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Datasheet for the decision of 24 September 2020

Case Number: T 1151/16 - 3.2.03

Application Number: 03784932.0

Publication Number: 1552031

IPC: C23C4/10

Language of the proceedings: EN

Title of invention:
PLASMA SPHEROIDIZED CERAMIC POWDER

Patent Proprietor:
Saint-Gobain Ceramics and Plastics, Inc.

Opponent:
H.C. Starck GmbH

Headword:

Relevant legal provisions:
EPC Art. 123(2), 56, 100(c), 100(a)

Keyword:
Grounds for opposition - subject-matter extends beyond content of earlier application (yes)
Inventive step - obvious combination of known features

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Decisions cited:

Catchword:

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Case Number: T 1151/16 - 3.2.03

D E C I S I O N
of Technical Board of Appeal 3.2.03
of 24 September 2020

Appellant: (Patent Proprietor)	Saint-Gobain Ceramics and Plastics, Inc. 1 New Bond Street, Box Number 15138 Worcester, MA 01615-0138 (US)
Representative:	Zimmermann & Partner Patentanwälte mbB Postfach 330 920 80069 München (DE)
Appellant: (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:	**Interlocutory decision of the Opposition Division of the European Patent Office posted on 8 March 2016 concerning maintenance of the European Patent No. 1552031 in amended form.**

Composition of the Board:

Chairman	G. Ashley
Members:	B. Miller
	E. Kossonakou

Summary of Facts and Submissions

I. European patent No. 1 552 031 (hereinafter: the patent) relates to a process for the production of a plasma spheroidized zirconia powder.

II. An opposition was filed against the patent, based on the grounds of Article 100(b) and (c) EPC and of Article 100(a) EPC together with both Articles 54 and 56 EPC.

In the interlocutory decision the opposition division found that the contested patent met the requirements of the EPC, on the basis of the claims of auxiliary request IV submitted during the oral proceedings on 11 December 2015.

This decision was appealed by both parties. As the patent proprietor and the opponent are appellants and respondents in the present proceedings, for simplicity, the Board will continue to refer to the parties as the proprietor and the opponent in this decision.

III. Requests

The proprietor requested that the decision under appeal be set aside and that the patent be maintained on the basis of claims 1 to 9 of the main request or alternatively on the basis of the claims of one of auxiliary requests I to IV submitted during the oral proceedings in opposition on 11 December 2015.

The opponent requested that the decision under appeal be set aside and the patent be revoked in its entirety.

IV. Wording of the claims

(a) Main request (claims as granted)

Claim 1 reads:

"A process for the production of a chemically uniform thermal spray powder which comprises the steps of:

a) electrofusing zirconia with up to 60% by weight of an oxide effective to stabilize the zirconia in the tetragonal phase;
b) quenching the electrofused stabilized zirconia to obtain particulate stabilized zirconia with at least 96% of the zirconia in the tetragonal or tetragonal/ cubic phase;
c) comminuting the stabilized zirconia particles of b) to a size smaller than 5 micrometers;
d) agglomerating the stabilized zirconia particles of c) by spray drying ;
e) heat treating the agglomerated stabilized zirconia particles to form substantially spherical hollow particles of stabilized zirconia with particles sizes of 200 micrometers or less."

Claim 6 reads:

"A zirconia thermal spray powder obtainable by the process of claim 1, wherein the thermal spray powder is chemically uniform and wherein at least 95 volume% of the stabilized zirconia is in the form of spheres with a particle size of less than 200 micrometres."

Claims 2 to 5 and 7 to 9 of the main request relate to preferred embodiments of the process according to claim 1 and the powder according to claim 6.

(b) Auxiliary request I

The claims of auxiliary request I correspond to the claims of the main request, wherein process step b) in claim 1 has been amended (indicated in bold) as follows:

"b) quenching the electrofused stabilized zirconia to obtain particulate stabilized zirconia with at least 96% **by weight** of the zirconia in the tetragonal ~~or tetragonal/cubic~~ phase;"

(c) Auxiliary request II

The claims of auxiliary request II correspond to the claims of auxiliary request I wherein process step a) in claim 1 has been amended as follows:

"a) electrofusing zirconia **by arc fusing the zirconia at a temperature range of 2750°C to 2950°C** with up to 60% by weight of an oxide effective to stabilize the zirconia in the tetragonal phase;"

(d) Auxiliary request III

The claims of auxiliary request III correspond to the claims of auxiliary request I wherein process step e) in claim 1 has been amended as follows:

"e) heat treating the agglomerated stabilized zirconia particles to form substantially spherical hollow particles of stabilized zirconia with particles

sizes of 200 micrometers or less
wherein in step e) the heat treatment is a plasma fusion process."

(e) Auxiliary request IV

The claims of auxiliary request IV correspond to the claims of the main request wherein process step b) in claim 1 has been amended as follows:

"b) quenching the electrofused stabilized zirconia **by rapidly cooling the molten material with water or air, wherein the melt flow is broken up into a flow of droplets** to obtain particulate stabilized zirconia with at least 96% **by weight** of the zirconia in the tetragonal ~~or tetragonal/cubic~~ phase;"

V. State of the art

The following cited documents are relevant for this decision:

D2: US 4 450 184 A;
D3: WO 02/45931 A;
D6: US 5 651 925 A.

VI. With the summons to oral proceedings, the Board sent a communication pursuant to Article 15(1) of the Rules of Procedure of the Boards of Appeal (RPBA) 2007 indicating to the parties its preliminary opinion of the case.

VII. With a letter dated 11 September 2020 the proprietor informed the Board that it would not attend the oral

proceedings and requested a decision according to the state of the file.

VIII. With a letter dated 20 August 2020 the opponent supplemented its arguments with regard to auxiliary request IV.

IX. Oral proceedings were held on 24 September 2020 in the absence of the proprietor pursuant to Article 15(3) RPBA 2020 and Rule 115(2) EPC.

X. The proprietor's arguments, as far as relevant for this decision, can be summarised as follows.

The application as filed disclosed on page 1, lines 16 to 19 that the zirconia could be in a cubic/tetragonal phase.

Starting from document D2 as the closest prior art, the process according to claim 1 of the patent differed by process steps a) to c).

Steps a) and b) provided a synergistic effect, i.e. an improved stabilization of the zirconia in the tetragonal phase.

D3 did not disclose a quenching step following the step of electrofusing. This was also not within the routine experimentation skills of the person skilled in the art. D6 did not describe that the specific quenching step disclosed therein could contribute to improve synergistically the stability of zirconia. Moreover, the necessity to combine three prior art documents to arrive at the subject-matter of claim 1 of auxiliary request IV was a clear indication for its inventiveness.

XI. The opponent's respective arguments can be summarised as follows.

The application as filed did not disclose that the zirconia could be in a cubic/tetragonal phase. Claim 1 of the main request therefore went beyond the teaching of the application as originally filed.

Starting from D2 as the closest prior art, the subject-matter of claim 1 of the main request was obvious when considering in addition D3 and the common general knowledge concerning quenching. The same argument applied to claim 1 of each of the auxiliary requests I to III. The specific quenching step required according to claim 1 of auxiliary request IV was further rendered obvious by D6.

Reasons for the Decision

1. Article 100(c) EPC - main request

Claim 1 of the main request is based on claim 5 as originally filed wherein the expression "or tetragonal/ cubic phase" has been added:

"particulate stabilized zirconia with at least 96% of the zirconia in the tetragonal **or tetragonal/cubic phase**".

The relevant passage on page 1, lines 16 to 19 of the application as filed reads (reference is made to the International A-publication of the patent: WO 2004/015158 A1, hereinafter: the application):

"There is another phase of zirconia which is also stable at temperatures above the monoclinic/tetragonal transition temperature, (the "cubic" phase), but since little or no volume change occurs on the transition from cubic to tetragonal, this is treated for the purposes of this description as a form of the tetragonal phase and is not distinguished therefrom."

From this paragraph in the application it follows that a cubic phase of zirconia exists which is stable at temperatures above the monoclinic/tetragonal transition temperature and that this cubic phase is encompassed in the term "tetragonal phase" for the purpose of the application.

Nevertheless, it cannot be derived therefrom that the spray powder can be made from zirconia which is in a tetragonal/cubic phase as required by claim 1. Since the ratio tetragonal phase/cubic phase is not defined, the term "tetragonal/cubic phase" also encompasses a cubic phase comprising minor amounts of tetragonal phase. However, a spray powder predominantly comprising zirconia stabilised in the cubic phase is not disclosed in the application. It follows that the addition of the expression "or tetragonal/cubic phase" extends beyond the teaching of the application.

The Board therefore sees to reason to deviate from the reasoning in point 2 of the contested decision and concludes that the ground of opposition pursuant to Article 100(c) EPC prejudices the maintenance of the patent as granted according to the main request.

2. Article 100(a) in conjunction with Article 56 EPC - auxiliary request I

2.1 The Board agrees with the submission of both parties that document D2 represents a suitable starting point for the assessment of inventive step, since it relates to the same technical field as the patent, namely a process for the production of a chemically uniform thermal spray powder.

2.2 D2 discloses in column 2, lines 28 to 47 a spray powder which is plasma-treated to fuse the particles in a partially or fully homogenized hollow structure, see column 3, lines 56 to 59. According to the embodiment described in column 3, lines 12 to 14, the particles comprise 93% by weight of zirconium oxide and 7% by weight of yttrium oxide (stabilising oxide). The final spray powder comprises substantially hollow particles having a particle size between 125 and 149 micrometers, see column 4, lines 39 to 42 and column 5, lines 12 to 16.

2.3 It is undisputed that the subject-matter of claim 1 differs from the process of D2 by steps a), b) and c).

2.4 A chemically uniform spray powder obtained by the process of claim 1 provides an improved durability of the thermal spray coatings, see paragraphs [0009] and [0024] of the patent.

2.5 The objective technical problem can be formulated as the provision of a process obtaining an improved zirconia thermal spray powder which achieves a higher durability in thermal spray coatings.

2.6 The Board agrees with the reasoning in point 6 of the impugned decision that the solution to this problem as defined in claim 1 is obvious in view of the combined teaching of documents D2 and D3 taking into account common general knowledge.

2.6.1 Document D3 relates to the use of pre-alloyed stabilized zirconia, such as yttria zirconia, as a raw material to form spray-dried powders for use in thermal spray applications of thermal barrier coatings and high temperature abradables, see page 1, lines 10 to 12.

D3 therefore belongs to the same technical field as D2 and would be considered by the skilled person.

2.6.2 D3 discloses from page 2, line 16 to page 3, line 13 that the chemical inhomogeneity of a spray powder has a negative impact on thermal and mechanical cycling performance of a thermal coating obtained therewith. According to the disclosure from page 4, line 22 to page 5, line 2, this effect can be minimised by pre-alloying yttria and zirconia. The alloying can be achieved by induction arc processing, see page 6, lines 3 to 4. The final alloyed powder has a particle size of less than or equal to about 10 micrometer, see page 6, lines 10 to 11. Further agglomeration by spray-drying results in particles having a particle size of 11 to 150 micrometer, see page 6, lines 12 to 17.

Hence, D3 describes in principle process steps a) and c) as defined in claim 1 of auxiliary request I for exactly the same purpose as the patent.

2.6.3 It comes within an obvious modification of the process according to D2, to apply the pre-alloying and

comminuting step of D3 for its known and predictable effects.

2.6.4 Thereby the skilled person arrives at a process according to claim 1 except for the particle size defined in step c) and the quenching step b).

Concerning the particle size, D3 discloses that the fused material is comminuted to a particle size of below 10 µm, whereas claim 1 requires that the particle size is below 5 µm. However, this limitation of the range of the particle size represents an arbitrary selection within the slightly broader range disclosed in D3, since the presence of particles having a particle size smaller than 5 µm is not linked to any particular technical effect compared to the broader range of smaller than 10 µm known from D3.

Concerning step b) of claim 1 the proprietor argues that D3 does not disclose a quenching step and that fusion and quenching together provide a synergistic effect. However, it is commonly known that quenching contributes to the stabilisation of a phase which is itself normally only stable at elevated temperature, since the material is "frozen" in the less stable phase. Hence, it is common practice to perform a quenching step after a fusion process when aiming at a material in a stabilised phase.

2.6.5 The Board therefore concludes that the subject-matter of claim 1 of auxiliary request I lacks an inventive step.

3. Article 100(a) EPC in conjunction with Article 56 EPC - auxiliary request II

Claim 1 of auxiliary request II differs from claim 1 of auxiliary request I in that the temperature of arc fusion is defined.

However, the temperature range defined in claim 1 is not linked to a particular effect. It comes within the experimental routine of the skilled person to choose an appropriate temperature dependent on the specific melting temperature of the oxides to be fused.

The added feature therefore does not contribute to establish the presence of an inventive step.

4. Article 100(a) EPC in conjunction with Article 56 EPC - auxiliary request III

Claim 1 of auxiliary request III differs from claim 1 of auxiliary request I in that the heat treatment in step e) is defined as a plasma fusion process.

D2 also discloses in claim 5 that the hollow spheres are produced by a plasma fusion process. Therefore the same argument applies as for claim 1 of auxiliary request I.

5. Article 100(a) EPC in conjunction with Article 56 EPC - auxiliary request IV

5.1 Claim 1 of auxiliary request IV differs from claim 1 of auxiliary request I in that the quenching conditions according to process step b) are defined in more detail.

As indicated above in point 2.6, it is common practice to perform a quenching step after a fusion process when aiming at a material in a stabilised phase which without further stabilisation only exists at higher temperatures.

5.2 Concerning the specific quenching conditions defined in process step b) of claim 1, the patent discloses in paragraph [0018]:

"Upon cooling to room temperature, the stabilizing oxide maintains this tetragonal state even below the normal transition temperature. To enhance this effect, the molten material is preferably rapidly cooled with water or air such that the melt flow is broken up into a flow of droplets and cooled to provide fine particles of stabilized zirconia with a very homogenous chemical composition."

The effect of process step b) therefore corresponds to the usual quenching effect, i.e. it contributes to the stabilisation of the thermodynamically unstable phase at room temperature by rapid cooling. No further effect is disclosed which goes beyond the known effect of quenching. In this regard it belongs to the general knowledge that the contribution to the stabilisation of a phase is the higher, the faster the cooling is achieved.

Hence, the Board does not agree with the argument of the proprietor that process steps a) and b) provide an unexpected synergistic effect.

5.3 The objective technical problem to be solved by the process of claim 1 of auxiliary request IV is therefore

the same as for the process according to claim 1 of auxiliary request I.

5.4 The specific way of quenching as defined in claim 1 is generally known in the art, and although D6 is a patent document, it relates in general to a process for quenching molten ceramics (see title), and thus would be considered by the skilled person. It discloses in claim 1 a process of quenching wherein a melt of inorganic oxides is broken up into droplets by feeding it into a turbulent air stream.

According to column 1, lines 43 to 45 of D6 the quenching process can be adapted to the production of ceramics including yttria-stabilized zirconia and can be used to stabilise the tetragonal form of zirconia, see column 2, lines 45 to 48. Hence, the quenching process according to D6 is not only suitable for the oxides used in the process described in D2 and D3 but is even described as apt to stabilize the tetragonal form of zirconia. Therefore, this quenching method would be taken into account by the skilled person when trying to find a suitable way of quenching while reworking the electrofusion step proposed by D3.

The subject-matter of claim 1 of auxiliary request IV is thus obvious when considering documents D2, D3 and D6.

5.5 The proprietor argued in this regard that the necessity to combine three documents to arrive at the subject-matter of claim 1 demonstrated that the process defined therein was not obvious. However, this argument is not convincing. There is nothing to prevent the skilled person in particular circumstances from consulting more than two documents. Starting from an obvious

combination of the two documents (D2 and D3) it comes within the usual procedure of the skilled person to consult further documents with regard to the implicit features or features within the domain of the common general knowledge. Therefore the skilled person would consult D6 within its experimental routine to supplement the implicit disclosure of D3 concerning the routine step of quenching.

5.6 In summary, the Board concludes, that the subject-matter of claim 1 of auxiliary request IV is obvious and the ground of opposition pursuant to Article 100 (a) EPC also prejudices the maintenance of the patent in amended form on the basis of claim 1 of auxiliary request IV.

Order

For these reasons it is decided that:

1. The decision under appeal is set aside.
2. The patent is revoked.

The Registrar: The Chairman:

C. Spira G. Ashley

Decision electronically authenticated