

THIS OPINION WAS NOT WRITTEN FOR PUBLICATION

The opinion in support of the decision being entered today (1) was not written for publication in a law journal and (2) is not binding precedent of the Board.

Paper No. 13

UNITED STATES PATENT AND TRADEMARK OFFICE

BEFORE THE BOARD OF PATENT APPEALS
AND INTERFERENCES

Ex parte REN XU and KUEIR-WEEI CHOUR

Appeal No. 96-3649
Application 08/344,509¹

ON BRIEF

Before KIMLIN, JOHN D. SMITH and WEIFFENBACH, Administrative Patent Judges.

JOHN D. SMITH, Administrative Patent Judge.

DECISION ON APPEAL

This is an appeal pursuant to 35 USC § 134 from the final rejection of claims 1 through 29.

Claim 1 is representative and is reproduced below:

1. A method for the chemical vapor deposition of a heterometaloxide film, comprising:

selecting a volatile precursor which contains

¹ Application for patent filed November 23, 1994.

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metal constituents in a stoichiometric ratio desired for said film;

vaporizing said precursor at a selected rate;

hydrolyzing a portion of said precursor in the vapor state to form a hydrolyzed precursor; and

reacting said hydrolyzed precursor on a substrate to form a film containing said metal constituents in said stoichiometric ratio.

The references of record relied upon by the examiner are:

Rothschild et al. (Rothschild)	4,888,203	Dec. 19, 1989
Purdy	H1170	Apr. 6, 1993

Sladek et al. (Sladek), "Low Temperature Metal Oxide Deposition by Alkoxide Hydrolysis", Journal article announcement No. GRAI7301, published in Proceedings of the International Conference on Chemical Vapor Deposition (3rd), Salt Lake City, Utah, April 24-27, 1972, pages 215-231.²

Claims 1 through 6 and 23 through 29 stand rejected under 35 USC § 103 over Sladek in view of Purdy. Additionally, appealed claims 7 through 22 stand similarly rejected under the same section of the statute further in view of Rothschild.

We affirm.

The subject matter on appeal is directed to a method for the chemical vapor deposition of a heterometaloxide film (e.g., a

² Only an abstract of this publication is physically present in the record.

double metal oxide) comprising the steps of selecting and vaporizing a volatile precursor (e.g., a double metal alkoxide) which contains metal constituents in a stoichiometric ratio desired for the ultimate deposited oxide film. Appellants' method employs the step of hydrolyzing the volatile precursor in the vapor state to form a hydrolyzed precursor followed by reacting the hydrolyzed precursor on a substrate to form a film containing the metal constituents in the desired stoichiometric ratio, i.e., the stoichiometric ratio provided by the originally selected volatile precursor.

In their "Background of the Invention" section of the specification at page 3, appellants indicate that metal oxide chemical vapor deposition (MOCVD) of multicomponent oxide films has successfully involved the use of separate metalorganic compounds containing the respective film components as precursors. As stated in this portion of the specification, the precursor compounds are ordinarily independently transformed to the vapor phase by evaporation or other means in a controlled manner to achieve the desired molar ratio between vapor phase species. This approach is said to inevitably require a trial-and-error approach to achieve a precisely stoichiometric film composition. At the top of page 7 of the specification,

appellants explain that when MOCVD is applied using double metal alkoxide precursors, useful films are not produced. Appellants explain that for all known double metal alkoxides, premature decomposition producing individual metal oxides occurs at temperatures much lower than the pyrolytic temperature of each individual metal alkoxide. Thus standard chemical vapor deposition pyrolysis reactions do not produce stoichiometric oxide films because the M' to M'' ratio is lost prior to film deposition. Appellants' invention overcomes this problem by using a vapor phase hydrolysis chemical vapor deposition technique. Appellants contend that prior to their invention, it was not known how to apply vapor deposition technology, notably vapor phase hydrolysis, to double metal alkoxide precursors to produce a film having the same stoichiometric ratio of metals as exist in the precursor. See the specification at page 4, lines 16 through 19.

As evidence of obviousness of the claimed invention, the examiner principally relies on Sladek and Purdy. Appellants acknowledge (Brief, page 4) that although the combination proposed (i.e., the use of a stoichiometric double metal alkoxide precursor as described by Purdy in Sladek's chemical vapor deposition process) might be expected to produce an oxide film,

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?it is not adduceable from the references that such a film would be a stoichiometric oxide film.? Thus appellants contend that neither reference contains any suggestion that a stoichiometric double metal oxide film is achievable, much less how one would produce such a film. More specifically, appellants contend that Sladek is specifically directed to processes and products ?in which stoichiometry is not a consideration.? We do not agree this interpretation of Sladek, however.

Initially, we point out that our review of Sladek is hampered because the examiner has provided only the abstract of this multipage publication for review. Nevertheless, the Sladek abstract indicates that films of a family of metal oxide materials were produced by passing water and alkoxide vapors in nitrogen over a substrate in an isothermal rectangular reactor. More specifically, Sladek indicates that films of Al_2O_3 , Nb_2O_5 , Sb_2O_3 , TiO_2 , and ZrO_2 were obtained as amorphous films which were annealed at high temperatures to cause crystallization with increases in the refractive index. Clearly, the above films are stoichiometric films i.e, chemical compounds having definite proportions of the respective elements. Moreover, that stoichiometry is a consideration of importance to Sladek is implied by Sladek's teaching that films described as VO_x and BO_x

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(films having undefined stoichiometry) were not further tested.

We also do not agree with appellants' argument that the references provide no suggestion as to how to produce a stoichiometric double metal oxide film. Admittedly, Sladek only describes the successful formation of single metal oxide films by using a vapor phase hydrolysis chemical vapor deposition technique. Importantly, however, Sladek indicates that the films were all obtained at low temperatures, i.e., from 25 to 130°C. Remembering that the problem of using a known double metal alkoxide in a standard MOCVD process is the premature thermal decomposition of the alkoxide into the respective individual metal alkoxide components because of the high temperature conditions required for pyrolysis (specification, page 7), one of ordinary skill in the art would have been led to utilize such a double metal alkoxide precursor in the low temperature vapor phase hydrolysis chemical vapor deposition technique of Sladek with a reasonable expectation of producing a stoichiometric double metal oxide film. Based on the above analysis, it is our view that a prima facie case of obviousness has been established for the subject matter defined by independent claim 1 and those claims dependent thereon.

Appellants have asked for separate consideration of several

groups of claims as set forth in the Brief at page 3. With the exception of appealed claim 12, we think that a prima facie case of obviousness has been established for the subject matter defined by these claims. Appealed claim 12, which is separately argued, requires control of the substrate temperature to within the range of about 400°C to about 800°C. Sladek's disclosure that a family of metal oxides CVD reactions were successfully achieved using vapor phase hydrolysis chemical vapor deposition techniques at temperatures from 25 to 130°C, in our view, is insufficient to provide a reasonable expectation that at the higher temperatures claimed, stoichiometric double metal oxide films would have been achieved. Thus, we reverse the examiner's rejection as it applies to dependent claim 12.

With respect to independent method claim 7, appellants argue that the prior art fails to suggest structure strategically positioned, and arranged to promote a polycondensation reaction of a hydrolyzed precursor on a substrate. Apparently, appellants are referring to the claim language defining a structure positioned within a reaction chamber ?in the proximity of said reaction zone, said structure being constructed and arranged to promote a polycondensation reaction of said hydrolyzed precursor on the substrate?. Based on Figure 1 of the application, this

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claim language apparently refers to graphite susceptor 52. However, Sladek's teaching of the vapor phase hydrolysis of a metal alkoxide by passing water and alkoxide vapors and nitrogen over a substrate in a isothermal rectangular reactor at least implies the presence of a substrate heating means structure for carrying out a hydrolysis/condensation reaction necessary to produce a stoichiometric family of metal oxide films.

Appellants contend that appealed claims 6, 7, 18, 23 and 29 distinguish over the prior art because they require an annealing step which differs from the annealing procedure described in the references. With respect to this argument, as the examiner has clearly pointed out, Sladek teaches air annealing at 350 to 1000°C which causes crystallization of an amorphous film. How appellants' claimed annealing step differs from the prior art annealing is not apparent. Thus, we think these claims are also properly rejected. Likewise, although appellants argue that appealed claims 18 and 23 require the formation of a "perfect" crystal structure, appellants have not addressed the disclosure in Sladek that the air annealing step, which caused crystallization, produced anything other than perfect crystalline films. Finally, while appellants indicate that none of the references

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suggests the vapor deposition of a stoichiometric hetero-metaloxide film by subjecting a vaporized precursor to an S_N2 reaction as in appealed claims 24 through 29 (appellants apparently refer to the reaction mechanism referred to as S_N2 and known as substitution nucleophilic bimolecular mechanism)³, it would appear that this mechanism would have been expected by a person of ordinary skill in the art for the hydrolysis reaction in question based on the structure of the known double metal alkoxides. Compare the specification at page 5, lines 7 through 15.

In passing, we observe that the thrust of many of the arguments in appellants' Brief and indeed the disclosure in appellants' specification is that prior art workers have not known how to apply vapor deposition technology, notably vapor phase hydrolysis, to double alkoxide precursors to produce large crystals containing the same stoichiometric ratio of metals that exist in the precursor. In the event of any subsequent prosecution of this application, the examiner may wish to consider the propriety of a rejection of the appealed claims under 35 USC § 112, first paragraph, *enablement requirement*,

³ See Organic Chemistry, Morrison and Boyd, pages 370-375, 1959, copy attached.

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particularly for the broadly drafted claims that are now present in this appeal.

The examiner's rejection of claim 12 is reversed. The rejection of all other claims is affirmed. Accordingly, the decision of the examiner is affirmed-in-part.

No time period for taking any subsequent action in connection with this appeal may be extended under 37 CFR § 1.136(a).

AFFIRMED-IN-PART

EDWARD C. KIMLIN)
Administrative Patent Judge)
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) BOARD OF PATENT
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Administrative Patent Judge) APPEALS AND

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