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**Datasheet for the decision
of 15 June 2010**

Case Number: T 1246/08 - 3.2.08

Application Number: 99962850.6

Publication Number: 1137820

IPC: C22B 34/24

Language of the proceedings: EN

Title of invention:

High purity tantalum and products containing the same like sputter targets

Patentee:

CABOT CORPORATION

Opponents:

OI H.C. Starck GmbH
OII Praxair, Inc.
OIV Plansee SE

Headword:

-

Relevant legal provisions:

-

Relevant legal provisions (EPC 1973):

EPC Art. 56

Keyword:

"Inventive step (yes - after amendments)"

Decisions cited:

-

Catchword:

-

Case Number: T 1246/08 - 3.2.08

DECISION
of the Technical Board of Appeal 3.2.08
of 15 June 2010

Appellant I:
(Opponent OI)

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Appellant II:
(Opponent OII)

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Appellant III:
(Patent Proprietor)

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Respondent:
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Decision under appeal: Interlocutory decision of the Opposition
Division of the European Patent Office posted
29 April 2008 concerning maintenance of the
European patent No. 1137820 in amended form.

Composition of the Board:

Chairman: T. Kriner
Members: R. Ries
A. Pignatelli

Summary of Facts and Submissions

I. Oppositions were filed against European patent No. 1 137 820 as a whole by the present appellants OI (H. C. Starck GmbH) and OII (Praxair Inc.) and further by opponent OIV (Plansee AG). The oppositions were based on Article 100 (a) EPC (lack of novelty and lack of inventive step).

In its interlocutory decision dispatched on 29 April 2008, the opposition division held that the subject matter of the claims according to the first auxiliary request then on file met the requirements of the EPC and that the patent could be maintained in amended form on the basis of this request.

II. Opponents OI and OII lodged an appeal against this decision on 30 June 2008 and on 1 July 2008, respectively. The appeal fees were paid on the same dates. The statements setting out the grounds of appeal by opponents OI and OII were both received on 9 September 2008.

An appeal was also lodged against this decision by the patent proprietor on 1 July 2009, and the appeal fee was paid on the same date. The statement setting out the grounds of appeal was received on 3 September 2008.

III. For the present decision, the following documents have played a major role:

D8: JP-A-01-290766 and

D8a: Translation into English of D8;

D9: C. Pokross: "Controlling the Texture of Tantalum Plate", Journal of Metals, October 1989, pages 46 to 49;

D11a:WO-A-87/07650 (translation into English language).

IV. Oral proceedings were held before the Board on 15 June 2010.

Appellants I and II (OI and (OII) requested that the decision under appeal be set aside and the patent be revoked.

Appellant III (the patent proprietor) requested that the decision under appeal be set aside and the patent be maintained on the basis of the set of claims filed as the main request at the oral proceedings.

V. Independent claims 1 and 15 of the main request read as follows:

"1. A sputtering target consisting of tantalum metal obtainable from a tantalum ingot by thermomechanical processing, said tantalum metal having a purity of at least 99.995%, and an average grain size of 75 •m (microns) or less, wherein said metal has

- a) a texture in which a (100) pole figure has a center peak intensity within any 5% incremental thickness of 0 to 5 random, and
- b) a natural log (Ln) ratio of (111):(100) center peak intensities within the same increment of -1.5 to 7 or -3 to 5."

"15. A process of making a sputtering target from tantalum metal having a purity of at least 99.995%, comprising:

- a) mechanically or chemically cleaning the surfaces of the tantalum metal, wherein the tantalum metal has a sufficient starting cross-sectional area to permit steps (b) through (g);
- b) flat forging the tantalum metal into at least one rolling slab, wherein the at least one rolling slab has sufficient deformation to achieve substantially uniform recrystallization after annealing in step (d);
- c) mechanically or chemically cleaning the surfaces of the at least one rolling slab;
- d) annealing the at least one rolling slab at a sufficient temperature and for a sufficient time to achieve at least partial recrystallization of the at least one rolling slab;
- e) cold or warm rolling the at least one rolling slab in both the perpendicular and parallel directions to the axis of the starting tantalum metal to form at least one plate;
- f) flattening the at least one plate; and
- g) annealing the at least one plate to have an average grain size equal to or less than 150 •m (microns) and a texture substantially void of (100) textural bands."

VI. The arguments of the appellants OI and OII can be summarized as follows:

Document D8a related to high purity tantalum targets of 5 to 6N (i.e. 99.999 to 99.9999 % purity; see D8a, page 3, "Prior art", 1st paragraph). However, D8a was

silent on the microstructure of the sputtering target, in particular the grain size and texture featuring in claim 1 of the patent at issue. On page 4, lines 6 to 12, D8a further pointed out that in order to form high quality films in a stable manner by sputtering, it was of utmost importance for the targets to meet the following requirements: high purity, uniformity and high density.

Starting from D8a as the closest prior art and looking for further information as to how these needs, in particular the high uniformity requirement could be satisfied, the skilled person would turn to document D9 which likewise related to a tantalum plate exhibiting a high purity of 99.994 or even better (see D9, page 47, first full paragraph). Specifically, document D9 disclosed e.g. in the paragraph "Introduction" that a texture of the (111) type yielded the finest and most uniform microstructure. The manufacturing schedules given in Figure 1 of D8 resulted - after cleaning and machining the surface of the sample to remove disturbing metal bands - in a grain size of 40 or 45 μm and the favourable (111) texture (see in particular Tables I to III and the paragraph on pages 47/48: Texture Analysis). Thus a skilled person, faced with the problem of improving the uniformity and in consequence thereof the sputtering efficiency of the Ta target given in D8a, would in an obvious manner consider document D9 to produce a favourable uniform (111) texture and fine grain size which both met features a) and b) of the Ta-target set out in claim 1.

Likewise, the combined teaching of documents D11a and D9 made the claimed sputtering target obvious. D11a

disclosed a Ta target having a purity higher than 99.999% Ta that was prepared by cold and hot isostatic pressing, electron beam melting and machining, including deformation processing, cutting and surface finishing (see D11a, pages 3/4, steps (d) to (f)). Nothing was said in D11a about the grain size and texture. So, for the same reasons as given above, the skilled person would apply the process disclosed in document D9 in order to produce a predominantly (111) texture and a fine grain size so as to improve the sputtering efficiency.

The process features of making a sputtering target from tantalum according to claim 15 were also obvious for the same reasons. As to put into practice the deformation processing step for producing a high purity Ta-sputtering target referred to in D11a, page 7, paragraph "Machining", the skilled person would consider the process which was described in detail in document D9 including ingot breakdown by upset forging, rolling, annealing at 1010°C/2h, cold rolling twice perpendicular (78% reduction) and parallel (46% reduction) to the ingot centre line and annealing the rolled product to achieve a (111) texture with an average grain size of less than 150 μm (see D9, e.g. Figure 1; Tables 1 and 2).

The subject matter of independent claims 1 and 15 therefore lacked an inventive step.

VII. The arguments of appellant III (patent proprietor) can be summarized as follows:

Document D8a taught the skilled person that for sputtering targets a high purity, uniformity and high density were important to form high quality films in a stable manner. Contrary to melting an ingot as claimed in the patent, the tantalum sputtering targets of D8a were prepared strictly by the powder metallurgical (PM) route by cold and hot isostatic pressing. The requirements of high density and uniformity thus related to the (PM) derived body and not to a cast and forged ingot which was further rolled down to bands of the desired thickness as disclosed by the process depicted in Figure 1 of document D9. Given that D9 failed to mention any specific use at all and was not concerned with sputtering or that the (111) texture and grain size of the rolled tantalum material could bring about any benefit with respect to the sputtering efficiency, the skilled person had no reason whatsoever to turn to this document.

The same reasoning applied to document D11a which disclosed melting a Ta-ingot which was further reduced by deformation processing, cutting and cleaning. Given the missing link to sputtering in D9 referred to above, the skilled person aiming at improving the sputtering efficiency of a Ta target had no motivation to consider this document. The same line of argument was also true for the process steps featuring in claim 15.

The subject matter of independent claim 1 and claim 15 therefore involved an inventive step.

Reasons for the Decision

1. The appeal is admissible.

2. *Article 123(2) EPC and novelty (Article 54 EPC):*

At the oral proceedings, appellants I and II had no objections under Article 123(2) EPC to the amended claims according to the main request, and the novelty of the subject matter of independent claims 1 and 15 was not questioned. The Board does not see any reason why this evaluation should be put into doubt.

3. *Inventive step:*

3.1 Like the patent at issue, document D11a is concerned with a high purity tantalum target and a method of its manufacture. To this end, tantalum powder having a purity of 99.999% or higher is formed by pressure moulding, sintered, repeatedly melted by electron beam melting and machined including deformation processing and cutting (see D11a, pages 3/4, points (d) to (f); page 6, last line to page 7, paragraph "VI Machining"; page 8, last paragraph; page 10, first paragraph). Therefore, document D11a qualifies as the closest prior art. However, D11a fails to give any information about the microstructure including the texture and grain size of the Ta sputtering target.

Starting from the disclosure of document D11a, the problem underlying the patent at issue resides in increasing the sputtering efficiency i.e. in providing a greater sputtering rate and an improved uniformity of the thickness of the sputtered deposited film (see the

patent at issue, paragraph [0004]). The solution to this problem is achieved by a specific grain size and by the features a) and b) of claim 1. Specifically, the claimed sputtering Ta target exhibits a fine average grain size of 75 μ m or less and a predominantly uniform (111) texture wherein the centre peak intensity of the undesired (100) structure within any 5% incremental thickness is between 0 and 5 random. This structure of the Ta target which is substantially devoid of the (100) texture leads to a very uniform sputtering erosion and to a uniform sputtered film as well (see the patent specification, paragraph [0023]).

The arguments of appellants OI and OII that document D9 related to a high purity Ta material which exhibited after rolling and annealing a substantially (111) texture and a grain size of about 45 μ m as shown in Tables 1 and 2 is not disputed. However, the problem of how to improve the sputtering efficiency when using a high purity Ta target is not addressed at all in document D9, and a suggestion does not even remotely exist in this document that the provision of a uniform (111) texture and a specific grain size could be helpful to solve it. Document D9 states on page 47, ("Texture Analysis") that the texture can be different at the rolled surface or near surface and, therefore, should be measured at least 13 mm from the rolled face. This leads one to conclude that the texture was measured only once somewhere in the middle of the sample. There is no proof whatsoever to be found anywhere in document D9 that the measured (111) texture actually exists uniformly over the whole thickness of the sample and fulfils the requirements of conditions a)

and b) within any 5% incremental thickness as set out in claim 1 of the patent.

Given this situation there is no reason for a skilled person facing the identified problem to pick features from document D9 to associate with the teaching of document D11a, and even if this were done, the subject matter of claim 1 would not be arrived at.

A similar situation exists with respect to the combination of the teaching of documents D8a and D9. The high purity Ta target according to D8a was produced by the (PM) route followed by hot isostatic pressing (HIPing) and machining to achieve a uniform high-density product. Baring in mind the problems associated with (PM) processing, the statement on page 4, second paragraph that the sputtering target should exhibit uniformity and a high density, has to be understood. It means to the expert reader that the pressed and sintered Ta product should be free of pores and voids and be uniform ("isotropic") in its microstructure. Put the other way, this statement would mean that a specific texture rendering the material in its properties "anisotropic" is not desired or aimed at. Like D11a, document D8a is far from giving any suggestion towards the problem solved by the patent in suit. Applying the same reasoning as given in connection with D11a, the skilled person was not prompted to start from the technical teaching of document D8a and to combine it with that of document D9, and even if nevertheless effected, the combined teaching would not lead in an obvious way to the sputtering target defined in claim 1 of the patent.

Hence the subject matter of claim 1 involves an inventive step.

3.2 As to the process of producing the high purity sputtering target set out in claim 15, the teaching of document D8a is far from giving any incentive to deform an ingot by forging and rolling as required by the claimed process, since this document is concerned with a (PM) product which is HIPed, heat treated and finished to a Ta sintered target through machining and surface polishing.

Although document D11a even considers the deformation and machining of a high purity Ta ingot, the skilled person has no reason to take into account the process disclosed in document D9 because this document fails to give any suggestion towards the problem to be solved by the claimed process. Reference is made in this context to the detailed reasoning given in paragraph 3.1.

Given this situation, the subject matter of claim 15 also involves an inventive step.

The dependent claims 2 to 14 and 16 to 19 relate to preferred embodiments of the sputtering target according to claim 1 and the process set out in claim 15, respectively. Therefore, these claims are also allowable.

Order

For these reasons it is decided that:

1. The decision under appeal is set aside.

2. The case is remitted to the first instance with the order to maintain the patent with claims 1 to 19 according to the main request filed during the oral proceedings, a description and drawings to be adapted.

The Registrar:

The Chairman:

V. Commare

T. Kriner