpatentable;

FURTHER ORDERED that Enthone's objections to exhibits of BASF are moot; and

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FURTHER ORDERED that, because this is a final written decision, parties to the proceeding seeking judicial review of the decision must comply with the notice and service requirements of 37 C.F.R. § 90.2.

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