

1 UNITED STATES PATENT AND TRADEMARK OFFICE
2 BEFORE THE PATENT TRIAL AND APPEAL BOARD

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MICRON TECHNOLOGY, INC.,

6

Petitioner

7

vs.

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PRESIDENT & FELLOWS OF HARVARD COLLEGE

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Patent Owner

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11

Case Nos. IPR2017-00662 (US 6,969,539)

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IPR2017-00663 (US 8,334,016)

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IPR2017-00664 (US 8,334,016)

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DEPOSITION OF WAYNE L. GLADFELTER, PH.D.

17

THURSDAY, JANUARY 11, 2018

18

9:07 A.M. - 6:03 P.M.

19

PEPPER HAMILTON LLP

20

125 HIGH STREET

21

BOSTON, MASSACHUSETTS

22

23

Reported by: Sandra A. Deschaine, CSR, RPR,

24

CLR, RSA

25

Job No. BO-157244

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1 January 11, 2018

2 9:07 A.M.

3 Deposition of Wayne L. Gladfelter,
4 Ph.D., held at the offices of Pepper
5 Hamilton LLP, 125 High Street, Boston,
6 Massachusetts, pursuant to Agreement before
7 Sandra A. Deschaine, Registered Professional
8 Reporter, Certified LiveNote Reporter,
9 Realtime Services Administrator and Notary
10 Public within and for the Commonwealth of
11 Massachusetts.

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1 Wayne L. Gladfelter, Deponent,
2 having first been satisfactorily identified
3 by the production of his Minnesota driver's
4 license and duly sworn by the Notary Public,
5 was examined and testified as follows:

6 EXAMINATION

7 BY MR. BOBROW:

8 Q. Good morning, Dr. Gladfelter.

9 A. Good morning.

10 Q. Nice to see you again.

11 A. Thank you. Nice to see you.

12 Q. You know that you're here for your
13 deposition today?

14 A. Yes.

15 Q. And do you understand that your
16 deposition is proceeding today in connection
17 with three intra partes review proceedings
18 that are pending at the Patent and Trademark
19 Office? Is that your understanding?

20 A. That is my understanding.

21 Q. As with the deposition in the
22 District Court proceeding, you understand
23 that you've been administered and obviously
24 you're obligated to testify fully and
25 truthfully here today.

1 Do you understand that?

2 A. I do.

3 Q. And you can see there's a court
4 reporter seated next to you who is taking
5 down verbatim what is being said during the
6 course of the deposition here today. The
7 court reporter will be preparing a transcript
8 of what is said, and we would ask, actually,
9 at the conclusion of the deposition, that you
10 review the transcript, and -- under the
11 applicable rules.

12 But I should caution you that if
13 you make changes to the transcript, a change
14 in your answers, for example, that we would
15 have an opportunity to comment upon that to
16 the Patent Trial and Appeal Board.

17 Do you understand that?

18 A. I do. Thank you.

19 Q. All right. Good.

20 If I ask a question today and it's
21 unclear to you, you don't understand the
22 question, please let me know, and I'll
23 rephrase it or repeat it.

24 Do you understand that?

25 A. I do.

1 Q. Okay. Over the course of the day,
2 I'm sure we'll be taking a break, you know,
3 every hour and 15 minutes, hour, what have
4 you. But if you need to take a break any
5 more frequently than that, just let us you
6 know and we'll accommodate that. Okay?

7 A. Thank you.

8 Q. Are you under the influence of any
9 medications, alcohol, anything of the kind
10 that would influence or impair your ability
11 to testify truthfully and fully today?

12 A. No.

13 Q. All right. Very good.

14 Did you have a chance to prepare
15 for the deposition today?

16 A. I did.

17 Q. What did you do to prepare for the
18 deposition?

19 A. I visited, starting on Tuesday
20 morning, with the group at Pepper Hamilton
21 and reviewed many of the documents that are
22 going to be discussed, are part of IPR
23 questions, petitions. And we talked by phone
24 last Friday as well, for a couple hours on
25 some of the material, and during the

1 course -- between then and now also, just
2 reading -- rereading documents.

3 Q. In connection with your
4 preparation this week, have you been here in
5 Boston since Tuesday?

6 A. I arrived Monday night.

7 Q. I see. And you prepared on -- all
8 day Tuesday and all day Wednesday for the
9 deposition?

10 A. Yes.

11 Q. I see. And then also in addition
12 to that, a few hours last Friday as well?

13 A. Correct.

14 Q. All right. Did you have a chance
15 to review the declarations that you signed in
16 connection with the IPR proceedings?

17 A. Yes.

18 Q. Did you have a chance to review
19 the exhibits that you referred to in those
20 declarations?

21 A. Yes.

22 Q. Okay. And I take it that among
23 the exhibits that you reviewed were the, if I
24 can call them this, the '539 patent and the
25 '016 patent; is that correct?

1 A. That's correct.

2 Q. All right.

3 MR. BOBROW: Why don't we mark as
4 Exhibit 1 the '539 patent.

5 (Exhibit 1, United States Patent 6,969,539,
6 marked for identification.)

7 BY MR. BOBROW:

8 Q. Dr. Gladfelter, you've been handed
9 Exhibit 1 for identification. This is U.S.
10 Patent No. 6,969,959, and I'll note for the
11 record that this is Exhibit 1001 in the 662
12 proceedings, 1010 in the 663 proceeding, and
13 1020 in the 664 proceeding.

14 Do you recognize this exhibit,
15 the '539 patent?

16 A. Yes. Excuse me. Yes, I do.

17 Q. All right. Let me ask you to
18 turn, if I might, to the very last page,
19 Column 32, and you'll see there Claim 24.

20 Do you see that?

21 A. I do.

22 Q. You understand that Claims 24, 26,
23 and 29 are the claims that are at issue in
24 these proceedings, correct?

25 A. I do understand.

1 Q. Now, with respect to the language
2 of Claim 24, there is a two-word phrase there
3 that I wanted to start with, and it's "metal"
4 and the next word is "amides."

5 Do you see what I'm referring to
6 there?

7 A. Yes, I do.

8 Q. So first off, how do you pronounce
9 a-m-i-d-e-s?

10 A. Amides.

11 Q. All right. Very good. I've heard
12 it pronounced a number of ways in this case.
13 I'll try to pronounce it the way you
14 pronounce it.

15 A. Okay.

16 Q. So let me ask you about that
17 phrase, metal amides. First of all, what
18 makes something an amide?

19 A. An amide is a ligand that binds to
20 a metal. It has a nitrogen that forms the
21 actual bond to the metal. And then it has
22 two substituents. These are organic
23 substituents. They are, typically in these
24 kind of compounds, in precursors for
25 processes -- are alkyls, in essence,

1 saturated hydrocarbons or closely related
2 substituents.

3 Q. Okay.

4 A. And I would continue. An amide by
5 itself typically has a negative one charge.
6 And as I said, that amide ligand is attached
7 to a metal.

8 Q. And there are amides that are not
9 metal amides; is that correct?

10 A. That is correct.

11 Q. So there could be something like a
12 silyl amide, for example, where you would
13 have a bond between the nitrogen and silicon?

14 A. That was, in my description,
15 included in the closely related category of
16 substituent, closely related to a metal
17 alkyl, yes. It's a saturated species.

18 Q. Okay.

19 A. You can also have amides in simple
20 organic chemistry that contain no metals
21 whatsoever, but it is one of the well-defined
22 organic functional groups.

23 Q. Are there classifications of metal
24 amides or categories of metal amides?

25 A. Sometimes you see people refer to

1 amides, depending on whether they have, I'd
2 say two alkyl groups or just one alkyl group
3 differently. Sometimes you will see that it
4 will be a secondary amide or primary amide,
5 but that's usually not done because it's part
6 of the description that usually is applied
7 into amides, is we simply include this is a
8 dialkylamide, which, by itself, explains that
9 it's a secondary amide that's being
10 discussed.

11 Q. One category of amides is
12 alkylamides; is that right?

13 A. Yes.

14 Q. Are there other categories of
15 metal amides besides alkylamides?

16 A. Yes. An alkyl group implies a
17 saturated hydrocarbon moiety. Saturated
18 meaning there are no double bonds associated
19 with it, that particular carbon. You can
20 have, as an example, an aryl group -- that's
21 spelled a-r-y-l -- in which you might have --
22 the most common component would be a
23 phenyl -- that's p-h-e-n-y-l -- as a
24 substituent. That would not be considered an
25 alkyl group. It's an aryl group.

1 Q. Okay. So there are metal aryl
2 amides?

3 A. Yes, there are.

4 Q. Are there other categories or
5 classifications of metal amides besides alkyl
6 amides and aryl amides?

7 A. What I'm doing is running through
8 in my mind all possible ligands that end and
9 the various definitions. Sometimes, as an
10 example, you will hear people refer to
11 compounds as a cyclic amide, in which the
12 amide has substituents that are primarily
13 carbon, that proceed around such that the two
14 ends of the substituent are bound to the
15 nitrogen and the entire group forms a cycle,
16 and those cycles can be alkylamides, but they
17 could also be unsaturated amides.

18 Q. Meaning there's a double bond?

19 A. Meaning there's a double bond or
20 some other substituent. You can go through
21 the organic literature and find many
22 additional components that you could add to
23 the carbon, to the alkyl group, to convert it
24 from an alkyl to some other functional group.

25 Q. Okay. Is there a category of

1 amides called allyl amides, a-l-l-y-l amides?

2 A. It's an unsaturated -- an allyl
3 group -- it's a-l-l-y-l -- is an unsaturated
4 group. It's an unsaturated hydrocarbon, so
5 it has one double bond, and there are three
6 carbons in an allyl group.

7 Q. Are there other classifications of
8 amides that you're aware of besides alkyl
9 amides, aryl amides, cyclic amides, and allyl
10 amides?

11 A. Yes. As they come to mind, you
12 could have a vinyl amide, which is a vinyl --
13 vinyl is spelled v-i-n-y-l -- and this is in
14 which -- an example where the nitrogen is
15 attached as one of the -- as bonded to one of
16 the carbons that's involved in the
17 carbon-carbon double bond. So like allyl, it
18 has a carbon-carbon double bond. But in this
19 case, the nitrogen is attached as one of the
20 four groups that are part of the
21 carbon-carbon double bond. An allyl group,
22 the vinyl component is one carbon removed
23 from the nitrogen.

24 Q. Did you say in an allyl group the
25 vinyl --

1 A. When you have -- so I can clarify.
2 An allyl amide would, in its simplest
3 example, would contain a nitrogen, as all of
4 the amides do. And then you would go to a
5 CH₂ substituent, and then you go to another
6 carbon-carbon bond, and then you put your --
7 the next group, between the second and third
8 carbon is a double bond.

9 Q. I see. Okay.

10 Other classifications of amides
11 that you can think of besides the ones you've
12 mentioned so far?

13 A. As I mentioned, you do have all
14 the organic compounds, but -- so maybe you
15 could clarify.

16 Are you referring specifically to
17 examples where amides can be bound to a
18 transition metal -- or excuse me -- a metal.

19 Q. To a metal, yes.

20 A. To a metal.

21 I would -- the field of organic
22 chemistry and the number of possibilities is
23 very large, so I don't want to consider what
24 I've written as an exhaustive list. It would
25 take me a while to get through all the

1 possibilities.

2 But the answer is I'm sure there
3 are other components that could be listed if
4 we look through the chemical literature.

5 Q. I mean, are there on the order of
6 hundreds of different amides?

7 A. That are used as ligands to
8 metals?

9 Q. Let's start there.

10 A. Yeah. I doubt that.

11 Q. Dozens?

12 A. I would agree with that.

13 And, again, just to clarify, when
14 we talk about any one of these, we're talking
15 about, in essence, a functional group; and as
16 in simple organic chemistry, a functional
17 group can be modified by lots of substituents
18 to create a new molecule, and that's true
19 in -- in metal amides as well. Simply as an
20 example, you can have a methanamide, and you
21 could have -- change that to an ethanamide,
22 so those would be two different compounds.

23 Q. In your last answer, did you say
24 methyl, m-e-t-h-y-l?

25 A. I did. I'm sorry I didn't spell

1 that.

2 Q. No, that's fine. I think the
3 reporter thought you said metal, m-e-t-a-l.

4 But in all events, you would agree
5 it's fair to say that there are dozens of
6 amides that would be bound to a metal. Is
7 that fair to say?

8 MR. LEN: Objection.

9 A. I would agree that you could have
10 some large number of different nitrogen-based
11 groups that would be classified as amides
12 that combine to a metal as a ligand.

13 Q. More than 20?

14 A. I'm hesitant to give a specific
15 value just because the magnitude of the field
16 is so large, and the classification of the
17 ligands and the number of examples that are
18 out there, it's hard to pin down a particular
19 number.

20 Q. I'm not asking you to pin down a
21 number. I'm asking you: Can you say with
22 confidence that there would be more than 20
23 metal amides?

24 MR. LEN: Objection.

25 A. Well, to be specific in answering

1 your question, no, I can't say with
2 confidence that there is more, but I would
3 not be surprised if there were more.

4 Q. I see. All right.

5 And what about amides that are not
6 bound to metals? Are there such things out
7 there?

8 A. I mentioned that in simple, or
9 another word, pure organic compounds, amide
10 is an important functional group. The
11 functional group that -- and there are
12 several different types of organic amides.
13 And there are examples, for instance, the
14 amide bond in a protein is an example of an
15 organic amide. That's often given a separate
16 name, called a peptide bond; but yes, there
17 are different types of amides.

18 Q. That are not bound to a metal?

19 A. That are not bound to a metal.

20 Q. And are there dozens of such
21 amides that are not bound to metals?

22 A. The category is more -- is usually
23 subdivided at the broadest possible term into
24 amide, a primary amide and a secondary amide.
25 A primary amide has a nitrogen. Attached to

1 the nitrogen is a carbonyl group, a carbon
2 with a double bound to an oxygen. And then
3 there is a organic substituent attached to
4 the other side of the carbonyl, to the other
5 side of the carbon.

6 In applying the parent name amide,
7 the parent has two hydrogens on the nitrogen.
8 In a primary amide, there is one of those
9 hydrogens that is converted to another
10 substituent. In a secondary amide, both of
11 those hydrogens are converted to a different
12 substituent.

13 You recall I was using primary and
14 secondary in referring just to the metal
15 amides as well. That's where those names
16 ultimately were derived from.

17 Now, back to your question, are
18 there more than three types of amides?

19 When we were listing different
20 types of substituents that could be bound to
21 the amides, nitrogens on metal amides, we
22 have the same type of category -- categories
23 that could be applied to organic amides.
24 Only the -- you can have -- just to step
25 back.

1 You can have alkyls, aryls; they
2 can be cyclic; they can be saturated, meaning
3 they have no double bonds or multiple bonds,
4 or unsaturated. So they can be part of a
5 ring. It's a big field. So in that one it's
6 a little bit easier to describe the number to
7 greater than a hundred.

8 Q. Okay. All right. Thank you.

9 Now, going back to the metal
10 amides for a minute. In a metal amide, is it
11 the case that metal atom is bonded to the
12 nitrogen atom?

13 A. In a metal amide, yes, the
14 nitrogen is bonded to the metal amide.

15 Q. And is it the case in the metal
16 amide that the metal atom is not bonded to
17 the carbon atoms?

18 MR. LEN: Objection.

19 A. There are examples of compounds in
20 which -- the more complex compounds in which
21 there is an amide nitrogen that has a
22 substituent, an organic substituent, that
23 ultimately ends up with a carbon directly
24 bound to the metal to create a ring in which
25 both a carbon and a nitrogen are bound to the

1 amide or -- excuse me -- to the metal.

2 Q. All right. Now, with respect to
3 metal amides, are there any metal atoms in
4 which one of the functional groups that is
5 part of the molecule is oxygen?

6 A. Well, yes, there are examples
7 where oxygen can be part of the functional
8 group.

9 Q. Can you list some examples of such
10 metal amides?

11 A. If you look at -- let's make an
12 example with a nitrogen. We'll start, and
13 I'm going to now move around -- move out from
14 the nitrogen. We could have, for instance,
15 two alkyl -- two carbon atoms, so we go
16 nitrogen, carbon, carbon, and then we may see
17 an oxygen and then to another carbon.

18 When we have a carbon oxygen,
19 carbon linkage, in which those carbons on the
20 other side of the oxygen are not part of a
21 carbonyl, what you would have identified is
22 something called an ether. So that is an
23 example of a metal -- again, a metal
24 alkylamide, which does contain a nitrogen
25 which is bound to the metal and a separate

1 oxygen.

2 And this design feature is
3 sometimes built into ligands, because that
4 oxygen, which is an ether, is a weak linkage,
5 but sometimes it can be -- can actually
6 arrange itself to bind weakly to the metal.
7 That's just one example. Again, it can go
8 into other types of substituents or carbon
9 based or sulfur based and so on.

10 Q. Does the presence of the oxygen in
11 that metal alkylamide change its properties
12 as compared to a metal alkylamide that lacks
13 the oxygen?

14 A. One of the reasons that this has
15 been included as a design feature in some
16 metal amides is to make use of that -- I'm
17 going to use the term chelation effect -- so
18 that's spelled c-h-e-l-a-t-i-o-n -- to help
19 stabilize, number one, the metal complex.
20 And two, to prevent it from -- or minimize
21 the potential for it to dimerize or
22 oligomerize.

23 The purpose in some of these cases
24 is to change the volatility of the -- so
25 that's a physical property. It changes the

1 volatility -- or can change the volatility of
2 a metal complex.

3 Q. By adding the oxygen, is the
4 purpose to make the metal complex more
5 volatile or less volatile?

6 A. The intention of people --
7 researchers doing this would be to -- related
8 to our discussions, to precursors, would be
9 to make them more volatile.

10 Q. Does the addition of the oxygen
11 change any of the other properties of the
12 metal alkylamide besides at least
13 intending to make it --

14 THE REPORTER: I'm sorry, of the
15 other properties of the metal alkylamide
16 intending?

17 MR. BOBROW: Sorry. I'll repeat
18 it or rephrase it.

19 BY MR. BOBROW:

20 Q. Is the addition of the oxygen atom
21 to the metal alkylamide complex intended to
22 change any of the other properties of the
23 metal alkylamide besides enhancing its
24 volatility?

25 MR. LEN: Objection.

1 A. I think it's hard to be quite so
2 expansive about this and respond accurately
3 to that question.

4 Changing anything in the molecule
5 can change a physical property or a chemical
6 reactivity and that may be part of the design
7 feature. So it's possible that other things
8 could happen, but I can't say with certainty.

9 Q. As opposed to whether things would
10 happen, necessarily, you spoke before about
11 the design intent. And my question was
12 focused on that.

13 Is the addition of the oxygen to a
14 metal alkylamide intended to do something
15 else besides increase its volatility?

16 MR. LEN: Objection.

17 A. I think I would have to look at
18 specific examples to respond to your
19 question. It becomes, I think -- the many
20 possibilities too nebulous to address by
21 itself.

22 Q. Just a couple of follow-ups. You
23 mentioned in one of your answers something
24 called a carbonyl.

25 Would you spell that for the

1 record?

2 A. C-a-r-b-o-n-y-l.

3 Q. You also referred in one of your
4 answers to stabilizing a metal complex to
5 minimize, I believe you said dimerization and
6 oligomerization. Did I get that correct?

7 A. That's correct.

8 Q. Is dimerization a species of
9 oligomerization? An example of it?

10 A. They are in the same category. It
11 is step one to species. Monomers come
12 together. Just two would make a dimer. If
13 you add a third, trimer and so on. And so
14 these are characterized as oligomers if
15 they're small. If it goes on to large
16 numbers, the word switches to polymers.

17 Q. Now, with respect to -- let's go
18 back to the metal amides that we talked about
19 before.

20 Do alkyl amides, aryl amides,
21 cyclic amides, allyl amides, vinyl amides, do
22 they all have the same chemical and physical
23 properties?

24 MR. LEN: Objection.

25 A. They will have different chemical

1 and physical properties.

2 Q. And what about the organic flavors
3 of the amides that you talked about, again,
4 with alkyl amides, cyclic amides, saturated
5 amides, et cetera, will those have different
6 chemical and physical properties?

7 MR. LEN: Objection.

8 A. Again, let me ask for
9 clarification. You're talking about the
10 compounds that don't have any metals? These
11 are just the organic?

12 Q. Correct.

13 A. Yes, they will have different
14 properties.

15 Q. Back to the metal amides.

16 Is one property that is evaluated
17 for purposes of chemical vapor deposition or
18 atomic layer deposition, when we're talking
19 about the metal amides, a property that I've
20 heard referred to as hydrolysis?

21 MR. LEN: Objection.

22 Q. Or hydrolyzation?

23 A. The hydrolysis is the reaction
24 with water of a species. And certainly in
25 the case where one wants to deposit a metal

1 oxide by CVD, the issue of reaction with
2 water of the precursor is important.

3 Q. Will different metal amides have
4 different reactivity to water?

5 MR. LEN: Objection.

6 A. Maybe you could explain. What do
7 you mean by "different reactivity"?

8 Q. I mean will they be more reactive
9 or less reactive to water, or do metal amides
10 all react identically to water?

11 MR. LEN: Objection.

12 A. Again, there is a broad category
13 of amides that we've been discussing thus
14 far. So one could see different reactions as
15 well as different reaction velocities,
16 different reaction rates.

17 Q. Okay. What about with respect to
18 oxidation? Would evaluating a metal amide
19 for purposes of chemical vapor deposition or
20 atomic layer deposition, do all metal amides
21 oxidize in the same way at the same rate?

22 MR. LEN: Objection.

23 A. So do you -- could you clarify
24 what you mean by oxidation?

25 Q. Why don't you tell me what you

1 mean by oxidation, as you use that term in
2 your field of chemistry.

3 A. Oxidation means that, to me, that
4 there is a change in the oxidation state of
5 the -- of an element, as you go from starting
6 material to the product. So the -- I only
7 hesitated because there can be a couple
8 things that can lead to oxidation or its
9 opposite, the reduction, and it depends
10 somewhat on the reagent.

11 So if we look at oxygen, which is
12 a common oxidizing agent -- if I could go
13 back. Could you ask your question again? I
14 lost my train of thought.

15 Q. Sure. Let me try to clarify a
16 little bit, then, in light of what I believe
17 you just said.

18 So with respect to all the
19 different metal amides that you were
20 discussing before, do those all oxidize in
21 the same way, at the same rate for a given
22 oxidizing agent?

23 MR. LEN: Objection.

24 A. I would say in general terms the
25 answer is similar; that molecules will have

1 potentially different products as well as the
2 different rates of reaction with a given
3 reagent.

4 Q. Are you familiar with a concept of
5 ring formation in the course of chemical
6 vapor deposition or atomic layer deposition?

7 MR. LEN: Objection.

8 A. I would have to ask you to specify
9 what you mean or perhaps give an example. I
10 know what a ring is.

11 Q. Yeah.

12 A. We've talked about that in the
13 discussion of amides and -- metal amides.
14 But in the context of ring formation, I need
15 a little bit more direction from you.

16 Q. Okay. In the context of chemical
17 vapor deposition and atomic layer deposition,
18 are there circumstances in which the reaction
19 that is taking place between precursors and
20 surfaces and the like, will create, as
21 by-products, a ring?

22 MR. LEN: Objection.

23 A. Here's another clarifying point.
24 A by-product do you mean a by-product that is
25 ultimately expelled from the reactor as part

1 of the overall reaction stoichiometry?

2 Q. Could be expelled from the
3 reactor, perhaps bits of it are incorporated
4 into the film as an impurity. Things like
5 that.

6 MR. LEN: Objection.

7 A. So with respect to -- and, again,
8 let me ask for another point of
9 clarification. What kind of starting
10 material would you be referring to?

11 Q. Could be a -- a metal amide is
12 what I had in mind from a question, I
13 believe, I think that was included in the
14 question.

15 A. Okay. But I'm now clarifying.
16 The structure of that amide, would it have
17 the cycle to begin with?

18 Q. It might or it might not, and
19 that's part of my question, is, is whether
20 all metal amides will, in the course of
21 chemical vapor deposition or atomic layer
22 deposition, be prone to ring formation in the
23 same way? That's really the question.

24 MR. LEN: Objection.

25 A. In the -- if the cycle is part of

1 the amide to begin with, and if one were
2 doing a reaction with water as a co-reaction,
3 as the oxidizing oxygen source, and that
4 leads to expulsion of the product amine, it
5 will continue to -- it will retain that
6 cyclic structure. On the other hand -- and
7 that would be expelled from the chamber
8 during the deposition process.

9 In the other example there are --
10 or I say the other category, which I think
11 you mentioned is not a by-product that is
12 swept from the reaction but referring to
13 something that's retained on the surface,
14 there are reports of that as well, that a
15 cyclic structure can be formed is -- these
16 have been more difficult to characterize
17 because the cyclic structure is part -- that
18 ring is part of the -- or part of the ring, I
19 should say, is an atom or two on the metal
20 surface, or three; I don't want to limit it.

21 But one could indeed see a
22 precursor that begins to react or reacts
23 leading to a cyclic structure, which part of
24 the nitrogen, if we're referring to amides
25 again, remains bound to the starting metal in

1 the precursor, and one of the carbons may
2 have been activated and formed a bond with
3 the surface, while the metal and the amide
4 remain intact. Then you have atoms through
5 the surface, through the metal, through the
6 nitrogen, and back through to create a ring.
7 That's a cyclic structure.

8 So those are sometimes -- not
9 sometimes. They're always more difficult to
10 define because our spectroscopic methods are
11 more difficult to assign such a structure
12 when you're working with a solid surface.

13 Q. Will different amides have
14 different properties and propensities in
15 terms of forming these sorts of rings in the
16 course of chemical vapor deposition and
17 atomic layer deposition?

18 A. Again, it's difficult to say for
19 certain without looking at specifics; but,
20 again, I think when you change the structure
21 of an amide, you're going to change -- you're
22 going to change physical and/or chemical
23 properties.

24 I can give an example, though. We
25 talked about having a vinyl amide or an allyl

1 amide that has a double bond present in the
2 substituents connected to the nitrogen. That
3 double bond can be a reactive component with
4 the surface as well, and it could also lead
5 to a cyclic structure on the surface.

6 Q. Now, going back again to the
7 category of amides, will -- are you familiar
8 with a concept that I've at least seen
9 referred to as ligand rearrangement? Is that
10 a concept that you're familiar with it?

11 A. Yes.

12 MR. LEN: Objection.

13 A. It can refer to a couple
14 different -- a couple different rearranging
15 processes.

16 Q. Okay. So tell me, then, what
17 ligand rearrangement is. And if there are a
18 couple examples, then please list those for
19 me.

20 A. The -- rearrangement of any sort
21 is a change in the -- if the molecule remains
22 the same, in other words, all the ligands are
23 the same, a rearrangement might refer to some
24 stereochemical transformation in the
25 molecule. I can give an example.

1 We talked about a -- about
2 compounds that contain a double bond. If
3 there are more than two carbons attached to
4 the four points on a double bond, one can
5 have isomers. These will have different
6 chemical and physical properties. Cis and
7 trans -- c-i-s and t-r-a-n-s -- are two
8 isomers that one sees in double bonds. And,
9 again, that changes a little bit -- the
10 properties of the molecules. That is one
11 example of a ligand rearrangement.

12 Q. Go ahead. I didn't mean to
13 interrupt.

14 A. There are other examples that
15 could be focused on a metal where the
16 stereochemistry of the metal, meaning the
17 distribution of the ligand surrounding the
18 metal, change in some fashion. That's a
19 rearrangement.

20 And you haven't clarified, and
21 this would be important to understand, in
22 the -- this discussion, in terms of ligand
23 rearrangements. Do the ligands always have
24 to remain the same? Let me clarify that.

25 If you take an example with four

1 amides attached to a -- excuse me -- attached
2 to a metal and these are all labeled with,
3 I'll just say substituents on the nitrogen,
4 we're going to give it a generic R1, a common
5 practice in organic chemistry. So I
6 didn't --

7 Perhaps you could clarify. Do you
8 want to keep all those as R1 in your question
9 regarding ligand rearrangement?

10 Q. And is your point that the R1
11 could be, in the course of being rearranged,
12 be changed to an R2 or R3 or something else?

13 A. If there were another source of
14 the ligand, yes. I probably would not refer
15 to such an example as a rearrangement. I
16 would refer to it as a -- as a reaction.

17 Q. Now, with respect to what you have
18 described as a rearrangement, ligand
19 rearrangement, do different amides have
20 different properties with respect to how
21 prone they are to a ligand rearrangement?

22 MR. LEN: Objection.

23 A. I think -- I find that difficult
24 to answer in a -- in a fully generic sense.
25 For instance, if you had, and we -- I, in

1 essence, used this example earlier. One of
2 the R groups of substituents be contained in
3 oxygen, I used the explicit example early in
4 an ether linkage, that that R group can
5 rearrange to flop around in the solvent
6 unbound to the metal, or it could form an
7 interaction, a bond to the metal, a weak bond
8 to the metal. That would be a ligand
9 rearrangement that involves the metal in one
10 part.

11 Q. I see. And if the oxygen were
12 absent from the amide, then that kind of
13 rearrangement at least would not happen?

14 MR. LEN: Objection.

15 A. That is correct. You need a long
16 pair of electrons typically to be donated to
17 the metal, and an oxygen has it. Other atoms
18 may also -- or will also have lone pairs, but
19 a saturated organic fragment will not.

20 Q. You -- in the course of your
21 answer and earlier, I had asked you some
22 questions about metal amides that included
23 oxygen atom or atoms.

24 Do you recall that?

25 A. Yes.

1 Q. You also used earlier a metal
2 complex. Can you tell me first of all what a
3 metal complex is?

4 A. A metal complex is a group of
5 atoms, often with the metal that is at the
6 center surrounded by some number of ligands
7 that are bonded to the metal.

8 Q. Thank you. Now, with respect to
9 amides that include one or more oxygen atoms,
10 were you familiar with those sorts of amides
11 back in the 19- -- I'm trying to remember
12 your CV -- back in the 1980s, let's say.

13 MR. LEN: Objection.

14 A. There was -- I simply am trying to
15 look at time here.

16 Q. Yeah, of course.

17 A. And I find it somewhat difficult
18 to identify when an -- when these were first
19 characterized, and what comes to mind is a
20 statement that they were not a shockingly new
21 compound. It was considered to be a variant
22 on a theme for making a metal amide, but I
23 cannot say when the first one of these
24 appeared in the literature. I just don't
25 remember.

1 Q. And as opposed to when the first
2 one appeared, which isn't my question, what I
3 would like to know is, do you recall such
4 amides with oxygen atoms in them, let's say
5 in the 1980s or 1990s?

6 MR. LEN: Objection.

7 A. I cannot point to a specific
8 example. But in generic -- or in general, I
9 would -- I would guess that they existed by
10 that point.

11 Q. And, you know, to the best of your
12 ability, had such amides with oxygen been
13 used in reactions, chemical reactions in the
14 1980s or 1990s, along the lines of chemical
15 vapor deposition or atomic layer deposition?

16 MR. LEN: Objection.

17 A. Again, I don't have in mind a
18 specific example; but, in part, I will say
19 that one of the motivations for incorporating
20 the potential of a chelation effect, as we
21 talked before, the potential for
22 incorporating that to minimize
23 oligomerization processes, that that idea has
24 been presented in the use of compounds for
25 vapor deposition or as vapor deposition

1 precursors.

2 Q. So staying with Claim 24 of
3 the '539 patent. In your view, does Claim 24
4 require a process in which the deposition is
5 self-limiting?

6 A. Again, I think I discuss this in
7 the declaration. The description here is
8 exposing heated surface alternately to the
9 vapor of the amides and then to the vapors of
10 an oxygen source, water or an alcohol.

11 And while the words in this do not
12 explicitly come out self-limiting, with the
13 additional information available in the
14 specification, it's clear that self-limiting
15 is an important component of the deposition.

16 Q. For Claim 24?

17 A. For Claim 24.

18 Q. In the opinions that you expressed
19 in your declarations, were -- did those
20 opinions assume or are they based on the
21 understanding that Claim 24 requires
22 deposition that is self-limiting?

23 MR. LEN: Objection.

24 A. Self-limiting is a basic component
25 of atomic layer deposition. And so, again, I

1 forget specifically the words that are listed
2 in the declaration, but this is a component
3 of Claim 24.

4 Q. And I'm simply asking the opinions
5 that you expressed in your declarations
6 regarding the patentability of Claim 24 -- or
7 based on that understanding, that
8 self-limiting is part of Claim 24?

9 A. Yes.

10 Q. Now, with respect to self-limiting
11 deposition, have you heard of a concept
12 called an ALD window?

13 MR. LEN: Objection.

14 A. Yes.

15 Q. What's an ALD window?

16 A. As described as far back as the
17 '70s, when Suntola, S-u-n-t-o-l-a, introduced
18 some of the defining features of this
19 process, at the time called atomic layer
20 epitaxy, this has been melded into atomic
21 layer deposition, so that you see these used
22 interchangeably.

23 The idea of an ALD window results
24 from a graph in which the vertical axis is
25 typically the growth per cycle, growth

1 meaning the thickness per cycle. So to
2 clarify, a cycle -- excuse me -- an ALD
3 process involves a number of cycles that are
4 conducted in sequence to give the desired
5 thickness of a film.

6 Back to the ALD window, the
7 vertical axis is the thickness per cycle.
8 The horizontal axis is temperature. And
9 Suntola then defines the ideal ALD window as
10 this relatively horizontal component of this,
11 in which the thickness per cycle does not
12 change a great deal as a function of
13 temperature.

14 Q. Okay. And is the idea that the
15 window will have sort of a lower bound to the
16 temperature and an upper bound to the
17 temperature?

18 MR. LEN: Objection.

19 A. The window, that's the picture,
20 yes. The magnitude of that lower bound and
21 upper bound can range from zero to a larger
22 number.

23 Q. Great.

24 And assuming that the number for
25 the window isn't zero --

1 A. Okay.

2 Q. -- but assuming that it's some
3 sort of positive number, is the general idea
4 of the ALD window that the temperature could
5 vary or change from the low end of the window
6 to the high end of the window but the
7 thickness of deposition per cycle will not
8 change as the temperature varies within that
9 window?

10 MR. LEN: Objection.

11 A. Once again, the idea is that it
12 won't change significantly. Indeed it --
13 temperature effects often are more
14 complicated than one simple window. So the
15 idea that Suntola was getting across is that
16 this has been official to see this limited
17 change in deposition rate, and it's
18 represented indeed as a straight line. It's
19 represented as a flat line. So that is
20 consistent with what you described.

21 But it's also understood that you
22 can see lines that tend down or tend up
23 during this window, but there's still a
24 relatively small variation in growth as a
25 function of temperature.

1 Q. All right. So with respect, then,
2 to an ALD window, if I understand what you
3 said, the idea is that I could vary the
4 temperature from the low end of the window to
5 the high end of the window and the deposition
6 thickness per cycle will exhibit a relatively
7 small variation of growth within that window?

8 MR. LEN: Objection.

9 A. That is the idealized picture,
10 yes.

11 Q. With respect to this concept of
12 self-limiting deposition, have you heard or
13 read in the literature that concept being
14 described in terms of self-control or
15 self-controlling deposition?

16 MR. LEN: Objection.

17 A. I do not recall reading those
18 words.

19 Q. All right. You mentioned earlier,
20 in the context of the work of Suntola, that
21 back in the day this process was called
22 atomic layer epitaxy or ALE; is that correct?

23 A. Yes.

24 Q. Is it the case that -- I think I
25 understood you to say but I want to make

1 sure, that over time, that those two concepts
2 are understood to represent the same thing,
3 ALD and ALE, I should say?

4 A. Yes. Epitaxy does have a very
5 specific connotation regarding the crystal
6 structure of an underlying substrate and the
7 overlying film, such that there is a
8 crystallographic relationship between these
9 two.

10 But even in the earlier days and
11 when people were still using ALE, that
12 requirement disappeared in the literature,
13 and I think this ultimately led to the
14 preference being given to using just atomic
15 layer deposition.

16 MR. LEN: Jared, we've been going
17 just over an hour, so whenever you get a
18 chance, just take a short restroom
19 break.

20 MR. BOBROW: Okay. No problem.
21 Let's just continue for just another
22 minute or so.

23 BY MR. BOBROW:

24 Q. Is it fair to say, though, in the
25 literature, that when one reads ALE, or

1 atomic layer epitaxy, that one is referring
2 to the same process as when one is referring
3 to atomic layer deposition or ALD?

4 A. Yes. I think, just to clarify
5 that particular point, it would depend on
6 looking at the specific system. There are
7 still people, researchers that will deposit
8 epitaxial films, and they may still use, in
9 my view properly, the term ALE, and in that
10 more constrained definition, it's an
11 appropriate -- it's an appropriate name.

12 Q. So -- but with respect to a
13 situation in which, you know, an alternating
14 pulse purge, pulse purge type of system is
15 being used to deposit an epitaxial film, that
16 would still be a type of atomic layer
17 deposition, would it not?

18 A. Yes.

19 MR. BOBROW: Great. Why don't we
20 take a short break.

21 (Recess taken at 10:21 a.m. to 10:32 a.m.)

22 MR. BOBROW: Let's go back on the
23 record.

24 BY MR. BOBROW:

25 Q. Dr. Gladfelter, before the break,

1 I was asking you about atomic layer
2 deposition and atomic layer epitaxy.

3 Do you recall that?

4 A. Yes.

5 Q. Now, with respect to ALD, would
6 you agree that ALD is a type of chemical
7 vapor deposition?

8 A. It's a vapor deposition process.
9 There's certainly a relationship between ALD
10 and CVD, which is the abbreviation for
11 chemical vapor deposition, but they have
12 different requirements.

13 Q. My question, though, is, is ALD a
14 type of CVD? Is it a type of chemical vapor
15 deposition?

16 A. I have seen literature that
17 suggests that category -- categ -- I can't
18 say the word -- categorization of ALD. It's
19 not the way I usually describe it when I'm
20 teaching and working with students. I treat
21 them as separate processes.

22 Q. Okay. Would you agree that ALD is
23 a form of chemical vapor deposition?

24 A. That seems like we're just
25 changing a word, but yeah, I don't really

1 have a problem with that.

2 Q. Okay. And with respect to the
3 '539 and the '016 patents, you'd agree with
4 me that they say that ALD is a form of CVD,
5 correct?

6 A. I believe that is in here at --

7 Q. Why don't you take a look at
8 Exhibit 1, Column 1, and I direct your
9 attention to the paragraph that begins at
10 line 40 of Column 1 and goes to line 49, and
11 again this is in Exhibit 1.

12 A. Yes, I see it.

13 Q. And you'd agree with me that the
14 '016 patent -- sorry -- the '539 patent and
15 therefore the '016 patent treat ALD as a form
16 of CVD, correct?

17 A. Yeah. So as I read this, it
18 describes CVD and specifically states the
19 form of CVD -- or considers ALD in the form
20 of CVD.

21 Q. And you wouldn't disagree with
22 that, would you?

23 A. No, I would not.

24 MR. BOBROW: Let's go ahead and
25 mark this as the next exhibit in order.

1 (Exhibit 2, MOCVD and ALD of High-k
2 Dielectric Oxides Using Alkoxide Precursors,
3 marked for identification.)

4 BY MR. BOBROW:

5 Q. Dr. Gladfelter, you've been handed
6 what's been marked as Exhibit 2 for
7 identification. This is a document entitled
8 "MOCVD and ALD of High-k Dielectric Oxides
9 using Alkoxide Precursors."

10 We've marked that as Exhibit 2 for
11 the deposition. I'll note that this is
12 Exhibit 2102 to the proceedings in the 662,
13 663, and 664 proceedings. This is one of the
14 exhibits to your declaration.

15 I take it, Dr. Gladfelter, that
16 you have read Exhibit 2 before?

17 A. Yes.

18 Q. This is a paper authored by
19 Anthony C. Jones and a number of others.

20 Do you see that?

21 A. I do.

22 Q. Do you know Dr. Jones?

23 A. I have met Dr. Jones.

24 Q. Have you met him -- in what
25 context have you met Dr. Jones?

1 A. I've met him at meetings.

2 Q. Of what type?

3 A. I'm trying to remember the
4 specifics. But scientific meetings that were
5 connected to using precursors and deposition
6 processes. I don't remember the specifics,
7 but it was a while ago.

8 Q. Precursors in the context of vapor
9 deposition processes?

10 A. Yes.

11 Q. Are you familiar with the journal
12 in which Exhibit 2 appeared, a journal called
13 Chemical Vapor Deposition?

14 A. Yes.

15 Q. Is this a journal that you read in
16 connection with your work as a chemistry
17 professor?

18 A. I have. It no longer exists, but
19 yes --

20 Q. And was this journal considered to
21 be a reputable one in the field?

22 A. I hope so. I published there.

23 Q. All right. And is -- maybe I
24 should use the past tense of that verb -- was
25 the Chemical Vapor Deposition journal, was

1 this a journal in which papers were submitted
2 and reviewed by peers in the field before
3 publication?

4 A. Yes.

5 Q. What was the context in which you
6 first read Exhibit 2? Was it in connection
7 with these proceedings between Harvard and
8 Micron or some other context?

9 A. In the course of -- excuse me --
10 in the course of reading the literature, this
11 was published a number of years ago, prior to
12 any knowledge of these proceedings. So it
13 was somewhat of a mini review article that
14 focused on a variety of different precursors.

15 Q. I see. So if I understand you
16 correctly, then, the first time you reviewed
17 Exhibit 2, the Jones paper, was in the
18 context of your work in the field some number
19 of years ago, unrelated to the dispute?

20 A. Yes.

21 Q. And with respect to -- back to
22 Dr. Jones. Is he considered to be a
23 respected person in the field?

24 A. Yes, I would say so.

25 Q. It says that he's a professor in

1 the Department of Chemistry at the University
2 of Liverpool. Do you know if that's still
3 his position?

4 A. No, I don't.

5 Q. Have you read other papers by
6 Dr. Jones?

7 A. Yes, I have.

8 Q. And has he published fairly
9 frequently in the field of precursors and ALD
10 and CVD?

11 A. Yes.

12 Q. All right. You mentioned that
13 this is a review article, and just for our
14 record, can you explain what a review article
15 is?

16 A. It is an article that summarizes
17 not just the recent results of a particular
18 set of experiments conducted by the authors
19 but also tries to give an overview of the
20 broader area.

21 Q. You're aware that Exhibit 2 was a
22 paper that was submitted by Harvard in
23 connection with the prosecution of the '016
24 patent; is that correct?

25 A. Yes.

1 Q. You have -- and I take it with
2 respect to that fact, you became aware of
3 that in the context of the disputes between
4 Harvard and Micron; is that fair?

5 A. Yes.

6 Q. Take a look, if you would, please,
7 at the second page of Exhibit 2 starting at
8 the left-hand column, and -- well, I guess
9 there and in other places in the paper
10 there's a reference to MOCVD.

11 Do you see that acronym there?

12 A. Yes.

13 Q. And does that stand for
14 metalorganic CVD?

15 A. Yes.

16 Q. What is metalorganic CVD?

17 A. Metalorganic is a designation
18 associated with the type of precursor used in
19 the deposition process, and it's simply a
20 statement that the precursor has a metal and
21 an organic component to it.

22 Q. And when you refer there to an
23 organic component to it, what do you mean?

24 A. In other words, the molecule
25 itself has both the metal and the end ligands

1 that include hydrocarbon groups of some sort,
2 organic groups.

3 Q. So groups with carbon, with
4 hydrogen, with oxygen, things like that?

5 A. Yes.

6 Q. Okay. Take a look -- there's a
7 paragraph at the bottom of the left-hand
8 column of the second page of Exhibit 2 that
9 runs over to the top of the second page.
10 Just take a moment to read that to yourself
11 and let me know when you have.

12 (Witness reviewing document.)

13 A. Yes.

14 Q. Okay.

15 A. Thank you.

16 Q. And in this paragraph Dr. Jones is
17 describing physical properties, chemical
18 properties that make precursors suitable or
19 unsuitable for MOCVD and ALD; is that right?

20 MR. LEN: Objection.

21 A. Certainly he's listing several
22 components here.

23 Q. And have you read this paper in
24 its entirety?

25 A. Yes.

1 Q. And does Dr. Jones list anywhere
2 else in the paper other properties or trades
3 that make precursors suitable for MOCVD and
4 ALD besides what he lists in this paragraph?

5 MR. LEN: Objection.

6 A. Well, memory -- okay. My
7 recollection is that there is some refinement
8 of some of the ideas that are presented in
9 that introductory paragraph, but I would
10 really need to take a -- need to take time to
11 review the paper to find these.

12 Q. Okay. Let me ask it differently
13 then?

14 A. Okay.

15 Q. Without taking the time to read a
16 20-page paper -- I couldn't hear.

17 A. It would take me a long time.

18 Q. Without reading this line for line
19 and word for word, do you recall any other
20 properties that Dr. Jones listed as being
21 properties that made a precursor suitable for
22 ALD or MOCVD besides the ones listed on the
23 second page?

24 A. The -- so I can go through some of
25 these and spot examples. Design of a ligand,

1 for instance, one might have to be concerned
2 about certain types of reactions that occur
3 at lower energy. One of these might be a
4 beta-hydrogen elimination. I saw that
5 towards the end.

6 Q. My question is limited to the
7 question of suitability. And that's why I'm
8 saying if you remember this, that's fine, if
9 you don't, I don't want to take the time to
10 read it.

11 Does Dr. Jones, in the paper,
12 describe any other properties or trades for
13 making a precursor suitable, which is a word
14 he uses on page 2, and my question is: Do
15 you recall him using that standard anywhere
16 else in the paper?

17 MR. LEN: Objection.

18 A. Well, I think I can answer that I
19 could not point to a particular spot. One of
20 the -- my interpretation of this paper is
21 that one important component is he is trying
22 to highlight the factors -- the design
23 features that are different in CVD precursor
24 versus one that's good for an ALD precursor.

25 Q. Okay. All right. Let's move on

1 then.

2 MR. BOBROW: Let's mark this next.

3 (Exhibit 3, Bates No. MIHA0039892, marked
4 for identification.)

5 BY MR. BOBROW:

6 Q. Sir, you've been handed Exhibit 3
7 for identification. This is a PCT patent
8 publication WO 001/5865, with inventors
9 Leskela, Ritala, and others. It bears a
10 production number MIHA0039892 to 39914. This
11 is not an exhibit to your declarations.

12 My question, first off,
13 Dr. Gladfelter, is whether you have seen
14 Exhibit 3 before?

15 A. May I take a moment to take a look
16 at it.

17 Q. Yes.

18 MR. LEN: Jared, just while he
19 looks at this, obviously, we'll allow a
20 leeway of questioning, but tie this to
21 the references asserted in the petitions
22 in his declaration, I assume?

23 THE WITNESS: So I can respond
24 that I've read a patent by Leskela that
25 covers this material. I don't remember

1 if it was exactly this particular
2 application. It looks very similar to
3 what I remember reading on deposition of
4 strontium, Titan 8, but I just wanted to
5 clarify that --

6 BY MR. BOBROW:

7 Q. So if I understand what you're
8 saying, you're not positive that you read
9 Exhibit 3 before, but you believe that you
10 have read the material that is included in
11 Exhibit 3 before; is that fair?

12 A. Yes.

13 Q. Okay. In what context had you
14 read the material included in Exhibit 3
15 before? Was it in the context of the
16 disputes between Micron and Harvard or some
17 other context?

18 MR. LEN: Objection.

19 A. Yes.

20 Q. Sorry. I asked a compound
21 question.

22 Was it in the context of the
23 Micron-Harvard dispute?

24 A. I did read it in context of the
25 Micron-Harvard dispute.

1 Q. Was that the first time that you
2 had read the material included in Exhibit 3?

3 MR. LEN: Objection.

4 A. There is material included in
5 Exhibit 3 on this use of a strontium
6 precursor that contains a cyclopentadienyl
7 derivative in a published article. That is
8 probably the first time I read information
9 about exhibit -- the similar content, which
10 is what your question was, not identical.

11 But the first time that I saw
12 this -- the patent associated with that
13 science was associated with this -- these
14 proceedings.

15 Q. Thank you.

16 A. These proceedings being the
17 Harvard-MIT dispute.

18 Q. Thank you.

19 Now, do you know Dr. Leskela?

20 A. Yes, I do.

21 Q. You met him before?

22 A. Yes.

23 Q. Have you met him in vapor
24 deposition conferences?

25 A. Yes.

1 Q. Have you met him at atomic layer
2 deposition conferences?

3 A. Yes.

4 Q. Have you heard him speak at those
5 conferences?

6 A. Yes.

7 Q. Is he considered to have a good
8 reputation in the field of atomic deposition?

9 A. Yes, he is.

10 Q. What about Dr. Ritala?

11 A. No.

12 Q. Do you know Dr. Ritala?

13 A. Yes, I do.

14 Q. Have you met him at conferences?

15 A. Yes.

16 Q. Have you met him at conferences on
17 atomic layer deposition?

18 A. Yes.

19 Q. Does Dr. Ritala have a good
20 representation in the field of atomic layer
21 deposition?

22 A. Yes.

23 Q. This patent refers to a process by
24 the acronym of atomic layer epitaxy or ALE.

25 Do you see what I'm referring to,

1 in the abstract on the second page? Do you
2 see that?

3 A. Yes.

4 Q. In the context of the material
5 that you had read before, even if you hadn't
6 read this patent, it was your understanding
7 that the process described as ALE is the same
8 as the atomic layer deposition?

9 MR. LEN: Objection.

10 A. My understanding is that they're
11 referring to -- that the work here is -- that
12 ALE, and this is an example of being
13 synonymous with ALD.

14 Q. ALE and ALD being synonymous?

15 A. Yes.

16 Q. In your understanding of the work
17 about which you read before and as reflected
18 in this patent, that this is a description of
19 a method for forming oxide thin films; is
20 that correct?

21 MR. LEN: Objection.

22 A. The patent is specifically
23 referring to the use of a particular set of
24 ligands to volatile -- that are effective,
25 according to the researchers, at volatilized

1 strontium and barium, and they use this to
2 make metal oxide films that contain strontium
3 and barium.

4 Q. And titanium as well?

5 A. Right. But at least the titanium
6 precursor used in their experiments was a
7 known precursor. These were -- I don't
8 believe these had been demonstrated to be
9 useful precursors, the barium and strontium
10 precursors prior to the work by Leskela and
11 Ritala.

12 Q. Let me ask you to turn a few pages
13 into the document, and directing your
14 attention to page 39900, and beginning at
15 line 13, there's a reference to some listed
16 titanium compounds.

17 Do you see what I'm referring to
18 there?

19 A. Yes, I do.

20 Q. And it describes these titanium
21 compounds as having vapor pressures that are
22 sufficient at a temperature lower than the
23 temperature of the deposition substrate, and
24 which are thus then suitable for use in the
25 method according to the present invention.

1 Do you see what I'm referring to?

2 A. Yes.

3 Q. First off, what is vapor pressure?

4 A. Vapor pressure is the equilibrium
5 concentration of the molecule in the gaseous
6 state in contact with the molecule in the
7 liquid or the solid state.

8 Q. When, in the field of vapor
9 deposition, someone is referring to vapor
10 pressure or a sufficient vapor pressure, what
11 is that a reference to?

12 A. That's somewhat broad, but a
13 question, so I will answer it as best as I
14 can.

15 In referring to a precursor for
16 vapor deposition processes, you have to have
17 a certain concentration of the molecule in
18 the gas phase to be transported into the
19 reaction chamber where it can interact with
20 the substrate.

21 MR. LEN: Sorry to interrupt.

22 Unless we're wrapping up soon, can we go
23 off the record, or do you have a lot
24 more questions on this document?

25 MR. BOBROW: I don't think a lot.

1 But a couple. Why don't we just keep
2 going and then we'll see where we go.

3 MR. LEN: Well, I would like to
4 have a discussion off the record to try
5 to understand how this is tying to the
6 IPRs at issue that Dr. Gladfelter is
7 here today to testify about and his
8 declarations.

9 MR. BOBROW: Right. And you'd
10 agree with me I'm not bound by the
11 exhibits that he attached to his
12 declaration for the scope of my
13 questioning, correct?

14 MR. LEN: Correct. But we're here
15 to discuss the IPRs.

16 MR. BOBROW: That's right.

17 MR. LEN: And this is not an
18 asserted reference in the IPRs.

19 MR. BOBROW: But I'm not bound to
20 show the doctor only those things that
21 are exhibits to the transcript. I'm
22 entitled to a broader scope than that,
23 and I'm simple asking some questions
24 about this reference.

25 I don't think it's going to be

1 that long. I think we're taking up a
2 lot of time. I don't appreciate that.
3 I have a scope to be able to go beyond
4 what is in the four corners of the
5 papers and that's what I'm doing.

6 MR. LEN: Understood.

7 MR. BOBROW: Thank you.

8 MR. LEN: But we're here also for
9 the IPRs.

10 MR. BOBROW: I understand that.

11 BY MR. BOBROW:

12 Q. So back to page 39900. Under the
13 paragraph that I directed your attention to
14 first, there is -- there's some dashed
15 paragraphs, I'll call them, quite a few of
16 them, underneath that paragraph that starts
17 at line 13.

18 Do you see what I'm referring
19 to?

20 A. Yes.

21 Q. All right. And the fourth
22 bulleted paragraph down begins with a phrase
23 "alkylamino complexes of titanium."

24 Do you see what I'm referring to
25 there?

1 A. I do.

2 Q. And what is an alkylamino complex
3 of titanium?

4 A. In the context listed here, it is
5 a -- it is synonymous with an alkylamido
6 compound, the titanium.

7 Q. And the '539 patent and the '016
8 patent list a number of alkylamido complexes
9 of titanium, correct?

10 A. Yes.

11 MR. LEN: Objection.

12 A. Yes.

13 Q. And the '539 patent refers to
14 metal amides having -- is it an amido, is
15 that how you pronounce it, or amido?

16 A. Amido.

17 Q. Go figure.

18 Having an amido group selected
19 from the group consisting of the dialkylamido
20 moieties.

21 Do you see where I'm referring to
22 there at Column 32 of the Exhibit 1, the '539
23 patent?

24 A. I see metal amide, and in number
25 24, that's line 17, it includes dialkylamido.

1 Q. Okay. And with respect to
2 Exhibit 3, page 7, which is 39900 by Bates
3 number, is tetrakis(diethylamino)titanium, is
4 that an example of a metal amide -- amide
5 having a dialkylamido moiety?

6 MR. LEN: Objection.

7 A. Yes, it is a -- it is.

8 Q. And staying with the '539 patent
9 and looking at Table 1, which starts on the
10 page with Columns 9 and 10 and runs all the
11 way to the pages with Columns 15 and 16, take
12 a look on -- sorry -- take a look at Column
13 13. And about halfway down the page there is
14 a compound and the compound seems to be
15 Ti(NEt, sub 2)4.

16 Do you see what I'm referring to
17 there?

18 A. Yes.

19 Q. And is that compound listed in the
20 '539 patent tetrakis(diethylamino)titanium?

21 A. Yes. Did you say amino or amido?

22 Q. Good point. I said amino.

23 A. I thought he used the word amido.

24 Q. Dr. Gordon?

25 A. Doctor -- being the authors, yes,

1 of the '539 patent. But I will also say that
2 in the context of this -- of metal complexes
3 of this sort, sometimes you see those two
4 being used interchangeably.

5 Q. I see. And in Exhibit 3 on page
6 7, you understand the reference to
7 tetrakis(diethylamino)titanium to be
8 referring to the same metal dialkylamide as
9 the titanium (NEt 2)4, in Column 13 of the
10 '539 patent?

11 MR. LEN: Objection.

12 A. Again, let me just clarify. You
13 specified the tetrakis(diethylamino)titanium?
14 Is that the one you were --

15 Q. That's the one.

16 A. That's my understanding, yes.

17 Q. And the
18 tetrakis(diethylamino)titanium in Exhibit 3,
19 you would understand that to be a metal
20 dialkylamide? Exhibit 3 is the one right in
21 front of you.

22 A. Right. That was the same compound
23 you were just discussing, the diethylamido.
24 And your question was again?

25 Q. Do you understand the

1 tetrakis(diethylamino)titanium to be a metal
2 dialkylamide?

3 MR. LEN: Objection.

4 A. Yes, it's one member of those, of
5 that broad category.

6 Q. The next -- staying with
7 Exhibit 3, at line 22, of page 7, there's a
8 reference to tetrakis(dimethylamino)titanium.

9 Do you see that?

10 A. Yes.

11 Q. Is that also a metal dialkylamide?

12 MR. LEN: Objection.

13 A. Yes, it is.

14 Q. Is that also referred to in the
15 '539 Gordon patent in Column 13?

16 A. Yes, it's referred to.

17 Q. And is that referred to by
18 $\text{Ti}(\text{NME } 2)_4$?

19 A. Correct.

20 Q. The next compound on page 7 of
21 Exhibit 3, the Leskela patent publication, is
22 tetrakis(ethylmethylanino)titanium.

23 Do you see that?

24 A. I do.

25 Q. Is that a metal dialkylamide?

1 MR. LEN: Objection.

2 A. Yes, I would clarify it as a
3 dialkylamide.

4 Q. Is the
5 tetrakis(ethylmethyamino)titanium
6 dialkylamide also referred to in the '539
7 patent, Table 1, Column 13? Actually, as I'm
8 looking at it, it doesn't appear to be.

9 A. Yeah, I don't see it.

10 Q. And is -- okay. And then on page
11 7, Exhibit 3, the next compound listed is
12 tetrakis(isopropanolmethyamino)titanium. Is
13 that a metal dialkylamide?

14 MR. LEN: Objection.

15 A. Yes.

16 Q. In preparing your declarations
17 that you submitted in the intra partes review
18 proceedings, you did not attach or submit
19 Exhibit 3, the Leskela patent publication,
20 correct?

21 A. Correct.

22 Q. Did you consider Exhibit 3, the
23 Leskela patent publication, in expressing and
24 forming your opinions in the declarations
25 that you submitted in the 662, 663, and 664

1 IPR proceedings?

2 MR. LEN: Objection.

3 A. For the IPR proceedings?

4 Q. Correct.

5 A. Memory. We have talked about
6 this, discussed this. "We" being the
7 attorneys at Pepper Hamilton. But I don't
8 remember explicitly when the discussion took
9 place and whether it was in reference to the
10 IPR proceedings that we're in now, or whether
11 this was included in discussion associated
12 with the claim construction proceedings or
13 other earlier proceedings. So I'm trying to
14 remember, and I don't, off the top of my
15 head, recall.

16 Q. Okay. I think I understand.

17 MR. LEN: If he finished his
18 answer, I appreciate -- he referenced
19 that he's generally aware of technology,
20 but I caution the witness not to refer
21 to specifics of discussions with Pepper
22 Hamilton attorneys.

23 But I don't think that's what you
24 were looking for, Jared, so.

25 MR. BOBROW: No, I'm not, but I do

1 need to ask some follow-up questions on
2 this.

3 BY MR. BOBROW:

4 Q. With respect to the IPR
5 proceedings and the declarations that you
6 submitted in connection with those
7 proceedings, did you consider what is
8 disclosed in Exhibit 3 in expressing and
9 formulating your opinions that you expressed
10 in those declarations?

11 MR. LEN: Objection. Just to
12 clarify, you're asking him if he
13 considered an exhibit that we already
14 established is not attached to his
15 declarations. Is that what you're
16 asking him?

17 MR. BOBROW: Well, I put it the
18 way that I put it, and I'll stand on my
19 question.

20 MR. LEN: Okay. Objection
21 stands.

22 THE WITNESS: Would you ask the
23 question again, please?

24 BY MR. BOBROW:

25 Q. Sure. In connection with the IPR

1 proceedings, 662, 663, 664 --

2 A. Yes.

3 Q. -- in which you submitted
4 declarations, my question is: Did you
5 consider the disclosure in Exhibit 3 in
6 formulating and expressing your opinions in
7 those declarations?

8 MR. LEN: Objection.

9 A. I think I already clarified that.
10 I recall reviewing a patent. It may have
11 been based on this application, and I also
12 recall reviewing publication by -- on the
13 same topic. But I'm afraid what I can't
14 recall is whether that was with -- associated
15 specifically with the IPR proceedings or
16 whether it was some of the earlier work.

17 Q. In connection with the IPR
18 proceedings and the declarations that you
19 submitted in those proceedings, did you
20 consider the publication that you just
21 referenced, and whatever patent it might have
22 been, that includes the similar or the same
23 subject matter in formulating and expressing
24 the opinions that you set forth in your
25 declarations?

1 MR. LEN: Objection.

2 A. Again, I must repeat my earlier
3 comment. I'm familiar with this research.
4 And specifically at what point I read these
5 and became more familiar with them as a
6 result of the dispute between Harvard and
7 MTI, I don't remember the exact time.

8 Q. Is it fair to say that you cannot
9 say with confidence that you considered
10 either Exhibit 3 or the disclosures of the
11 subject matter in Exhibit 3 in formulating
12 and expressing the opinions that you did in
13 the IPR declarations?

14 MR. LEN: Objection.

15 A. Yep. I don't recall. Again, I
16 don't want to say what I discussed, but I
17 don't recall the point at which I was reading
18 and evaluating the significance of these --
19 of this research topic from Leskela and
20 Ritala.

21 Q. Right. What I'm trying to
22 understand specifically is the following:
23 Based upon what you just said, I'm
24 understanding you to say that you can't say
25 that you considered Exhibit 3 or the related

1 publication or patent in expressing and
2 formulating your opinions in the IPR
3 declarations.

4 MR. LEN: Objection.

5 BY MR. BOBROW:

6 Q. Is that fair?

7 MR. LEN: Objection. Asked and
8 answered.

9 A. Yes, I think that is correct.

10 BY MR. BOBROW:

11 Q. Okay. Thank you.

12 Now, you had mentioned --

13 MR. BOBROW: And, Mr. Len, I just
14 want to give you a heads-up out of
15 courtesy. I'm just going to ask a
16 couple of foundational questions about
17 the discussion that Dr. Gladfelter had
18 with you and your firm.

19 MR. LEN: Okay. But in the
20 preface to that, if there's something
21 that refreshed your recollection or
22 general facts of documents, that's fine.
23 But the substance of the discussions
24 that you may have had with Pepper
25 Hamilton attorneys is an area that you

1 should not be going into. I caution the
2 witness not to answer the substance of
3 the questions about discussions, but
4 we'll see where it goes.

5 MR. BOBROW: Yeah. Exactly.

6 BY MR. BOBROW:

7 Q. I'm not trying to find out in
8 detail what you and Mr. Len or others at his
9 firm talked about with respect to Exhibit 3.
10 So I just wanted to give you a heads-up about
11 that, which is why I gave a heads-up to
12 Mr. Len.

13 MR. LEN: I object. The witness
14 never said that -- discussed Exhibit 3,
15 but with that reservation, let's move
16 forward.

17 MR. BOBROW: Okay.

18 BY MR. BOBROW:

19 Q. So the questions -- let me ask
20 this: First question is: At some point
21 prior to today, had you discussed -- just yes
22 or no -- had you discussed Exhibit 3 with
23 Mr. Len or anyone at the Pepper Hamilton
24 firm?

25 MR. LEN: Objection.

1 A. Yes.

2 Q. Now, can you place in time how
3 long before today you had a discussion with
4 Mr. Len or anyone at the Pepper Hamilton firm
5 about Exhibit 3?

6 MR. LEN: Objection.

7 A. Within the last year.

8 Q. Okay. And can you be more
9 specific about the time in which you had a
10 discussion with Mr. Len or someone in the
11 Pepper Hamilton firm about Exhibit 3?

12 MR. LEN: Objection.

13 A. I think not.

14 Q. Now, just to refresh your memory,
15 possibly refresh your memory, the last time
16 that I took your deposition was some number
17 of months ago in September of 2017. Do you
18 recall -- I think we were in the conference
19 room across the hall for your deposition.

20 Do you remember that?

21 A. I recall.

22 Q. Okay. Had you reviewed Exhibit 3
23 or the paper, or patent, or publications with
24 similar overlapping subject matter prior to
25 the time that I took your deposition on the

1 claim construction issues in the District
2 Court proceeding?

3 MR. LEN: Objection.

4 A. As actually I think I said
5 earlier, is the -- the review of this content
6 may have been with -- in reference to the
7 claim construction deposition back in
8 September.

9 Q. All right.

10 MR. BOBROW: Let's mark this as
11 Exhibit 4.

12 (Exhibit 4, Cp2Me2Zr document, marked for
13 identification.)

14 BY MR. BOBROW:

15 Q. Sir, you've been handed a document
16 that we have marked as Exhibit 4 for
17 identification. Exhibit 4 contains some
18 typed material with some names of some
19 chemical compounds, four bullet points of
20 them.

21 And I wanted to ask you about the
22 compounds that are listed here. First of
23 all, there's a compound Cp2Me2Zr.

24 Do you see that?

25 A. Yes.

1 Q. Do you know what that compound is?

2 A. I believe so.

3 Q. What is it?

4 A. It is a zirconium complex with two
5 cyclopentadienyl ligands that is a -- and
6 probably two methyl ligands. So a total of
7 four ligands attached to the zirconium. The
8 formula itself is a little unclear about
9 that. It's not inconceivable that those
10 metals are supposed to be indicated on -- to
11 be attached to the Cp, but I doubt that
12 that's the case.

13 Q. Have you seen in the literature
14 any references to this complex before, in the
15 first bullet point?

16 MR. LEN: Objection.

17 A. Yes, I have seen reference to that
18 compound. If, again, it's the dimethyl
19 bis(Cp) complex.

20 Q. Have you seen that complex used in
21 chemical vapor deposition?

22 A. I can't specifically remember.
23 This is a vague memory, but I believe people
24 have used this to deposit zirconium carbide
25 in a so-called single-source CVD process.

1 But that is not a certain statement.

2 Q. Okay. Is this complex Cp₂Me₂Zr,
3 is that a complex -- do you know whether
4 that's a complex that can be purchased
5 commercially?

6 MR. LEN: Objection.

7 A. I think so. Usually, if we were
8 to do that, we would buy the corresponding
9 bis(Cp)zirconium dichloride and make it, but
10 I think there have been times where this has
11 been a commercially available material.

12 Q. Have you synthesized that complex
13 in your lab before?

14 A. We have not synthesized this
15 compound.

16 Q. Have you seen in the literature
17 others having synthesized this compound?

18 MR. LEN: Objection.

19 A. I have seen in the literature that
20 others have made this compound.

21 Q. Okay. All right.

22 Next bullet down there are a
23 number of compounds that are listed there.
24 Are you familiar with those compounds?

25 MR. LEN: Objection.

1 A. I'm sorry. I'm just going through
2 the list.

3 Q. Sure.

4 A. I'm familiar with a class of
5 compounds that contain four alkoxy ligands
6 attached to the metals. I'm familiar with
7 some of these compounds themselves, but
8 others I'm -- I don't recall ever seeing
9 reference to them. Or perhaps there is
10 reference to them; but, you know, it's an
11 example where I perhaps don't recall.

12 Q. Are the compounds in the second
13 bullet on Exhibit 4 examples of alkoxides?

14 A. Yes, that is my understanding of
15 that.

16 Q. Now, the third bullet down lists
17 tetrakis(dimethylamino)zirconium or hafnium.
18 Do you see that?

19 A. I do.

20 Q. Are you familiar with those
21 compounds?

22 A. Yes.

23 Q. Have you ever purchased those
24 compounds before?

25 A. We might have. I don't remember,

1 to be honest.

2 Q. Have you synthesized those
3 compounds before?

4 A. Again, we might have. We didn't
5 work a great deal with the zirconium and
6 hafnium dimethylamido complexes ourselves.
7 So I'm a little bit -- I'm hesitant to say we
8 either purchased or made specifically these
9 materials.

10 Q. You've seen those materials
11 referred to in the literature?

12 A. Yes.

13 Q. In the fourth bullet there's a
14 reference to tetrakis(diethylamino)zirconium
15 or hafnium.

16 Do you see that?

17 A. I do.

18 Q. Are you familiar with that
19 compound?

20 A. Yes.

21 Q. Have you synthesized it before?

22 A. The answer is more or less the
23 same as I reported for the dimethyl
24 derivative.

25 Q. You've seen the

1 tetrakis(diethylamino)zirconium or hafnium
2 referred to in the literature?

3 A. Yes.

4 Q. Is tetrakis(diethylamino)zirconium
5 or hafnium available for purchase?

6 A. I believe that it is available for
7 purchase.

8 Q. Is that also true for the
9 tetrakis(diethylamino)zirconium or hafnium?

10 MR. LEN: Objection.

11 A. I also believe that that's
12 available.

13 Q. Okay. Now, if we wind the clock
14 back to the 1990s time frame, that decade,
15 were these compounds listed on Exhibit 4; did
16 they exist?

17 MR. LEN: Objection.

18 A. The bis(Cp)dimethylzirconium, your
19 first bullet point did exist. The third and
20 fourth bullet points did exist. Several of
21 the alkoxides on the second bullet point I
22 know to have existed. Some of the compounds
23 on the second bullet point, specifically the
24 fluorinated examples, I don't know to have
25 existed back in the 1990s. They may have,

1 but I don't remember off the top of my head.

2 Q. All right. Thank you.

3 With respect to the second bullet
4 point, can we go through them, and I'd like
5 you to list for me the ones that you know did
6 exist in the 1990s.

7 MR. LEN: Objection.

8 A. The methoxy derivative, that's the
9 first one in the list, the ethoxy derivative.

10 Q. The second one?

11 A. The second item on the list. The
12 isopropoxy derivative. The tert, t-e-r-t,
13 butoxy derivative. I can't specifically say
14 whether the isobutoxy derivative existed. I
15 think the tetrabutoxy derivative did. And
16 with respect to the fluorinated derivatives,
17 the next three, I'm not sure when they first
18 were prepared and characterized.

19 The next item on the list, in
20 essence, is a redundant one. That is the
21 tetra-tert-butoxy and that did exist. The
22 silyl derivative, the trimethylsilyl
23 derivative, that's tetra -- that's the same
24 as the last derivative. Those two are
25 identical. I'm sorry. They are not

1 identical. They are not. One is the
2 tristrimethylsilyl butoxy derivative, and the
3 other is the trimethylsiloxo derivative. So
4 they are different.

5 And what I would have to say is,
6 again, I'm not sure exactly when they were
7 first prepared or -- and characterized.
8 Likewise, with the one in between them, the
9 tristrifluoro compound, I don't remember when
10 and -- these first came on the scene. They
11 are all part of a group of compounds.
12 Nothing is particularly surprising, in
13 differentiating them, that falls in the
14 category of metal alkoxides.

15 And, again, I am responding to the
16 point, I don't recall when they were first
17 prepared, and some of them -- in fact, I
18 don't recall when any of them were first
19 prepared and characterized. I'm recalling
20 when I may have first seen them.

21 Q. In the 1990s, which is the time
22 period that I asked you about, had the types
23 of -- had alkoxides generally in the art of
24 CVD and ALD been characterized?

25 MR. LEN: Objection.

1 A. Yes. I think we already discussed
2 in an earlier exhibit the Jones review
3 article that indeed talks about alkoxides as
4 precursors. That's just one example of these
5 molecules.

6 Q. Okay. Now, talking about
7 Exhibit 4 broadly and the various compounds
8 that are referred to on Exhibit 4, in the
9 decade of the 1990s or earlier, were these
10 compounds -- let me ask it a little
11 differently.

12 In the 1990s, with respect to the
13 compounds listed in Exhibit 4, were the
14 compounds that you described being -- knowing
15 to have existed at that time, had their vapor
16 pressures been characterized in the 1990s?

17 MR. LEN: Objection.

18 A. I don't know when the vapor
19 pressures were first reported. Some of the
20 compounds, such as the tetramethoxy
21 derivative is, if I recall, oligomeric, maybe
22 even polymeric, and I don't think that has a
23 very good vapor pressure, and that may be
24 true also for the ethoxy derivative.

25 I know that the tert-butoxy

1 derivative at temperature -- again, it was
2 certainly measured, and it has been used as a
3 precursor, but I don't recall when the first
4 report of the vapor pressures appeared for
5 these.

6 Q. With respect to the compounds
7 listed on Exhibit 4, in your view, would
8 determining the vapor pressure of these
9 compounds have been something that would be
10 difficult or beyond the skill of a person of
11 ordinary skill?

12 MR. LEN: Objection.

13 A. No, I don't believe so. The
14 determination of vapor pressure used to be a
15 common characterization method for new
16 compounds that fell by the wayside as the
17 quality and -- and capabilities of other
18 types of methods for characterizing compounds
19 became more prevalent, but measurement of
20 vapor pressure is a relatively routine
21 component of studies that make use of
22 molecules as precursors for vapor deposition
23 processes.

24 Q. What about the bond strengths of
25 the chemical bonds of these compounds? Would

1 that have been, in the 1990s, a relatively
2 routine thing to have determined?

3 MR. LEN: Objection.

4 Q. By a person of ordinary skill in
5 the art?

6 MR. LEN: Same objection.

7 A. Well, in response to your
8 question, no.

9 Q. And were the bond strengths of the
10 compounds that are listed here, were these
11 things that could be looked up in a chemical
12 reference book?

13 MR. LEN: Objection.

14 A. There are, of course, in these
15 molecules many bonds. So perhaps you could
16 clarify what bonds you're interested in.

17 Q. Well, for example, in the
18 tetrakis(diethylamino)zirconium compound, are
19 there bonds there which would be -- you know,
20 which you would be unable to look up in a
21 chemical text?

22 A. There are some reports of the --
23 of bond strengths. Again, something like the
24 tetrakis(dimethylamino) as it's listed here,
25 zirconium compound, has carbon hydrogen bonds

1 and you can look up characteristic carbon
2 hydrogen bonds in standard tables. One can
3 look up carbon nitrogen bonds, which are also
4 present in standard tables.

5 But it is not so common to see
6 metal ligand bond strengths. Not unknown by
7 any means, but I believe there was work, at
8 least with titanium or some of the elements,
9 to sort that out. But that's trying to
10 remember back to the 1990s.

11 Q. So back in the 1990s, was it a
12 routine matter for a person of ordinary skill
13 to look up a carbon hydrogen bond strength?

14 MR. LEN: Objection.

15 A. I guess it would have been routine
16 to be able to look that up, and it's taught
17 in organic texts. Usually the compounds that
18 are used to create those numbers are simple
19 organics. They don't contain metals.

20 Q. What about the carbon nitrogen
21 bond, is that a bond that a person of
22 ordinary skill in the 1990s would have been
23 able to look at in a routine matter, the bond
24 strength?

25 A. Absent of a metal, yes.

1 Q. And then with respect to bonds
2 between nitrogen and metals, you know, amide
3 bonds, back in the 1990s, was that
4 something -- were those bonds something that
5 appeared in the literature -- or the bond
6 strengths, I should say, that appeared in the
7 literature?

8 MR. LEN: Objection.

9 A. I recall some examples. It is
10 much more difficult to be quantitative about
11 the metal nitrogen bonds using thermodynamic
12 methods.

13 Q. If I understand you, though, your
14 recollection is that at least some nitrogen
15 metal bonds strengths had been reported in
16 the literature; is that fair?

17 MR. LEN: Objection.

18 A. That is correct. I agree.

19 Q. What about oxygen carbon bonds, is
20 that something that a person of ordinary
21 skill in the 1990s would have the ability to
22 look up routinely in a reference book or the
23 literature?

24 MR. LEN: Objection.

25 A. In the 1990s, one could look this

1 up for organic compounds.

2 Q. What about oxygen silicon bonds?
3 Is the bond strength of oxygen silicon bonds
4 something that a person of ordinary skill
5 could have looked up routinely in reference
6 books or in the literature, in the 1990s?

7 A. Yes.

8 Q. And what about carbon fluorine
9 bonds? Were carbon fluorine bonds -- let me
10 ask it a little differently.

11 Back in 1990s, would a person of
12 ordinary skill in the art have been able to
13 look up the bond strength of carbon fluorine
14 bonds?

15 MR. LEN: Objection.

16 A. For organic fluorinated compounds,
17 there were some of these available, yes.

18 Q. And would that have been a routine
19 matter to go to a handbook or a textbook or
20 the like and look up the strength of a bond
21 between --

22 A. Carbon and fluorine.

23 Q. -- carbon and fluorine?

24 MR. LEN: Same objection.

25 A. I believe so.

1 Q. In your work in and before the
2 1990s, in your lab, had you been involved in
3 attempting to either quantify or evaluate
4 relative strength of bonds between, you know,
5 nitrogen and metal?

6 MR. LEN: Objection.

7 A. You included in that statement the
8 relative strengths. Are you implying that I
9 would have studied the strength of one ligand
10 versus another?

11 Q. I guess what I had in mind, and I
12 wasn't being very clear, was, I could imagine
13 the bond strength between an aluminum atom
14 and a nitrogen atom being different than the
15 bond strength between a titanium atom and a
16 nitrogen atom. And so my question was trying
17 to cover both, so maybe we should break it
18 up.

19 So in the first instance for any
20 given metal, were you aware -- sorry -- for
21 any given metal, did you, in your lab,
22 attempt to quantify a bond strength between a
23 particular metal and a nitrogen?

24 A. No.

25 MR. LEN: Objection.

1 Q. Back in the 1990s and earlier,
2 were you aware of reports in the literature
3 that attempted to quantify, you know, such
4 bond strength between a metal and a nitrogen,
5 even if you didn't do that yourself?

6 MR. LEN: Objection.

7 A. I mentioned already that there
8 were reports of metal nitrogen bond strengths
9 in the literature.

10 Q. All right. And in the literature
11 in the 1990s and earlier, were you aware that
12 there had been reported information on the
13 bond strength between zirconium metal and
14 nitrogen?

15 A. That I can't remember.

16 Q. You can't remember one way or the
17 other?

18 A. Whether that existed at the time.

19 Q. Or not?

20 A. Or not.

21 Q. What about hafnium? In the 1990s
22 and earlier, were there reports in the
23 literature on the bond strength between
24 hafnium metal and nitrogen?

25 MR. LEN: Objection.

1 A. My answer is the same. I don't
2 recall or not the existence of data for that.

3 Q. With respect to the compounds
4 listed on Exhibit 4, back in the 1990s and
5 earlier, was information available in the art
6 about the melting points of these compounds?

7 A. I believe there were.

8 Q. What about decomposition
9 temperatures of these compounds, was there
10 information available in the 1990s and
11 earlier in the literature about that?

12 MR. LEN: Objection.

13 A. I suspect there were for at least
14 some of them.

15 Q. And what about evaporation
16 temperatures? Was information available
17 about the temperatures at which these
18 compounds would evaporate in the 1990s and
19 earlier?

20 MR. LEN: Objection.

21 A. It is possible the information
22 existed as a result of synthesizing some of
23 these compounds. One often has to purify
24 them, and one of the techniques to purify a
25 compound is to sublime it, to sublime it,

1 and that requires temperature and pressure to
2 be known. It's not a direct measure of the
3 vapor pressure. It's used frequently as a --
4 a rough measure that the molecule has a
5 finite volatility.

6 Q. Okay. And back in the 1990s and
7 earlier, would you expect that the compounds
8 listed on Exhibit 4 could have been isolated
9 by a person of ordinary skill?

10 A. I agree with that.

11 Q. And back in the 1990s and earlier,
12 would you agree that a person of ordinary
13 skill in the art could synthesize the
14 compounds listed on Exhibit 4 in some sort of
15 a stable fashion?

16 MR. LEN: Objection.

17 A. I think so, yes.

18 MR. BOBROW: Let's mark this next.
19 (Exhibit 5, United States Patent No.
20 6,984,591, marked for identification.)

21 BY MR. BOBROW:

22 Q. All right. Dr. Gladfelter, you've
23 been handed Exhibit 5 for identification.
24 This is a copy of U.S. Patent No. 6,984,591.
25 This is Exhibit 1005 in both the 662 and 663

1 proceedings.

2 I take it that you have read
3 Exhibit 5, the Buchanan patent, before,
4 right?

5 A. Yes.

6 Q. You reviewed the Buchanan patent
7 in connection with your work in the IPRs,
8 correct?

9 A. Correct.

10 Q. Did you have a chance over the
11 last couple of days to review the Buchanan
12 patent, Exhibit 5?

13 A. Yes.

14 Q. Take a look, if you would for me,
15 at Column 16 of the Buchanan patent,
16 Exhibit 5. There's a heading in that column
17 that says, "Precursor Source Mixtures for
18 Nitrate-Containing Compound."

19 Do you see what I'm referring to
20 there?

21 A. I do.

22 Q. All right. Can you -- are you
23 familiar with nitrate-containing compounds?

24 A. Yes.

25 Q. What is a nitrate-containing

1 compound?

2 A. A nitrate is a ionic compound, has
3 a negative charge, has one nitrogen and three
4 oxygens. The nitrogen is in the center of
5 these three oxygens. It creates a triangle.
6 The three oxygens create a triangle, and the
7 nitrogen is in the center of that.

8 Q. Beneath that heading there is a
9 formula for nitrate-containing compounds I
10 believe at line 6 of Column 16 of the
11 Buchanan patent.

12 Do you see that?

13 A. I do.

14 Q. It says "MR1, sub x, R2, sub y, A
15 sub z.

16 Do you see what I'm referring to
17 there?

18 A. Yes.

19 Q. Do you know how to interpret that?

20 A. The description that's provided
21 is -- I think it begins line -- what is that,
22 12 perhaps, 12 or 13. R1 is identified as a
23 nitrate. R2 is a ligand that's one of many
24 that are listed. I won't read through all of
25 them, unless you wish to do so. And the "A"

1 is another ligand that would be bound to the
2 metal.

3 Q. All right. Now, if you take a
4 look towards the bottom of that column -- oh,
5 and I take it that "M" is metal, right?

6 A. I'm sorry. Yes.

7 Q. If you take a look at -- towards
8 the bottom of that Column 16, around line 51
9 running to 55, the Buchanan patent lists what
10 are called preferred precursor source
11 mixtures and what they're comprised of.

12 Do you see what I'm referring to
13 there?

14 A. I do.

15 Q. All right. And if I've got this
16 right, the first compound listed there is a
17 titanium nitrate; is that correct?

18 A. Yes.

19 Q. And the second is a zirconium
20 nitrate, correct?

21 A. Yes.

22 Q. The third is a hafnium nitrate; is
23 that correct?

24 A. Yes.

25 Q. What is the fourth one, the

1 silicon one?

2 A. The fourth one, the compound is
3 trimethylsilyl nitrate. So it has an oxygen
4 of the nitrate bound to silicon and also
5 three methyl groups.

6 Q. All right. Now, with respect to
7 the titanium compound listed there, I take it
8 that the only preferred titanium nitrate
9 compound is the $\text{Ti}(\text{NO}_3)_4$; is that right?

10 MR. LEN: Objection.

11 A. Okay. Just from reading what is
12 described in this paragraph, there are no
13 other titanium compounds referred to.

14 Q. No other titanium nitrate
15 compounds?

16 A. Or any titanium compound. But
17 yes.

18 Q. All right. And is it also the
19 case that there are no other zirconium
20 nitrate-based compounds that are listed under
21 that preferred precursor source mixture
22 heading?

23 A. Correct.

24 Q. There are no other hafnium
25 nitrate-based compounds other than the one --

1 specific one under the heading "Preferred
2 Precursor Source Mixtures," correct?

3 A. Correct. In that paragraph,
4 that's correct.

5 Q. In one of your earlier answers,
6 this was some time ago, but I believe it was
7 in the context of Exhibit 4, you used the
8 phrase "single-source CVD."

9 Do you recall using that phrase?

10 A. I do.

11 Q. What is a single-source CVD?

12 A. It refers to a precursor that can
13 be used without any additional reactants to
14 deposit a compound film -- excuse me -- a
15 compound film or an elemental film.

16 Q. Okay. All right.

17 Have you heard of the phrase
18 "binary chemical vapor deposition reaction"?

19 A. Yes, I have heard of it. It's not
20 one that I commonly use as opposed to saying
21 two-precursor process, but those two
22 approaches mean the same thing, in my
23 understanding.

24 Q. In your understanding, is a binary
25 CVD process different than a single-source

1 CVD process?

2 A. Yes.

3 Q. And how do those differ?

4 A. As I noted, a single-source
5 precursor has, within its molecular
6 structure, all elements necessary to deposit
7 a compound or an elemental film. A
8 two-source system is an example where you
9 have a precursor that will -- for deposition
10 of a film and another required precursor,
11 sometimes, as an example, you were depositing
12 a metal nitride, you might use a reactant for
13 the second -- for the nitrogen precursor as
14 ammonia, and you might use a metal-containing
15 precursor to deposit the metal. Sorry. Not
16 might. You would.

17 Q. So just by way of example, I could
18 take, for example, a titanium-bearing element
19 alkylamide, and I could use that for
20 single-source CVD, essentially if I just use
21 that compound by itself and form some kind of
22 titanium nitride compound film; is that
23 right?

24 MR. LEN: Objection.

25 A. There are examples of using

1 titanium alkylamides to deposit compound
2 films, yes.

3 Q. Of titanium nitride, for example?

4 A. The resulting film, in such cases,
5 is usually -- in fact, I think more
6 accurately is never a pure nitride. It
7 includes impurities, in particular carbon, in
8 the film. Also, typically hydrogen, but
9 that's less of an easily spotted impurity.

10 Q. And the idea of the two-source
11 precursor, the binary chemical vaporization
12 deposition, is I can take that same metal
13 precursor but instead of reacting -- causing
14 reaction by itself, I could add something to
15 the mix, like ammonia, to deposit the
16 compound film?

17 MR. LEN: Objection.

18 A. So it is the case that you could
19 add -- you may, depending on the precise
20 system, a source for ammonia. I already used
21 that particular example to deposit a metal
22 nitride.

23 Q. And, for example, I could use a
24 metal-bearing alkylamide with ammonia,
25 combine those together in CVD and form a

1 titanium nitride compound; is that right?

2 A. That has been done, yes.

3 Q. In the context of the binary CVD
4 that we were talking about earlier, have you
5 heard, in that context, of half-reactions?

6 A. Yes. I'm sorry. Would you ask
7 your question again?

8 Q. Yes. In the context of binary
9 chemical vapor deposition, have you heard of
10 the concept of separating such a binary
11 chemical vapor deposition reaction into
12 half-reactions?

13 A. So I need some clarification. Are
14 you referring to this just as a way to
15 explain the chemistry that is involved, or
16 are you talking about -- are you referring to
17 doing an actual deposition?

18 Q. Well, let's start with explaining
19 the chemistry. Have you heard of this
20 concept of separating a binary chemical vapor
21 deposition reaction into two half-reactions?

22 A. Well, to be honest, no. I
23 certainly, in explanation, recognize, and
24 it's an important component that these
25 reactions often involve many microscopic

1 events to convert starting material into the
2 desired product. There is no way to, in an
3 explanation, to -- I know of no way -- let me
4 clarify that -- to say this is half -- the
5 first half and this is the second half.

6 Q. Okay. In the context of
7 describing atomic layer deposition reactions,
8 have you heard them described in terms of
9 splitting or separating a binary chemical
10 vapor deposition reaction into two halves?

11 MR. LEN: Objection.

12 A. So your comment was, if I
13 understand or heard you correctly, you talked
14 about ALD, but then as being used to -- the
15 events in an ALD process being used to
16 explain the two halves, or two halves of a
17 CVD reaction?

18 Q. No.

19 A. Oh, okay.

20 Q. I'm sorry. I might have
21 misspoken. Let me try it again.

22 Have you heard of an ALD reaction
23 being described essentially as separating a
24 binary chemical vapor deposition reaction
25 into half-reactions?

1 A. Oh, I see.

2 So in a typical -- I think the
3 brief answer to this is yes.

4 Q. All right. And can you explain
5 what it means to a person of ordinary skill
6 to separate a binary chemical vapor
7 deposition reaction into two half-reactions?

8 A. Again, in an ALD process?

9 Q. Yes.

10 A. Okay. I can try.

11 The ALD process for materials, for
12 depositing in film, is temporally broken into
13 two sequential steps. And in one step you'll
14 have a reaction between a precursor and the
15 surface. Again, you transport the precursor
16 through the gas phase. It bounces around and
17 eventually hits and sticks on the surface,
18 and there's a chemical reaction associated
19 with forming a strong bond to the surface.

20 After that surface has been
21 saturated, then no further process will occur
22 between the surface and the excess precursor,
23 that is what is known to limit -- or lead to
24 self-limited growth in an ALD process. And
25 step one is done. In your terminology, that

1 would be the first half-reaction.

2 Now you switch to the second
3 reactant, transport it through the gas phase,
4 it bounces around and reacts with the
5 absorbed component from the first precursor,
6 and converts that into, in essence, the -- I
7 don't want to say the final product, but it
8 removes some of the additional ligands that
9 are still attached to the first precursor and
10 leaves you with a surface that is now
11 available to, once again, continue the cycle
12 by going back to step one.

13 And that second -- the addition of
14 that second reactant to the surface, once
15 again, will go through the process of -- let
16 me say it's saturated and leads to -- that
17 leads to the self-limited behavior of the
18 ALD. When that's done in an ALD process,
19 then you can go back and repeat that -- the
20 chemistry in those two steps as many times as
21 you wish to grow a given thickness of the
22 film.

23 Q. Would you describe the addition of
24 the second reactant, and went on after that,
25 is that the second of these half-reactions in

1 the binary CVD case?

2 A. Yes.

3 MR. BOBROW: It's about 12:10. I
4 was going to switch to another exhibit.
5 But perhaps we should take lunch, given
6 that we have a 12:45 call.

7 MR. LEN: Yeah.

8 MR. BOBROW: Why don't we take our
9 lunch break now.

10 (Recess taken at 12:10 p.m. to 1:19 p.m.)

11 MR. BOBROW: Back on the record.

12 Let's mark this as next in order.

13 (Exhibit 6, The Journal of Physical
14 Chemistry, marked for identification.)

15 BY MR. BOBROW:

16 Q. Dr. Gladfelter, you've been handed
17 Exhibit 6 for identification. This is the
18 cover page to the Journal of Physical
19 Chemistry, along with an article called
20 Surface Chemistry for Atomic Layer Growth by
21 S.M. George and others.

22 This exhibit, which we've marked
23 in this deposition is Exhibit 6, is
24 Exhibit 1011 in the 662 proceeding, 112 in
25 the 663 proceeding, and 1011 in the 664

1 proceeding.

2 Dr. Gladfelter, have you reviewed
3 Exhibit 6 before?

4 A. Yes.

5 Q. Was the first time that you
6 reviewed Exhibit 6 in the context of the
7 dispute between Micron and Harvard?

8 A. No.

9 Q. You had reviewed this in
10 connection with your work as a professor of
11 chemistry at the University of Minnesota?

12 A. Professor of chemistry.

13 Q. What did I say?

14 A. It sounded like history.

15 Q. Oh, I thought I said chemistry.

16 A. It might have been.

17 Yes, I reviewed it as part of my
18 research program.

19 Q. Is the Journal of Physical
20 Chemistry a publication that you review in
21 the course of your work in research at the
22 University of Minnesota?

23 A. Yes.

24 Q. Is it a respected publication?

25 A. Yes.

1 Q. Is this a publication in which the
2 submissions are peer reviewed?

3 A. Yes.

4 Q. With respect to the authors of the
5 paper, do you know S.M. George?

6 A. Yes.

7 Q. Is that Steve or Steven George?

8 A. Correct.

9 Q. And at the time of this article,
10 it appears he was a professor of chemistry --
11 or he was in the chemistry and biochemistry
12 department at the University of Colorado. Is
13 that how you know him?

14 A. Yes, it is.

15 Q. And is he still a professor in
16 that department?

17 A. Last I knew. It's relatively
18 recent, yes.

19 Q. And is Dr. George respected in the
20 field of chemical vapor deposition and atomic
21 layer deposition?

22 A. Yes, he is.

23 Q. And to your knowledge, has he been
24 working in those fields, you know, at least
25 since the 1990s?

1 A. Yes.

2 Q. And is Professor George, to your
3 knowledge, knowledgeable about ALD precursors
4 and CVD precursors?

5 A. Yes.

6 Q. And is he knowledgeable about ALD
7 reactions and CVD reactions?

8 A. Yes.

9 (Exhibit 7, Proceedings of the Twelfth
10 European Conference on Chemical Vapour
11 Deposition, Volume II, marked for
12 identification.)

13 BY MR. BOBROW:

14 Q. Dr. Gladfelter, before you now is
15 Exhibit 7 for identification. This is the
16 cover page of certain proceedings of the
17 Twelfth European Conference on Chemical
18 Vapour Deposition along with an attached
19 article from those proceedings entitled "ALD
20 precursor chemistry: Evolution and future
21 challenges."

22 This exhibit, Exhibit 7, is
23 Exhibit 1012 in the 662 proceeding, 1013 in
24 the 663 proceeding, and 1012 in the 664
25 proceeding.

1 Dr. Gladfelter, have you reviewed
2 Exhibit 7 before?

3 A. Yes.

4 Q. And when was the first time that
5 you reviewed Exhibit 7?

6 A. It would have been a number of
7 years ago, prior to these proceedings, but as
8 part of our research program.

9 Q. Are you familiar with the -- this
10 European Conference on Chemical Vapour
11 Deposition?

12 A. Yes, I am.

13 Q. Have you attended the proceedings
14 of that conference before? Sorry. Have you
15 attended that conference before, I should
16 say?

17 A. I've attended -- I didn't attend
18 this particular conference in Barcelona, but
19 I have attended other conferences. Other
20 EuroCVD conferences.

21 Q. At the EuroCVD Conference, does
22 the conference discuss not only the chemical
23 vapor deposition but also the type of
24 chemical vapor deposition called atomic layer
25 deposition or atomic layer epitaxy?

1 MR. LEN: Objection.

2 A. Well, papers that are contributed
3 in that particular area to this conference as
4 well.

5 Q. And the paper before you,
6 Exhibit 7, is one such paper regarding atomic
7 layer deposition submitted to the conference
8 on chemical vapor deposition, correct?

9 A. Correct.

10 Q. I think in the context of another
11 exhibit I asked you about the authors of this
12 paper. The authors are listed as M. Leskela
13 and M. Ritala.

14 Do you see what I'm referring to
15 there?

16 A. Yes.

17 Q. Right. And these are the same two
18 individuals who are listed on -- as inventors
19 on Exhibit 3, the PCT publication; is that
20 correct?

21 A. Yes.

22 MR. BOBROW: Let's mark this next.
23 (Exhibit 8, Synthesis of Thin Films by
24 Atmospheric Pressure Chemical Vapor
25 Deposition Using Amido and Imido Titanium,

1 (IV) Compounds as Precursors, marked for
2 identification.)

3 BY MR. BOBROW:

4 Q. Dr. Gladfelter, you've been handed
5 what was marked as Exhibit 8 for
6 identification. This is a paper entitled
7 "Synthesis of Thin Films by Atmospheric
8 Pressure Chemical Vapor Deposition Using
9 Amido and Imido Titanium (IVm)," Roman
10 Numeral IV, "Compounds as Precursors."

11 This exhibit is marked in the IPR
12 proceedings as Exhibit 2103 in the 662, 663,
13 and 664 proceedings.

14 You have reviewed Exhibit 8
15 before, have you not?

16 A. Yes.

17 Q. When was the first time that you
18 reviewed Exhibit 8?

19 A. After it came out. When it was
20 published.

21 Q. Okay. So in or around the late
22 1980s?

23 A. Well --

24 Q. Or 1990s?

25 A. Early '90s.

1 Q. Okay. Was this article of
2 interest to you at the time?

3 A. Yes.

4 Q. Why so?

5 A. For a couple reasons. It produces
6 the -- a relatively clean reaction to --
7 actually, let me just take a look. There are
8 a couple papers that came out at the time.
9 But I want to verify whether this was the one
10 with a coreactant of ammonia or not. And it
11 was not, so.

12 The primary reason that I was
13 interested in this was that they were able to
14 look at using these precursors, and they saw
15 indeed that there was a substantial amount of
16 carbon that was included in the film as a
17 result of degradation of the precursor.

18 Q. And why was that of interest to
19 you at the time?

20 A. I was -- at the time, one of my
21 research projects was focused on precursor
22 design for CVD processes, and we were
23 looking -- we were focused on metal nitrides
24 and trying to develop an effective route to
25 things like aluminum nitride and gallium

1 nitride at the time.

2 Q. I see. Now, if I understand this,
3 Exhibit 8 describes a CVD reaction of the
4 type that you previously characterized as a
5 single-source CVD reaction; is that correct?

6 MR. LEN: Objection.

7 A. As I understand it -- yes, this is
8 a reaction -- a deposition that did not use a
9 coreactant, ammonia, for instance.

10 Q. Making it a single-source CVD
11 reaction?

12 A. Yes.

13 Q. Take a look at page 3 of the
14 exhibit at the top, and there's a listing
15 there of precursors.

16 Do you see what I'm referring to
17 there?

18 A. Yes.

19 Q. Table 1.

20 A. Okay.

21 Q. And the first listed precursor in
22 the table -- let me ask it a little bit
23 differently.

24 All of the compounds listed as
25 precursors there in the table are titanium

1 compounds; is that correct?

2 A. Yes.

3 Q. Are all of the compounds listed
4 metal dialkylamides?

5 A. Yes. It has several of the
6 entries in that table, are what we would
7 refer to as a homoleptic metal to
8 dialkylamide -- so that's
9 h-o-m-o-l-e-p-t-i-c -- and it refers to the
10 fact that all the ligands are the same,
11 nothing more elaborate than that. And two of
12 the ligands in that table are not -- they
13 have a different ligand for the fourth
14 ligand.

15 Q. And which are those that have
16 the -- that are not homoleptic?

17 A. On that table, they would be the
18 fourth one down. Actually, there's only one
19 compound, but they're listed twice. So it's
20 the fourth one down.

21 Q. Okay. It's the fourth and fifth
22 entries?

23 A. The fourth and fifth entries.

24 Q. And that's the same compound,
25 you're saying?

1 A. Yes.

2 (Griffin Mesmer entered the deposition.)

3 BY MR. BOBROW:

4 Q. And when -- in a single-source CVD
5 reaction such as this, is the idea that you
6 flow the precursor into a deposition reaction
7 chamber at an elevated temperature and then
8 the precursor breaks down in some fashion to
9 hopefully form a desired film?

10 MR. LEN: Objection.

11 A. More or less that's the idea.

12 Q. Now, with respect to the metal
13 dialkylamides in Table 1, because these are
14 metal alkylamides, I take it that you have
15 the titanium bonded to the nitrogen; is that
16 right?

17 A. Correct.

18 Q. And the nitrogen, in turn, is
19 bonded to, I think what you called earlier
20 some hydrocarbons; is that right?

21 A. Substituents, yes.

22 Q. Hydrocarbon substituents?

23 A. Right.

24 Q. And in the case of the first
25 reactant or the first precursor, you have

1 methyls that are attached to the nitrogen or
2 bonded to the nitrogen, correct?

3 A. Yes.

4 (Griffin Mesmer exited the deposition.)

5 BY MR. BOBROW:

6 Q. And in the sixth one it looks like
7 you have ethyls bonded to the nitrogen; is
8 that correct?

9 A. Yes.

10 Q. Okay. Now, let's --

11 A. Well, there is one additional one
12 there. Didn't recognize it. It was longer,
13 at the bottom. But yes, there is a -- the
14 seventh one is not a homoleptic, just to
15 clarify an earlier response that I made. It
16 has -- it does have alkylamido ligands but
17 also imidos, i-m-i-d-o, an imido ligand.

18 Q. Let me ask you, for the
19 dialkylamide that's listed as the fourth and
20 fifth entry in the parentheses, it says lower
21 case "t" and then hyphen, capital "B," lower
22 case "u." Do you see that?

23 A. Yes.

24 Q. Is that a reference to a
25 tert-butoxy?

1 A. No.

2 Q. What is t-Bu?

3 A. It's a tertiary, sometimes spoken
4 as tert, t-e-r-t, butyl group, not a butoxy.

5 Q. And what is a butyl group?

6 A. It is a four-carbon fragment.

7 Q. No oxygen?

8 A. No oxygen.

9 Q. Okay. Now, going back to where I
10 was, if we look at the first precursor, the
11 tetrakis(dimethylamido)titanium, with respect
12 to that precursor, I think I was asking you,
13 we have the titanium metal bonded to the
14 nitrogen and the nitrogen in turn bonded to
15 carbon; is that correct?

16 A. Yes.

17 Q. Now, is the idea of the
18 single-source CVD reaction, when you're
19 trying to form the titanium nitride from the
20 tetrakis(dimethylamido)titanium, that you are
21 trying to break the nitrogen carbon bonds but
22 not the titanium nitrogen bond?

23 A. That's correct.

24 MR. LEN: Objection.

25 Q. Go ahead.

1 A. Yes.

2 Q. Okay. Now, changing things up a
3 bit. I know that this isn't described in
4 this paper, but if I were to try to form
5 titanium nitride using the
6 tetrakis(dimethylamido)titanium using ammonia
7 and using a binary type CVD reaction, in that
8 case, am I breaking the nitrogen carbon bonds
9 of the alkylamide as well as the titanium
10 nitrogen bonds of the alkylamide?

11 MR. LEN: Objection.

12 A. What is taught in the literature
13 is that, in essence, you have two competing
14 routes in such a case to produce the desired
15 titanium nitride. The addition of ammonia to
16 the system leads to a higher percentage of
17 nitrogen in the final film, and a smaller --
18 somewhat smaller amount of impurities, carbon
19 impurities.

20 Q. Okay. Now, you mentioned there
21 were two routes, but I may not have
22 understood what -- your response when you
23 said that. What did you mean by two routes?

24 A. Well, we had already mentioned
25 that the -- in the use of one of these

1 precursors by itself, the carbon nitrogen
2 bond can be cleaved, leaving behind some
3 nitrogen, some carbon, but sometimes that
4 carbon is trapped into the film as evidence
5 when you look at some of the results that
6 they report in Table 3, which shows the
7 atomic percent of carbon and the titanium
8 nitride films, which is, in many cases,
9 actually a larger concentration of that
10 element relative to nitrogen.

11 That chemistry doesn't
12 automatically just stop when you incorporate
13 an ammonia source into the reactor. What
14 ultimately comes out will be a film that will
15 be -- will have titanium nitride units
16 that -- some of which will come from this
17 process, some of which will come from the
18 direct reaction of ammonia. So you can get a
19 combination.

20 Q. Okay. When you're referring to
21 the direct reaction with the ammonia, when
22 that's the mechanism, are the bonds between
23 the original titanium nitrogen bond in the
24 alkylamide broken as well?
25 (Griffin Mesmer entered the deposition.)

1 A. There will be some -- so I'm going
2 to step back for a second. What we may know
3 and what is conjecture regarding mechanism is
4 important to clarify. I do not know that the
5 amide -- the dialkylamide bond is broken or
6 not during this process. What I may know is
7 that I get a film that contains both titanium
8 and nitrogen in the final result. And when I
9 don't have -- if I were to run this without
10 ammonia, I would also have some carbon
11 incorporated into the film as well.

12 I do not know exactly where that
13 carbon is bound, to what atoms. I don't know
14 whether there is a carbon nitrogen bond
15 retained in that thin film or whether it is
16 only bound to titanium. So there's --
17 there's a lot that may go on that's invisible
18 to us.

19 So I guess I want to come back and
20 make sure I understand your question. Again,
21 so if you could repeat, I'd appreciate it.

22 Q. Well, I think you answered the
23 question, because I was asking simply whether
24 when you do the -- so let's take a step back.

25 This exhibit, Exhibit 8, does not

1 describe forming a titanium nitride film by
2 CVD using a titanium dialkylamide and
3 ammonia; is that correct?

4 A. That's correct.

5 Q. Instead it describes forming a
6 titanium nitride film using a single-source
7 CVD route --

8 A. Correct.

9 Q. -- or process, correct?

10 A. Yes.

11 Q. Now, what I was asking you
12 earlier, and which I think you answered but
13 we can verify that, I wanted to know whether
14 in terms of the mechanism of forming titanium
15 nitride, when you use a titanium dialkylamide
16 and ammonia, whether that mechanism for the
17 chemical reaction includes breaking the
18 titanium nitrogen bond in the dialkylamide?

19 MR. LEN: Objection.

20 A. From this paper -- well, of
21 course, this paper doesn't include ammonia,
22 but in other literature studies that have
23 been reported, the majority, if I
24 understand -- I think I understand correctly,
25 is the majority of the nitrogen comes from

1 ammonia when ammonia is used as a coreactant.
2 So if I understand your question,
3 that means that the titanium nitrogen bond in
4 a certain portion of the precursor is
5 cleaved.

6 Q. Thank you. Earlier, when I first
7 showed you Exhibit 8, and I asked you why you
8 were interested, you used a phrase regarding
9 deposition and you said something about a
10 relatively clean film.

11 Do you recall that?

12 A. Was that in reference to a -- the
13 incorporation of ammonia into the --

14 Q. It was simply when I asked you --
15 give me just a second.

16 Dr. Gladfelter, earlier -- shortly
17 after we had marked this as an exhibit, I had
18 asked you why you were interested in this
19 paper at the time, and you said for a couple
20 of reasons, and then you said it produces
21 the -- a relatively clean reaction to --
22 actually, let me take a look, and then you
23 went from there.

24 A. Yeah.

25 Q. And so the question that I posed

1 to you earlier, before we undertook this
2 investigation to find what you had said, was
3 simply what you had meant by the phrase
4 "relatively clean reaction."

5 A. So when this first appeared, I
6 thought this was the example where they were
7 using ammonia as a coreagent -- a coreactant,
8 which does produce a film with a smaller
9 amount of carbon impurity than this film. So
10 the "relatively clean" comment does not refer
11 to the paper that you've given me listed as
12 Exhibit 8.

13 Q. I see. So the "relatively clean
14 reaction" comment referred to papers that
15 talk about forming titanium nitride by
16 reacting metal dialkylamide precursor with
17 ammonia?

18 A. Yes.

19 Q. Now, Exhibit 8 is one of the
20 papers that you refer and discuss in your
21 declarations in these IPR proceedings,
22 correct?

23 A. Yes.

24 (Exhibit 9, Low Temperature Deposition of
25 Metal Nitrides by Thermal Deposition of

1 Organometallic Compounds, marked for
2 identification.)

3 BY MR. BOBROW:

4 Q. Dr. Gladfelter, you've been handed
5 Exhibit 9 for identification. Exhibit 9
6 appears to be a paper by Sugiyama,
7 S-u-g-i-y-a-m-a, and others entitled "Low
8 Temperature Deposition of Metal Nitrides by
9 Thermal Deposition of Organometallic
10 Compounds."

11 And Exhibit 9 for your deposition
12 is Exhibit 2114 in the 662, 663, and 664
13 proceedings.

14 Do you recognize Exhibit 9?

15 A. I do.

16 Q. This is one of the papers that you
17 refer to in your declaration?

18 A. Correct.

19 Q. Declarations, I should say.
20 Correct?

21 A. Yes.

22 Q. Okay. And this is a paper by
23 Sugiyama in which the authors are attempting
24 to form a compound film such as a titanium
25 nitride film by chemical vapor deposition; is

1 that correct?

2 A. Yes.

3 Q. And is it the case that the
4 decomposition reactions that are under study
5 in this paper are deposition reactions of
6 dialkylamides?

7 A. I believed every one is a
8 dialkylamide. I know that titanium is -- and
9 I think -- yeah, actually, it says it in the
10 first line at the abstract for several
11 different elements, but yes.

12 Q. Among the precursors that were
13 studied included both titanium dialkylamides
14 and zirconium dialkylamides, correct?

15 A. Yes.

16 Q. The Sugiyama paper, Exhibit 9,
17 uses a single-source CVD process to form the
18 titanium nitride films; is that correct?

19 A. That's how I interpret this paper.

20 Q. You don't see in Exhibit 9, the
21 Sugiyama paper, any reactions in which
22 ammonia or some other nitrogen-bearing
23 reactant is reacted with the dialkylamides;
24 is that correct?

25 A. I do not see anything. Have not

1 seen anything, no.

2 (Exhibit 10, Infrared Studies of the
3 Surface and Gas Phase Reactions Leading to
4 the Growth of Titanium Nitride Thin Films
5 from Tetrakis(dimethylamido) Titanium and
6 Ammonia, marked for identification.)

7 BY MR. BOBROW:

8 Q. Dr. Gladfelter, before you now is
9 Exhibit 10, which appears to be a paper by
10 Dubois and others entitled "Infrared Studies
11 of the Surface and Gas Phase Reactions
12 Leading to the Growth of Titanium Nitride
13 Thin Films from Tetrakis(dimethylamido)
14 Titanium and Ammonia."

15 This is Exhibit 2105 in 662, 663,
16 and 664 proceedings.

17 Dr. Gladfelter, do you recognize
18 Exhibit 10?

19 A. Yes.

20 Q. This is one of the papers that you
21 refer to in your declarations in these
22 proceedings; is that correct?

23 A. Yes.

24 Q. When was the first time that you
25 reviewed Exhibit 10?

1 A. Again, shortly after it appeared
2 in the literature.

3 Q. And that was apparently in the
4 early 1990s; is that right?

5 A. Correct.

6 Q. And was that also true of
7 Exhibit 9, that you reviewed Exhibit 9 at or
8 around the time that it came out in the
9 academic literature, which apparently was in
10 the 1970s?

11 A. So the answer is no.

12 Q. When did you first review
13 Exhibit 9?

14 A. I first reviewed Exhibit 9 as a
15 result of dealings with this dispute.

16 Q. The Harvard-Micron dispute?

17 A. Yes.

18 Q. All right. So back to Exhibit 10.
19 As I understand Exhibit 10, it describes two
20 different reactions using a titanium
21 dialkylamide: one single source and one
22 using ammonia; is that correct?

23 A. Yes.

24 Q. Now, in the -- if I'm interpreting
25 the paper correctly, as I had read it, it

1 appears that the authors concluded that there
2 is less carbon in the titanium nitride film
3 when you use the reaction of the titanium
4 dialkylamide with ammonia as compared to the
5 single-source dialkylamide reaction; is that
6 correct?

7 A. Yes.

8 Q. And is that consistent with other
9 literature that you have read in the field?

10 MR. LEN: Objection.

11 A. Well, that I was referring to
12 regarding other literature when you raised
13 the question regarding Fix and Gordon's
14 paper, Hoffman and Gordon, that was
15 Exhibit 8, was, in fact, this paper.

16 Q. Now, if I might direct your
17 attention to the fourth page of the exhibit.
18 And on the right-hand column there is a
19 heading that says reactions of
20 tetrakis(dimethylamido)titanium, but not
21 spelled out in words but just the formula.

22 Do you see what I'm referring to
23 there?

24 A. I do.

25 Q. Now, as I understand the paper,

1 it, at least in part, it describes exposing
2 what it calls a clean polycrystalline
3 titanium disilicide surface to ammonia at
4 temperatures between 300 and 500 K.

5 Do you see what I'm referring to?
6 That's right underneath the heading.

7 A. The title?

8 Q. Yeah.

9 A. Yes.

10 Q. All right. And then beneath that
11 it says, "Similarly, if the same surface is
12 saturated with a layer of
13 tetrakis(diethylamino)titanium, and then
14 exposed to NH₃," ammonia, "at temperatures
15 between 300 and 600 K, no reaction is seen."

16 Do you see what I'm referring
17 to?

18 A. Yeah, let me -- so the first
19 question was when a clean silicide --
20 titanium disilicide surface is exposed,
21 that's what says there's no reaction under
22 their experimental conditions.

23 Is that what you're referring to?

24 Q. There seem to be two surfaces
25 here, correct?

1 A. And then -- then it goes
2 "Similarly, if the same surface is
3 saturated..."

4 So I read that to mean that the
5 titanium disilicide surface is saturated with
6 the tetrakis(dimethylamido)titanium and then
7 exposed to ammonia, under those temperature
8 conditions there's no reaction seen.

9 I think that's what you were
10 asking. So I'm agreeing that those words are
11 there.

12 Q. And that's what I wanted to direct
13 your attention to.

14 At what temperatures did you
15 understand this paper was using to deposit
16 the titanium nitride film using the titanium
17 barium diakylamide and ammonia?

18 A. And ammonia. This was -- I don't
19 remember the terms they were using for the
20 depositions. My understanding was that they
21 were certainly in that regime.

22 Q. Okay. Now, what does it mean in
23 that column to saturate the, as I read it,
24 this titanium disilicide surface to a layer
25 of tetrakis(dimethylamido)titanium?

1 A. So you're asking -- let me make
2 sure I understand. You're asking what does
3 it mean to saturate the silicide surface, the
4 titanium disilicide surface with
5 the precursor --

6 Q. Correct.

7 A. -- the tetrakis?

8 Those are good words. It means
9 that the available sites on the titanium
10 disilicide surface are going to be occupied
11 with the molecule -- the precursor molecule
12 itself or some fragment thereof.

13 Q. And then after that saturation
14 occurs in this paper, then at that point some
15 nitrogen gas is introduced to react with that
16 surface that was formed using the titanium
17 dialkylamide?

18 MR. LEN: Objection.

19 A. Not nitrogen gas.

20 Q. Did I say nitrogen? I meant to
21 say ammonia.

22 A. Ammonia is what is the second
23 reactant in this particular set of surface
24 experiments.

25 Q. All right. So just so I'm clear,

1 then, there's a surface, the dialkylamide is
2 introduced, reacts with that surface, and
3 then after that reaction happens, then the
4 ammonia is introduced to react with the
5 titanium dialkylamide surface; is that right?

6 A. Yes. That was the experiment they
7 did, and what they concluded is also written
8 there.

9 Q. Okay. And were they able then
10 to -- I see. And it says that no reaction is
11 seen. Is that's what --

12 A. Yes, that's what I was referring
13 to.

14 Q. Yes. And then there's also a
15 description of gas phase study.

16 Do you see what I'm referring to
17 under that?

18 A. Yes.

19 Q. And here in the gas phase studies,
20 is it correct that the dialkylamide and the
21 ammonia are introduced into a reaction
22 chamber at the same time?

23 A. That's how I understand this
24 experiment to be done.

25 Q. Okay. And that's different than

1 the surface reaction test that was done in
2 the preceding paragraph; is that correct?

3 A. That is correct.

4 Q. So in the gas phase experiment
5 where you introduce both of the reactants,
6 there's a sentence towards the bottom of the
7 page that says, "No partial decomposition
8 species or adducts containing both titanium
9 and ammonia were detected."

10 Do you see what I'm referring to
11 there?

12 A. I do.

13 Q. What is an adduct?

14 A. It's typically referring to a
15 relatively weak, so-called donor-acceptor
16 bond formed between a molecule that contains
17 a pair of electrons, such as ammonia, and an
18 element, in this case titanium they're using,
19 that can serve as an acceptor. So often,
20 without being more explicit, these are called
21 Lewis acids and Lewis basis. And it's just
22 suggesting that -- I think this is suggesting
23 that they don't see any evidence for this in
24 the gas phase.

25 Q. And what about decomposition

1 species, what is that referring to, in that
2 same sentence?

3 A. Well, let me see that sentence.
4 So you're looking at the last sentence on
5 page -- on the fourth page?

6 Q. Correct.

7 A. So the actual sentence says, "No
8 partial decomposition species or adducts
9 containing titanium and ammonia were
10 detected."

11 I'm not sure I can augment.

12 Q. Okay. But what does it mean to be
13 a decomposition species? What is that?
14 Because it's referring to partial
15 decomposition species, so I'm trying to
16 understand what that means.

17 A. That some chemistry had occurred
18 to change the formula of the molecules,
19 either -- well, both nitrogen and ammonia, as
20 well as the titanium amide precursor.

21 Q. On the previous page, page 3,
22 there's a reference in connection with the
23 single-source experiment -- single-source CVD
24 experiment, on the right-hand column, to
25 something that is called, and it's at the

1 very bottom -- near the very bottom of page
2 3, a metallacycle.

3 Do you see what I'm referring to
4 there?

5 A. Yes.

6 Q. What is a metallacycle?

7 A. It's a cyclic grouping of atoms
8 that includes a metal. So in this case it's
9 indicated as a titanium to nitrogen to
10 carbon, back to the titanium.

11 Q. Yes. And so there's something in
12 that preceding sentence that's referred to as
13 a titanium nitrogen carbon three-membered
14 ring.

15 Do you see what I'm referring
16 to?

17 A. Yes.

18 Q. And that's an example of a
19 metallacycle?

20 A. Yes.

21 Q. And if I understand this
22 correctly, that metallacycle is something
23 that the authors found when they attempted to
24 form the titanium nitride film using the
25 single-source CVD technique; is that correct?

1 MR. LEN: Objection.

2 A. I recall they inferred the
3 presence of a metallacycle in this, in part
4 because the ability of molecules or ligands
5 with beta-hydrogens, hydrogens on the second
6 element that's attached to the metal. So I
7 have a metal; I have a nitrogen, that's
8 alpha, because it's the first element, and
9 then attached to the alpha atom is the
10 beta-atom, and a well-known process in
11 organometallic chemistry is beta-hydrogen
12 elimination. That hydrogen that is on a beta
13 carbon can be lost, and it can lead to
14 species of -- drawn as shown on page 4,
15 left-hand column.

16 Q. And my question is simply that the
17 authors of Exhibit 10 either inferred or
18 believed that using the single-source CVD
19 technique using the metal dialkylamide for
20 CVD, that this titanium nitrogen carbon
21 three-membered ring was something that was
22 produced in that process; is that correct?

23 MR. LEN: Objection.

24 A. That's how I understand this to
25 be -- what I understand it, that they

1 inferred that is an intermediate.

2 Q. And what do you mean by "an
3 intermediate"?

4 A. A species that exists somewhere
5 between the starting compound and the final
6 film.

7 (Exhibit 11, Model Studies of Low
8 Temperature Titanium Nitride Thin Film
9 Growth, marked for identification.)

10 BY MR. BOBROW:

11 Q. Dr. Gladfelter, you've been handed
12 Exhibit 11 for identification. Exhibit 11,
13 for purposes of your deposition, is
14 Exhibit 2106 in the 662, 663, and 664
15 proceedings. It's a paper by Lawrence Dubois
16 titled "Model Studies of Low Temperature
17 Titanium Nitride Thin Film Growth."

18 You've seen this exhibit before, I
19 trust?

20 A. Yes.

21 Q. This is a paper that you cite and
22 refer to in your declarations in these
23 proceedings, correct?

24 A. Yes.

25 Q. And when was the first time that

1 you reviewed Exhibit 11?

2 A. Back in the mid-'90s. Once again,
3 shortly after it appeared.

4 Q. Do you understand that Exhibits 10
5 and 11 are related one to the other?

6 A. Yes.

7 Q. And as I read Exhibit 11, it also
8 appears to discuss both a single-source CVD
9 process using a metal dialkylamide to form a
10 titanium nitride film as well as a process --
11 a dual source process in which the titanium
12 dialkylamide and ammonia are used.

13 Is that how you read this?

14 A. There are aspects of both of those
15 that are discussed.

16 Q. And the alkylamide source in the
17 titanium nitride film is TDMAT; is that
18 correct?

19 A. Yes.

20 Q. That's
21 tetrakis(dimethylamido)titanium?

22 A. Correct.

23 Q. And that's a metal alkylamide,
24 correct?

25 A. Yes.

1 Q. On page 2 of this Dubois paper,
2 Exhibit 11, there's title called "Pyrolysis
3 of Pure TDMAT and Carbon Incorporation."

4 Do you see what I'm referring
5 to?

6 A. Yes.

7 Q. What is pyrolysis?

8 A. It's a thermal reaction referring
9 typically to heating a single material to
10 elevated temperature.

11 Q. And referring to the right-hand
12 column of page 2 of Exhibit 11, there's a
13 reference to -- around the middle of the
14 page -- to pyrolysis of TDMAT.

15 You see what I'm referring to
16 there?

17 A. Yes. Let me make sure I do. It's
18 in the sentence "Further, mass spectral
19 analysis"?

20 Q. I'm glad you asked. Go down a
21 little bit further and it says "We have
22 probed..."

23 A. Oh.

24 Q. And then a new paragraph goes on
25 to page 3; and, again, talks about

1 three-membered rings over in the left-hand
2 column.

3 Do you see what I'm referring to
4 there?

5 A. Yes.

6 Q. Okay. Now, with respect to the
7 discussion that begins at "We have probed" on
8 page 2, and continues on to page 3 until the
9 next heading, which says "Source of Nitrogen
10 in Ti Nitride," that discussion is talking
11 about a single-source CVD process using the
12 pyrolysis technique?

13 A. Yes.

14 Q. And where it says "Source of
15 nitrogen in Ti Nitride" on page 3, that's at
16 the top of page 3 of Exhibit 11, the ensuing
17 discussion there is referring to the
18 different CVD technique, one that is using
19 the metal dialkylamide, TDMAT, along with
20 ammonia; is that correct?

21 A. That's the reaction that they talk
22 about, yes.

23 Q. There's a reference on page 3 of
24 Exhibit 11 --

25 A. On page?

1 Q. Page 3 of Exhibit 11. And you'll
2 see about halfway down the page there's a
3 reference to Fix, et al. and then there's a
4 Footnote 23.

5 Do you see what I'm referring
6 to?

7 A. Yes.

8 Q. Is the Fix, et al. paper that's
9 referred to on page 3 of Exhibit 11 what
10 we've marked here as Exhibit 8 here in your
11 deposition?

12 A. Let me verify. It's the same set
13 of authors in the same order. Let me check
14 the page order. It's also the same journal.
15 And that is 235. Yes, it's the same paper.
16 (Exhibit 12, IOP Science, Characterization
17 of CVD-TiN Films Prepared with Metalorganic
18 Course, marked for identification.)

19 BY MR. BOBROW:

20 Q. Dr. Gladfelter, Exhibit 12 is now
21 before you. This appears to be a copy of a
22 paper in something called IOP Science called
23 a "Characterization of CVD Ti Nitride Films
24 Compared with Metalorganic Source."

25 This Exhibit 12 to your deposition

1 is authored -- first authored by Ishihara,
2 I-s-h-i-h-a-r-a, and is Exhibit 2104 in the
3 662, 663, and 664 proceedings.

4 You've seen this paper before?

5 A. Yes.

6 Q. When was the first time that you
7 saw Exhibit 12?

8 A. First time I saw it was when we
9 started these proceedings -- the proceedings
10 in general between MTI and Harvard.

11 Q. I see. And it appears that the
12 paper came out roughly in the 1990s time
13 frame, and this was not something that you
14 reviewed back at that time; is that correct?

15 A. I did not, no.

16 Q. Do you know what IOP Science is?

17 A. Well, the actual journal is not
18 that. That's the company that now
19 distributes the journal. The journal is The
20 Japanese Journal of Applied Physics, and
21 that's a known journal.

22 Q. Sure. And is that a journal that
23 you read back in the 1990s?

24 A. Yes, I have read articles from
25 that.

1 Q. Now, as I read this paper, it
2 appeared to be investigating Ti Nitride films
3 formed by a dual-source CVD technique using a
4 metal dialkylamide and ammonia; is that
5 right?

6 A. Yes.

7 Q. And the metal dialkylamide in this
8 case is tetrakis(dimethylamido)titanium; is
9 that right?

10 A. Yes.

11 Q. Now, take a look at Figure 2,
12 which is on page 3 of Exhibit 12. There
13 appears to be a graph and on the horizontal
14 axis there appears to be -- the horizontal
15 axis, I should say, is in terms of units of
16 binding energy, electron volts; is that
17 right?

18 A. Correct.

19 Q. Okay. And what is the vertical
20 axis?

21 A. It's intensity. This is, as it
22 states in the caption, an XPS, an x-ray
23 photoelectron spectrum. Actually, two
24 spectra. And what you typically have on a
25 vertical axis in this spectroscopy is counts

1 or count rate. It's an x-ray. You're
2 measuring x-ray counts.

3 Q. And you said it's a measure, I
4 thought, of intensity. What do you mean by
5 that?

6 A. It's -- intensity is the -- how
7 many counts per second or how many total
8 counts coming out with the energy that's
9 listed on the horizontal axis.

10 Q. And in that context, what do you
11 mean by a count?

12 A. X-rays are detected by a variety
13 of methods, but one way is to have a detector
14 that will provide a count every time an x-ray
15 photon impinges on the detector, and that is
16 what is being observed and plotted here.

17 Q. Okay. And is -- I see. So remind
18 me again -- x-ray -- I'm sorry -- XPS stands
19 for?

20 A. X-ray photoelectron spectrum or
21 spectrometry, however.

22 Q. Okay. Then -- I'm remembering
23 another table -- let me ask you to turn your
24 attention back to Exhibit 8. And please turn
25 to page 3 of Exhibit 8 towards the bottom.

1 A. Yes.

2 Q. And you had alluded earlier in
3 your testimony to Table 3. Do you have Table
4 3 there before you?

5 A. Yes.

6 Q. And in Table 3 there appears to be
7 a -- what's set forth appears to be
8 percentages of material obtained using these
9 different precursors.

10 Do you see what I'm referring
11 to?

12 A. I will look -- Table 3 is a
13 representation of the concentration of
14 elements within the films. So it's not a
15 yield or a fraction of material formed. It's
16 the analysis of the elements that are present
17 in the film that was deposited using that
18 precursor.

19 Q. Yes. And, for example, just
20 looking at the column headings in this table,
21 precursor, I think we know what that means,
22 right? It's simply a listing of the chemical
23 precursor that was used to form the film,
24 correct?

25 A. Yes.

1 Q. And then in the next column that
2 appears to be temperature, is that correct,
3 in centigrade?

4 A. Yeah. If you clarify the T as --
5 or superscript A, which is referring to the
6 substrate temperature.

7 Q. Yes. And then the next column
8 says "Ti" and then little "b," and
9 then "at.%."

10 Do you see where I'm referring to?

11 A. Yes.

12 Q. Is that a reference to the atom
13 percent composition of titanium that was
14 found in the film?

15 A. Yes.

16 Q. And what does atom percent
17 composition mean?

18 A. The atomic percentage is going to
19 be, assuming we have 100 atoms of this film,
20 the fraction of those atoms that will be
21 titanium, would then be converted into a
22 percentage by multiplying times a hundred.

23 Q. And it appears that these -- that
24 the atomic percentage numbers for titanium,
25 nitrogen, carbon and oxygen were calculated

1 using something called an electron microprobe
2 analysis; is that right?

3 A. Yes.

4 Q. What is -- are you familiar with
5 that technique?

6 A. Yes.

7 Q. That technique is not the XPS
8 technique we were just looking at a minute
9 ago, is it?

10 A. No.

11 Q. Okay. What is the electron
12 microprobe analysis technique?

13 A. This is an experiment that is
14 typically -- that is conducted in an electron
15 microscope, and the electron beam that's used
16 to create the images also causes some degree
17 of electrons and x-rays to be emitted from
18 the atoms. Those emitted particles have
19 characteristic energies that can be detected
20 and that can be correlated to the -- a
21 particular element.

22 Q. Okay. And is that a well-accepted
23 technique?

24 A. Yes.

25 Q. And with the XPS technique that

1 you were describing with respect to
2 Exhibit 12, are the graphs that are set forth
3 on page 3 either calculating or counting the
4 atomic percent of different elements or
5 compounds in a film?

6 MR. LEN: Objection.

7 A. Well, we don't know exact --
8 there's not a vertical axis in this
9 particular graph, but in a typical x-ray
10 photoelectron spectrum, you will use the
11 intensity of that peak related to a standard
12 that gives you a standard intensity for a
13 known composition to extract a value of the
14 concentration in your unknown.

15 Q. All right. And was that done with
16 respect to Figures 2 and 3 in this Ishihara
17 paper?

18 A. I cannot say. I'd have to -- I'm
19 not sure if they clarified whether they used
20 the standard or not. So I would have to read
21 a little bit more. I don't remember off the
22 top of my head.

23 Q. All right. And the Exhibit 12,
24 the Ishihara paper, doesn't attempt to
25 calculate an atomic percent of nitrogen, or

1 titanium, or carbon, or other materials in
2 the film, correct?

3 A. No, I don't see any.

4 MR. LEN: Whenever you get to a
5 good breaking point, we've been going
6 over an hour and 15.

7 MR. BOBROW: Time flies.

8 MR. LEN: We're having fun. Just
9 for a restroom break.

10 MR. BOBROW: That's fine. I was
11 about to switch to a new exhibit. So if
12 this is a good time, I'm happy to break,
13 if you'd like.

14 (Recess taken at 2:35 p.m. to 2:45 p.m.)

15 (Exhibit 13, Properties of Titanium Nitride
16 Thin Films Deposited by Rapid-Thermal-Low-
17 Pressure-Metalorganic- Chemical-Vapor-
18 Deposition Technique Using
19 tetrakis(dimethylamido)titanium precursor,
20 marked for identification.)

21 BY MR. BOBROW:

22 Q. Dr. Gladfelter, the court reporter
23 has handed you Exhibit 13 for identification.
24 This is a paper by Katz and others from 1991
25 entitled "Properties of Titanium Nitride Thin

1 Films Deposited by
2 Rapid-Thermal-Low-Pressure-Metalorganic-Chemi
3 cal-Vapor- Deposition Technique Using
4 tetrakis(dimethylamido)titanium precursor."

5 It is Exhibit 2126 in the IPR
6 proceedings.

7 Dr. Gladfelter, do you recognize
8 Exhibit 13?

9 A. I do.

10 Q. This is one of the papers that you
11 refer to and cited in your declarations in
12 these proceedings, correct?

13 A. That is correct.

14 Q. When was the first time that you
15 reviewed Exhibit 13?

16 A. I saw this back in the '90s.

17 Q. Okay. Let's see. Just to get our
18 bearings here, this paper is describing an
19 experiment in which a titanium nitride film
20 was formed; is that correct?

21 A. Yes. The titanium nitride film,
22 it certainly is contaminated with a certain
23 amount of carbon, just to clarify.

24 Q. Sure. And the technique that the
25 authors use to deposit the titanium film was

1 a single-source CVD technique; is that
2 correct?

3 A. Yeah, that's my understanding.

4 Q. And there's nothing in Exhibit 13
5 that describes an experiment in which the
6 titanium nitride film was formed using metal
7 alkylamide and ammonia gas, is there?

8 A. No.

9 Q. Is it the case that a titanium
10 dialkylamide precursor was used in the
11 single-source CVD system?

12 A. Yes.

13 Q. And that precursor was titanium --
14 I'm sorry -- the precursor was
15 tetrakis(dimethylamido)titanium?

16 A. Yes.

17 Q. And in the single-source CVD
18 technique, was the deposition accomplished by
19 flowing the dialkylamide precursor into some
20 type of heated deposition chamber?

21 MR. LEN: Objection.

22 A. I don't -- the substrate was
23 heated. I don't remember if the deposition
24 chamber, as an entirety, was heated, but you
25 would get at the hot substrate.

1 Q. And is that akin to the pyrolysis
2 technique that we discussed earlier in the
3 diagnosis?

4 MR. LEN: Objection.

5 A. The activation is a thermal
6 activation in both the pyrolysis and then --
7 in this process.

8 Q. The process Katz describes?

9 A. That's in Katz's paper, yes.
10 (Exhibit 14, Electrical Properties of
11 ultrathin hafnium oxide films for
12 replacement metal gate transistors,
13 fabricated by atomic layer deposition using
14 methylethylamido hafnium and ozone, marked
15 for identification.)

16 BY MR. BOBROW:

17 Q. Doctor, you have Exhibit 14 before
18 you. This is a paper by Kamiyama,
19 K-a-m-i-y-a-m-a, and others entitled
20 "Electrical Properties of ultrathin hafnium
21 oxide films for replacement metal gate
22 transistors, fabricated by atomic layer
23 deposition using -- I think I'll get this
24 right -- tetrakis ethyl -- or
25 "methylethylamido hafnium and ozone"; is that

1 correct?

2 A. We would put those in alphabetical
3 order. So it would be ethylmethyramido, so
4 that's how you get the TMA.

5 Q. Okay. And in the way that it's
6 written out in the title, it's looking like
7 the methyl is listed before the ethyl; is
8 that right?

9 A. Yes.

10 Q. Okay. In all events, this is a
11 metal dialkylamide, correct?

12 A. Correct.

13 Q. Okay. When was the first time
14 that you reviewed Exhibit 14?

15 A. I think that was part of the --
16 looking at background for this case.

17 Q. I see. So the Kamiyama paper was
18 not one that you reviewed in the course of
19 your research in working at the university;
20 is that correct?

21 A. Yes.

22 Q. Now, it appears from my review of
23 this that the authors did some experiments
24 forming hafnium oxide films at different
25 temperatures; is that right?

1 A. I'm just going to look at the
2 abstract and see what it says, because off
3 the top of my head, I don't remember the
4 experimental details of this.

5 So they do talk about the
6 measurement of physical properties of the
7 films that were deposited at different
8 temperatures. So I think that's correct.

9 Q. And in the abstract there is
10 something called "V," as in Victor, and I
11 imagine it's voltage, and then underneath
12 that a subscript "f" as in Frank, and "b" as
13 in boy.

14 Do you see that?

15 A. In the abstract?

16 Q. Yes, about the fifth line down
17 over to the right-hand side of the line. The
18 Vfb with 200 degrees C?

19 A. Oh, yes.

20 Q. And what is Vfb?

21 A. It's the flat-band voltage.

22 Q. And referring back to what I had
23 alluded to earlier, the paper appears to
24 perform deposition of the hafnium oxide film
25 at least at 200 degrees C and at 275 degrees

1 C; is that right?

2 A. At least, yes.

3 Q. Take a look at page 3 of
4 Exhibit 14 in the right-hand column. There's
5 a discussion of Figure 2 and something called
6 a SIMS, S-I-M-S, depth profile.

7 Do you see what I'm referring
8 to?

9 A. I see the figure. You want to
10 look at the right-hand side --

11 Q. Yeah, let me be clearer.

12 A. Kind of the first photograph.

13 Q. In the text over to the right-hand
14 column on page 3, it refers to Figure 2 --

15 A. Yes.

16 Q. -- filling a SIMS depth profile.

17 Do you see what I'm talking about
18 there?

19 A. Yes.

20 Q. And then the actual Figure 2 is in
21 the left-hand column on page 3 at the bottom;
22 is that right?

23 A. Correct.

24 Q. And this is saying, among other
25 things, that the authors carried out film

1 depositions at 200 degrees C, 250 degrees C,
2 and 275 degrees C; is that right?

3 A. Yes.

4 Q. And as you understand the paper,
5 these SIMS profiles, profile A shows the
6 profile of the film at 200 degrees C; B shows
7 the profile at 250 degrees C; and C shows the
8 profile at 275 degrees C; is that right?

9 A. Yes. I'm reading that from the
10 figure caption.

11 Q. Okay. And from what you read in
12 Exhibit 14 in connection with this matter,
13 did you understand the authors to be saying
14 that the concentration of carbon and nitrogen
15 impurities went down as a function of
16 increasing temperature?

17 MR. LEN: Objection.

18 A. I'm going to have to take a
19 careful look. I don't remember the exact
20 trend. SIMS is used as a more sensitive
21 analytical probe of composition. And so they
22 are indeed interested in both nitrogen and
23 carbon impurities in these metal oxide films.
24 I forget the trend as a function of the
25 deposition temperature.

1 Q. All right. Take a look, if you
2 would, on the right-hand column on page 3.

3 A. Yes.

4 Q. About halfway down there's a
5 sentence that begins, "By increasing the ALD
6 process temperature..."

7 Do you see that?

8 A. Yes.

9 Q. Okay. Why don't you read that
10 sentence and the two sentences following that
11 and let me know when you've read those to
12 yourself.

13 (Witness reviewing document.)

14 A. I think I've read everything you
15 were referring to.

16 Q. Okay. And based upon your review
17 of Exhibit 14, do the authors appear to be
18 reporting that the concentration of carbon
19 and nitrogen impurities in the film -- in the
20 hafnium oxide film went down as the
21 temperature was increased from 200 to 250 and
22 went down even further as you went from 250
23 to 275?

24 A. That's what is stated in this
25 paragraph.

1 Q. What is a SIMS depth profile?

2 A. SIMS is secondary ion mass
3 spectrometry. It's an analytical tool in
4 which you fire a cesium ion beam at a surface
5 causing some billiard ball type of process
6 causing the atoms that are present on the
7 film to bounce off; they go off into the gas
8 phase and are analyzed by a mass
9 spectrometer.

10 Q. Now you -- thank you. And you
11 said cesium, c-e-s-i-u-m?

12 A. Cesium is c-e-s-i-u-m, correct.

13 Q. Now, in this paper by Kamiyama,
14 this paper appears to be dated in 2005.

15 Do you see that?

16 A. I do.

17 Q. All right. And you thought
18 that -- you understand that these patents
19 were filed for at least the provisionals in
20 2000 and then the application in 2001,
21 correct?

22 A. I do.

23 Q. Okay. Did you nonetheless think
24 that it was appropriate to use Exhibit 14 for
25 purposes of your declarations even though it

1 was written after -- or published after the
2 patent-filing dates at issue in the case?

3 MR. LEN: Objection.

4 A. As I said, SIMS is a more
5 sensitive method for probing carbon and
6 nitrogen impurities than some of the others.
7 So this is a -- granted, it is after 2000,
8 but it just continues to confirm that you
9 can -- that the use of dialkylamido metal
10 precursors can lead to incorporation of these
11 impurities.

12 Q. And these authors in Exhibit 14
13 ended up -- or I'm sorry -- their design
14 intent in conducting these experiments was to
15 develop a process that could form a thin film
16 that could be used as a gate dielectric,
17 correct?

18 A. Yes.

19 Q. And at the conclusion of this,
20 you'll agree with me that they determined
21 that indeed -- that the process temperature
22 of 275 degrees was suitable for fabrication
23 of hafnium oxide gate dielectrics, correct?

24 A. That's correct.

25 I guess I would clarify on that.

1 For their purposes -- their experimental
2 purposes, they were satisfied with the 275
3 centigrade deposition temperature and
4 composition. I think it's just important to
5 emphasize there is a finite amount of carbon.
6 Whether this is important to a given process
7 or a given device depends on that device and
8 the design of those devices; and, again, they
9 were satisfied with it for their device.
10 Whether this is going to work effectively for
11 everybody's FETs, field effect transistors,
12 or not is up to the individuals and companies
13 that are going to make them.

14 Q. Thank you.

15 MR. BOBROW: Let's mark this next,
16 please.

17 (Exhibit 15, United States Patent No.
18 5,389,401, marked for identification.)

19 BY MR. BOBROW:

20 Q. All right. Sir, you've been
21 handed Exhibit 15 for identification. This
22 is a United States patent issued to Roy
23 Gordon, U.S. Patent 5,389,401.

24 Have you seen Exhibit 15 before?

25 A. I've seen reference to it, to the

1 work of using this, but I don't recall
2 reading this.

3 Q. When you say you've seen reference
4 to the work using this, what did you mean by
5 that?

6 A. In -- maybe it was -- I'm not sure
7 exactly where, but maybe during the course of
8 these deliberations, the course of the -- the
9 work associated with this dispute. I vaguely
10 remember seeing that Gordon had a number of
11 other patents and one of these involved the
12 alkoxide, but I don't remember reading it in
13 detail.

14 Q. Had you read any of Dr. Gordon's
15 patents before you got involved in the
16 dispute between Micron and Harvard?

17 A. No.

18 Q. You read some of his papers?

19 A. I read many of his papers.

20 Q. Now, with respect to this patent,
21 Exhibit 15, this is not an exhibit -- sorry.
22 Strike that.

23 Exhibit 15 is not a paper that you
24 refer to or cite in your declarations in
25 these IPR proceedings; correct?

1 A. Yes, that is correct.

2 Q. At the time that you submitted
3 your declarations in this matter, had you
4 read Exhibit 15?

5 MR. LEN: Objection.

6 A. This one.

7 Q. Yes, exactly, this patent. The
8 '401 patent.

9 A. I think I already said I had not
10 read this.

11 Q. And then it sounded as though you
12 had read something that had referred to it.
13 So maybe I'm unclear.

14 What had you read or reviewed
15 which led you to have some familiarity with
16 the subject addressed in the '401 Patent?

17 A. Again, I just vaguely recall
18 seeing a list of earlier patents by the
19 Gordon group at Harvard.

20 Q. And you see that the title of the
21 patent is "Chemical Vapor Deposition of Metal
22 Oxides," correct?

23 A. Yes.

24 Q. And you understand that this
25 patent relates to methods performing metal

1 oxide by chemical vapor deposition?

2 A. That's as much as I can tell from
3 the title, yes.

4 Q. And take a look, if you would, on
5 the Column 1 and running over to 2, there's
6 something called the "Summary of The
7 Invention."

8 Do you see that?

9 A. Yes.

10 Q. And you'll see that this says, "A
11 principal object of the present invention is
12 to deposit metal oxides having high purity,
13 not contaminated by elements remaining from
14 the metal ligands, such as carbon, nitrogen
15 or chlorine."

16 Do you see what I'm referring to
17 there?

18 A. I do.

19 Q. If you go further down in the
20 Summary of the Invention, you'll see at line
21 27 a reference by Dr. Gordon to, "Many
22 different volatile metal compounds..."

23 Do you see where I'm referring to
24 there?

25 A. I do.

1 Q. And Dr. Gordon there is saying
2 that there are many different metal compounds
3 that can be used as the metal source for
4 metal oxide deposition; is that fair?

5 MR. LEN: Objection.

6 A. It's specifically saying that
7 alkoxides, halides and dialkylamides -- all
8 functions in --

9 THE REPORTER: Keep your voice up,
10 please.

11 THE WITNESS: Sorry. I'm just
12 reading slowly as I'm speaking.

13 -- "in the invention as sources of
14 the metal," yes.

15 BY MR. BOBROW:

16 Q. So one of the sources of the metal
17 for purposes of the invention in this patent
18 is dialkylamide metals, correct?

19 A. I'm a little bit at a loss here
20 because I haven't read through the entire
21 document. There is, as I read the abstract,
22 no discussion of metal dialkylamides. So I'm
23 hesitant to be more specific than that until
24 I go through the rest of the -- read the rest
25 of the specification in the claims.

1 Q. Let me ask you a little bit of a
2 different question.

3 Were you aware that Dr. Gordon had
4 secured patents relating to the deposition of
5 metal oxides by CVD using metal
6 dialkylamides?

7 MR. LEN: Objection.

8 A. I was familiar prior to starting
9 the proceedings associated with this dispute
10 of the papers, the publications, but I had
11 not reviewed, prior to this dispute, the
12 patents by Gordon.

13 Q. Okay. Were you aware that
14 Dr. Gordon had published papers on the
15 subject of forming metal oxides using metal
16 dialkylamides as the metal source, prior to
17 the time he filed the patents, the '016 and
18 '539 patents?

19 A. Yeah, so the publications I'm
20 familiar with.

21 Q. Right. And were you aware one way
22 or the other as to whether Dr. Gordon was
23 developing techniques for reducing carbon,
24 nitrogen, and chlorine contamination in metal
25 oxide films performed by metal dialkylamides,

1 prior to the time that he filed the '016 and
2 '539 patents?

3 MR. LEN: Objection.

4 A. I am not aware of all the
5 motivations that Professor Gordon had for
6 conducting the sequence of studies.

7 Q. Okay. Prior to reading that
8 sentence in the Summary of the Invention that
9 talks about an object of the invention, is to
10 deposit metal oxides having high purity not
11 contaminated by elements remaining from the
12 metal ligands, such as carbon, nitrogen, or
13 chlorine, had you been aware that Dr. Gordon,
14 in his research, had attempted to form metal
15 oxides using metal, such as metal
16 dialkylamides, in an effort to reduce carbon,
17 nitrogen, and chlorine in those films?

18 MR. LEN: Objection.

19 A. I'm sorry. Could you ask that
20 again?

21 Q. Yes. But before the time that
22 you, just now, read the sentence at lines 67
23 to 68 of Column 1 and lines 1 to 2 of
24 Column 2 of Exhibit 15, the '401 Patent, had
25 you been aware that one object of

1 Dr. Gordon's research in the area of metal
2 oxides was to reduce carbon, nitrogen, and
3 chlorine contamination in films, such as
4 metal oxide films, formed using alkoxides,
5 halides, and dialkylamides?

6 MR. LEN: Objection.

7 A. Again, I think what I said was,
8 and perhaps this was the immediately
9 presiding question, was that I don't -- I
10 didn't know what the specific motivation that
11 Gordon had for a given sequence of studies.
12 I do understand that in our work, and I think
13 in the work of many people that were working
14 in the precursor area, that is one of the
15 generic goals, is to deposit films of the
16 highest possible purity. So in that regard,
17 I can only assume that Gordon had that as one
18 of his goals.

19 Q. Did you, as part of your work in
20 these IPR proceedings, conduct any sort of
21 prior art search for patents dealing with
22 deposition of metal oxides by CVD or ALD in
23 which a dialkylamide precursor was used?

24 MR. LEN: Objection.

25 A. So when I conduct a literature

1 search these days, I make extensive use of a
2 methodology available to us called SciFinder.
3 It's run by the chemical abstract service of
4 the American Chemical Society, and it does,
5 for a given search, input, give you both
6 papers and patents, and I would scan both of
7 these for relevant -- relevant literature.

8 Q. Did you do such a search in
9 connection with the IPR proceedings?

10 MR. LEN: Objection.

11 A. I guess what I can say is I do
12 searches almost -- well, I wouldn't say
13 daily, but I do a lot of searches on this
14 whenever I have question to see whether
15 there's precedent. So I'm sure that I did
16 some searches that were directly related to
17 these IPR proceedings.

18 Q. Okay. In connection with any
19 prior art searching that you did, did the
20 '401 Patent of Gordon, Exhibit 15, surface in
21 connection with your searches?

22 MR. LEN: Objection.

23 A. I don't remember. It's actually
24 conceivable that that's where I saw it. So
25 I'm sorry. I just can't remember.

1 Q. Going back to Exhibit 3 that I had
2 asked you some questions about before, the
3 PCP publication to Leskela and Ritala. Did
4 Exhibit 3 surface in connection with any
5 prior art searching that you had done in
6 connection with your work on these IPRs?

7 MR. LEN: Objection.

8 A. I believe so. The only reason I'm
9 hesitant about Exhibit 3 is I saw this
10 international application, and perhaps you
11 can correct me or clarify that this
12 ultimately did turn into a patent.

13 Q. I can't answer that question right
14 this minute. So I don't know. I'm sorry.

15 A. Okay.

16 Q. Maybe I'm taking the thread of
17 your question the wrong way. But are you
18 stating that you believe that you found, in
19 the course of your prior art searching for
20 purposes of the IPRs, a patent that
21 ultimately issued from Exhibit 3?

22 MR. LEN: Objection. Assumes
23 facts not in evidence. Go ahead.

24 A. I think the first time I saw
25 this -- I'm trying to think back -- was a

1 result of reading some of the documentation
2 that was presented by MTI that called this
3 into review.

4 Q. All right. So as best as you can
5 recall, then, in terms of prior art searches
6 that you conducted, Exhibit 3 was not one of
7 the pieces of prior art that surfaced from
8 your own searching?

9 MR. LEN: Objection.

10 A. I can't say that it did or did not
11 come up. The more I think about it, the more
12 I recall that this was a document that was
13 part of the information coming forward from
14 MTI.

15 Q. And did this information also come
16 forward from a company called
17 GlobalFoundries?

18 MR. LEN: Objection.

19 A. I don't remember.

20 Q. Okay.

21 A. It could have, but.

22 Q. Okay. I should say you were
23 involved in connection with the dispute
24 between Harvard and GlobalFoundries; is that
25 correct?

1 A. Yes.

2 MR. LEN: I'll let it go.

3 Q. I'm finished with the Leskela
4 exhibit. Maybe you can take a look back with
5 me, though, at Exhibit 15. I'm going to
6 phrase this in a particular way, so bear with
7 me.

8 Was either Exhibit 15 or the work
9 that you alluded to that Dr. Gordon has
10 published over the years on forming metal
11 oxides using metal alkylamides something that
12 you considered and relied on in forming and
13 expressing your opinions in the IPR
14 proceedings specifically?

15 MR. LEN: Objection.

16 A. I can say that I did not consider
17 this particular patent.

18 Q. Exhibit 15?

19 A. Yeah, Exhibit 15, in preparing
20 declarations.

21 (Exhibit 16, Chemistry of Materials, marked
22 for identification.)

23 BY MR. BOBROW:

24 Q. Dr. Gladfelter, you have been
25 handed Exhibit 16. This is a paper in the

1 journal called Chemistry of Materials. The
2 paper is titled "Chemical Vapor Deposition of
3 Titanium, Zirconium, and Hafnium Nitride Thin
4 Films." The authors are Fix, Gordon, and
5 Hofman from 1991.

6 This Exhibit is 1034 in the 662
7 proceeding, 1032 in the 663 proceeding, and
8 1022 in the 664 proceeding.

9 You have seen Exhibit 16 before,
10 Dr. Gladfelter?

11 A. Yes.

12 Q. And when was the first time that
13 you reviewed Exhibit 16?

14 A. Shortly after it appeared.

15 Q. And you're familiar with these
16 authors, right, Fix, Gordon, and Hofman?

17 A. Yes. I don't -- I've never met
18 Fix.

19 Q. But you met Gordon and you've met
20 Hoffman?

21 A. Yes.

22 Q. Now, take a look, if you would,
23 please, at page 8 of the exhibit, which is
24 internal page 1141 of the article, and
25 there's a heading called "Results."

1 Do you see what I'm referring
2 to?

3 A. Yes, I do.

4 Q. And in the first sentence there's
5 a discussion of growing nitride-thin films of
6 titanium nitrides, zirconium nitride, hafnium
7 nitride.

8 You see what I'm referring to?

9 A. Yes.

10 Q. And these were grown along with
11 ammonia as a reactant; is that right?

12 A. Yes.

13 Q. And the metal precursors that were
14 used for purposes of this paper were metal
15 dialkylamides, correct?

16 A. Yes.

17 Q. And there's a titanium
18 dialkylamide, a zirconium dialkylamide, and a
19 hafnium dialkylamide, correct?

20 A. Yes.

21 Q. And those were used in -- at least
22 in this paper, in CVD experiments, correct?

23 A. That's correct.

24 Q. Now, I noted that in the -- in
25 the -- on page 9 towards the bottom there's a

1 discussion of the titanium nitride film
2 deposited by CVD.

3 Do you see what I'm referring to?
4 It's sort of the lower portion of page 9.

5 A. Yes.

6 Q. There's a reference there to a
7 technique called RBS.

8 Do you see what I'm referring
9 to?

10 A. Yes.

11 Q. And it appears that RBS was used
12 to determine the percentage of carbon and
13 oxygen that was present in the film.

14 Do you see what I'm referring
15 to?

16 A. Yes.

17 Q. Okay. What is RBS?

18 A. It's Rutherford Backscattering
19 Spectrometry.

20 Q. Can you describe that technique,
21 please?

22 A. A small accelerator, or an
23 accelerate -- ion accelerator is used to
24 create a beam of atoms -- or actually to
25 ionize and create a beam of ions that bombard

1 a film with energies in the megaelectron volt
2 range, and some of these ions, which is
3 typically helium ions, He1 or He2, that's the
4 charge on the ion, a 1+ or a 2+. A 2+ is
5 actually an alpha particle. It contains no
6 electrons. And that particle beam hits
7 nuclei in the film.

8 The nuclei are more massive than
9 the alpha particle. So the alpha particle is
10 scattered back out in the back direction, and
11 the number of particles that are observed by
12 the detector is proportional to the number of
13 nuclei, and the energy of the backscattering
14 beam is proportional to the mass of the
15 nuclei of which it was scattered.

16 Q. Okay. Thank you.

17 And is that a technique that was
18 used to, for example, determine the atomic
19 percentage of the material in a given film?

20 A. Yes.

21 Q. Further down from that there's a
22 reference to XPS.

23 Do you see that?

24 A. I do.

25 Q. You'll see there there's a

1 reference to XPS depth profile data.

2 Do you see that?

3 A. Yes.

4 Q. What is depth profile data, XPS
5 profile data?

6 A. The XPS is a technique that can
7 also measure the concentration of an atom in
8 a film. But it can also measure the chemical
9 state of that particular atom, and it does
10 that -- it can do that as a function of depth
11 by a couple different methods, but often it's
12 used in conjunction with a -- either a
13 sputtering process, which is a way to drill
14 away top layers of a material, and you
15 reanalyze, drill away a little more, analyze
16 again, and create a profile, the depth
17 profile. That's one way to do it.

18 There's another way to do it by
19 doing something called angular resolved XPS,
20 but that's not what they used here. You can
21 see in the sentence that follows in that
22 paragraph, the third sentence, that the
23 layers were sputtered using an argon ion gun.

24 Q. And it appears in the titanium
25 nitride film that using the RBS technique, it

1 says that the carbon and oxygen contamination
2 was below the detection limits of RPS.

3 Do you see what I'm referring
4 to?

5 A. Where are you at?

6 Q. Towards the bottom of page 9,
7 column -- the low right-hand column under
8 titanium nitride.

9 A. Oh, yes. Okay. I see it right at
10 the very top of that section, yes.

11 Q. Does the conclusion that's drawn
12 from that, that these carbon and oxygen were
13 present at less than two to three atom
14 percent, is that consistent with your
15 understanding of what the limits of RPS were
16 back at this time, in the early 1990s?

17 MR. LEN: Objection.

18 A. It's not inconsistent. Again,
19 such declarations of sensitivity depend on
20 the actual instrument, and, quite frankly,
21 the operator, as to how good it can be. But
22 it's often stated that a few percent for
23 light elements, such as carbon and nitrogen,
24 the sensitivity is not as good as for heavy
25 elements.

1 Q. I see. And then if you turn to
2 page 10, there's further discussion of the --
3 what was found by the XPS technique where it
4 says, on the left-hand side, that "The
5 highest purity films were obtained at 200
6 degrees C; and they contained less than one
7 atom % carbon and oxygen."

8 Do you see where I'm referring
9 to?

10 A. Right at the top. I see.

11 Q. And back in the early 1990s, was
12 XPS depth profile technologies sufficient
13 such that one could measure a film and reach
14 the determination that it contained less than
15 one atom percent carbon and oxygen?

16 A. Yes.

17 Q. In the zirconium and hafnium
18 nitride section of the discussion on page 10,
19 in the second paragraph under that heading,
20 it says, "No carbon or oxygen was detected in
21 the zirconium or hafnium nitride films by
22 RBS. XPS depth profile data showed that, in
23 general, these films contain less than 1 atom
24 % of carbon and oxygen."

25 Do you see what I'm referring

1 to?

2 A. I do.

3 Q. Okay. And, again, is this
4 conclusion in evaluating a zirconium or a
5 hafnium nitride film in using XPS, was it
6 understood in the art that XPS would allow
7 one to make the conclusion here that the
8 percentage was less than one percent -- one
9 atom percent carbon and oxygen in a film?

10 A. I would simply clarify, as I did
11 with Rutherford backscattering, that this
12 will depend on the instrument and the
13 operator. You know, how careful the operator
14 is in making the measurements and in
15 particular whether or not they used a
16 standard to correctly calculate the
17 concentration. I have no knowledge as to
18 exactly how they processed the data here.

19 Q. And would you expect that
20 Dr. Gordon and those in his lab would have
21 done a good job of conducting these
22 experiments?

23 MR. LEN: Objection.

24 A. My understanding is that they
25 probably would have.

1 Q. In connection with forming the
2 opinions that you expressed in your
3 declarations, did you consider or rely upon
4 Exhibit 16?

5 A. That's what we're looking at now?

6 Q. That's correct.

7 A. Was that the question?

8 Q. That was.

9 A. Yes, it was included.

10 Q. And my recollection was that
11 Exhibit 16 was not one of your exhibits, but
12 it was an exhibit that Dr. Banerjee supplied
13 in these proceedings.

14 So my question was directed to
15 whether you reviewed Exhibit 16 and
16 formulated your opinions that you expressed
17 in those declarations and your reliance upon
18 the data in Exhibit 1?

19 MR. LEN: Objection.

20 A. Could you ask this again?

21 Q. Yes.

22 A. It's convoluted.

23 Q. I apologize. This exhibit, which
24 has an exhibit number, for example, of 134,
25 was an exhibit supplied in these proceedings

1 by Dr. Banerjee.

2 A. Okay.

3 Q. And my question to you is whether
4 you relied upon the experiments and the
5 results of the experiments that are set forth
6 in Exhibit 16 in forming and expressing the
7 opinions that you did in your declaration?

8 A. Yes, this is an example of running
9 a deposition in the presence of ammonia to
10 reduce the amount of carbon and nitrogen
11 relative to running it in the absence of
12 this.

13 This was also a particularly
14 interesting paper at the time that it
15 appeared because they also did characterize
16 the amount of hydrogen in a film, which was
17 something that is far more difficult to do,
18 and they were able to find that there was a
19 large amount of hydrogen in these materials.

20 Q. Let's move on to another exhibit.
21 (Exhibit 17, United States Patent No.
22 5,178,911, marked for identification.)

23 BY MR. BOBROW:

24 Q. Dr. Gladfelter, before you is
25 Exhibit 17. It's a copy of U.S. Patent No.

1 5,178,911 to Gordon, Hoffman, and Riaz, with
2 a issue date of January 12th, 1993. Take a
3 moment, if you would, to look at it
4 sufficiently to let me know whether you have
5 seen Exhibit 17 before.

6 A. I don't recall seeing 17 before.

7 Q. I take it from that, that this was
8 not one of the patents that you reviewed in
9 connection with forming and expressing your
10 opinions in this -- the IPR proceedings; is
11 that fair?

12 MR. LEN: Objection.

13 A. As I said, I don't recall
14 reviewing this patent.

15 (Exhibit 18, Korean Shin Patent, marked for
16 identification.)

17 BY MR. BOBROW:

18 Q. Dr. Gladfelter, you have
19 Exhibit 18 for identification. This is a
20 copy of a translation and also the original
21 Korean Patent to Shin, S-h-i-n, in the IPR
22 proceedings. It is Exhibit 1007 in the 663,
23 and 1007 in the 664 proceeding.

24 Is Exhibit 18 an exhibit that you
25 reviewed in connection with these

1 proceedings?

2 A. Yes.

3 Q. Did you understand that Shin also
4 conducted experiments on CVD using metal
5 alkylamide precursors?

6 A. Yes.

7 Q. And you also understand that Shin
8 formed a titanium nitride film in two ways.
9 One way a single source, and one way using
10 the metal alkylamide plus ammonia, correct?

11 A. Yes.

12 Q. And would you agree that
13 consistent with some of the other papers that
14 we looked at, that the percentage of carbon
15 in the film using the single-source CVD
16 technique was higher than using the technique
17 with ammonia?

18 A. Yes.

19 Q. And that was one of the findings
20 in Shin, correct?

21 A. Yes.

22 Q. Let me ask you -- I have to dig
23 this out of the pile. Take a look, if you
24 would, at Exhibit 1, which is the '539
25 patent. You have that in front of you. And

1 I'm going to mark the '016 as the next in
2 order.

3 (Exhibit 19, United States Patent No.
4 8,334,016, marked for identification.)

5 BY MR. BOBROW:

6 Q. All right. You have Exhibit 19 in
7 front of you. It is a U.S. Patent 8,334,016,
8 which in these proceedings is Exhibit 1028 in
9 the 662 IPR, 1001 in the 663, and 1001 in the
10 664 IPR.

11 And I trust that you've had an
12 opportunity and have read the '016 patent,
13 correct?

14 A. Yes.

15 Q. Take a look back at the claims, if
16 you would for me. They appear in Column 30.

17 A. Of '016?

18 Q. Yes. And I guess, if you could,
19 I'm going to ask you to open Exhibit 1,
20 the '539 patent, and also turn to the last
21 page of the claims that are involved in these
22 IPR proceedings. I might have some questions
23 for you about both of these.

24 One question I had for you has to
25 do with the word in the '016 patent, Claim 1

1 at lines 20 to 21, there's a reference to a
2 metal alkylamide.

3 Do you see what I'm referring
4 to?

5 A. Yes.

6 Q. And in the '539 patent, there's a
7 reference to a metal amide that has a
8 dialkylamido moiety.

9 You see what I'm referring to
10 there?

11 A. In 24?

12 Q. Yeah, exactly.

13 A. Claim 24?

14 Q. Correct.

15 A. Yes.

16 Q. Okay. Can you tell me what the
17 difference is between a metal alkylamide and
18 a metal dialkylamide?

19 A. It's a short writing of -- there
20 has to be considered two answers to this. If
21 it is a monoalkylamide, meaning that the
22 amide attached to the metal, that nitrogen
23 attached to the metal has two substituents,
24 one is a hydrogen and the other is an alkyl,
25 or silyl, or some other saturated

1 substituent, that would be a monoalkylamide.
2 A dialkylamide is one in which you have two
3 alkyl groups, and it's not uncommon to see
4 people just say --

5 THE REPORTER: To see people?

6 THE WITNESS: To see people use
7 metal alkylamides to cover the broader
8 class of these compounds.

9 BY MR. BOBROW:

10 Q. Okay. Thank you.

11 Now, in the '016 patent, also in
12 Claim 1, there's a reference to making an
13 insulator in a microelectronic device.

14 Do you see what I'm referring
15 to?

16 A. At the very top?

17 Q. Yeah.

18 A. Yes.

19 Q. What is an insulator, in this
20 context?

21 A. It's a reference to -- it's an
22 electrical property of a material. In this
23 case a thin film, in which the material has a
24 high electrical resistance. It's not a
25 effective conductor of electricity.

1 Q. And what about a microelectronic
2 device, what's that?

3 A. A very tiny electronic device.

4 Q. Okay. And are there metes and
5 bounds of what qualifies as tiny?

6 A. There may be by the people that
7 process these. You do see some people go to
8 the term nanoelectronic device, but the
9 generic term is going to be, that I've always
10 seen, is microelectronic device be a broad
11 coverage of these -- of these devices.

12 Q. Okay. So I wanted to ask you a
13 couple more questions about the claims. One
14 thing I wanted to confirm is that as you
15 understand these claims, both the '539 patent
16 and the '016 patent that are as issue in
17 these this proceedings, the claims do not
18 require that any sort of metal oxide film
19 being deposited have a particular K value,
20 permativity value.

21 A. Permativity.

22 Q. Would you agree with that?

23 MR. LEN: Objection.

24 A. I'm not sure -- can you ask that
25 again?

1 Q. Yeah. Do the claims in your view
2 require some minimum permativity value? Must
3 it be a permativity of a particular quantity?

4 A. I would say -- the answer is
5 there's nothing that specifies a value of K.
6 The purpose of developing many of these
7 processes was to make a material that has a
8 higher value of K than silicon dioxide, which
9 was the current -- or the existing gate
10 dielectric material in field effect
11 transistors prior to -- or within the '90s.

12 Q. And the claims, however, in your
13 view, don't specify that the -- that the
14 permativity of any film being deposited has
15 to exceed or be higher than silicon dioxide
16 or silicon nitride, for example?

17 A. No, I don't see any reference --
18 any quantitative reference to that.

19 Q. Would you agree with me that the
20 claims also do not place any requirement on
21 the amount of carbon or nitrogen or other
22 impurities in the film?

23 MR. LEN: Objection.

24 A. There's no statement within these
25 claims. It's certainly -- you know, a goal

1 that is to be achieved during these
2 deposition processes, that goal being to
3 reduce these as much as possible.

4 Q. But you would agree with me that
5 the claims don't specify that there's some
6 maximum level of contaminant that could be
7 present in the film being deposited using the
8 claim technique?

9 A. Yeah, I --

10 MR. LEN: Objection.

11 A. I don't see any written words that
12 express that.

13 Q. Now, you understand that in the
14 662, 663, and 664 proceedings that we're
15 involved in, Claim 31 is not involved in
16 those proceedings, correct?

17 A. That's my understanding.

18 Q. Okay. Now, with respect, then, to
19 the claims that are at issue here in these
20 proceedings, those claims don't require that
21 the film be deposited on any particular
22 structure, via a trench, a hole or the like,
23 correct?

24 A. I understand that these are -- I
25 would agree, this is a process -- I

1 understand it -- you know, they talk about,
2 in this deposition process, the only
3 reference to a structure and to coverage
4 really came in Claim 31 of the '539 patent.

5 Q. Would you agree, then, that in
6 terms of the claims that are at issue in the
7 662, 663, and 664 proceedings, that the
8 claims don't require any particular level or
9 degree of conformality of a film being formed
10 and deposited?

11 MR. LEN: Objection.

12 A. I think maybe -- well, I would say
13 it's implicit that -- and it's described as
14 one of the characteristics of atomic layer
15 deposition, I believe in the specification,
16 that these -- that atomic layer deposition
17 processes are particularly good, given
18 conformal coverage. There's not -- outside
19 of Claim 31, I don't recognize any statement
20 that delves further into that, but certainly
21 there's information in the specification that
22 talks about conformal coverage.

23 Q. Right. But in your understanding
24 and your opinion, any particular level of
25 conformality or step coverage is not being

1 claimed in the claims that are at issue in
2 the 662, 663 and 664 proceedings?

3 A. I don't -- yes, I'm in agreement,
4 outside of Claim 31 in '539, I don't have
5 any -- not had any further discussions about
6 a quantitative measure of conformality.

7 Q. Now, unlike the '539 -- sorry --
8 strike that.

9 Unlike the '016 patent, the
10 claims of the '539 patent do not require that
11 the film be deposited for a microelectronic
12 device, correct?

13 A. You said for -- for '539 it does
14 not. Yeah, for '016 that's part of it, but
15 the '539 does not.

16 Q. Just more generally for a minute,
17 would you agree that there are uses in
18 applications for atomic layer deposition that
19 are outside of the microelectronics field?

20 MR. LEN: Objection.

21 A. I understand that there are other
22 applications, yes.

23 Q. What other applications outside of
24 microelectronics are you aware of for which
25 ALD has been used as a technique to deposit

1 metal oxide films?

2 A. You can go to some of the work
3 where you create a thin film on a -- on a
4 glass to serve as an antireflective coating
5 or another type of coating that reflects
6 thermal infrared radiation. These are thin
7 films. That's one place where people have
8 talked about it. Then there are other
9 applications that people discuss in the
10 literature, and it's unclear whether these
11 have made it into any industrial process.

12 Q. And what applications do you have
13 in mind?

14 A. Applications that include using
15 atomic layer deposition, for instance, to
16 uniformly coat a membrane that might be
17 porous, that one could then use to filter out
18 water or different species in an application.
19 Applications that allow you to take, for
20 instance, a particle and coat the surface of
21 a particle with a material that might be
22 particularly effective at a catalytic
23 reaction. So these are a couple examples.

24 Q. Okay. Have you personally been
25 involved in any ALD research to deposit films

1 outside of an electronic context?

2 A. One of our more recent patents was
3 to deposit films on a polymer surface, a
4 structured polymer surface.

5 Q. Okay. Back to the claims of these
6 patents, again, putting aside Claim 31, which
7 isn't at issue in these proceedings, would
8 you agree that the claims of the '539 patent
9 and the '016 patent do not require that the
10 metal oxide film be formed on a surface or a
11 structure that has a particular aspect ratio?

12 MR. LEN: Objection.

13 A. The claims themselves don't
14 identify the structure of the underlying
15 substrate.

16 Q. Okay. And nor do they specify any
17 such -- any aspect ratio or geometric
18 property associated with the surface on which
19 the film is being deposited, correct?

20 A. That's correct.

21 I might add, though, just to -- if
22 you don't have some sort of structured film
23 to -- or structured substrate to do a
24 deposition, choosing to do atomic layer
25 deposition might not be the wisest choice.

1 It's a wonderful process for what it
2 achieves, but it's a very slow process.

3 So if you have a uniform surface
4 that doesn't have complicated surfaces --
5 structures on the surface, using regular CVD
6 or even some other physical vapor deposition
7 process might be a more economical and
8 effective alternative.

9 Q. Now, the claims of the '539 patent
10 and the '016 patent don't require that the
11 film be formed for some industrial or
12 commercial purpose, correct?

13 MR. LEN: Objection.

14 A. Well, I think the inclusion in the
15 '016 patent of -- on the first claim to make
16 an insulator for a microelectronic device
17 does imply that it's important for -- or it's
18 potentially going to be used in that
19 industry.

20 Q. What about Claim 24? Claim 24
21 does not require the film be deposited for
22 any particular commercial or industrial
23 purpose, correct?

24 A. There's nothing in Claim 24 that
25 alludes to the purpose. In the specification

1 for these patents, you see more information
2 written about the types of materials that are
3 being deposited.

4 Q. Now, with respect to that phrase
5 "microelectronic device" in this introductory
6 paragraph of Claim 1 of the '016 patent, your
7 review is that microelectronic device
8 that's -- you view that as being a
9 requirement of the claim? In other words,
10 that's something that if you deposit -- if
11 you use this method to coat this stapler,
12 that that would not be covered by the claim?

13 MR. LEN: Objection.

14 A. I would not interpret these
15 patents as being limited to a microelectronic
16 device.

17 Q. Okay. I saw a reference in, I
18 think, one of your declarations to something
19 called an ITRS road map?

20 A. Yes.

21 Q. Are you familiar with what I'm
22 referring to?

23 A. Yes.

24 Q. In terms of the acronym ITRS, is
25 that like an industry consortium or a group?

1 What is ITRS?

2 A. That's my understanding. This was
3 an industry -- industrial consortium that got
4 together to try to solve common problems
5 associated with keeping up with what is
6 called Moore's law.

7 Q. Have you ever been part of that
8 ITRS group?

9 A. No.

10 Q. Have you been consulted by members
11 of that group for purposes of contributing to
12 the work or the output of that group?

13 A. No.

14 Q. Are you part of any -- sorry. Are
15 you a member, I should say, of any
16 semiconductor industry groups?

17 A. No.

18 Q. Have you ever been?

19 A. We were funded. When I say "we,"
20 my colleague Steve Campbell and I. Professor
21 Campbell is a faculty member in the
22 electrical and computer engineering
23 department at the University of Minnesota,
24 and we've coauthored more than 20
25 publications with one another. Many on high

1 K dielectrics, and some of that work was
2 funded by the Semiconductor Research
3 Corporation.

4 Q. Okay. And although you received
5 funding from SRC, you are not and haven't
6 been a member of SRC?

7 A. That's correct.

8 MR. BOBROW: Shall we take a short
9 break? I don't know how long we've been
10 going, but it feels like a long time.

11 (Recess taken at 4:07 p.m. to 4:20 p.m.)

12 MR. BOBROW: Let's go back on the
13 record.

14 BY MR. BOBROW:

15 Q. You still have the '539 and '016
16 patents in front of you?

17 A. Yes.

18 Q. I think -- let me just ask you to
19 take a look at the '539 patent. Your
20 understanding is that the specifications of
21 the '539 and the '016 patent are the same,
22 right?

23 A. Yes.

24 Q. And in the specifications of these
25 two patents, there are a number of what the

1 authors of the patent call examples.

2 Do you know what I'm referring
3 to?

4 A. Yes.

5 Q. And I believe in the '539
6 patent -- those begin at the bottom of
7 Column 23 and they run through Column 29
8 right where the claims begin.

9 Do you see that where I'm
10 referring to?

11 A. I do.

12 Q. Okay. And with respect to these
13 examples, it appears that the authors recite
14 24 examples of depositions that are
15 conducted.

16 Do you see what I'm referring to?
17 Including some control -- comparative
18 controls.

19 A. I was just going to comment, yes,
20 they did -- they included some that are
21 called comparative examples; the depositions
22 by competing technology.

23 Q. And in each of the comparative
24 examples, there is no film that was
25 deposited, correct?

1 A. Yes, that is correct. I agree.

2 Q. Okay. So in the 24 examples where
3 there was some film that was deposited, you
4 would agree with me that those examples did
5 not use all of the amides that are listed in
6 Table 1 of the '539 patent and '016 patent,
7 correct?

8 MR. LEN: Objection.

9 A. Let me take a look. Table 1 is a
10 list of amides, and no, not all of these were
11 used in the deposition. They used a number
12 of examples of the amides but not all of
13 them.

14 Q. Okay. And you would agree with me
15 that in the examples that are set forth in
16 the '539 and '016 patents, the named
17 inventors on the patents did not test all of
18 the metal alkylamides or dialkylamides that
19 are listed in Table 1, correct?

20 MR. LEN: Objection.

21 A. It's certainly my understanding
22 that there was not a need to go through and
23 test each one of these to recognize that they
24 are potential precursors for the process
25 claimed, the atomic layer deposition

1 processes.

2 Q. Regardless of need, my question
3 was simply that you would agree that the
4 examples set forth in these patents do not
5 recite tests or experiments on all of the
6 metal alkylamides and dialkylamides that are
7 listed in Table 1. Would you agree with
8 that?

9 A. I agree with that.

10 Q. Now, with respect to the claims,
11 Claim 24 of the '539 and Claim 1 of the '016,
12 you'd agree with me that Claim 1 of the '016
13 is not restricted to a particular subset of
14 metal alkylamides.

15 Would you agree with that?

16 MR. LEN: Objection.

17 A. I -- there's no restriction that I
18 can read into -- into the words in Claim 1.

19 Q. And with respect to Claim 24 of
20 the '539 patent, you would agree that there's
21 no limit on which metal dialkylamide is used
22 in performing Claim 24.

23 Would you agree with that?

24 A. Are you referring --

25 MR. LEN: Objection. Sorry.

1 THE WITNESS: I'm sorry.

2 MR. LEN: Objection. Go ahead.

3 THE WITNESS: Are you referring to
4 the variation in the metal?

5 BY MR. BOBROW:

6 Q. Correct. In other words,
7 there's -- it refers to the metal amides that
8 include a dialky- --

9 A. Right.

10 Q. -- a dialkylamido moiety; right?
11 And my question was: There isn't
12 a restriction on whether that metal
13 alkylamide is titanium, or zirconium, or
14 hafnium, or tin, or lead, or any other metal.
15 Would you agree with that?

16 A. I agree with that.

17 Q. Would you agree with me that the
18 metal dialkylamides that fall within the
19 scope of Claim 24 of the '539 patent, given
20 that it's not limited to any particular
21 metals, cover a group of dialkylamides that
22 have different chemical and physical
23 properties?

24 MR. LEN: Objection.

25 A. Well, first of all, just make sure

1 we're being inclusive here. There are the
2 dialkylamides, the disilylamides, and the
3 alkylsilylamido compounds that are included.
4 So when I speak of this as the alkylamides, I
5 want to recognize that those are included in
6 this.

7 So now I have to go back to the
8 specific question. Please, if you could
9 repeat.

10 Q. Yes. My question is that Claim 24
11 of the '539 patent and Claim 1 of the '016
12 patent encompass metal dialkylamides and
13 metal alkylamides respectively, that include
14 compounds that have different physical and
15 chemical properties?

16 MR. LEN: Objection.

17 A. Yes, it would include materials
18 that -- the precursors themselves, that
19 you're referring to, the precursors
20 themselves will be -- will have different
21 properties.

22 Q. And not all those precursors were
23 tested by the named inventors in connection
24 with these two patents, correct?

25 MR. LEN: Objection.

1 A. Not all the precursors -- the
2 precursors?

3 Q. All the precursors that would be
4 encompassed within Claim 1 of the '016 and
5 Claim 24 of the '539?

6 A. No. And it's my understanding
7 that it's not a requirement for the patent to
8 have tested every individual precursor that
9 would fit into this generic category.

10 Q. And would you agree that a person
11 of ordinary skill reading the '539 and '016
12 patents would be able to make and use the
13 inventions claimed in Claim 24 of the '539
14 and Claim 1 of the '016 for any metal
15 alkylamide or dialkylamide based upon the
16 disclosure of the specific example
17 experiments, 1 through 24, that's set forth
18 in those patents?

19 A. The subset of when you look at the
20 literature of inorganic compounds, the subset
21 of compounds that are dialkyl -- let's just
22 say the metal amido complexes are -- the
23 metal alkylamido complexes constitute a
24 relatively small overall class of materials
25 as these things -- as in comparison to, you

1 know, looking at an example -- at examples
2 where you can vary all the ligands and see a
3 huge number of materials being covered. The
4 alkylamides will have some important similar
5 properties, in terms of their reactivity, in
6 particular the reactivity is relevant to an
7 ALD process.

8 Q. And what important similarities in
9 terms of reactivity do you have in mind?

10 A. In atomic layer deposition, if you
11 recall earlier in our discussion, we talked
12 about half-reactions, you need a high
13 reactivity for the initial precursor to bond
14 with the surface and then a high -- when I
15 say "high," I'm not talking about an
16 elevation. I'm talking about a reaction that
17 is relatively fast but, more importantly,
18 that is prone to go to completion, meaning
19 every possible species on the surface that is
20 available can be reacted with the amides.
21 That's one of the requirements set forth by
22 the early researchers in atomic layer
23 deposition. In addition to some of the
24 desirable features, such as volatility and so
25 on. You have to have every active compound.

1 And many of the examples in the
2 table -- I'm referring to Table 1 in the
3 specification of these patents -- fulfill
4 this characteristic.

5 Q. And when you were referring
6 earlier to the relatively small number in a
7 class, were you referring to a class of metal
8 alkylamides?

9 A. Yes.

10 Q. And was it known in the 1970s, and
11 1980s, 1990s, that metal alkylamides had this
12 property of being reactive?

13 MR. LEN: Objection.

14 A. The chemists that originally
15 synthesized these materials, going back to
16 the earlier work in the late '50s, early
17 '60s, reported that they were reactive, and
18 as the body of literature increased, we see
19 that the alkylamides, as a class, had a
20 reactivity pattern that was characteristic of
21 that material.

22 Q. When you were referring to the
23 work in the 1950s and early '60s, were you
24 referring to the work by Bradley?

25 A. Don Bradley was one of the people

1 that -- was one of the leading researchers in
2 this field at the beginning.

3 Q. So given what you just described,
4 is it your view, then, that a person of
5 ordinary skill reading the specific number of
6 examples with specific metal alkylamides
7 precursors in the '016 and '539 patents,
8 would then be able to make and use the
9 inventions of the '016 and '539 on other
10 metal alkylamide precursors without having to
11 experiment unduly?

12 MR. LEN: Objection.

13 A. Well, that's a pretty broad
14 statement to try and cover. The -- there are
15 a large number of examples where these are
16 demonstrated, and my view is that the
17 patent -- the patents can be extended to
18 incorporate deposition using precursors,
19 other than the ones provided in specific
20 examples.

21 Q. And one would be able to do that
22 without having to do a lot of
23 experimentation, just routine testing?

24 A. No, I don't agree with that. I
25 think any time you change the precursor

1 there's going to be -- you know, there's
2 different volatilities you have to deal with,
3 there's different -- the chemical reactivity
4 that I refer to as being high, it doesn't
5 mean that they're always the same. So one
6 needs to spend particular time dealing with
7 changing things like the deposition
8 temperature, changing the flux of the
9 precursor. The flux is the amount of
10 precursor in the gas phase that collides with
11 the surface, an area of the surface per unit
12 time. So all of those factors have to be
13 addressed.

14 Q. And, in your view, in order to use
15 and make the claimed inventions of the '539
16 and the '016 patent, using a metal alkylamide
17 or dialkylamide precursor that was not the
18 subject of one of the specific examples, in
19 your view that would take a person of
20 ordinary skill extensive experimentation with
21 a design of experiment, would take
22 considerable time and effort and skill, or
23 would it be more routine in nature?

24 MR. LEN: Objection.

25 A. I think it's best to say that you

1 would understand -- a person skilled in the
2 art would understand what variations have to
3 be tested and what kind of studies would have
4 to be done, but you will not know the -- some
5 of the critical results.

6 We've spent a fair amount of time
7 in our discussion this afternoon dealing with
8 something associated with impurities. We've
9 seen some experiments that work to reduce the
10 amount of purities in depositions and other
11 examples where that doesn't work. A person
12 looking at these will not only be looking at
13 the question of does the deposition work at
14 all, but they're going to characterize the
15 deposition. Is it conformal? Is it uniform
16 across many wafers or within wafers in terms
17 of the thickness? Does it incorporate
18 impurities? Is there structure on the
19 substrate? Is it a good conformal coverage
20 over the structure? So these are -- they're
21 are time-consuming.

22 I think one skilled in the art
23 would understand what experiments need to be
24 done, but it's not -- it's not something that
25 is fully predictable. Within a class it's

1 probably a little bit easier to predict going
2 beyond classes, which we haven't discussed,
3 becomes a much more significant issue.

4 Q. And when you were just referring
5 to a class, one such class would be metal
6 alkylamides, for example?

7 A. Yes. Again, these metalorganic
8 compounds are often classified by the ligand
9 set that's associated with them.

10 Q. And is the predictability that you
11 just described improved when one takes a
12 class, such as alkylamides, and then narrows
13 it down to a subclass, for example, metals
14 that are in a particular column of the
15 periodic table?

16 MR. LEN: Objection.

17 Q. Does that enhance the
18 predictability?

19 A. I would agree that it enhances the
20 predictability, but there are some -- always
21 some surprises when you are doing an
22 experimental science like chemistry that you
23 may encounter.

24 Q. A few minutes ago when you were
25 providing some testimony about things that a

1 person of ordinary skill in the art would do
2 to take the deposition beyond the question of
3 whether it would work at all but to consider
4 other factors.

5 In that context, let me ask you
6 this: Would a person of ordinary skill in
7 the art reading the '539 and the '016 patent
8 specifications and reading the examples, the
9 examples 1 through 24 that are set forth
10 therein, be able to extend that to cover --
11 or to use the claimed inventions with a
12 precursor that is not used in those examples,
13 at least to demonstrate that it works at all?

14 MR. LEN: Objection.

15 Q. Without having to experiment
16 unduly.

17 MR. LEN: Same objection.

18 A. Experiment unduly is a relative
19 term that might mean different things to
20 different people. In my view, if you wanted
21 to demonstrate a different deposition with a
22 different precursor, it will always require
23 experimentation that we'll be looking at
24 changing temperature, changing -- and
25 demonstrating, particular when you're looking

1 at an atomic layer deposition process,
2 whether this does form -- you know, undergo a
3 self-limiting deposition.

4 And, again, I think the work in
5 Gordon's patents that demonstrates that there
6 are good examples of creating a self-limited
7 deposition process with ALD is important and
8 suggestive that you can do this with other
9 metal alkylamides. It doesn't suggest that
10 you can do this with alkoxides or other
11 classes of compounds.

12 Q. And when you say it suggests that
13 you could do this with other alkylamides,
14 what sort of -- or what level or extent of
15 testing would be involved in order to use one
16 of the metal alkylamides in Table 1 that was
17 not one of the examples that's described in
18 the patent?

19 MR. LEN: Objection.

20 A. Again, I think this is a -- to
21 demonstrate what's involved one would have to
22 have the compound. If it's not commercially
23 available, it has to be synthesized. One of
24 the beauties of Table 1 is that there are
25 references to a large number of metal

1 alkylamides that provide that information.
2 But it can be time-consuming and not
3 necessarily easy to synthesize the compounds,
4 so one has to worry about providing the
5 source for the material that you're going to
6 go in to attempt to use.

7 But I think the Gordon patent
8 teaches that once there, there's a pretty
9 good likelihood that you're going to be able
10 to, if you can volatilize the material and
11 expose it to a surface, that you'll be able
12 to do it. I agree that the Gordon patent has
13 the ability to cover the class of compounds
14 as precursors known as metal alkylamides.

15 Q. Okay.

16 MR. BOBROW: Let's mark this next.
17 (Exhibit 20, United States Patent No.
18 4,923,717, marked for identification.)

19 THE WITNESS: Do we need the
20 patents remaining?

21 BY MR. BOBROW:

22 Q. Yeah, I would keep them at hand.

23 Dr. Gladfelter, you have been
24 handed U.S. Patent 4,923,717 to, of all
25 people, yourself, and someone named David C.

1 Boyd. This was a patent issued in May of
2 1990.

3 Do you recognize this patent?

4 A. I do.

5 Q. Who is David Boyd?

6 A. He was a postdoc -- postdoctoral
7 associate in my research group.

8 Q. And do you recall the research
9 that led to this patent?

10 A. I do.

11 Q. Did you review this patent in
12 connection with your work on these
13 proceedings, these IPR proceedings?

14 A. I didn't really have to.

15 Q. Okay. Fair enough.

16 Let's see. All right. So take a
17 look, if you would, at Claim 1 of the '717
18 Patent, which is at Columns 11 and 12.

19 A. Yes.

20 Q. All right. And one thing that you
21 mention here is exposing a surface to a first
22 vapor comprising a Group IVB or VB metal
23 complex.

24 Do you see what I'm referring to
25 there?

1 A. I do.

2 Q. All right. And those references
3 there are two columns of the periodic table?

4 A. Yes.

5 Q. All right. And the Group IVB,
6 that would include titanium, hafnium,
7 zirconium, Rutherfordium?

8 A. That's true.

9 Q. Okay. Have you ever --

10 A. Never worked with Rutherfordium
11 myself, but the other three, yes.

12 Q. Is Rutherfordium something that
13 can be synthesized in a stable way?

14 A. No, it's got a finite lifetime. A
15 short, finite lifetime.

16 Q. And then what about VB, vanadium,
17 niobium, tantalum, anything else?

18 A. Not at the time that we published
19 this. So that whole fourth row of the
20 transition metal series has been filled in by
21 the nuclear chemists and nuclear physicists
22 working on the synthesis of new atoms. But,
23 again, they are all -- the transuranium
24 elements of finite lifetimes in many cases
25 require -- because of their radioactivity,

1 the ones that do have longer lifetimes, like
2 plutonium, you don't want to -- special
3 requirements needed.

4 Q. Sure. And I guess what I'm
5 driving at is that with respect to what you
6 describe in element A of Claim 1, Group IVB
7 and VB, metal complexes, there's a large
8 number of those. Would you agree?

9 A. Yes.

10 Q. And then in paragraph (b) of that,
11 there's a second vapor where you have a
12 formula for that that includes a vapor that
13 has an alkyl, alkenyl, et cetera, et cetera.

14 You see what I'm referring to?

15 A. Yes.

16 Q. And would you agree that there's a
17 large number of compounds that would fit
18 within that formula?

19 A. Yes.

20 Q. All right. Now, in the patent, if
21 I'm reading this correctly, you provided one
22 example of your claimed invention, correct?

23 A. I believe that's the correct case.

24 Q. And as I understand it -- and I
25 don't fully -- as I understand it, you were

1 using titanium tetrachloride, TiCl_4 , and
2 something else that looks like it has some
3 metals in it?

4 A. Yeah. So trimethylamine, as an
5 example. And if you look in example one, I
6 think that was always trimethylamine.

7 Q. Is that a metal alkylamide?

8 A. No. Just to make you crazy, it's
9 an amine compound. It sounds a little bit
10 like the others but it's not.

11 Q. How does it differ chemically from
12 a metal alkylamide?

13 A. Well, I would point out that the
14 generic formula shown in Claim 1 contains
15 three groups, three R groups. These are --
16 and defined a little bit more extensively
17 later on in the claim, but these are
18 typically alkyl groups, such as methyl, ethyl
19 and other possibilities.

20 Q. Okay. But specifically with
21 respect to the other vapor that you use in
22 example one, could you explain to me why --
23 or how that -- what appears to be an
24 aluminum-bearing compound does not -- is not
25 an alkylamide?

1 A. You can see within the square
2 bracket the full formula contains three R
3 groups attached to a Y, and Y is a nitrogen
4 in the actual example provided. It can also
5 be a phosphorous. And if you recall, we were
6 talking about examples where things are donor
7 molecules, donor-acceptor molecules. I think
8 you used the word "adduct" in one of your
9 questions. These are trimethylamine adducts.
10 The aluminum species referred to as alane,
11 a-l-a-n-e.

12 Q. So one part --

13 A. I should just continue. You asked
14 how this differs from an amide?

15 Q. Correct.

16 A. An amide has two substituents, and
17 there is a formal negative charge on the
18 nitrogen, and in the case of an amine, it has
19 three substituents, and it is a neutral
20 moiety.

21 Q. No positive or negative charge?

22 A. No positive or negative charge,
23 correct.

24 Q. And with respect to the second
25 group of vapors there that's in Claim 1,

1 paragraph (b), can you give me a sense of how
2 many compounds would meet that formula?

3 A. In the second --

4 Q. 1(b).

5 A. 1(b).

6 Q. Yep.

7 A. How many of which compounds?

8 Q. Where it says the "second vapor
9 comprising a compound of the formula," and
10 then it provides the formula.

11 A. Okay.

12 Q. I'm trying to get a sense of how
13 many different compounds there are in the
14 world that need that formula.

15 A. Existing in the literature at the
16 time of this work were probably a relatively
17 small number. If I remember correctly, we
18 would see things like trimethylamine alane,
19 which we, of course, use. Triethylamine
20 alane was, I believe also in the literature.
21 Trimethylphosphine alane, that's where the Y
22 group is a phosphorous, and that word is
23 phosphine; as an example, I think that
24 existed at the time of the submission of the
25 patent. So it's a relatively small subset of

1 compounds that existed at the time.

2 Q. And then in the context of this
3 '717 Patent, and it appears in the examples
4 that you provided, Columns 7 and 8, that in
5 terms of the metal vapor from a Group IVB or
6 VB complex, the one you provided was the
7 titanium tetrachloride, correct?

8 A. That's correct.

9 Q. And did you expect that a person
10 of ordinary skill reading the patent and
11 reading this one example would be able to
12 make and use the claimed invention with the
13 full scope of metal vapors covered by Claim
14 1, that is, all the Group IVB metal complexes
15 and Group VB metal complexes?

16 MR. LEN: Objection.

17 A. I will comment the vary -- to
18 begin, the variation of changing one of those
19 substituents on a nitrogen or phosphorous,
20 keeping that relatively small subset, will
21 not change the properties of the -- and
22 structure of the alane itself. As in all
23 cases, there will be change in some of the
24 physical properties.

25 So you would expect physical

1 and -- properties such as volatility or
2 chemical properties such as stability to be
3 impacted by that to some extent. They are
4 different compounds. A different compound
5 will have somewhat different properties.

6 Q. And given that, did you
7 nonetheless expect that a person of ordinary
8 skill reading your patent would be able to
9 practice its full scope, that is, using any
10 Group IVB or VB metal complex to practice
11 your inventions, you know, without having to
12 experiment unduly?

13 MR. LEN: Objection.

14 A. Well, I think you're getting the
15 experiment unduly again, and I'll come back
16 to my statement that I made with respect to
17 the Gordon patents. That any time you change
18 something, you are going to have to spend
19 time -- excuse me -- dealing with refining
20 the process.

21 But, again, I think that what we
22 encompassed here is a relatively small number
23 of species that could be used as the alane
24 source. It's grown a bit since then. But it
25 is still -- the aluminum source is still used

1 extensively in the literature.

2 Q. Okay. And in connection with
3 your -- well, let me ask it a little bit
4 differently.

5 In your understanding, as part of
6 the process of securing a patent, is it your
7 understanding that an inventor is required to
8 describe in the specification in his or her
9 patent application enough to enable a person
10 of ordinary skill to practice the full scope
11 of the inventions that are claimed?

12 A. I think that's my understanding.

13 Q. And your understanding includes
14 that you're supposed to, as a patent
15 applicant, supply enough information so that
16 a person of ordinary skill can make and use
17 the claimed inventions without undue
18 experimentation, correct?

19 A. Yeah. Again, we had that undue
20 experimentation clause that, in my view, you
21 have still have to run these reactions and
22 optimize them if you make any change.

23 Q. And with respect to the '717
24 Patent, you believe that you fulfilled your
25 obligation to describe in the specification

1 sufficient information to enable a person of
2 ordinary skill to practice the full scope of
3 your inventions without undue experimenting?

4 MR. LEN: Objection.

5 A. I believe that, yes.

6 Q. In -- I'll call your patent a
7 chemical patent. I don't mean to disparage
8 it, but I'll say a chemical patent, such as
9 the '717 Patent. You include this general
10 formula, I'll call it, with R1, R2,
11 et cetera. Is that a common practice in your
12 field to include a general description of a
13 formula in that fashion, in these sorts of
14 patents?

15 MR. LEN: Objection.

16 A. Yeah, this is a relatively -- when
17 you're simply varying, for instance, the
18 alkyl substituent on a ligand, that is indeed
19 a common practice.

20 Q. Okay. And then going back to the
21 Gordon patents. In your view, did the Gordon
22 inventors in the specification provide enough
23 information for a person of ordinary skill to
24 practice the full scope of the claims of the
25 '539 and '016 patents without having to

1 unduly experiment to practice the full scope?

2 MR. LEN: Objection.

3 A. I believe these are valid patents
4 that provide the basis to conduct
5 depositions, atomic layer depositions of thin
6 films using precursors other than titanium
7 tetrakis amides or hytetrakis,
8 dimethylamido--

9 THE REPORTER: You need to speak
10 up.

11 THE WITNESS: Sorry. My voice is
12 starting to go away.

13 THE WITNESS: Titanium amides,
14 alkylamides, or zirconium alkylamides,
15 and hafnium alkylamides.

16 BY MR. BOBROW:

17 Q. Without undue experimentation?

18 MR. LEN: Objection.

19 A. Again, I will emphasize that the
20 amount of experimentation, whenever you
21 change a precursor, there is going to be some
22 experimentation that has to be done, and this
23 is simply because there is not perfect
24 predictability for any of these reactions.
25 You have, I think, in the case of the amides,

1 a relatively -- a uniform group of ligands
2 attached to the central element that give
3 rise to a better predictability than if you
4 started varying the ligands extensively.

5 Q. All right. And given that, would
6 you agree that the Gordon patents, the '539
7 and '016 patents, provide in the
8 specification sufficient information to allow
9 a person of ordinary skill to make and use
10 the claimed inventions with metal
11 dialkylamides that are not set forth in the
12 examples of the patent without undue
13 experimentation?

14 MR. LEN: Objection, asked and
15 answered.

16 A. Yeah, again, the -- I think it's
17 similar to what I just said. That I think
18 that these patents put forward a deposition
19 process using a set of precursors that will
20 allow the -- one skilled in the art to design
21 a process and then have a reasonable hope
22 that -- a reasonable chance for achieving a
23 deposition using those metals.

24 Q. Without undue experimentation?

25 MR. LEN: Objection.

1 A. Yeah, undue experimentation is
2 dependent on, apparently, the eye of the
3 beholder. I think that there's still going
4 to be the requirement to do such things, such
5 as vary the -- for ALD the pulse width and
6 time for the individual pulses to verify that
7 you're going to see self-limiting behavior.
8 You're going to have to vary temperature to
9 scope the -- let's say the temperature
10 windows, the region where you can get good
11 deposition. You're going to have to look at
12 the impact of the precursor flux on those
13 factors.

14 These are not short experiments.
15 It will still take some time. But it's
16 using, as the basis, the patents that Gordon
17 and the methodology that Gordon described in
18 his two patents.

19 Q. So I understand you to have just
20 described for me some of the experimental
21 work that you would see as being involved.
22 But that's not really my question.

23 My question is: As yourself being
24 a person of ordinary skill and understanding
25 that the Gordon patent specifications were

1 required to describe and disclose sufficient
2 information to enable the full scope of the
3 invention without experimentation. I need
4 you to answer my question about whether the
5 experimentation would be undue or not, to
6 extend -- to allow a person of ordinary skill
7 to make and use the invention using a metal
8 precursor that is not one that is set forth
9 specifically in the listed examples.

10 MR. LEN: Same objection.

11 A. I believe that -- I think we're
12 going around and around here. But I believe
13 that the amount of experimentation is still
14 going to be something that can be planned and
15 run in these processes with a reasonable hope
16 that you will be successful in depositing the
17 desired metal oxide film.

18 Undue, as I said, is something
19 that is going to be dependent on an
20 individual's perspective, and I guess my
21 perspective is more easily summed up by -- in
22 the following: If I were to deposit a metal
23 oxide film by ALD using a dialkylamide that
24 was not specifically listed in the Gordon
25 patent, I would not be running off to try to

1 patent that separately because it's already
2 covered in the patent.

3 Q. All right. I think, given the
4 risk that we'll continue to go around and
5 around, I need to move on to another subject,
6 which I will.

7 Let me ask you just -- I know you
8 had told me in your prior deposition you have
9 a lab, and I'm assuming you're still
10 continuing to supervise doctoral students.

11 A. Yes, I do have a lab, yes, and a
12 research group.

13 Q. Right. Now, taking yourself back
14 to the time period between 2000, you had a
15 lab in the 1990s at the University of
16 Minnesota, did you not?

17 A. Yes.

18 Q. You had doctoral students that you
19 were supervising at that time, correct?

20 A. Yes.

21 Q. All right. Now -- and you were
22 doing work in the area of ALD in the 1990s?

23 A. No.

24 Q. Okay. And when did that work
25 start?

1 A. I think we started ALD in perhaps
2 the late '90s early 2000s.

3 Q. Okay. So, you know, at that
4 time -- let me ask a little bit differently.

5 In connection with your work to,
6 you know, investigate thin film deposition,
7 has there ever come a circumstance in which
8 you have conducted an experiment to form the
9 thin film, even though you had a very high
10 degree of confidence that the experiment
11 would work?

12 MR. LEN: Objection. Same
13 objections.

14 A. Could you ask -- repeat the
15 question again, please?

16 Q. Yes. In connection with work that
17 you've done in forming thin films using vapor
18 deposition technology, have you ever
19 performed an experiment to test a reaction
20 that you were very confident, prior to the
21 reaction and prior to the experiment, would
22 work, in order to confirm that it would work?

23 MR. LEN: Objection.

24 A. I'm sorry. I'm smiling because,
25 as a research advisor, we give advice many --

1 many bits of advice to students. They make
2 fun of us because that's an advisor
3 suggestion. This shouldn't take long. And
4 the students are always -- they learned
5 quickly to be aware -- or I should say to be
6 aware of such advice. That being aside, I
7 think you look at any potential experiment
8 with a question of whether it might be -- of
9 how successful, or what is the chance of
10 success. So I think we have certainly done
11 some experiments that we thought were a high
12 chance of success, of depositions.

13 Q. And partly to confirm your
14 hypothesis that a film could be formed using
15 that technique?

16 A. It might be -- it might be for
17 that particular reason. It might also be
18 because we needed that film for some other
19 experiment application.

20 Q. And flipping it around, have you
21 ever performed an experiment or have your
22 students performed experiments in the lab to
23 test a, you know, vapor phase reaction that
24 you weren't quite sure would work?

25 MR. LEN: Objection.

1 A. I think that is perhaps the more
2 common of the scenarios. Yes.

3 Q. Okay.

4 (Exhibit 21, Declaration of Wayne L.
5 Gladfelter, Ph.D., in Support of Patent
6 Owner's Response, Patent No. '539, marked
7 for identification.)

8 MR. BOBROW: Thank you.

9 BY MR. BOBROW:

10 Q. Sir, you've been handed
11 Exhibit 21, which I understand is your
12 declaration in support of the patent owners'
13 response in the 662 proceeding.

14 Do you recognize it as such?

15 A. I do.

16 Q. We covered a lot of ground today
17 so it will take me a minute to flip because I
18 don't have questions about everything that I
19 flagged here.

20 A. I was looking at that.

21 Q. So let me ask you to turn to
22 page 5 of your declaration, Exhibit 21,
23 paragraph 12. In the first sentence of that
24 paragraph you refer to "controlled, stepwise
25 growth of a thin film on a heated surface" in

1 ALD deposition process.

2 Do you see what I'm referring
3 to?

4 A. I do, yes.

5 Q. And you underline the words
6 "controlled" and "stepwise."

7 Do you see that?

8 A. Yes.

9 Q. And when you're referring there to
10 controlled and stepwise growth of a thin
11 film, is that in any respect to depositing a
12 film in a self-limiting manner?

13 A. Yes.

14 Q. Take a look for me, if you would,
15 at paragraph 66 of your declaration, which is
16 on page 33.

17 A. Yes.

18 Q. Thank you. And in the second
19 sentence of that paragraph you wrote, "The
20 common definition of the organometallic
21 compounds requires that one or more of the
22 carbon atoms of a ligand is bonded directly
23 to the metal atom."

24 Do you see that where I'm
25 referring to?

1 A. Sure.

2 Q. What is the -- what source are you
3 relying upon for that definition?

4 A. My understanding of organometallic
5 chemistry.

6 Q. And is the definition you provide
7 there, you say that it's common.

8 Do you see that?

9 A. Yes.

10 Q. Is this a definition that you
11 teach your students, for example, at the
12 university?

13 A. Yes. I teach a course in
14 organometallic chemistry, yes.

15 Q. Have you seen these words or words
16 that are highly similar to these words appear
17 in textbooks or, you know, chemistry
18 handbooks to define what an organic --
19 sorry -- what an organometallic compound is?

20 A. Yes.

21 Q. Take a look, if you would, now,
22 at paragraph 115 of your declaration, which
23 appears at pages 54 and 55 of your
24 declaration, Exhibit 21. You refer there
25 to -- towards the bottom of that paragraph on

1 page 54 and the top of page 55 -- some
2 information about the purity of precursors
3 supplied by chemical companies.

4 Do you see what I'm referring to
5 there?

6 A. Yes.

7 Q. And you mentioned that a company
8 called Sigma-Aldrich has a technical grade of
9 trimethylaluminum and an electronic grade of
10 trimethylaluminum.

11 Am I interpreting your sentence
12 there correctly?

13 A. Yes.

14 Q. Now, with respect to those purity
15 levels, one says technical grades, one says
16 electronic grade.

17 What does technical grade mean?

18 A. I think I clarified, if you look
19 at the -- this is put in parentheses adjacent
20 to each one of those descriptors, the
21 indicated purity that Sigma-Aldrich, you
22 know, puts in their catalogue.

23 Q. Fair enough. I didn't ask a very
24 good question.

25 I actually meant when something is

1 called a technical grade, what does the
2 technical meaning in technical grade?

3 A. It is a phrase that has been
4 applied to indicate a general purity of
5 solvents, of chemicals, in general, for as
6 long as I've been in the field, and it's a
7 lower purity grade than something called
8 reagent grade, which is above that or
9 spectroscopic grade, which is above that.
10 When that practice first began I do not know.

11 Q. And is reagent grade a higher
12 grade than technical but lower than
13 electronic?

14 A. Yes.

15 Q. And spectroscopic, where does it
16 fall in the spectrum?

17 A. Spectroscopic is probably below
18 electronic grade.

19 Q. But above the other two?

20 A. But above the other two.

21 Q. Now, with respect even to the
22 electronic grade of the trimethylaluminum,
23 that electronic grade of trimethylaluminum
24 will have -- the trimethylaluminum has carbon
25 in it, right?

1 A. That's correct.

2 Q. Okay. So it's not as though the
3 electronic grade is stripping off carbons
4 from the trimethylaluminum molecule, right?

5 A. That's right.

6 Q. Okay. And that's true in the
7 technical grade as well, correct?

8 A. Yes.

9 Q. Okay.

10 MR. BOBROW: Just -- we're
11 planning to -- I think we can wrap up
12 soon.

13 MR. BOBROW: Here we go.
14 (Exhibit 22, Declaration of Wayne L.
15 Gladfelter, Ph.D. in Support of Patent
16 Owner's Response, Patent No. '016, marked
17 for identification.)

18 BY MR. BOBROW:

19 Q. Sir, you've been handed
20 Exhibit 22. This happens to be the
21 declaration that you submitted in support of
22 the patent owner's response in the 663 and
23 664 proceedings.

24 Is that what this is?

25 A. Yes.

1 Q. Let me ask you to turn to
2 paragraph 182 of your declaration,
3 Exhibit 22, which is on page 89.

4 A. Yes.

5 Q. And in that paragraph you make
6 reference to a paper by Min.

7 You see what I'm referring to
8 there?

9 A. Yes.

10 Q. Let me give you a copy of Min.
11 (Exhibit 23, Japanese Journal of Applied
12 Physics, Volume 37, marked for
13 identification.)

14 BY MR. BOBROW:

15 Q. Sir, you've been handed Exhibit 23
16 for identification. This is a cover page
17 from the Japanese Journal of Applied Physics,
18 along with an article by Min and others
19 called "Atomic Layer Deposition of Ti Nitride
20 films by alternate supply of
21 tetrakis(ethylmethyldamino)titanium and
22 ammonia." This is Exhibit 1007 in 662
23 proceedings, 1006 in the 663 proceedings, and
24 1006 in the 664 proceedings.

25 You're familiar with the Min

1 paper?

2 A. Yes, I am.

3 Q. Now, in this paragraph of your
4 declaration, Exhibit 22, you make reference
5 to Min at Abstract, 8.

6 Do you see where I'm referring
7 to?

8 A. Yes.

9 Q. And as I read paragraph 182,
10 you're saying that Min is seeking to find a
11 stable temperature with an ALD window.

12 You see what I'm referring to
13 there, in your declaration?

14 A. Oh, I'm sorry. In my declaration.

15 Q. Yes, you're saying that Min is
16 seeking to find a stable temperature, and
17 then you cite Min at abstract and then eight.

18 Do you see that?

19 A. I see that.

20 Q. And what I wanted you to do is
21 point out to me specifically where in the
22 abstract you believe that Min is stating that
23 Min is that seeking to find a stable
24 temperature within an ALD window?

25 A. Is the search right at the first

1 sentence where you're looking at. The
2 statement that atomic layer deposition of
3 amorphous titanium nitride on silicon dioxide
4 between 170 and 210 has been investigated
5 using the tetrakis(ethylmethyldamido)titanium,
6 that's the aspect that was, as you
7 understand -- as I understand this paper, it
8 was determined experimentally so.

9 Q. Okay. And what about at page 8 of
10 Min? What were you relying on at page 8
11 in -- to support your statements in paragraph
12 182 of your declaration, Exhibit 22?

13 A. This is the -- if you look at
14 Figure 3 on page 8, is a figure that shows
15 the temperature dependence of the film
16 thickness per cycle.

17 Q. And how does Figure 3 support your
18 opinion that Min is seeking to find a stable
19 temperature within an ALD window?

20 A. Looking at the results, you see
21 that there are three lines represented in
22 this particular graph. On the left-hand side
23 they're plotting the film thickness per
24 cycle, and on the right side they are
25 plotting the growth rates in nanometers per

1 minute.

2 The triangles, and you can see in
3 the -- in the figure caption the triangles
4 refer to the -- the filled triangles refer to
5 item C, which is conventional MOCVD using
6 TEMAT -- that's T-E-M-A-T -- plus ammonia,
7 that's a CVD process, and it's reporting the
8 growth rate as a -- at five different
9 temperatures.

10 B is also a CVD process, which is
11 reporting the growth rate at another set of
12 five temperatures, but this is without
13 ammonia. So those two are different in their
14 behavior.

15 And then the third line in Min
16 shows the process conducted in an ALD-type
17 approach that has a lot more data points in
18 it, and Min reports finding a temperature
19 window, an ALD window that corresponds to --
20 what is it? -- 170, I think, to three --
21 two -- it was a 230 I think. Yeah. Sorry,
22 210. 210 centigrade. That's -- the
23 temperature windows is that flat region where
24 the thickness per cycle does not change as
25 significantly as the temperature changes.

1 Q. It appears that there are five
2 data points in what Min is calling the ALD
3 window.

4 Do you read it that way?

5 A. That's what I see.

6 Q. So, in other words, the thickness
7 was measured in this ALD process at five
8 different temperatures?

9 A. That's -- yeah, that's how I
10 interpret that.

11 Q. And is there something in Figure 3
12 that is saying that Min wants to pick a
13 particular temperature within that window and
14 hold the temperature at that particular
15 temperature in the window.

16 A. I don't recall that he
17 specifically stated that he wanted to do a
18 deposition at any one of those. I think --
19 but he could have a particular reason to do
20 one that's slightly higher or slightly lower
21 that I don't remember.

22 Q. Now, in what we've marked as
23 Exhibits 21 and 22, that is, your
24 declarations, you formed opinions, I take it,
25 that the grounds that Micron has advanced in

1 these IPRs for anticipation and obviousness
2 do not render the claims anticipated or
3 obvious, correct?

4 A. Yes.

5 Q. All right. Now, in connection
6 with forming your opinions on obviousness,
7 did you have some kind of standard in mind as
8 to how certain a person of ordinary skill in
9 the art would need to be that a prior art
10 combination would work to perform a claimed
11 invention? Did you have some standard in
12 mind as to what that level of certainty is?

13 MR. LEN: Objection.

14 A. Could you clarify what you might
15 mean as a standard?

16 Q. What I mean by that is in your
17 view, did you -- let me ask it a little
18 differently.

19 When you came to the conclusion
20 the patents were not obvious to a person of
21 ordinary skill, did you apply, when you
22 looked at the proposed combinations of prior
23 art, some sort of standard as to how certain
24 or uncertain a person would need to be about
25 how likelihood -- how likely the combinations

1 would be successful?

2 MR. LEN: Objection.

3 A. Are you referring to the
4 combination of Min with --

5 Q. Min with Buchanan.

6 A. -- or Min with Sensaki.

7 Q. Min with Sensaki.

8 A. So not a specific one?

9 Q. Precisely. I'm lumping all of the
10 obviousness opinions together and asking
11 whether you had in mind a standard for how
12 certain a person of ordinary skill would have
13 to be in order to find that the combination
14 would render the inventions obvious.

15 A. I would -- I did look at what the
16 individual goals for the -- the individual
17 goal, for instance, for the work by Min or
18 any one of these, I looked at what they were
19 attempting to do. And I tried to address the
20 question whether that was the same goal, and
21 the -- in perhaps one of the other patents,
22 and whether it made sense -- sorry, that's a
23 very nebulous term -- but I'm not sure how to
24 quantify an opinion in much greater detail,
25 that it would be obvious to choose to combine

1 them.

2 Q. In looking at the obviousness
3 combinations, did you essentially, you know,
4 ask yourself whether, with respect to, you
5 know, reference A and reference B, whether I
6 could essentially physically incorporate the
7 teaching or disclosure of B into A? Is that
8 something that you did?

9 MR. LEN: Objection.

10 A. The -- I mean, that's what I
11 understand the combination to mean, that you
12 somehow are able to put these two together to
13 make it obvious that a result will occur.

14 Q. Got it. Now, in your view, in
15 assessing obviousness and anticipation, was
16 it your view that if a person of ordinary
17 skill would look at the reference or look at
18 the combination of references and need to do
19 an experiment in order to determine whether
20 the disclosure would work or the combination
21 would work, in your view, did that defeat the
22 anticipation or obviousness ground?

23 MR. LEN: Objection.

24 A. Well, if there was, as a result of
25 the combination to make a prediction whether

1 this was going to work or be useful, a
2 substantial amount of research, such that it
3 would involve a new -- the way I look at it,
4 as a research advisor as a new project, then
5 I would not concluded this to be a --
6 obvious.

7 Q. And tell me what you mean by a new
8 project as a research advisor.

9 A. A project that would lead to a new
10 product or a new idea or ultimately a
11 publication -- idea is probably not the best
12 word -- but publication, which is what --
13 it's kind of my currency as a faculty member
14 and perhaps discovery.

15 Q. And with respect to this idea of a
16 new publication, tell me what standard you
17 were applying as to whether something would
18 or wouldn't merit a publication?

19 MR. LEN: Objection.

20 A. So I understand what the field is,
21 and if there is new knowledge that comes out
22 of a set of experiments that would be
23 beneficial to others, that is what would
24 merit the publication.

25 Q. Now, with respect to your opinions

1 on the question of anticipation, in
2 expressing and formulating your opinions in
3 the declarations in these IPR proceedings,
4 was it your understanding that a prior artist
5 had to actually carry out or use the claimed
6 invention in the prior art in order for a
7 prior art reference to anticipate the claims
8 at issue?

9 MR. LEN: Objection.

10 A. So say this again.

11 Q. Sure.

12 A. Go ahead.

13 Q. With respect to your opinions on
14 the question of anticipation --

15 A. Yes. Yes.

16 Q. -- in expressing and formulating
17 your opinions in the declarations in the IPR
18 proceedings, was it your understanding that a
19 prior artist had to actually carry out or use
20 the claimed invention in the prior art in
21 order for the prior art reference to
22 anticipate claims at issue?

23 MR. LEN: Objection.

24 A. I'm going through my mind
25 searching for a -- specific examples of this,

1 and it's certainly the case -- it is, in my
2 view of the case, that when a patent includes
3 a specific example, it's easier to see, it
4 becomes more nebulous and a little bit more
5 difficult to clarify in broad terms if you're
6 extrapolating from an experiment that's known
7 to work to one that is not specified.

8 I'm sorry. I know specification
9 means something to you that I should be
10 cautious about that may not be part of a
11 specific example.

12 Q. Okay. And what I was trying to
13 drive at was whether you viewed that as being
14 a requirement for anticipation. In other
15 words, in evaluating whether a prior art
16 reference anticipates a later claimed
17 invention, did you view it as something that
18 was -- would be a requirement that, hey, the
19 prior art reference actually used or made the
20 claimed invention?

21 MR. LEN: Objection.

22 A. In the cases where the prior art
23 did demonstrate, it makes it so much easier.
24 The question that becomes more subject to
25 opinion is when there is a -- when you have

1 to extrapolate or look at a suggestion that
2 doesn't have experimental background
3 associated with it.

4 Q. And my question is: In performing
5 your work in the IPR proceedings --

6 A. Right.

7 Q. -- and expressing your opinions on
8 anticipation, did you say to yourself, in
9 substance, the prior art does not anticipate
10 the claims because the prior art didn't
11 actually carry out an experiment that made or
12 used the claimed invention?

13 MR. LEN: Objection.

14 A. Not in every case.

15 Q. And in some cases did you do that?

16 A. In some cases I -- if you go to an
17 example, I would be hard pressed. I'd have
18 to go back and look at the declaration to
19 individually proceed through it, but in some
20 cases, it was more characteristic of the
21 decision.

22 Q. And was that characteristic of the
23 decision, the case with respect to your
24 analysis of whether Buchanan anticipated the
25 claims of the '539 patent?

1 MR. LEN: Objection.

2 A. So the Buchanan patent teaches the
3 preparation of a precursor source mixture
4 that can be used in vapor deposition process,
5 in a known vapor deposition process, such as
6 a chemical vapor deposition or an atomic
7 layer deposition process.

8 So it teaches that, you know, that
9 it is useful to take these precursors, which
10 there are many, mix them with an inert
11 solvent and use that combination, the
12 so-called precursor source mixture, as an
13 effective way to deliver the precursor into
14 the reaction zone.

15 Q. The question I was driving at --
16 and I may not have asked the question well --
17 when you were looking at Buchanan and you
18 said to yourself, I don't think that Buchanan
19 anticipates the claims of the '539 patent,
20 when you came to that conclusion, did you say
21 to yourself, it doesn't anticipate because
22 Buchanan didn't actually carry out the
23 experiment, you know, of a metal dialkylamide
24 and water or alcohol, and therefore since
25 that isn't set forth it doesn't anticipate?

1 MR. LEN: Objection.

2 A. Well, the Buchanan patent does not
3 incorporate, I think, the critical elements
4 of the Gordon patents, which is to make use
5 of dialkylamide in an ALD process to deposit
6 the metal oxide film. That's the problem
7 with the -- with using the Buchanan patent.

8 Q. Right. And so was it the case
9 that you formed the opinion that because
10 Buchanan doesn't say that an experiment was
11 carried out between -- to do ALD using a
12 dialkylamide and water or alcohol, because it
13 doesn't say that and it wasn't done as an
14 experiment, that it can't anticipate?

15 MR. LEN: Objection.

16 A. No. It doesn't get close enough
17 in any of its descriptions and examples to
18 get there.

19 Q. Okay. I understand.

20 MR. BOBROW: Let me just, if you
21 would, give me a couple minutes to see
22 if I have anything else, and then I
23 think we'll be done.

24 (Recess taken at 5:48 p.m. to 5:55 p.m.)

25 MR. BOBROW: Let's go back on the

1 record.

2 MR. BOBROW: Dr. Gladfelter, thank
3 you for your time today. I don't have
4 any further questions for you at this
5 time.

6 MR. LEN: So if you could -- I
7 apologize, if you could bear with us
8 for, like, one minute, 30-second break,
9 so I can go off and confer with my
10 colleague, and we'll come back.

11 MR. BOBROW: I thought you had
12 done that.

13 MR. LEN: I didn't realize it was
14 going to be that short. If we do have a
15 question or two, it's going to be very,
16 very, short. So If you could bear with
17 us for, like, 60 seconds.

18 MR. BOBROW: Sure.

19 MR. LEN: Thank you. Go off the
20 record.

21 (Recess taken at 5:57 p.m. to 5:59 p.m.)

22 MR. LEN: Let's go back on the
23 record.

24 EXAMINATION

25 BY MR. LEN:

1 Q. Welcome back, Doctor. I just have
2 a couple -- couple short questions.

3 If I could direct your attention
4 to Exhibit 1 and Exhibit 19, which should be
5 the '539 and the '016 patents.

6 Do you have those in front of
7 you?

8 A. I do.

9 Q. Okay. If I can direct your
10 attention to the last page of Exhibit 1 and
11 Exhibit 19 for the claims.

12 A. Okay.

13 Q. Do you recall earlier this
14 afternoon counsel from Micron asked you
15 questions about the structures of the claims,
16 excluding Claim 31?

17 A. I do.

18 Q. If I can direct your attention to
19 Exhibit 19, the '016 patent, page 21, Column
20 30, Claim 2.

21 Do you see Claim 2?

22 A. Yes.

23 Q. Could you read for me Claim 2?

24 A. "The process as in Claim 1,
25 wherein the insulator insulates a gate or a

1 capacitor."

2 Q. What, if any, opinion do you have
3 about a structure of a gate or a capacitor?

4 A. A gate or a capacitor, these are
5 structures, multiple layers that are present
6 on a surface of typically a silicon substrate
7 of some sort. So they indeed will have a
8 topography other than flat.

9 MR. LEN: So thank you, Doctor.
10 Subject to Mr. Bobrow's recross, I don't
11 have any further questions for you at
12 the moment.

13 MR. BOBROW: I don't have any
14 questions.

15 MR. LEN: Jared, before we go off
16 the record, can I note one item for
17 housekeeping. We would like to note --
18 let's see here, get the right numbers.
19 We'd like to note that with respect to
20 the exhibits that were marked today,
21 Exhibit 3, Exhibit 15, Exhibit 17, and
22 Exhibit 20, that we'd like to raise the
23 objection that these are impermissible
24 introduction of new evidence that may
25 not be relevant to this case, and we

1 would like to reserve our right to move
2 to exclude if necessary.

3 MR. BOBROW: Understood. We --
4 obviously if you make that motion, we
5 would think, based upon what we know and
6 what you've said, that it would not be
7 well taken, and we would oppose the
8 motion, but that seems as if it's for
9 another day.

10 MR. LEN: Just putting you on
11 notice. Thank you.

12 (Deposition concluded at 6:03 p.m.)
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1 COMMONWEALTH OF MASSACHUSETTS

2 SUFFOLK SS:

3

4 I, Sandra A. Deschaine, Registered
5 Professional Reporter, Certified LiveNote
6 Reporter and Notary Public (credentials)
7 within and for the Commonwealth of
8 Massachusetts at large, do hereby certify:

6

7 That Wayne L. Gladfelter, Ph.D., the
8 witness whose deposition is hereinbefore set
9 forth, was duly sworn by me and that such
10 deposition is a true record of the testimony
11 given by the witness.

10

11 Before completion of the deposition,
12 review of the transcript was requested; If
13 requested, any changes made by the deponent
14 during the period allowed are appended
15 hereto.

13

14 I further certify that I am not related
15 to any of the parties to this action by blood
16 or marriage, and that I am in no way
17 interested in the outcome of this matter.

16

17 IN WITNESS WHEREOF, I have hereunto set
18 my hand this 16th day of January 2018.

18

19



20

21 Sandra A. Deschaine
22 Registered Professional Reporter

22

23

24 My Commission Expires
25 July 5, 2024

25

1 SIGNATURE PAGE
2 MICRON TECHNOLOGY, INC., vs. PRESIDENT &
3 FELLOWS OF HARVARD COLLEGE
4 WAYNE L. GLADFELTER, PH.D.
5

6 I the undersigned, declare under
7 penalty of perjury that I have read the
8 foregoing transcript, and I have made any
9 corrections, additions or deletions that I
10 was desirous of making; that the foregoing is
11 a true and correct transcript of my testimony
12 contained therein.
13

14 Executed this _____ day of
15 _____,
16
17 at _____, _____.
18 (CITY) (STATE)
19

20 -----
21 WAYNE L. GLADFELTER, PH.D.
22
23
24
25

1 ERRATA SHEET
2 MICRON TECHNOLOGY, INC., vs. PRESIDENT &
3 FELLOWS OF HARVARD COLLEGE
4 WAYNE L. GLADFELTER, PH.D.
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