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**Datasheet for the decision  
of 30 January 2007**

**Case Number:** T 0216/02 - 3.3.05

**Application Number:** 96910577.4

**Publication Number:** 0819104

**IPC:** C04B 35/111

**Language of the proceedings:** EN

**Title of invention:**

Firing sol-gel alumina particles

**Patentee:**

Saint-Gobain

**Opponent:**

3M Innovative Properties Company

**Headword:**

Abrasive grits/Saint-Gobain Industrial Ceramics

**Relevant legal provisions:**

EPC Art. 100 b)

**Keyword:**

"Sufficiency of disclosure (no)"

**Decisions cited:**

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**Catchword:**

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Case Number: T 0216/02 - 3.3.05

**DECISION**  
of the Technical Board of Appeal 3.3.05  
of 30 January 2007

(Opponent) 3M Innovative Properties Company  
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**Decision under appeal:** Interlocutory decision of the Opposition  
Division of the European Patent Office posted  
13 December 2001 concerning maintenance of the  
European Patent No. 0819104 in amended form.

**Composition of the Board:**

**Chairman:** M. Eberhard  
**Members:** E. Waeckerlin  
S. Hoffmann

## Summary of Facts and Submissions

- I. The appeal is from the decision of the opposition division posted on 13 December 2001 concerning the maintenance of the European patent No. 0 819 104 in amended form on the basis of claims 1 to 32 according to the main request filed during the oral proceedings.
- II. The independent claims 1, 12, 17 and 22 of said request read as follows:

*" 1. A process for explosively comminuting a composition comprising a dried but unfired sol-gel alumina said composition having a volatilizable content of at least 5% by weight, which comprises feeding particles of the composition directly into a furnace held at a temperature from about 400°C to 1600°C and controlling the residence time in the furnace to produce explosively comminuted alumina particles.*

*12. A process for explosively comminuting a composition comprising a dried but unfired seeded sol-gel alumina, said composition comprising from about 20 to about 40% by weight of volatilizable material which comprises feeding the composition into a tubular rotary furnace having a hot zone maintained at a temperature of from about 600°C to about 1500°C, in which the composition is fed directly at a point adjacent the hot zone, and the tube is inclined at an angle of from about 2° to about 20° to the horizontal and rotated at from about 2 to about 20 rpm.*

*17. A process for explosively comminuting a dried but unfired unseeded sol-gel alumina comprising from about*

*20 to about 40% of volatilizable material which comprises feeding the dried sol-gel into a tubular rotary furnace having a hot zone maintained at a temperature of from about 600°C to about 1650°C in which the composition is fed directly at a point adjacent the hot zone, and the tube is inclined at an angle of from about 3° to about 20° to the horizontal and rotated at from about 1 to about 20 rpm.*

*22. Alumina abrasive grits that are non-symmetrical about their longitudinal dimension and, as produced and within any grit size fraction thereof, comprise more than 25% of grits with an aspect ratio of at least 2:1 and have a density that is at least 95% of the theoretical density. "*

III. In the decision under appeal the opposition division held that none of the grounds for opposition invoked by the opponent prejudiced the maintenance of the patent as amended. In particular the opposition division held that the description and Figure 1 of the patent in suit provided adequate information to enable the skilled person to put into practice without undue burden the required "volatilizable content" by drying the unfired sol-gel material to the amounts as claimed, and to achieve explosive comminution. Thus, the disclosure was sufficient.

IV. The appellant (opponent) lodged an appeal against this decision. In the grounds of appeal, the appellant submitted for the first time that the alumina abrasive grits according to Claim 22 lacked novelty having regard to three public prior uses of commercially

available products, namely "SWSK-L", "NorZon F826" and "3M 984C".

He maintained his previous objection that the amendment of claims 12 and 17, respectively, was not in conformity with Article 123(2) EPC. In his view the feature according to which "*the composition is fed **directly** at a point adjacent the hot zone*" was not disclosed in the application as originally filed.

Moreover claims 12 and 17 lacked clarity, because it was unclear how far away from the hot zone the area "*adjacent the hot zone*" was.

The appellant further argued in particular that the requirement of sufficiency of the disclosure was not met. Claim 1 required a "*volatilizable content of at least 5% by weight*" in the sol-gel alumina composition. The patent did not mention, however, how the amount of the volatilizable components was determined.

The disclosure of the patent was also insufficient in respect of the feature of "*explosively comminuting*", because the patent did not give any indication how the skilled person could recognise whether explosive comminution had taken place, or not, particularly in the case of the formation of "*weakness lines*".

- V. In a communication the board made various observations regarding the alleged public prior uses.
  
- VI. Oral proceedings took place on 30 January 2007. During the oral proceedings the respondent (proprietor of the patent) submitted an amended set of claims as the first auxiliary request. Claim 1 of this request differs from

Claim 1 of the main request by the incorporation of the feature "*at a point adjacent to the hot zone*" after the word "*directly*". Claims 12 and 17 of the first auxiliary request differ from those of the main request by the insertion of the word "*directly*" before the word "*feeding*".

- VII. The respondent denied that the amended claims were not in conformity with Article 123(2) EPC. He argued that the skilled person recognised readily that the expression "*adjacent the hot zone*" meant any location just outside the hot zone, at the borderline of the hot zone, or just inside the hot zone.

Concerning the sufficiency of the disclosure, the respondent held that there exists no difficulty for the person skilled in the art to determine the content of volatilizable material. This could be done by means of calculations or on the basis of routine analytical testing. Such testing could be based *inter alia* on the description and Figure 1 of the patent. Moreover the skilled person knew many analytical techniques for determining the content of volatilizable material, including the standard test methods set out in ASTM C 323-56 and ASTM E 1131-03. With regard to the issue of recognising explosive comminution, the respondent argued that the meaning of the expression "*explosively comminuting*" was plain from the description. When dried sol-gel alumina particles were fed into the furnace and were at least partially broken up into smaller particles without the use of any externally imposed force, then explosive comminution had taken place.

VIII. The appellant requested that the decision under appeal be set aside and that the European patent No. 0 819 104 be revoked.

The respondent requested that the appeal be dismissed or, in the alternative, that the decision under appeal be set aside and that the patent be maintained on the basis of the first auxiliary request filed during the oral proceedings.

### **Reasons for the Decision**

#### *Allowability of the amendments - Article 123(2), (3) EPC*

1. The question whether the amendments in the independent claims of the main request and the first auxiliary request are in conformity with the provisions of Article 123(2), (3) EPC does not need to be decided, since the patent has to be revoked due to insufficiency of disclosure for the reasons indicated hereinafter.

#### *Sufficiency of the disclosure - Article 100 b) EPC*

##### Main request

2. The question arises whether the patent discloses the invention in a manner sufficiently clear and complete for it to be carried out by a person skilled in the art (Article 100 b) EPC).
3. In this respect the appellant submitted that the skilled person was unable to determine the required "volatilizable content of at least 5% by weight" (see

Claim 1), or the amount of "volatilizable material" of "from about 20 to about 40%" (see claims 12 and 17), because the method for the determination of the amount of the volatilizable components was not indicated in the patent.

Moreover the skilled person could not recognise whether the process led to explosive comminution, or not. It was completely unclear how the creation of "weakness lines" could be determined.

4. As far as the expressions "volatilizable content" and "volatilizable material" are concerned, the board notes that the claims contain no definition of what is meant by the term "volatilizable". There is no general definition in the description either, although some guidance is given by section [0009] of the patent in suit (see page 2, line 57 to page 3, line 8). There it is explained that in the case of dried boehmite, each molecule "has an associated molecule of water, with perhaps some residual water not completely removed in the drying. In addition advantageous modifiers such as magnesia, yttria, rubidia, caesia, or rare earth or transition metal oxides are often added to the sol-gel in the form of their soluble nitrates and these too will contribute volatilizable components, (such as nitrogen oxides), to the dried gel. If an acid such as nitric or acetic acid has been used to peptize the sol-gel there may also be residues of this acid in the dried sol-gel." It follows from this explanation that in the case of boehmite the dried but unfired sol-gel alumina composition used as the starting material in the claimed process contains volatilizable material from one or more of the following sources:



- (i) crystal water;
- (ii) residual water from the drying process;
- (iii) modifiers in the form of their soluble nitrates;  
and/or
- (iv) residues of acids.

All these components contribute to the "*volatilizable content*" or the amount of "*volatilizable material*" set out in claims 1, 12 and 17, respectively.

5. There remains the question how the amount of "*volatilizable material*" can be determined. The claims are silent in this respect, and the description does not refer either to a specific method. The board has therefore examined whether the skilled person is able to determine the amount of "*volatilizable material*" of the sol-gel alumina compositions on the basis of the contents of the description in combination with common general knowledge. In particular the board has investigated whether the skilled person can determine the temperature at which the measurement has to be made.
  
6. It is common general knowledge that the amount of volatilizable components contained in a sample may be determined by heating the sample to a specific temperature  $T_m$  and determining the difference between the initial weight of the sample before heating and its weight after heating. Thus, in the case of a dried but unfired sol-gel alumina composition the "*volatilizable content*" will depend *inter alia* on the types of volatile components contained in the composition and the temperature  $T_m$ .

7. The patent in suit does not indicate how the temperature  $T_m$  has to be selected. Although it is stated in section [0009] of the patent that *"drying is usually conducted at a temperature below about 200 °C and more usually at temperatures far lower"* (see page 3, lines 6-7), and although it is further stated that *"generally the dried gel has an overall vaporizable content of from about 5 to about 50 %, preferably from about 10 to about 45 %, and more preferably from about 20 to about 40 % by weight"* (see page 3, lines 4-6), there is no indication to which temperature the dry products have to be heated in order to determine the percentage of volatilizable material set out in the claims. It is evident for the skilled person, however, that the *"volatilizable content"* depends on that temperature. For the specific case of a seeded sol-gel material this is exemplified by section [0025] of the patent in suit, according to which there is a *"loss of volatiles including water and acid and salt decomposition products"* at about 400°C. This loss of volatiles is said to cause the explosive comminution (see page 4, lines 36-37). Further it is observed in section [0025] that by about 600°C *"the amount of volatiles to be removed has significantly diminished and conversion to the anhydrous phases of alumina such as gamma alumina is complete"* (see page 4, lines 35-40). This means that, depending on whether the measuring temperature is 400°C or 600°C or even higher, different levels of the content of volatilizable material will be found. But there is no disclosure in section [0025], taken separately or in combination with Figure 1 of the patent in suit, which would allow a conclusion as to what the temperature of the measurement should be.

8. In any case section [0025] in combination with Figure 1 of the patent in suit cannot be regarded as a general disclosure, because it concerns a specific composition comprising a seeded sol-gel alumina (see page 4, line 29). According to section [0025] the Differential Thermal Analysis trace of an unseeded sol-gel material is different from that of a seeded sol-gel material in that the alpha conversion peak occurs at a higher temperature (see page 4, lines 41-43). Furthermore, since the starting material of the process according to Claim 1 of the main request may be any composition comprising "*a dried but unfired sol-gel alumina*" it is by no means limited to the material investigated in section [0025], let alone to boehmite, but may include various other components which may or may not contain volatilizable materials (see page 3, lines 1-3). The conclusion is, therefore, that section [0025] of the patent in suit does not provide sufficient guidance as to the temperature at which the "*volatilizable content*" of the dried but unfired composition comprising the sol-gel alumina has to be measured.
9. The examples contained in the patent in suit are of no help either. Whereas examples 1 and 2 are silent regarding the content of volatilizable material, it is mentioned in example 3 that the gel was "*dried to a water content of about 30 - 35 % by weight*" (see section [0049], page 7, lines 7-8). There is no indication, however, as to how the water content has been determined.
10. The board observes that, even under the assumption that the skilled person would conclude from section [0025] and the other information contained in the patent that

the temperature for determining the "*volatilizable content*" is slightly below the temperature at which explosive comminution occurs, there would still remain the question what this temperature is precisely. In fact the temperature at which explosive comminution takes place depends on many factors, in particular the chemical analysis of the composition comprising the sol-gel alumina, the velocity of the loss of volatiles (i.e. the speed of heating, see sections [0025] and [0026]), the size of the particles and the point at which the alumina composition is fed into the furnace (see page 5, lines 5-6). According to section [0029] the particles explode when they enter the hot zone within the furnace (see page 5, lines 5-6); however the firing temperatures in the hot zone given in section [0034] of the patent (see page 5, lines 31-34) do not permit to conclude at which temperature explosive comminution occurs. Therefore the said assumption would not provide a precise teaching of the temperature which has to be used for measuring the "*volatilizable content*".

11. According to the respondent, different methods may be applied to determine the "*volatilizable content*". In certain situations theoretical calculations will already lead to a result. In the case of boehmite, for example, two moles of boehmite of the formula  $\text{AlO}(\text{OH})$  are required to form one mole of alumina  $\text{Al}_2\text{O}_3$  and one mole of water  $\text{H}_2\text{O}$ . This corresponds to about 15 % by weight of "*volatilizable content*" in the form of "*chemically bound water*", i.e. crystal water. The content of volatiles will be higher than the calculated result, whenever the starting boehmite-material is not

completely dried (see respondent's letter dated 28 December 2006, page 9, second paragraph).

12. As far as such theoretical considerations are concerned, the board concurs with the respondent's view that the amount of "*chemically bound water*" can be calculated in the case of boehmite from the chemical formula. The board observes, however, that such calculations are only possible for certain specific materials (e.g. for boehmite), as the respondent has pointed out himself. Claim 1 is by no means limited to such materials but covers any compositions comprising a dried but unfired sol-gel alumina. Therefore calculations are not feasible over the whole scope of Claim 1.
  
13. The respondent argued further that the skilled person "*would know many analytical techniques for measuring volatilizable materials content in the sol-gel materials before they are explosively comminuted*" (see letter dated 28 December 2006, page 11, first paragraph). As examples of such analytical techniques the respondent referred to the standard test methods ASTM "*C 323-56 (Reapproved 2006)*" and ASTM "*E 1131-03*", respectively.
  
14. The board observes that document C 323-56 (Reapproved 2006) has been published in February 2006, i.e. after the priority date of the patent in suit. It deals with methods for chemical analysis of ceramic whiteware clays, in particular with the determination of "*moisture*" of samples at a drying temperature not under 105 nor over 110 °C (see C 323-56, page 3, section 7), and with the determination of "*loss on ignition*" of moisture-free samples at a temperature of 900°C to

1000°C (see C 323-56, page 3, section 8). The patent in suit is not concerned with loss on ignition, however, but with the "*volatilizable content*" of compositions comprising a dried sol-gel alumina.

Document E 1131-03, which has been published in April 2003, i.e. also after the priority date of the patent in suit, concerns a standard test method for compositional analysis by thermogravimetry "*to determine the amount of highly volatile matter, medium volatile matter, combustible material, and ash content of compounds*" (see E 1131-03, paragraph 1.1). The temperature range of test is typically room temperature to 1000°C (see E 1131-03, paragraph 1.3). Low boiling components having a boiling point of 200°C or less are designated as "*highly volatile matter*", whereas materials that degrade or volatilize in the temperature range 200°C to 750°C are designated as "*medium volatile matter*" (see E 1131-03, paragraphs 3.2.1 and 3.2.2). The mass of a sample, heated at a controlled rate in an appropriate environment, is recorded as a function of time or temperature. Mass loss over specific temperature ranges provides a compositional analysis of the sample. Various test parameters, including in particular the temperature range and the heating program within the desired temperature range, have to be set when carrying out the method (see E 1131-03, paragraph 4.1; section 11, particularly paragraphs 11.9 and 11.9.2; tables 1 and 2). This confirms that the results obtained by the method depend on the specific test conditions, particularly on the selection of the temperature range. E 1131-03 provides no specific information regarding the analysis of the

"volatilizable content" of sol-gel alumina, or compositions comprising the latter.

The board concludes, therefore, that in the case of the patent in suit, neither C 323-56 (Reapproved 2006) nor E 1131-03 provide sufficient guidance.

15. As argued by the respondent the "volatilizable content" may be determined by measuring the loss of weight during the heating of the composition comprising the sol-gel alumina. However, it is evident that the results will depend to a large degree on the composition of the samples and the measurement conditions, in particular the temperature. For example, if the measurements are carried out at temperatures of 400°C or higher, then the amount of the crystal water contained in the samples will have an impact on the results (see Figure 1). At temperatures of 200°C or below this is not the case, however, because at such low temperatures the crystal water is not volatilized. The board notes that there is no indication in the patent in suit which specific measuring conditions, in particular which temperature, have to be observed.
  
16. At the oral proceedings the respondent's representative has stated that in the case of the patent in suit, the amount of volatilizable material has to be determined in the following manner: A sample of undried material containing free water, bonded water and/or vaporizable additives is steadily heated at a fixed predetermined drying temperature below 200°C until its weight is constant. The loss of weight is recorded as a function of the drying time. For each specific drying time the loss of weight is expressed as a percentage, based on

the weight of the sample before drying. The graphic curve of loss of weight as a function of drying time provides the information after which time a given loss of weight (e.g. 5 %) is reached. If the skilled person wishes to set a specific "volatilizable content", the only thing he has to do consists in observing the corresponding drying conditions, namely the proper drying time at the predetermined drying temperature. In the view of the respondent's representative this is the right method to be applied when putting the claimed process into practice. The respondent's representative admitted that this method is not stated in the patent, but in his view it forms part of the common general knowledge of the skilled person, so that there is no need for a specific disclosure in the patent.

17. The board notes that the explanations regarding the method for determining the "volatilizable content", as given by the respondent's representative at the oral proceedings, are not in line with the statements contained in the letter dated 28 December 2006. In fact, it cannot be derived from the patent or the arguments presented in writing that the amount of the volatilizable material is determined at a temperature below 200°C, i.e. at a temperature which is typical for the drying process, especially since the endotherm peak at about 400°C in Figure 1 of the patent is said to indicate the loss of volatiles including water and acid and salt decomposition products (see page 4, lines 35-37 of the patent in suit). Furthermore, the argument that the temperature range of below 200°C used for the determination of the volatilizable content (see point 16 above) forms part of the common general



knowledge of the skilled person represents a mere allegation which was not supported by any evidence.

18. The process of Claim 1 being characterised by the "*volatilizable content of at least 5 % by weight*", the skilled person must be able to determine this parameter by means of indications in the description of the patent in suit and/or by well-known procedures. However, since on the one hand the essential measurement conditions for determining this parameter, in particular the temperature, are left open in the patent in suit, and on the other hand the temperature range indicated in point 16 above was not shown to form part of common general knowledge, the board concludes that the process as defined in Claim 1 of the main request does not meet the requirement of sufficiency of disclosure within the meaning of Article 100 b) EPC.

Auxiliary request

19. The finding of lack of sufficient disclosure applies likewise to the auxiliary request, since the feature concerning the content of volatilizable material set out in Claim 1 of the auxiliary request is the same as in Claim 1 of the main request.

**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. Vodz

M. Eberhard