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**Datasheet for the decision
of 8 April 2011**

Case Number: T 0370/09 - 3.4.01

Application Number: 98966979.1

Publication Number: 0953847

IPC: G01R 31/26

Language of the proceedings: EN

Title of invention:

Capacitor with an electrode composed of a sintered body of partially nitrided niobium powder

Patentee:

SHOWA DENKO K.K.

Opponent:

H.C. Starck GmbH

Headword:

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Relevant legal provisions:

-

Relevant legal provisions (EPC 1973):

EPC Art. 54(1)(2)

Keyword:

"Novelty (no, all requests)"

Decisions cited:

-

Catchword:

-



Case Number: T 0370/09 - 3.4.01

D E C I S I O N
of the Technical Board of Appeal 3.4.01
of 8 April 2011

Appellant: SHOWA DENKO K.K.
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Respondent: H.C. Starck GmbH
(Opponent) Im Schleeke 78-91
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Representative: Ackermann, Joachim
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Decision under appeal: Decision of the Opposition Division of the
European Patent Office posted 19 December 2008
revoking European patent No. 0953847 pursuant
to Article 101(2) EPC.

Composition of the Board:

Chairman: B. Schachenmann
Members: H. Wolfrum
F. Neumann

Summary of Facts and Submissions

- I. The contested European patent No. 0 953 847 arises from European patent application EP 98 966 979.1, which was published as international publication WO 98/038660.
- II. The opposition was based on the grounds of Articles 100(a) EPC 1973 for lack of novelty and inventive step as well as on Articles 100(b) and 100(c) EPC 1973. The opposition division had to decide on a main request comprising a set of claims 1 to 12 filed with letter of 2 January 2007 and an auxiliary request comprising a set of claims 1 to 12 filed with letter of 13 October 2008. In its decision, the opposition division held that the subject-matter of claim 1 of the main request lacked novelty (Article 100(a) EPC 1973 in combination with Articles 54(1) and (2) EPC 1973) and that the subject-matter of claim 1 of the auxiliary request lacked an inventive step (Article 100(a) EPC 1973 in combination with Article 56 EPC 1973).
- III. The appellant (patent proprietor, SHOWA DENKO K.K.) lodged an appeal against the decision of the opposition division, dispatched on 19 December 2008, revoking the patent. The notice of appeal was received on 2 February 2009 and the prescribed fee was paid on the same day. On 14 April 2009 a statement of grounds of appeal was filed. The appellant requested that the appeal be set aside and the patent be maintained in amended form according to the main request and the auxiliary request on which the contested decision was based.
- IV. The respondent (opponent, H.C. Starck GmbH) filed observations by letter dated 3 November 2009,

reiterating, amongst other objections, novelty objections with respect to several documents filed in opposition, including document :

D2: Y. V. Levinskii, A. B. Zaitsev, Y. M. Polyakov, Y. B. Patrikeev : "Interrelationship between the properties of powders, the conditions of pressing and sintering, and the structure of porous niobium; part I", Soviet Powder Metallurgy and Metal Ceramics, vol. 29, no. 3, 1991, pages 197 to 200; English translation of Poroshkovaya Metallurgiya, no. 3(327), 1990, pages 30 to 33.

As to the correct technical understanding of the teaching of document D2 (and others), reference was further made to document :

D15: P. Ettmayer, W. Lengauer : "Nitrides" in Ullmann's Encyclopedia of Industrial Chemistry, Wiley-VCH, Weinheim, 7th edition, 2005, pages 1 to 24.

V. In response thereto and to a communication of the Board of 24 January 2011 annexed to summons for oral proceedings, the appellant filed further arguments in support of novelty of the claimed subject-matter and made reference in this context to document :

D17: L. Tsetseris et al. : "Structure and interaction of point defects in transition-metal nitrides", Physical Review B, vol. B 76, 2007, pages 224107-1 to 224107-7.

VI. Oral proceedings were held on 8 April 2011. Apart from a brief debate of the questions of insufficiency of disclosure (Article 100(b) EPC 1973) and added subject-matter (Article 100(c) EPC 1973), the main point of

discussion was lack of novelty (Articles 100(a) and 54(1)(2) EPC 1973).

VII. The appellant requested that the decision under appeal be set aside and that the patent be maintained in amended form on the basis of the main request or the auxiliary request on file.

VIII. The respondent requested that the appeal be dismissed.

IX. The independent claims of the appellant's **main request** read as follows :

"1. *A partially nitrided niobium powder for a capacitor electrode characterized by having a bound nitrogen content in the range of 10 ppm to 50,000 ppm by weight, and having an average particle diameter in the range of 0.5 μm to 100 μm .*"

"2. *A process for preparing the partially nitrided niobium powder as claimed in claim 1, characterized by maintaining powdery niobium in a nitrogen gas atmosphere at a temperature of not higher than 2,000°C.*"

"4. *A sintered body of the partially nitrided niobium powder as claimed in claim 1.*"

"6. *A process for producing a sintered body, characterized by sintering the partially nitrided niobium powder as claimed in any one of claims 1 to 3 [sic !] at a temperature in the range of 1,000°C to 2,000°C under a pressure in the range of 13.3 Pa to 13.3 $\cdot 10^{-5}$ Pa (10^{-1} Torr to 10^{-6} Torr).*"

"7. A capacitor comprising a pair of electrodes and a dielectric substance intervening between the electrodes, characterized in that one of said electrodes is composed of a sintered body of the partially nitrided niobium powder as claimed in claim 1."

Claims 3, 5 and 8 to 12 are dependent claims.

The **auxiliary request** differs from the main request in that claim 1 requires the "*bound nitrogen content*" to be "*in the range of 100 ppm to 50,000 ppm by weight*".

- X. In the appellant's view, the subject-matter of the independent claims of both requests on file was novel and inventive because none of the documents of the prior art cited in the opposition and appeal disclosed a partially nitrided niobium powder for a capacitor electrode having a content of bound nitrogen and an average particle size in the respectively claimed ranges. Rather, the cited documents referred to powders or sintered bodies of niobium in which nitrogen occurred in insignificant amounts as an undesired superficial or interstitial impurity but was not chemically bound to the niobium, as was required by the present patent.
- XI. According to the respondent, the type of chemical bonding in niobium nitride was metallic bonding, manifested in the fact that the material was an electrical conductor. As was apparent, for instance, from document D15, the nitrogen atoms in niobium nitride occupied interstitial sites in a lattice formed

by the niobium atoms. Therefore, the "interstitial nitrogen" which was present as an impurity in a niobium powder as known, for instance, from document D2 had to be considered to constitute "bound nitrogen" within the meaning of the present patent. Since the nitrogen content and the grain size of the niobium powder known from document D2 fell within the claimed ranges, the subject-matter of claim 1 of both the main request and the auxiliary request lacked novelty.

Reasons for the Decision

1. In the light of the entry into force of the EPC 2000, reference is made to Article 7(1), 2nd sentence of the Revision Act of 29 November 2000 ("Act revising the Convention on the Grant of European Patents (European Patent Convention) of 5 October 1973, last revised on 17 December 1991") and the transitional provisions for the amended and new provisions of the EPC (Decision of the Administrative Council of 28 June 2001), from which it may be derived which Articles of the EPC 1973 are still applicable and which Articles of the EPC 2000 shall apply.
2. The appeal complies with the requirements of Articles 106 to 108 EPC and Rule 99 EPC and is, therefore, admissible.
3. Novelty (Articles 100(a) and 54(1) and (2) EPC 1973)
 - 3.1 Document D2 refers to a study of niobium powders and some of their physical properties of particular interest for sintering. There are three powder lots

under study which have differing average particle sizes in the range from 1 μm to 13 μm (D2 : Table 2). Moreover, the niobium powder lots contain *inter alia* nitrogen described as an "interstitial impurity" in a content of 0.7 weight% (7000 ppm by weight), 0.5 weight% (5000 ppm by weight) and 0.21 weight% (2100 ppm by weight), respectively (D2 : Table 1).

- 3.2 The known niobium powder would, once subjected to the appropriate manufacturing steps, eg sintering, be suitable for a capacitor electrode. Moreover, both the nitrogen content and the average particle diameter of the niobium powder known from document D2 fall squarely within the respective ranges claimed in claim 1 of both requests on file.

Thus, the decisive question to be answered is whether or not the nitrogen present as an impurity in the known niobium powder qualifies as "bound" nitrogen and renders the niobium powder "partially nitrided" within the meaning of claim 1 of both the main request and the auxiliary request on file.

- 3.3 The appellant has argued that the term "bound nitrogen" in the claims under consideration had to be understood as referring to nitrogen which was chemically bound to the niobium. The claimed nitrogen content was the result of a purposeful nitridding step in which niobium powder was kept at elevated temperatures in a pure nitrogen atmosphere for a long period of time, so as to allow the nitrogen to react chemically with the niobium whereby the niobium and nitrogen atoms would become covalently bound to each other.

In distinction thereto, document D2 aimed at producing pure niobium powders. The minute amounts of nitrogen present were an undesired impurity. The use of the term "interstitial impurity" meant that the nitrogen atoms existed in some kind of pores or at the surfaces of niobium powder particles where they were merely adsorbed but not chemically bound.

In this context, document D17 provided evidence for the fact that in transition-metal nitrides nitrogen existed in two different configurations, *ie* nitrogen atoms being (chemically) bound in and forming part of the nitride lattice and nitrogen interstitials forming point defects within the lattice. The latter configuration, which corresponded to the situation found in document D2, showed a limited stability and migration of the nitrogen atoms within the nitride lattice and thus had to be clearly distinguished from the configuration of chemically bound nitrogen as claimed in the present patent.

3.4 These arguments are unconvincing for several reasons.

First of all it is to be noted that the patent description does not contain an explanation of the term "bound nitrogen". Thus it is justified to interpret the word "bound" in the broadest technically meaningful sense.

Moreover, in actual fact, nitrogen may either adhere to the surfaces of niobium powder particles (in which case nitrogen atoms are bound to niobium by adsorption) or may be incorporated within the crystal lattice of the powder particles (in which case the nitrogen atoms

occupy interstitial sites in the lattice formed by the niobium atoms; for confirmation see document D15, chapter 4.2 "Transition-Metal Nitrides - General Properties"). That it is the latter case which applies to the nitrogen present in the niobium powders of document D2 is apparent from the designation "interstitial impurities" and confirmed by the fact that the nitrogen content was observed in powders which had been subjected to a dehydrogenation step that would have removed any surface-bound nitrogen.

Electrically conductive materials, such as for instance niobium nitride, require the presence of electrons which are free to move in a lattice; the type of chemical bonding occurring in such materials is known as 'metallic bonding'. In contrast thereto, in 'covalent bonding' or in 'ionic bonding' the binding electrons are not free to move in a lattice, but instead, remain fixedly associated with the participating atoms; the resulting materials are, consequently, electrically insulating. Therefore, the notion that in a partially nitrated niobium nitride according to the present patent nitrogen would be bound to niobium by covalent or ionic bonding, as brought forward by the appellant and erroneously mused by the opposition division in the decision under appeal (cf Reasons, paragraphs 2.4.4 and 2.9.1), is technically incorrect.

Even the appellant's reference to document D17 cannot cast doubt on this finding. Document D17 discusses point defects in some examples of transition-metal nitrides and distinguishes between two classes of defects, *ie* 'vacancies' and 'interstitials'. The

'nitrogen interstitials' studied in document D17 are nitrogen atoms which are bound in the regular lattice of over-stoichiometric nitride samples (cf the chapters "B. N interstitials" and "IV: Discussion" in D17), ie in nitride samples in which the regular sites for nitrogen atoms are all occupied. In distinction thereto, both the presently claimed subject-matter and document D2 concern niobium nitrides with sub-stoichiometric contents of nitrogen in which the "interstitials" examined in document D17 do not exist. Only for the sake of the argument it is added that the over-stoichiometric nitrogen interstitials of document D17, regardless of the fact that their binding in the crystal lattice is weaker than that of the regular stoichiometric nitrogen atoms, would nevertheless have to be considered 'bound' nitrogen atoms in the broad meaning of the present patent. Indeed, chapter "B. N interstitials" in D17 refers to the bonds holding the nitrogen atoms in the interstitial locations.

- 3.5 It follows from the above observations that the interstitial nitrogen present in the niobium powder known from document D2 qualifies as "bound" nitrogen and renders the niobium powder "partially nitrided" within the meaning of claim 1 of both the main request and the auxiliary request on file.

Therefore, the subject-matter of claim 1 of both the main request and the auxiliary request lacks novelty with respect to the prior art according to document D2.

4. In conclusion, the Board has found that the appellant's main request and auxiliary request do not comply with

the requirement of Article 54(1) and (2) EPC and thus
are not allowable.

Order

For these reasons it is decided that :

The appeal is dismissed.

The Registrar

The Chairman

R. Schumacher

B. Schachenmann