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DECISION of 22 February 2000

Case Number:

T 0517/96 - 3.3.5

Application Number:

89311687.1

Publication Number:

0373765

IPC:

C04B 35/10

Language of the proceedings: EN

Title of invention:

Ceramic alumina abrasive grains seeded with iron oxide

MINNESOTA MINING AND MANUFACTURING COMPANY

Opponent:

H.C. STARCK GmbH & Co. KG

Headword:

Abrasive grains/3M

Relevant legal provisions: EPC Art. 54, 56

Keyword:

"Novelty (yes)"

"Inventive step (yes)"

Decisions cited:

Catchword:



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Beschwerdekammern

Boards of Appeal

Chambres de recours

Case Number: T 0517/96 - 3.3.5

DECISION of the Technical Board of Appeal 3.3.5 of 22 February 2000

Appellant:

H.C. STARCK GmbH & Co. KG

Im Schleeke 78-91 (Opponent)

D-38642 Goslar (DE)

Representative:

Steiling, Lothar, Dr.

Bayer AG

Konzernbereich RP Patente und Lizenzen

D-51368 Leverkusen (DE)

Respondent:

MINNESOTA MINING AND MANUFACTURING COMPANY

(Proprietor of the patent)

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Representative:

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Decision under appeal:

Decision of the Opposition Division of the European Patent Office posted 9 April 1996

rejecting the opposition filed against European patent No. 0 373 765 pursuant to Article 102(2)

EPC.

Composition of the Board:

Chairman:

R. K. Spangenberg M. M. Eberhard M. B. Günzel

Members:

## Summary of Facts and Submissions

- I. European patent No. 0 373 765 based on application No. 89 311 687.1 was granted on the basis of nine claims. Granted claim 1 reads as follows:
  - "1. A process for forming abrasive grains, comprising the steps of:
  - a. preparing a dispersion of alpha alumina monohydrate particles;
  - b. seeding said dispersion with crystalline iron oxide particles;
  - c. gelling said dispersion;
  - d. drying the seeded gelled dispersion to form a solid;
  - e. calcining the solid;
  - f. crushing the dried or calcined solid to form particles; and
  - g. sintering the calcined particles, further characterized by said crystalline iron oxide particles having an average particle size of less than 150 nm and said seeding being achieved by adding said crystalline iron oxide particles to said dispersion."
- II. The appellant (opponent) filed a notice of opposition requesting revocation of the patent on the grounds of lack of novelty and lack of inventive step. The appellant relied on the following documents in support of his arguments:

D1: EP-A-0 200 487

D2: Lehrbuch der Lacke und Beschichtungen, H. Kittel, vol. 2, 1974, pages 108 to 121

D3: J. Am. Ceram. Soc., 68(9), C-238-C-240, 1985.

- The opposition division decided to reject the III. opposition. It held that the process according to granted claim 1 was new over the disclosure of D1 since the  $\alpha$ -ferric oxide seed particles used in Example 14 of D1 had a particle size falling outside the limit defined in claim 1. Furthermore, the in-situ formation of  $\alpha$ -ferric oxide seed particles from a precursor added in the form of a solution as disclosed in Examples 11 to 13 of D1 differed from the addition of crystalline iron oxide particles or particulate iron oxide precursors as defined in claim 1. Concerning inventive step, the opposition division considered that D1 was the closest prior art and that the improvement in abrasive performance obtained by the claimed method could not be foreseen in the light of the cited prior art. D1 contained no suggestion to use materials with a particle size of less than 150 nm. The statement in D3 that  $\alpha\text{-Fe}_2\text{O}_3$  seed crystals of 1  $\mu\text{m}$  might have been too large to be effective in lowering the sintering temperature was not relevant. It did not refer to abrasive properties but to the sintering temperature and could not be interpreted as an indication for using seed particles of less than 150 nm.
- IV. The appellant lodged an appeal against this decision.

  He referred to five additional documents at the appeal stage in support of his arguments concerning the novelty issue. Oral proceedings were held on 22 February 2000.

V. The appellant's submissions in writing and at the oral proceedings can be summarised as follows:

D1 disclosed a range of 80 to 700 nm for the particle size of α-alumina seed particles. According to page 7, the nucleating agent could be  $\alpha$ -alumina as well as α-ferric oxide. Although D1 gave no specific information for the particle size of the α-Fe<sub>2</sub>O<sub>3</sub> seed particles, the skilled person would have taken the same size as for the alumina seeds. If the particle size had to be different, this would have been indicated in D1. In Example 14, the particle size of 200 nm lay outside the claimed range and, thus, did not destroy the novelty thereof. However, it showed that in the case of  $\alpha\text{-Fe}_2\text{O}_3$  seeds, the size to be chosen was the same as for α-alumina seed particles. Taking into account that the range of particle size disclosed in D1 overlapped the claimed range, the claimed subject-matter was not novel over the disclosure of D1.

D1 further taught that precursors of  $\alpha$ -ferric oxide having a conversion temperature lower than the conversion temperature of boehmite to α-alumina were also useful as nucleating agent and that a material could, in principle, be used as a nucleating agent if it was crystallographically similar to  $\alpha$ -alumina. It directly followed therefrom that a conversion to  $\alpha$ -Fe<sub>2</sub>O<sub>3</sub> took place before formation of  $\alpha$ -alumina in Examples 9 and 11 to 13. The newly cited documents confirmed that crystalline goethite-seeds were formed in an aqueous solution of ferric nitrate and that heating an aqueous solution of ferric nitrate led to the formation of very small  $\alpha\text{-Fe}_2\text{O}_3$  seed particles in the first step. Therefore, the skilled person would have directly derived the subject-matter of claim 1 from the said examples.

If novelty were acknowledged with respect to D1, then the claimed process would lack an inventive step over the teaching of this document alone. Example 14 of D1 and the more general statements on pages 6 and 7 of the description represented the closest prior art. The skilled person confronted with the problem of producing improved abrasive grains would have worked under the conditions disclosed in D1, in particular within the preferred range of 80 to 700 nm given for the seed particle size. As this range overlapped the claimed range, the skilled person would have easily and automatically arrived at the claimed particle size. The skilled person whose purpose was to achieve an improvement would not have been hindered from performing tests within all the different ranges disclosed in D1. The value given in Table VI for the abrasive performance of Example 14 was not the best value but also not the worst. It lay in between the values given for the other examples. No conclusion could be drawn from the experiments reported in Table VI of D1 since they were not based on a systematic testing and furthermore only four examples concerned ferric oxide. In view of the teaching of D1 that smaller as well as greater particles could be used, the skilled person would have performed further tests with ferric oxide. The tests reported in the patent in suit were in fact the logical consequence of the teaching of D1.

VI. The respondent put forward inter alia the following arguments:

The skilled person would have understood the expression "particle size" stated in claim 1 as meaning the longest dimension since it was very common to define the particle size of abrasive grains by their longest dimension. The range of particle size disclosed in D1 concerned the  $\alpha$ -alumina seed particles, not the iron

oxide particles. The particle size given in Example 14 fell outside the claimed range and, thus, this example did not anticipate the claimed process. The examples using an iron nitrate solution did not destroy the novelty since no particles were added to the dispersion. A skilled person would not have used the same range of particle size for  $\alpha$ -ferric oxide and  $\alpha$ alumina seeds. D3 showed that, at the time of D1 (1985), completely different ranges of particle size were used for different kinds of seed particles such as  $\alpha-Al_2O_3$ ,  $\alpha-Fe_2O_3$  and spinel. Furthermore,  $\alpha-Al_2O_3$  did not dissolve in an acidic medium contrary to  $\alpha\text{-Fe}_2O_3$  so that the risk of dissolution was higher with very fine particles of α-Fe<sub>2</sub>O<sub>3</sub>. Therefore, there was no unambiguous disclosure of the claimed particle size in D1.

Concerning inventive step, the respondent argued that the experiments filed with the letters of 15 May 1995 and 8 February 1996 showed that the claimed process led to abrasive grains having a better hardness and a better grinding performance than the abrasive grains of Examples 12 and 14 of D1. The performance was so good that MgO could be left out. The skilled person would have inferred from Tables II and VI of D1 that a better abrasive performance was achieved with a solution of iron nitrate than with  $\alpha$ -Fe<sub>2</sub>O<sub>3</sub> particles. Examples 15 to 17 of Table VI taught that finer particles did not lead to better abrasive properties. Therefore, the skilled person faced with the problem of improving the abrasive performance would have tried to improve the embodiment involving the use of an iron nitrate solution. It was surprising that iron oxide seed particles with a particle size of less than 150 nm led to an improved abrasive performance and made it possible to leave out MgO. The obtaining of a better homogeneity than with an iron oxide solution was a further unexpected advantage.

VII. The appellant requested that the decision of the opposition division be set aside and that the patent be revoked. The respondent requested that the appeal be dismissed and that the patent be maintained unamended.

# Reasons for the Decision

- The appeal is admissible.
- The question arises whether or not the claimed subject-2. matter meets the requirement of novelty with respect to D1. This document discloses a process for forming abrasive grains, comprising all the steps (a) to (g) indicated in the preamble of claim 1 of the patent in suit (see D1, claims 1, 5 and 8, Example 14). The  $\alpha$ -ferric oxide seed particles used in Example 14 have a dimension of 0.2 x 0.02  $\mu m$ , ie 20 x 200 nm (see page 16, line 32). At the oral proceedings the appellant pointed out that this particle size did not destroy the novelty of the claimed range, the particle size indicated in claim 1 being the longest dimension of the particles. In reply to a question of the board regarding the expression "average particle size" used in claim 1, the respondent explained that it was very common to define the particle size of abrasive grains by their longest dimension and that the skilled person would, therefore, have understood the expression "average particle size" as meaning the longest dimension of the particles. This explanation is in agreement with the appellant's construction of claim 1 and was not contested by him. In addition, it also corresponds to the definition given on page 4, lines 57 to 58, of the patent in suit. In these circumstances, the board can accept that the skilled person would

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understand the expression "average particle size" used in claim 1 as referring to the longest dimension of the iron oxide particles. Therefore, Example 14 of D1 does not anticipate the claimed subject-matter.

2.1 The appellant argued that the general information given on page 6 (lines 11 to 26) and page 7 (lines 4 to 13) destroyed the novelty of the claimed subject-matter taking into account that the claimed range overlapped that given in D1 (see point V above). These arguments are not convincing for the following reasons:

According to page 6, lines 11 to 26, one of the most preferred nucleating agents is particulate  $\alpha$ -alumina and the particle size of the α-alumina particles may vary considerably. "Particle size varying from about 80 to about 700 nm have been found to be useful but smaller and larger particles are also thought to be useful". The appellant did not contest at the oral proceedings that this range clearly relates to the α-alumina particles. On page 7, D1 discloses that other nucleating agents include α-ferric oxide (Fe<sub>2</sub>O<sub>3</sub>), or precursors of α-alumina or α-ferric oxide which convert respectively to α-alumina or α-ferric oxide at a temperature below the temperature at which alumina monohydrate would transform to α-alumina. Materials which convert to α-alumina or materials which are crystallographically similar to  $\alpha$ -alumina, as is the case with α-ferric oxide, are said to be useful nucleating agents. In Examples 11 to 13 a solution of ferric nitrate is used as the precursor for the nucleating agent ( $\alpha$ -ferric oxide) and in Example 14 α-ferric oxide particles having a length of 200 nm are added to the dispersion as such instead of as a precursor. Example 14 is the sole example out of 61 examples in which  $\alpha$ -ferric oxide seed particles are added as such to the dispersion. Neither the passages on pages 6 and 7 of D1, nor the rest of the description

contain information from which it could be directly and unambiguously derived that in the case of the addition of a-ferric oxide seed particles, the appropriate particle size thereof would be the same as for  $\alpha$ alumina seed particles. The appellant's arguments that the skilled person would have taken the same particle size as for  $\alpha$ -alumina and that the particle size of the α-ferric oxide particles would have been stated in D1 if it had been different, do not convince the board. As pointed out by the respondent, the solubility of  $\alpha$ ferric oxide in an acidic medium substantially differs from that of  $\alpha$ -alumina so that the skilled person would have had no reason to think that in both cases the particle size of the seed particles was the same. Furthermore, D3 which was published in September 1985, ie the same year as the priority date of D1, shows that seed particles having very different ranges of particle size were used in the case of  $\alpha$ -alumina seed particles (200 to 400 nm), spinel seed particles (≈50 to 100 nm) and  $\alpha$ -ferric oxide (1000 nm), (see page C-238, righthand column). Therefore, it appears that in 1985, the skilled person would not have automatically chosen the same range of particle size when using different kinds of seed particles such as  $\alpha$ -alumina or  $\alpha$ -ferric oxide. The reasons for not giving a specific range of particle size in the case of  $\alpha$ -ferric oxide particles is not given in D1, but it is not excluded that further experiments with  $\alpha$ -ferric oxide particles were not performed as the use of  $\alpha$ -alumina seed particles was the most preferred embodiment. For the preceding reasons, the board considers that a range of 80 nm to 700 nm for the  $\alpha$ -ferric oxide seed particles is not directly and unambiguously derivable from D1.

- 2.2 In Examples 9 and 11 to 13 of D1, ferric nitrate was added to the dispersion of alumina monohydrate in the form of a **solution**. Therefore, there was no addition of crystalline iron oxide **particles** or of **particulate** iron oxide precursors to the dispersion as required in claim 1 (see the definition of "crystalline iron oxide particle" on page 3, lines 8 to 9). Thus, the claimed subject-matter is novel over the disclosure of these examples. As the question whether or not goethite seed particles and  $\alpha$ -Fe<sub>2</sub>O<sub>3</sub> seed particles were formed in situ before conversion to  $\alpha$ -alumina is not relevant for the novelty and inventive step issues, it can remain open.
- 2.3 It follows from the above that the process as defined in claim 1 is novel over the disclosure of D1. The claimed process is also new with respect to the disclosure in the remaining documents cited by the appellant. This was not in dispute so that reasons in this respect need not be given.
- 3. The board considers in agreement with the parties that D1 represents the closest prior art, in particular the embodiment of D1 involving the addition of  $\alpha$ -ferric oxide particles as the nucleating agent to the dispersion of alumina monohydrate.

Starting from this prior art, the technical problem underlying the claimed process can be seen in the provision of a process for making abrasive grains, which leads to abrasive grains having an improved abrasive performance.

The patent in suit proposes to solve this problem by the process as defined in claim 1, which comprises seeding the dispersion of alumina monohydrate particles by addition of crystalline iron oxide particles having an average particle size of less than 150 nm to the said dispersion. As indicated above, this process differs from that of the closest prior art by the lower average particle size of the added crystalline iron oxide particles. In view of the comparative examples enclosed with the respondent's letter dated 8 February 1996 and of the information and examples of the patent in suit, it is credible that this problem has actually been solved by the claimed process. This was not in dispute.

As already indicated above, D1 discloses no specific 3.1 range for the average particle size of the  $\alpha$ -ferric oxide seed particles added to the dispersion of alumina monohydrate. D1 teaches that, in the case of  $\alpha$ -alumina seed particles, particle sizes varying from about 80 nm to 700 nm have been found to be useful but smaller and larger particles are also thought to be useful. The skilled person would not have inferred from this teaching that the choice of the seed particle size within a specific range might be important for achieving an improved abrasive performance. D1 contains no further information which would have suggested to the skilled person that in the case of the addition of  $\alpha$ -ferric oxide seed particles, the abrasive performance might be improved by selecting a particle size of less than 150 nm. Table VI reports the abrasive performance achieved with the products prepared in the examples and Table II indicates the corresponding concentrations of components and the particle size of the seed particles. It cannot be inferred from these Tables that fine  $\alpha$ -alumina seed particles (83 nm in Example 17) would lead to an improved abrasive performance compared to  $\alpha$ alumina seed particles with a greater particle size such as 280 nm (Example 5), since not only the particle size of the  $\alpha$ -alumina seeds but also other parameters were changed simultaneously. A comparison of Example 14 with Example 17 is also not relevant since both the kind of seed material and the concentration thereof are

different, in addition to the different particle sizes of the seed material. A comparison of Examples 12 and 14 shows that the use of a solution of ferric nitrate as the precursor for  $\alpha$ -ferric oxide in an amount such that the final product contains 1 wt% ferric oxide leads to a better abrasive performance than the addition of 2 wt% q-ferric oxide seeds having a particle size of 200 nm, all other conditions being the same. The skilled person would not have contemplated performing further experiments based on the addition of α-ferric oxide seed particles having particle sizes different from, and in particular lower than that stated in Example 14 since, in view of the teaching of D1 indicated above, he would not have expected that an improvement of the abrasive performance might be achieved by modifying the size of the ferric oxide seed particles.

3.2 The appellant's arguments that the skilled person wishing to achieve an improvement would have performed tests under the conditions and within all the different ranges disclosed in D1, in particular within the particle size range of 80 to 700 nm, cannot be accepted by the board. Firstly, it is observed that a great number of parameters other than the seed particle size could have been modified, including the kind of seed material. Furthermore, according to the established case law of the boards, the relevant question is not whether the skilled person could have arrived at the claimed invention by modifying the closest prior art, but whether he would have done so in the expectation of solving the underlying technical problem or in the expectation of an improvement. In the present case, the technical problem to be solved is not simply the provision of another process for producing abrasive grains, but the provision of a process which leads to abrasive grains having an improved abrasive

performance. In this case, the skilled person would not have performed experiments in all possible directions but would have chosen among the great number of possibilities that or those for which, on the basis of the information given in the prior art, a reasonable chance of obtaining the desired improved abrasive performance existed. As already indicated above, D1 does not contain any suggestion that the abrasive performance might be improved by using  $\alpha$ -ferric oxide seed particles having a particle size different from, in particular lower than that stated in Example 14. In these circumstances, the skilled person would have had no reason to perform experiments in this direction, all the more as the embodiment in which a ferric nitrate solution is used as the precursor for  $\alpha$ -ferric oxide (Example 12) appeared to be promising. Therefore, the appellant's argument that the skilled person would have worked within the range of 80-700 nm disclosed for  $\alpha$ -alumina is based, in the board's view, on an ex post facto approach. For these reasons, the board considers that the claimed process is not obvious in view of the teaching of D1 alone.

3.3 At the appeal stage the appellant no longer relied on the disclosure of D3 in connection with the inventive step issue, but based his objection only on the teaching of D1. The board is also of the opinion that D3, which does not refer to the abrasive performance of the ceramic powder, contains no further information which, in combination with the teaching of D1, would hint towards the claimed solution. Further explanations in this respect are not necessary since this was not disputed. The same remarks apply likewise to the disclosure of D2. The additional prior art documents referred to by the appellant at the appeal stage were

referred to by the appellant at the appeal stage were cited only in connection with the novelty issue and do not relate to the production of alumina-based ceramic abrasive grains. There is also no information in these documents pointing to the claimed process.

- 3.4 It follows from the above that the subject-matter of claim 1 involves an inventive step over the disclosure of the prior art cited by the appellant. Thus, claim 1 meets the requirements of patentability set out in Article 52(1), 54 and 56 EPC.
- 4. Claim 1 being allowable, the same applies to dependent claims 2 to 9 whose patentability is supported by that of claim 1.

#### Order

## For these reasons it is decided that:

The appeal is dismissed.

The Registrar:

The Chairman:

S. Hue

R. Spangenberg