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Datasheet for the decision of 21 July 2017

Case Number: T 2225/15 - 3.3.05

Application Number: 07867118.7

Publication Number: 2038448

IPC: C04B35/486

Language of the proceedings: EN

Title of invention:
High purity powders and coatings prepared therefrom

Patent Proprietor:
PRAXAIR TECHNOLOGY, INC.

Opponent:
H.C. STARCK GmbH

Headword:
High purity zirconia/PRAXAIR

Relevant legal provisions:
EPC Art. 83

Keyword:
Sufficiency of disclosure - enabling disclosure (no)

Decisions cited:
T 0409/91, T 0435/91, T 1743/06

Catchword:

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Case Number: T 2225/15 - 3.3.05

D E C I S I O N
of Technical Board of Appeal 3.3.05
of 21 July 2017

Appellant: (Patent Proprietor)	PRAXAIR TECHNOLOGY, INC. 39 Old Ridgebury Road Danbury, CT 06810 (US)
Representative:	Schwan Schorer & Partner mbB Patentanwälte Bauerstrasse 22 80796 München (DE)
Respondent: (Opponent)	H.C. Starck GmbH Im Schleeke 78-91 38642 Goslar (DE)
Representative:	dompatent von Kreisler Selting Werner - Partnerschaft von Patent- und Rechtsanwälten mbB Deichmannhaus am Dom Bahnhofsvorplatz 1 50667 Köln (DE)
Decision under appeal:	**Decision of the Opposition Division of the European Patent Office posted on 19 October 2015 revoking European patent No. 2038448 pursuant to Article 101(3)(b) EPC.**

Composition of the Board:

Chairman	H. Engl
Members:	J.-M. Schwaller
	P. Guntz

Summary of Facts and Submissions

I. European patent EP-2 038 448 B1 relates to high purity yttria or ytterbia stabilized zirconia powders (YSZ, YbSZ) and to thermally sprayed coatings made therefrom.

II. The present appeal lies from the decision of the opposition division to revoke the European patent on the ground that the requirements of Article 83 EPC were not met.

The opposition division held in particular that the patent did not disclose how a stabilized zirconia powder having the desired high purity and morphology could be obtained. Nor were such powders known from the prior art.

Document D16: Purchase order (3 pages) of "ZRO-175 1-5u 8YZ POWDER" from CERES CORP (dated 14 July 2004) and the corresponding specification of the zirconia powder (report date 26 August 2004) were not accepted as a proof that such zirconia powders could be purchased before the relevant date of the opposed patent.

III. With its statement of grounds of appeal, the proprietor ("the appellant") submitted a main request and auxiliary requests 1 and 2.

The respective claims 1 of the main and first auxiliary requests are identical and read as follows:

"*1. A thermally sprayed coating of a high purity yttria or ytterbia stabilized zirconia powder for a substrate, said high purity yttria or ytterbia stabilized zirconia powder comprising from 0 to 0.15 weight percent impurity oxides, from 0 to 2 weight percent hafnium*

oxide (hafnia), from 6 to 25 weight percent yttrium oxide (yttria) or from 10 to 36 weight percent ytterbium oxide (ytterbia), and the balance zirconium oxide (zirconia), wherein the impurity oxides comprise from 0 to 0.02 weight percent silicon dioxide (silica), from 0 to 0.005 weight percent aluminum oxide (alumina), from 0 to 0.01 weight percent calcium oxide, from 0 to 0.01 weight percent ferric oxide, from 0 to 0.005 weight percent magnesium oxide, and from 0 to 0.01 weight percent titanium dioxide."

Claim 1 of the second auxiliary request is further characterised by the feature:

"*1. ..., wherein a bond coating is deposited between the substrate and the thermally sprayed coating, said bond coating comprising a MCrAlY+X coating applied by a plasma spray method, a detonation spray method or an electroplating method, where M is Ni, Co or Fe or any combination of the three elements, and X includes the addition of Pt, Ta, Hf, Re or other rare earth metals, or fine alumina dispersant particles, singularly or in combination.*"

IV. In response to a communication in which the board expressed its doubts whether the invention met the requirements of Article 83 EPC, further submissions were received from the parties. The appellant submitted in particular the following documents:

D20: Ceramic Industry 2001 Materials Handbook, 9 pages

D21: Excerpt from Concise Encyclopedia of Advanced Ceramic Materials, 1991 (7 pages)

D22: K. Nassau, *"Cubic Zirconia: An Update"*, Gems & Gemmology, Spring 1981, pages 9 to 19.

V. At the oral proceedings, the discussion focused on the question whether or not the invention met the requirements of Article 83 EPC.

VI. After closure of the debate, the chairman established the parties' requests to be as follows:

The appellant requested that the decision under appeal be set aside and the patent be maintained in amended form on the basis of the claims of the main request, or in the alternative, on the basis of the claims according to the first or second auxiliary requests, all filed with the statement of grounds of appeal.

The respondent (opponent) requested that the appeal be dismissed.

VII. Insofar as they are relevant for the present decision, the parties' arguments can be summarised as follows:

Appellant: The purchase order D16 was proof that the powder used in the invention was publicly available in 2004. Further evidence for the public availability of the high purity yttrium-stabilised zirconia powders was found in D20 to D22. In particular, the advertisement of MEI (Magnesium Elektron Inc.) in D20 showed that highly pure zirconia powders were sold before the priority date of the patent. Suitable production techniques for such high purity zirconia powders were mentioned in D22.

Respondent: The specification sheet in D16 indicated an amount of SiO_2 which was not necessarily inside the

claimed range. There was no evidence that the product according to D16 had actually been delivered. Furthermore, D16 related only to one particular embodiment and thus did not enable the skilled person to carry out the invention over the entire breadth of the claims. The appellant's assertion that highly pure yttria or ytterbia stabilized zirconia powders were publicly available before 2004 was in contradiction to statements in the opposed patent itself claiming such zirconia powders with a unique morphology as novel. The opposed patent neither disclosed a supplier for this powder nor a method of manufacturing it.

Reasons for the Decision

1. Main request - Sufficiency of disclosure

1.1 It is established case law that the requirements under Article 83 EPC for sufficiency of disclosure are met if the claimed invention could be performed at the filing date of the application by a person skilled in the art in the whole area claimed without undue burden, using common general knowledge and having regard to further information given in the patent in suit (see e.g. T 0409/91, OJ 1994, 653, point 3.5 of the reasons; T 0435/91, OJ 1995, 188, point 2.2.1 of the reasons; T 1743/06, point 1.1 of the reasons).

1.2 In the case at issue, the question arises whether at the priority date of the patent the skilled person was able to produce high purity yttria or ytterbia stabilised zirconia powders (YSZ, YbSZ) comprising up to 0.15 weight percent impurity oxides, among them up to 0.02 weight percent silica, up to 0.005 weight percent alumina, up to 0.01 weight percent calcium

oxide, up to 0.01 weight percent ferric oxide, up to 0.005 weight percent magnesium oxide, and up to 0.01 weight percent titanium dioxide. It is noted that said high purity zirconia powders are the essential starting materials for the claimed thermally sprayed coatings (see claim 1).

1.3 In the patent specification (paragraphs [0034] and [0043] to [0046]), methods are described how to control the particle size of the high purity yttria or ytterbia stabilised zirconia powders, namely by one of the conventional methods known as "fuse and crush", "spray dry and sinter", "sinter and crush", "cast and crush", or "densification".

Howewer, the patent in suit does not disclose how high purity yttria or ytterbia stabilised zirconia powders having a low impurity content as per claim 1 could be obtained. The patent neither discloses a supplier for such powders nor a method of making them.

1.4 The question thus arises whether the production of such high purity stabilised zirconia powders belonged to the common general knowledge. For the board, there is no concrete evidence that this was the case at the priority date of the patent.

Document D3: R. Vaßen et al, "*Influence of impurity content and porosity of plasma sprayed yttria-stabilized zirconia layers on the sintering behaviour*", Surface and Coatings Technology 141, pages 135-140 (2001)

is a scientific paper investigating the influence of the impurity content of various yttria-stabilized zirconia (YSZ) powders on the sintering behaviour of

plasma-sprayed coating layers (see abstract; page 136, left hand column, second paragraph). Four different YSZ powders (a standard powder A1, a new powder A2 and two powders obtained from a nondisclosed supplier) were examined. Their respective chemical compositions, in particular of the impurities are presented in Table 2 of D3 (page 137, top). Quite conspicuously, although the impurity levels are generally very low, none of these four YSZ powders meet the stringent purity criteria of claim 1 of the patent in suit (in particular as regards the levels of SiO_2, Al_2O_3 and TiO_2).

This corroborates the board's view that stabilised zirconia powders having the required low impurity content were not readily available in 2001 and certainly not part of common technical knowledge. There is also no evidence that this was any different at the priority date of the patent in suit (2006).

1.5 Under these circumstances it would have been in the board's opinion necessary to indicate in the patent specification at least one generally applicable method of manufacturing said highly pure powders, and/or to identify at least one reliable source for obtaining them.

1.5.1 The advertisement of Magnesium Elektron, Inc., NJ, USA, in document D20 discloses a range of yttria-doped zirconia powders. The stabilised zirconia powder closest to the ones used in the invention is MELox 8Y, which is described as containing Y_2O_3, SiO_2, TiO_2, Fe_2O_3 in respective amounts which fall within the ranges defined in claim 1 at issue. Said advertisement however does not disclose the amount of other impurities, namely calcium oxide, magnesium oxide and aluminum

oxide. Therefore, the content of D16 cannot be held as evidence that the high-purity yttria stabilised zirconia powder as defined in claim 1 at issue was commonly known or publicly available at the priority date of the invention.

1.5.2 The same conclusion arises from documents D21 and D22, which disclose high-purity stabilised zirconia powders and their production, without however any indication of the amounts of residual impurities, let alone of the amounts of the individual oxides of Si, Al, Ca, Mg, Fe and Ti, as defined in claim 1 at issue.

1.5.3 Document D16 comprises a purchase order (3 pages) issued by Praxair for a high purity yttria-stabilised zirconia powder apparently having the characteristics of the powder defined in claim 1 at issue. According to the appellant, this purchase order was evidence that the high purity yttria-stabilised zirconia powder used as starting material for the preparation of the claimed coating had been available from Ceres Inc. before the priority date of the patent.

The board does not accept this argument. Even if one accepted that the batch of stabilised zirconia purchased by the appellant (date of purchase 14 July 2004) had in fact been delivered to the buyer before the priority date of the patent (26 May 2006), for which there is no independent evidence, the patent itself neither indicates the source of said material nor suggests to the skilled person that such a high purity stabilised zirconia powders were available on the market. On the contrary, paragraph [0026] of the patent specification as filed (WO 2008/054536 A2) and paragraph [0022] of the patent specification state that "*a new morphology of yttria-stabilized zirconia powder*

has been discovered, [...]. The new high purity yttria or ytterbia stabilized zirconia powders, combined with new plasma and detonation gun thermal spray conditions, have been found to have much higher deposition efficiency, density, standoff tolerance for coating, and are capable of forming desired segmentation cracking pattern for strain tolerance" (emphasis added). The skilled reader of these passages and of claim 1 of the patent as granted is clearly taught that the high-purity yttria or ytterbia stabilised zirconia powder as defined in the claims was a novel product (cf. also paragraphs [0007], [0011], [0022], [0038] to [0040], [0081], third sentence, and claim 1 of the patent in suit). In view of this, the skilled person would have had no reason at all to look for a supplier of the said powder.

Even if, by coincidence, the skilled person would have come across Ceres Inc. as a potential supplier, the specification sheet D16 provided by Ceres Inc. for the ZRO-175 zirconia powder indicates a maximum SiO_2 content of 0.030 wt%, which is higher than the upper limit (0.02 weight percent) required by claim 1 at issue. It is thus not clear from D16 whether the ZRO-175 zirconia powder marketed by Ceres Inc. had a silica content falling within the claimed range.

1.6 It follows from the above that despite appellant's assertion that it used a YSZ having the characteristics as per claim 1, crucial information as to the method of manufacture or the source of said YSZ is missing in the patent in suit. The board also judges that in the absence of any indication in the patent that the high purity YSZ powder had been purchased, the skilled person attempting to perform the invention would not be in a position to reproduce the invention without undue

burden, since the information in the patent that the claimed product was novel is misleading. Moreover, in the absence of any information regarding the availability on the market of the stabilised zirconia powder essential to the invention, the skilled person would have to conduct his own investigations for possible sources of this material, which again amounts to undue burden.

Therefore, the board is of the opinion that the claimed invention is not disclosed in a manner sufficiently clear and complete for it to be carried out by a person skilled in the art, contrary to the requirements of Article 83 EPC.

2. Auxiliary requests 1 and 2 - sufficiency of disclosure

Claim 1 of the first auxiliary request is identical to claim 1 of the main request and claim 1 of the second auxiliary request contains the same features as claim 1 of the main request. Therefore, the same reasons as above apply to the respective subject-matters of these claims, which consequently do also not meet the requirements of Article 83 EPC.

3. The appellant's proposal during the oral proceedings to restrict the claimed subject-matter to *"a thermally sprayed coating of a high purity ytterbia stabilized zirconia powder ..."* instead of *"... yttria or ytterbia ..."* would not change the decision to be taken, since the same arguments apply to this embodiment. In fact, there is no evidence on file that a high purity ytterbia stabilized zirconia powder having the claimed purity characteristics belonged to common technical knowledge or was readily available on the market before the priority date of the patent in

suit. D20, for the reasons given above (point 1.4.1), provides no proof that a powder with the claimed quality was readily available to the market, no matter whether containing ytterbia or yttria stabilised zirconia.

4. As none of the sets of claims underlying the requests on file meets the requirements of the EPC, the appeal cannot succeed.

Order

For these reasons it is decided that:

The appeal is dismissed.

The Registrar: The Chairman:

C. Vodz H. Engl

Decision electronically authenticated