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Datasheet for the decision of 26 June 2012

Case Number: T 1518/10 - 3.2.03

Application Number: 01934455.5

Publication Number: 1291100

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Language of the proceedings: EN

Title of invention:
Niobium or tantalum powder and method for production thereof

Patentee:
Cabot Supermetals K.K.

Opponent:
H.C. Starck GmbH

Headword:
-

Relevant legal provisions:
EPC Art. 123(2), 54

Relevant legal provisions (EPC 1973):
-

Keyword:
"Amendments - added subject-matter (no)"
"Novelty (yes)"

Decisions cited:
-

Catchword:
-

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Beschwerdekammern | Boards of Appeal | Chambres de recours

Case Number: T 1518/10 - 3.2.03

D E C I S I O N
of the Technical Board of Appeal 3.2.03
of 26 June 2012

Appellant: (Patent Proprietor)
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Decision under appeal:
Decision of the Opposition Division of the European Patent Office posted 3 May 2010 revoking European patent No. 1291100 pursuant to Article 101(3)(b) EPC.

Composition of the Board:

Chairman: U. Krause
Members: G. Ashley
K. Garnett

Summary of Facts and Submissions

I. Grant of European patent EP-B1-1 291 100 was opposed on the grounds of lack of novelty and inventive step (Article 100(a) EPC), insufficient disclosure (Article 100(b) EPC) and added subject-matter (Article 100(c) EPC).

II. The opposition division considered that claims of the main and auxiliary requests either did not meet the requirements of Article 123(2), Article 84 EPC, were not sufficiently disclosed or lacked novelty. Hence the opposition division decided to revoke the patent. The decision was posted on 3 May 2010.

III. The appellant (the patent proprietor) filed notice of appeal on 9 July 2010, paying the appeal fee on the same day. A statement containing the grounds of appeal was filed on 13 September 2010.

IV. In a letter dated 21 January 2011 the opponent stated that it withdrew the opposition and would not be filing a response to the grounds of appeal.

V. Requests

The appellant requests that the above decision be set aside, and that the case be remitted to the opposition division for further prosecution on the basis of the sets of claims filed with the grounds of appeal as the main or first to third auxiliary requests.

Oral proceedings are requested should the Board be considering an adverse decision.

VI. Claims

(a) Claim 1 of the main request is as follows

"1. A tantalum powder comprising aggregates in which primary particles of tantalum are aggregated, and having a pore distribution having a peak in the range from 1 to 20 μm and a peak in the range 0.05 to 0.3 μm as measured by mercury porosimetry."

Independent claim 4 is directed to a production method:

"4. A production method of a niobium or tantalum powder having a pore distribution having a peak in the range from 1 to 20 μm as measured by mercury porosimetry comprising the steps of:

(1) adding a pore forming material which is removable from niobium powder or tantalum powder by decomposition or sublimation by heat and which is at least one of a particle having a diameter in a range from 2 to 6 μm, a film or foil having a thickness in a range from 1 to 20 μm, and a fiber having a diameter in a range from 1 to 20 μm and a length in a range from 100 μm to 1 cm to primary particles of niobium and tantalum, wherein the adding amount of pore forming material relative to the niobium or tantalum powder is 3% by volume or greater; and adding an amount of water such that the whole powder is uniformly wet while the powder is vibrated; and

(2) heat treating and thereby removing the pore forming material and forming aggregates."

Independent claim 5 also concerns a production method:

"5. A production method of a niobium or tantalum powder having a pore distribution having a peak in the range from 1 to 20 μm as measured by mercury porosimetry comprising the steps of:

(1) adding a pore forming material having an acid solubility and being at least one of a particle having a diameter in a range from a range from 2 to 6 μm, a film or foil having a thickness in a range from 1 to 20 μm, and a fiber having a diameter in a range from 1 to 20 μm and a length in a range from 100 μm to 1 cm to primary particles of niobium and tantalum and wherein the adding amount of pore forming material relative to the niobium or tantalum powder is 3% by volume or greater; and adding an amount of water such that the whole powder is uniformly wet while the powder is vibrated; and

(2) heat treating and acid treating and thereby removing the pore forming material and forming aggregates."

Dependent claims 2, 3 and 9 concern preferred embodiments of the powder of claim 1. Dependent claims 6 and 8 relate to preferred embodiments of production method 4, and dependent claim 7 relates to a preferred embodiment of the method of claim 5.

(b) Claim 1 of the application as originally filed (published as EP-A1-1 291 100) is as follows:

"1. A niobium or tantalum powder comprising aggregates in which primary particles of niobium or tantalum are aggregated, and having a pore distribution having a peak in the range 1 to 20 µm as measured by mercury porosimetry."

VII. Cited Prior Art and Relevant Documents

The following documents, amongst others, were referred to in the contested decision:

D1a: WO-A-00/67936
(cited as prior art under Article 54(3) EPC)
D1b: DE-A-198 31 280
D5: DE-A-31 30 392
D6: English translation of Amosov et al.: "Production of porous tantalum anodes of electrolytic condensers", Poroshkovaya Metallurgie, No.1 (109), pages 53 to 59, January 1972;
D7: JP-A-08-031700
D8, D8a: Versuchsbericht 1, submitted by the opponent;
D13: Versuchsbericht for example 2 of D5, submitted by the opponent;
D14: Versuchsbericht (continued) for example 2 of D5, submitted by the opponent.
D16: US-B2-7 066 979

VIII. Submissions of the Appellant

The submissions of the appellant can be summarised as follows.

(a) Article 123(2) EPC

The appellant submitted amended claims which address the objections set out in the contested decision. In particular, the product claims have been restricted to tantalum powders.

(b) Novelty

The appellant submitted that the tantalum powder of claim 1 is novel over D1a.

D1a does not explicitly mention the pore distribution defined in claim 1, but the opponent had submitted test results (D8) showing that examples 2 and 3 of D1a lead to tantalum powders having the characteristics of claim 1. The appellant alleged that these powders could not have been derived from the teachings of D1a alone, and that the opponent was able to carry out the tests only because it was the author of D1a and in possession of additional information. In particular, D1a does not disclose the morphology of the tantalum pentoxide starting materials, and this is essential information for producing the desired powders, as evidenced by D16.

Since D1a does not provide an enabling disclosure for the examples 2 and 3, the appellant submits powders of these examples do not belong to the prior art.

Reasons for the Decision

1. The appeal is admissible.

Main Request

2. Article 123(2) EPC

2.1 Claim 1

2.1.1 Whereas claim 1 before the opposition division referred to niobium or tantalum powders, the present claim 1 has been limited to a tantalum powder, so there is no objection under Article 123(2) EPC regarding this amendment.

2.1.2 During the opposition proceedings granted claim 1 was also amended to define the pore distribution as having a second peak in the range from 0.05 to 2 µm. The opposition considered that this range related to the size of vacancies in general without referring specifically to a peak, hence concluded that the amendment did not meet the requirements of Article 123(2) EPC.

Claim 1 of the main request before the Board has now been amended to define the pore distribution as having a second peak in the range 0.05 to 0.3 µm. This feature can be derived from paragraph [0028] of the published application. Here it is said that the peak in the distribution of vacancies (4) between particles is in the range 0.05 to 0.3 µm (peak A) and, in the case of the powder of the invention, there is a further peak (B)

due to the pores (3) between the aggregates, which is in the range 1 to 20 µm.

2.2 Claim 4

Independent claim 4 concerns a production method for making either tantalum or niobium powders, and is based mainly on original claims 4, 9 and 11.

Claim 4 also requires that the pore forming material is removable by decomposition or sublimation by heat. This feature is disclosed in paragraph [0032] of the application, and is presumably in response to the finding of the opposition division that there was no disclosure of a pore forming material removable by "heat treatment", this being a more general expression than those disclosed (paragraph 3.3 on page 6 of the disputed decision). The feature that water is added while the powder is vibrated is disclosed in paragraph [0037] of the application.

2.3 Claim 5

Independent claim 5 concerns a production method in which the pore material has acid solubility, and is based on claims 5, 10 and 12, and on paragraph [0037] of the application.

2.4 Claim 9

Claim 9 is new and concerns a preferred embodiment of the powder of claim 1 in which the diameter of the aggregates is defined. Support for this amendment can be found in paragraph [0029] of the application.

2.5 Consequently the requirements of Article 123(2) EPC are met for the claims of the main request.

3. Sufficiency of Disclosure

The opposition division considered that it was not possible to obtain the desired product over the whole ambit of the claimed method of the main and auxiliary requests 1, 3 and 4. These method claims defined the shape of the pore forming material.

The present method claim 4 has been amended to define in addition the size of the pore forming material. Similar method claims before the opposition division were considered to be sufficiently disclosed (see page 7, point 4 of the contested decision and auxiliary requests 2, 5, 6, 7), and the Board does not see any reason to depart from this view.

4. Novelty (Article 54 EPC)

Claim 1 - Tantalum Powder

4.1 The opposition division considered that the powder of claim 1 of the eighth auxiliary request, which defined a tantalum or niobium powder having the two pore distribution peaks of the present claim 1, to lack novelty with respect to D1a (cited as prior art under Article 54(3) EPC).

4.2 D1a does not explicitly mention the pore distribution defined in claim 1. However, the opponent submitted

test results (D8) showing that examples 2 and 3 lead to tantalum powders having the characteristics of claim 1.

4.3 The appellant alleges that D1a does not provide an enabling disclosure for the examples 2 and 3, and the powders of these examples do not belong to the prior art. In particular, D1a does not disclose the morphology of the tantalum pentoxide starting materials, and this is essential information for producing the desired powders.

4.3.1 In support of this argument, the appellant refers to Examples 2, 3 and 4 of D1a. Example 2 on page 23 describes a method for converting tantalum pentoxide into tantalum powder having a surface area of 37,600 cm^2/g. The tantalum powders of Examples 3 and 4 (page 25, lines 17 to 18 and page 26, lines 9 to 10) were also made according to the same procedure of Example 2, but have different surface areas of 57,000 cm^2/g and 133,000 cm^2/g respectively. It is therefore apparent that the surface area of the resulting powder does not arise just from the method steps given in Example 2.

4.3.2 The appellant also filed D16, which was published after the priority date of the disputed patent, but is not submitted as evidence of prior art or common knowledge at the filing date, but of the fact that there exists a relationship between a starting powder and the end product. D16 relates to a process for producing valve metal powders (tantalum or niobium) by reducing their oxides, and explains that there is a direct relationship between the morphology of the oxide used and the resulting valve metal powder (see column 2,

lines 16 to 18, column 3, lines 52 to 57 and column 4, lines 52 to 54).

4.3.3 Thus, it is apparent that the properties of tantalum powder are affected significantly by the nature of the tantalum pentoxide starting powder. In other words, without knowing the nature of the starting material it is not possible determine the properties of the end product.

4.3.4 The opposition division argued (point 2.1 on page 3 of the contested decision) that at page 12, lines 15 to 21 of D16, the final powder is said to be spherical, hence it is clear to the skilled person that spherical tantalum pentoxide powders should be used. In addition, it is stated at page 15, lines 21 to 26 of D16 that the pentoxides should be preferably 50 to 300 µm and 2 to 3 times bigger than the desired primary grain size of the final metal powder. The view of the opposition division was that skilled person is thus provided with sufficient information to select appropriate starting materials.

4.3.5 However, as argued by the appellant, Examples 2 and 3 of D16 do not give the primary grain size of the resulting tantalum powder, so it is not possible to choose the size of the starting material on this basis. It is also not certain that merely knowing the particle size of the starting material would inevitably result in a tantalum powder as defined in claim 1, as the following demonstrates.

4.3.6 Consider Example 16 (page 41), which provides details of the nature of the tantalum pentoxide powders by citing the following parameters:

- average particle diameter (FSSS);
- bulk density (Scott);
- specific surface area (BET);
- particle size distribution.

These seem to be the most important parameters for defining the starting powder, and are also identified as such in D16 (column 7, lines 25 to 35 and the examples). Proper characterisation of the starting powder thus requires more than merely knowing the particle size.

4.3.7 The opponent carried out the method of Example 16 (Test 1/9 of D8), so it is assumed that a tantalum pentoxide powder having the properties defined in Example 16 was used as the starting powder. A comparison of the properties of the resulting tantalum powder given in Example 16 and those obtained by Test 1/9 (see D8a) is as follows:

	BET m^2/g	Bulk Density (Scott) $g/inch^3$
Example 16	2.2	25.5
Test 1/9	2.4	27.4

Although not identical, the results seem to be within experimental scatter, as was argued by the opponent in the first instance proceedings. It is apparent from the results presented in D8 that, despite reproducing

Example 16, there is no porosity peak in the range 0.05 to 0.3 µm, as required by claim 1.

4.4 In summary, the properties of a tantalum powder depend on those of the starting material, and these are not sufficiently disclosed in D1a. The one example (Example 16) which does provide details of the starting powder does not result in the claimed tantalum powder. It cannot be said with any certainty that by following the teaching of D1a alone, a powder having the characteristics of claim 1 is produced. The subject-matter of claim 1 is thus novel over D1a.

4.5 Document D1b relates to niobium powders, hence is not relevant for assessing the novelty of claim 1.

Claim 4 - Method of Production

4.6 The opposition division was of the view that the method of claim 4 before the division lacked novelty with respect to D5, and in particular example 2 on page 12 of D5. The opponent reproduced example 2 and the results are presented in D13 and D14.

4.6.1 According to D13, 250 g tantalum powder was mixed with 2 wt% magnesium powder and then heated; the resulting MgO was washed out with sulphuric acid to give a porous product having pore distribution with a peak at about 3 µm (see D14). The method of present claim 4, which is an amended version of the claim considered by the opposition division, differs from the method disclosed in D5 in that:

- it requires a given particle shape and size, whereas that of the Mg powder of D5 is not known;
- the tantalum and magnesium powders are vibrated to uniformly wet the powder with water;
- the pore forming material is removed by heat treatment, whereas in D5 the magnesium is heated to form an oxide which is removed by acid.

4.6.2 The method of claim 4 is therefore novel over D5.

Claim 5 - Method of Production

4.7 The method of claim 5 differs from that of D5 in terms of the defined particle shape and size, and by vibrating in water (as above), and hence is also novel.

5. Remittal

5.1 Inventive step has not been considered by the opposition division, and the appellant requests that should the claims be found to be novel, the case be remitted to the opposition division for consideration of the question of inventive step.

5.2 The Board agrees that it is necessary to remit the case to the opposition division for further prosecution, including consideration of inventive step. Although the opponent has withdrawn its opposition, attention of the opposition division is drawn to Rule 84(2) EPC, which provides for continuation of opposition proceedings under such circumstances should the opposition division consider that any of the outstanding issues are prejudicial to the maintenance of the patent.

6. Oral Proceedings

Since claim 1 of the main request has been found to be novel and the case is to be remitted to the opposition division in accordance with the request of the appellant, it is not necessary to hold oral proceedings.

Order

For these reasons it is decided that:

1. The decision under appeal is set aside.

2. The case is remitted to the opposition division for further prosecution.

The Registrar: The Chairman:

A. Counillon U. Krause