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Datasheet for the decision of 10 July 2006

Case Number: T 0191/02 - 3.3.05

Application Number: 98300472.2

Publication Number: 0857703

IPC: C04B 35/622

Language of the proceedings: EN

Title of invention:
Recycling of refractory material

Applicant:
VESUVIUS CRUCIBLE COMPANY

Opponent:
-

Headword:
Refractory material/VESUVIUS

Relevant legal provisions:
EPC Art. 54, 56, 123(2)

Keyword:
"Novelty (yes)"
"Inventive step (yes)"

Decisions cited:
-

Catchword:
-

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Case Number: T 0191/02 - 3.3.05

D E C I S I O N
of the Technical Board of Appeal 3.3.05
of 10 July 2006

Appellant: VESUVIUS CRUCIBLE COMPANY
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Decision under appeal: **Decision of the Examining Division of the European Patent Office posted 2 April 2001 refusing European application No. 98300472.2 pursuant to Article 97(1) EPC.**

Composition of the Board:

Chairman: M. Eberhard
Members: E. Waeckerlin
J. Willems

Summary of Facts and Submissions

I. The appeal was lodged against the decision of the examining division refusing patent application No. 98 300 472.2 concerning a refractory material.

II. The examining division held that the subject-matter of claims 1, 3 and 10 of the set of claims submitted with a letter dated 19 May 2000 lacked novelty having regard to document US-A-4 140 745 (D1).

III. In the decision the following reasons were given:

D1 concerns the recovery of MgO from scrap lining materials. According to the example scrap brick is crushed so that 80 % of the resulting particles are finer than approximately 5 mm. The material is then steam autoclaved at 15 atm and 205 °C for 16 hours. The application acknowledges that MgO is a hydratable material. The recovered MgO can be reused for making bricks. Therefore claims 1, 3 and 10 are not novel.

IV. The appellant submitted together with the grounds of appeal dated 27 June 2001 an amended version of claim 1 as main request. On 12 July 2001 he submitted in addition a further version of claim 1 as auxiliary request.

The appellant argued that the claimed process was novel and involved an inventive step having regard to the disclosure of D1, since in his view the skilled person had no objective reason to take D1 into consideration.

The appellant requested the reimbursement of the appeal fee by reason of substantial procedural violations.

V. In an annex to the summons to oral proceedings the board raised objections under Article 123(2) EPC as well as objections of lack of clarity under Article 84 EPC against claim 1 of the main and auxiliary request. Furthermore the board expressed doubts under Article 83 EPC regarding the sufficiency of the disclosure of the process as defined in claim 1 of the main and auxiliary request. The board acknowledged the novelty of the process having regard to documents D1 and D2 (DD-A-100 445).

VI. By letter dated 23 May 2006 the appellant submitted his comments, together with three sets of amended claims as the main and two auxiliary requests. He developed and enlarged his argumentation in support of novelty and inventive step.

VII. During oral proceedings, which were held on 10 July 2006, the claims filed with the letter of 23 May 2006 were discussed, as well as various claims containing further amendments. The appellant submitted a new set of claims as main request and withdrew all auxiliary requests as well as the request for reimbursement of the appeal fee under Rule 67 EPC.

Claim 1 of the single request reads as follows:

"*1. A process for the treatment of magnesia-graphite material containing aluminium carbide and aluminium nitride reclaimed from a refractory lining after the end of its useful life comprising applying superheated*

steam to particulate feedstock material formed from the reclaimed material whilst maintaining the feedstock material at a temperature in the range 100 °C to 600 °C in a suitable vessel and wherein a common maximum particle size is 5 mm and the material remains within the furnace for not less than 10 minutes but not greater than 90 minutes."

The independent claim 8 reads as follows:

"*8. Use of a refractory material produced by the process of any one of Claims 1 to 7 as a contribution to a batch of materials for the production of compacts or bricks.*"

VIII. The arguments submitted by the appellant during the appeal procedure can be summarised as follows:

Before the invention was made it was known in the prior art to recycle magnesia-graphite bricks reclaimed from a refractory lining after the end of its useful life by removing front face contamination picked up in service, crushing the bricks to suitable particle sizes and using the particulate material as a component in the current production of fresh bricks.

When this recycling route was followed with magnesia-graphite bricks containing aluminium powder as an antioxidant for the protection of the graphite and the binder system, problems occurred due to cracking of the fresh bricks primarily during the curing process of organic binder systems.

The invention overcomes this problem by treating the reclaimed refractory material with superheated steam under the operating conditions set out in claim 1.

D1 relates to a process for recovering magnesia from refractory materials reclaimed from refractory linings. The reclaimed material is treated with saturated steam, whereby the magnesia contained in the material is hydrated. Subsequently the steam treated material is subjected to a water-leaching process in the presence of carbon dioxide. In the leaching process the impurities of the reclaimed material (silicate, iron oxide) remain in a solid leaching residue when the leaching solution is subjected to filtering, decanting or any other separating process. In other words the impurities are removed from the leaching solution.

In contrast to that the treatment by superheated steam according to the claimed process leads to the preferred hydration of the contaminants originating from reactions of the aluminium, rather than to the hydration of magnesia. Due to the hydration of aluminium carbide and aluminium nitride these contaminants are no longer an obstacle to the recycling of the material from refractory linings.

The skilled person would never have considered D1 when looking for a solution of the problem underlying the invention, because D1 relates to a process wherein the magnesia is hydrated, but not the impurities, and because D1 describes a method of purification which provides for the separation of the impurities from the magnesia.

The same argumentation applies *mutatis mutandis* to the claimed use of the refractory material. The product of the first step of the process of D1, i.e. the material obtained after the treatment with saturated steam, cannot be used directly as a contribution to a batch of materials for the production of compacts or bricks. On the contrary the magnesia, which is in hydrated form, has to be extracted by leaching in the form of the hydrocarbonate, before being precipitated and then regenerated by calcination.

IX. The appellant requested to set aside the decision under appeal and to grant a patent on the basis of the set of claims of the main request submitted during oral proceedings.

Reasons for the Decision

1. *Allowability of the amended claims under Article 123(2) EPC*

The process claims 1 to 7 have the following basis in the application as originally filed:

Claim 1: Claims 1, 3, 4 and 6; page 1, lines 15-23; page 2, lines 17-22; page 4, lines 9-10 and 18-20; page 7, lines 17-19.
Claim 2: Claim 2 as originally filed.
Claim 3: Claim 5 as originally filed.
Claim 4: Claim 7 as originally filed.
Claim 5: Claim 8 as originally filed.
Claim 6: Claim 9 as originally filed.
Claim 7: Claim 10 as originally filed.

The independent claim 8 is based on claim 11 as originally filed.

2. The objections under Articles 84 and 83 EPC (lack of clarity and insufficiency of disclosure, respectively) raised in the annex to the summons to oral proceedings against the claims then on file do not apply to the invention as now defined in the present claims.

3. *Novelty*

3.1 D1 discloses a method of recovering magnesia from impure magnesia-containing scrap material reclaimed from refractory linings (see claims 1, 5 and 19; col. 1, line 66 to col. 2, line 35) and comprising silicate-containing impurities and iron oxide containing impurities. The scrap lining material is crushed to particulate form (see col. 3, lines 1-5; claim 6), for example to a size where 80 % of the particles are finer than approximately 5 mm (see claim 5), and treated in a first step with saturated steam at a temperature within the range of 100 °C to 300 °C for a period of 1 to 50 hours to form $MgO.H_2O$ (see col. 2, lines 4-9 and 22-25; claim 1). Ranges of time and temperature of 6 to 30 hours and 160 to 250 °C, respectively, are said to be sufficient, whereby times and temperatures of approximately 16 hours and 205 °C are preferred (see col. 2, lines 40-47). Subsequently the steam-treated product is leached in a second step with water in the presence of carbon dioxide under pressure to form magnesium hydrocarbonates, whereafter the leaching solution is separated from the solid leaching residues containing the impurities, followed by the

precipitation of the magnesium-hydrocarbonates and the calcination of the material obtained to form highly pure magnesia (see col. 2, lines 9-34).

The claimed process as set out in claim 1 is distinguished from the process of D1 by the following features:
(i) the scrap material which is reclaimed from a refractory lining after the end of its useful life is magnesia-graphite based;
(ii) it contains aluminium carbide and aluminium nitride as contaminants;
(iii) it is treated with superheated steam instead of saturated steam.

3.2 D2 relates to a method for the "activation" of magnesium oxide, viz. the improvement of the sintering ability of magnesium oxide by means of a treatment with steam in the presence of oxygen (air) at elevated temperatures of, typically, about 350 °C for a period of about 15 to 20 minutes, followed by cooling of the product in the wet stream of oxygen (air) (see col. 1, line 30 to col. 2, line 2; col. 2, lines 6-18; claim 1).

The process of claim 1 is distinguished from the method of D2 by the material, viz. magnesia-graphite based material containing aluminium carbide and aluminium nitride. Moreover superheated steam is used for the treatment instead of steam in the presence of oxygen.

4. *Inventive step*

4.1 The board considers that the statements of prior art contained in the application, as further explained by

the appellant during the oral proceedings, represent the closest prior art. According to said statements it was known in the prior art to reclaim magnesia-graphite bricks from a refractory lining after the end of its useful life, to apply a minimal treatment to these bricks to remove front face contamination picked up in service, to crush the bricks to suitable particle sizes and to use the particulate material as a component in the production of fresh products (see page 1, lines 10-13 of the application as originally filed). Furthermore magnesia-graphite bricks containing aluminium powder as an antioxdant to protect the graphite and any other readily oxidisable components, for example carbon black and the binder system, were also comprised in the prior art (see page 1, lines 16-18 of the application as originally filed). When such aluminium containing magnesia-graphite bricks were recycled after the end of their useful life, following the minimal treatment indicated above, problems occurred due to cracking of the products, primarily during the curing process of organic binder systems (see page 1, lines 13-15).

4.2 Starting from this prior art, the problem to be solved by the process of claim 1 can be seen in providing a process for the treatment of waste magnesia-graphite based materials reclaimed from a refractory lining, which enables their recycling and consequent use in the production of fresh batches of material, while avoiding cracking of the products during processing (see page 1, lines 19-21; page 2, lines 8-10).

4.3 It is proposed to solve the above problem by the process as defined in claim 1. In view of the

information given in the description and the drawings, it is credible in the absence of evidence to the contrary, that by treating the material containing the impurities defined in claim 1 with superheated steam and choosing the parameters within the ranges set out in claim 1, the problem as stated above has been effectively solved (see in particular page 7, line 17 to page 8, line 11).

It remains to be investigated whether the claimed solution involves an inventive step.

4.4 As has been explained by the appellant during oral proceedings, the following technical aspects are particularly important in respect of the claimed process:
Under the operating conditions recited in claim 1 the aluminium carbide and aluminium nitride contaminants are hydrated and thus converted into a form that does not cause cracking of the bricks during re-use of the magnesia-graphite material, whereas the magnesium oxide phase, which is in principle also susceptible to hydration, is not affected, at least not to an unacceptable degree (see, in this respect, page 2, lines 1-7; page 2, line 20 to page 3, line 4; page 5, line 24 to page 6, line 14). Thus, the problem contaminants are eliminated or reduced to a level that has no adverse effect on the subsequent use of the material in the production of fresh products. In these circumstances there is no need to remove the contaminants from the magnesia-graphite material, so that they may remain in the material.

4.5 In contrast to that, D1 provides a teaching which points away from the claimed process. According to D1 the purpose of the treatment of the magnesia-containing scrap material by saturated steam consists in the hydration of the magnesium oxide (see col. 2, lines 22-25), because this is a prerequisite condition for the following conversion of the steam treated product into magnesium-hydrocarbonates (see page 2, lines 9-15). Furthermore in D1 the magnesia-containing scrap material is treated with saturated steam. There is no information which could give the skilled person an incentive to replace this treatment by a treatment with superheated steam under the conditions set out in claim 1 in order to solve the technical problem stated above. The board observes that D1 does not even mention the presence of aluminium contaminants such as aluminium carbide and aluminium nitride, and it does not deal with the problem of how cracking of the products during processing can be avoided. According to the present application the cracking is related to the presence of certain aluminium compounds in the waste refractory materials. In order to arrive at the claimed process, the skilled person had first of all to recognise that the aluminium was the cause of the cracking problems, in particular the aluminium carbide and the aluminium nitride formed during the use of the refractory materials. Thereafter the skilled person had to recognise that the cracking of bricks during processing occurs because the aluminium carbide and aluminium nitride contaminants undergo an expansile hydration reaction with the water content or condensation products of the binder systems (see page 1, lines 15-21). And, after having identified the cause of the cracking, the skilled person had to find a way to

overcome this unfavourable phenomenon. None of these steps is disclosed or at least foreshadowed in D1. For these reasons D1 does not provide any indications which would lead the skilled person towards the solution of the technical problem posed.

4.6 D2 is even more remote from the claimed process than D1, since it relates to both a completely different technical problem, namely the activation of magnesium oxide having a low sintering ability, and a different solution, i.e. the treatment of magnesium oxide with steam in the presence of oxygen. Therefore the skilled person had no objective reason to take D2 into consideration when trying to solve the problem underlying the claimed process. But even in the hypothetical case that he would have combined D2 with the closest prior art, he would still not have arrived at the claimed process, since D2 does not teach to use superheated steam.

4.7 Under these circumstances the board considers that neither D1 nor D2, taken alone or in combination with the closest prior art, suggest the combination of operating conditions set out in claim 1. Therefore the claimed process involves an inventive step as required by Articles 52(1) and 56 EPC.

4.8 Concerning the use claim 8 the problem to be solved is also based on the difficulties which arise when magnesia-graphite materials reclaimed from a refractory lining after the end of its useful life are recycled. As mentioned above, it has been found that cracking of the fresh refractory products occurs during processing. Thus, the technical problem underlying the claimed use

with respect to the closest prior art is to overcome these difficulties.

4.9 The proposed solution consists in using magnesia-graphite material, which has been treated by the process of claim 1, as a contribution to a batch of materials for the production of compacts or bricks. In the board's view the proposed solution solves the problem credibly (see point 4.3 above).

4.10 The board holds that the solution is not suggested by the prior art for the following reasons: Both D1 and D2 are completely silent on the problem of cracking. Hence, neither D1 nor D2 contains any information regarding the existence of the unfavourable cracking phenomenon, let alone its causes or any ways and means for overcoming it. The occurrence of cracking and the identification of its causes form part of a disclosure that is presented for the first time in the application and was not known before.
Furthermore the preceding considerations in connection with the process of claim 1 (see point 4.5 above) apply likewise to the use claim 8, which refers to the magnesia-graphite material produced by the process of claim 1. This process leads to a product which enables the recycling of waste magnesia-graphite based materials reclaimed from a refractory lining and solves at the same time the problem of cracking during processing. Neither that process nor the product resulting therefrom is suggested in D1 or D2. Therefore the claimed use cannot be regarded as being obvious to the skilled person. Accordingly it involves an inventive step as required by Articles 52(1) and 56 EPC.

4.11 The process claims 2 to 7 all depend on claim 1. Thus, the patentability of the subject-matter of these claims is supported by claim 1.

Order

For these reasons it is decided that:

1. The decision under appeal is set aside.

2. The case is remitted to the first instance with the order to grant a patent on the basis of the main request, filed during the oral proceedings, the drawings as filed with letter dated 12 February 1998 and a description to be adapted.

The Registrar: The Chairman:

C. Vodz M. Eberhard