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D E C I S I O N
of 19 September 2005

Case Number: T 0133/03 - 3.3.5

Application Number: 95114196.9

Publication Number: 0703198

IPC: C04B 35/528

Language of the proceedings: EN

Title of invention:
Carbon refractory for blast furnace and method for manufacturing such carbon refractory

Patentees:
Nippon Steel Corporation
Nippon Electrode Company, Ltd.

Opponent:
SGL CARBON AG

Headword:
Carbon refractory/NIPPON STEEL

Relevant legal provisions:
EPC Art. 114(2), 56

Keyword:
"Late filed evidence (no)"
"Inventive step (yes)"

Decisions cited:
T 0939/92

Catchword:
-

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Case Number: T 0133/03 - 3.3.5

D E C I S I O N
of the Technical Board of Appeal 3.3.5
of 19 September 2005

Appellant: (Opponent)
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Decision under appeal:
Decision of the Opposition Division of the European Patent Office posted 6 December 2002 rejecting the opposition filed against European patent No. 0703198 pursuant to Article 102(2) EPC.

Composition of the Board:

Chairman: M. M. Eberhard
Members: J.-M. Schwaller
J. H. P. Willems

Summary of Facts and Submissions

I. This appeal lies from the decision of the opposition division rejecting the opposition against European patent No. 0703198.

II. Claims 1 and 2 of the published patent, on which the main request of the present decision is based, read as follows:

"1. A carbon refractory for a blast furnace containing a compound of coarse grains, fine grains, and particulates of carbon aggregates, wherein the coarse-grains aggregate comprises an artificial coarse-grain aggregate, which is obtainable by adding an organic binder to 100 parts of a mixture composed of
- 70- 90 parts of carbon material having high thermal conductivity and a graphite content of 70 % or more, wherein said graphite is in the form of artificial graphite with a particle size of 1 mm or less and/or in the form of flake graphite,
- 5-15 parts of alumina particulates, and
- 5-15 parts of metallic silicon particulates, and
then kneading, molding, baking, crushing, and screening the binder-containing mixture.

2. A method for manufacturing a carbon refractory for a blast furnace by compounding coarse grains, fine grains, and particulates of carbon aggregates, said method using, as the coarse-grains aggregate, an artificial coarse-grain aggregate, which is obtained by adding an organic binder to 100 parts of a mixture composed of

- 70- 90 parts of carbon material having high thermal conductivity and a graphite content of 70 % or more, wherein said graphite is in the form of artificial graphite with a particle size of 1 mm or less and/or in the form of flake graphite,
- 5-15 parts of alumina particulates, and
- 5-15 parts of metallic silicon particulates, and

then kneading, molding, baking, crushing, and screening the binder-containing mixture."

III. According to the decision, the closest prior art was represented by D10 (English translation of JP-B-61003299), relating to the manufacture of graphite-based carbon refractory material for blast furnaces, graphite being used therein both in the form of natural flake graphite and in the form of artificial graphite. The problems associated with artificial graphite were its erosion by dissolution into molten iron and the presence of large pores into which molten iron penetrates easily, whereas flake graphite had a strong orientation problem which caused lamination, spring back during moulding and cracks. The inclusion into carbon-refractory material of an artificial coarse-grain aggregate as defined in claims 1 and 2 was considered as solving the above problems. The opposition division held that as a result of moulding and crushing the flake graphite was isotropically contained in the artificial grains, thus solving the problem linked with flake graphite. Furthermore, as a result of keeping the particle size of artificial graphite at 1 mm or less (and the inclusion of silicium and alumina), the pit-like erosion was avoided and therewith the problem associated with artificial graphite.

The claimed subject-matter was not obvious in view of the combination of D10 with D9 (DE-A-3714398) because *inter alia* the latter dealt with the problem of preventing segregation of materials when the carbon content was high, i.e. a problem which had no close relationship with the problem of orientation associated with flake graphite.

The opposition division, although noting the broad wording of the claims, considered that the stated problem was solved over their whole breadth, mainly because the skilled person had to interpret the claims as essentially limited to compositions containing a proportion of artificial coarse-grain aggregate sufficient for significantly/measurably providing its beneficial properties to the composition (as compared to the properties of the composition without artificial coarse-grain aggregate).

IV. With the grounds of appeal, the appellant (opponent) disputed the inventive step of the claimed subject-matter in the light of D10 in combination with D9. He further filed a test report accompanied by copies of photographs intended to show the damage generated by molten iron on a carbon refractory containing various amounts of artificial coarse-grain aggregate prepared according to Example 1 of the patent in suit.

V. With a letter dated 26 August 2005, the respondents (proprietors) asked the board to disregard said test report and filed three sets of amended claims as 1st to 3rd auxiliary requests. In a communication of the board, the amendments to claims 1 and 2 of set of claims III

were objected to under clarity and the allowability of the said amendments under Article 123(2) EPC was put in question. The respondents submitted six new sets of claims IV-IX as 4th to 9th auxiliary requests with a letter dated 8 September 2005.

VI. Oral proceedings took place on 19 September 2005.

VII. The appellant presented mainly the following arguments:

The test report filed with the grounds of appeal was a direct response to the content of the decision, therefore it could not be considered as late filed. The graphite grains "Graphitkörner" used in the test report were commercially available graphite grains, which usually are made of artificial graphite. The said report demonstrated that no improvement in erosion resistance to molten iron was observed when only minor quantities of an artificial coarse-grain aggregate prepared according to Example 1 of the patent in suit were introduced in a conventional carbon refractory material. Thus, since some of the claimed embodiments failed to solve the problem addressed in the patent in suit, they lacked an inventive step (see e.g. T 939/92).

The closest prior art was represented by D10 which disclosed a graphite-based carbon refractory including SiC as an additive. Although D10 was silent about alumina, this compound was disclosed as an additive for graphite-based carbon refractories in US-A-4282288 (D1). D1 claiming the same priority of the Japanese application 53-149661 as the Japanese publication No. 55-085461 referred to in D10, the teaching of D1 may

thus be considered as suggested in D10. D9 disclosed a process for producing a carbon-based refractory in which artificial coarse-grain aggregates were prepared by mixing refractory raw materials, additives, humidity, liquid hydrocarbons and a chemical binder. A plastic mass, which may also contain graphite, was made from said mixture, let harden, separated into pieces (i.e. crushed) and classified according to the size (i.e. screened). The molding and the baking steps were implicit to the skilled person. It did not matter that the purpose of the artificial coarse-grain aggregate in D9 was to prevent segregation problems occurring with high carbon contents in the refractory whereas the problem of orientation of graphite was acknowledged to be important in the patent in suit, since the orientation reduction would be automatically achieved during the crushing operation in D9, thereby creating a one-way street situation. Thus the subject-matter claimed lacked an inventive step.

VIII. The respondents (patentees) essentially argued as follows:

The appellant's test report should have been filed earlier, namely at the opposition stage, and should in consequence be disregarded as not being submitted in due time. Said test report was furthermore not clearly comprehensible, because on the one hand, it was unclear whether the samples tested were indeed carbon refractories according to claim 1, and on the other hand, no information was given as to why the particle size of the artificial coarse grain aggregate used in the samples was different to that prepared in Example 1 of the patent in suit. Moreover, the respondents could

not prepare counter experiments because the information that the graphite used in the tested samples was a commercial artificial one was obtained only at the oral proceedings. With respect to the copies of photographs filed with the test report, nothing meaningful could be seen thereon or deduced therefrom. As regards specifically the samples with low amounts of artificial coarse-grain aggregate, the effect of the latter in the refractory being essentially concentrated around the individual aggregate grains, this microscopic effect could not be visualised by means of photographs. The test report was therefore of no relevance for the assessment of patentability. The essential feature of the invention being the artificial coarse-grain aggregate itself, there was therefore no necessity of unduly limiting the subject-matter claimed because an effect may be obtained already with the very first grain of the inventive aggregate.

Document D9 could not be combined with D10 because D9, on the one hand, was not concerned with carbon refractories, and on the other hand, addressed a different problem to that of the patent in suit.

IX. The appellant requested that the decision under appeal be set aside and that the patent be revoked.

The respondents requested that the appeal be dismissed and that the patent be maintained as granted (main request) or, alternatively, that the case be remitted to the first instance for further prosecution or, alternatively that the patent be maintained on the basis of the set of claims filed with letter of 26 August 2003 as auxiliary requests I to III, or the

sets of claims filed with letter of 8 September 2005 as auxiliary requests IV to IX.

Reasons for the Decision

1. The appeal is admissible.

2. Admissibility of the test report.

2.1 In the annex to the summons to oral proceedings in the opposition proceedings, the opposition division pointed out that the claims neither set any limits to the proportion of coarse grains nor to their size and raised an objection of lack of inventive step against the subject-matter of claims 1 and 2, arguing that it was doubtful whether the beneficial effects of the artificial coarse-grain aggregate could also be detected in carbon refractories containing very little coarse aggregate (e.g. only 1 % of the coarse grains) or containing a very fine "coarse" aggregate.

2.2 The patentee argued in a letter dated 30 September 2002 that the essential feature of the invention was the artificial coarse-grain aggregate itself (and not the proportion thereof in the carbon refractory) and that the improvement over prior art carbon refractories already began with the addition of the first grain of said artificial coarse-grain aggregate.

2.3 Since the lack of inventive step objection referred to in item 2.1 *supra* was raised for the first time in the summons to oral proceedings, the opponent could have reasonably expected a decision in its favour at this

stage of the procedure and had thus no reason to file a test report before receipt of patentee's submissions of 30 September 2002. Since these were sent by the EPO only on 11 October 2002, i.e. less than one month before the oral proceedings scheduled for 5 November 2002, in the board's view, if the opponent intended to react e.g. by means of experimental tests, this lapse of time was relatively short for organising and carrying out such tests. Furthermore, a favourable decision could still be expected by the opponent at this time. In these circumstances, the board considers that the test report filed with the grounds of appeal can be regarded as a direct response to the arguments on which the decision to reject the opposition was based. The test report cannot thus be held as not having been filed in due time within the meaning of Article 114(2) EPC.

3. *Inventive step of the main request*

3.1 D10, which - as acknowledged by all the parties - represents the closest prior art, discloses a carbon refractory for blast furnaces prepared by a process comprising mixing and kneading an organic solvent with a carbon refractory material aggregate, then adding a phenol resin powder as an organic binder and kneading, forming, drying and baking these materials. The aggregate may comprise 40-60 parts of flake graphite with a particle size of 0.3-3 mm, 15-30 parts of coke type artificial graphite with a particle size of 0.1-4.5 mm, 10-20 parts of carbide with a particle size of 0.074 mm and 5-15 parts of metal powder (see claims 1 and 6); the carbon refractory thus contains the three kinds of particles broadly defined in claim 1 as

"coarse grains, fine grains and particulates of carbon aggregates". In embodiment 1, ethylene glycol is used as the organic solvent and admixed with a mixture of 50 parts of said flake graphite, 25 parts of said above coke type artificial graphite, 15 parts of said silicon carbide and 10 parts of metallic silicon before adding the phenol resin powder. The board notes that it is common general knowledge that graphite is a carbon material having high thermal conductivity.

3.2 The appellant argued that alumina was suggested in D10 as an additive for graphite-based carbon refractories because such an additive was disclosed in D1, a document claiming the same priority of the Japanese application 53-149661 as the Japanese prior publication 55-085461 referred to in D10. The board does not share this view, because although the Japanese publication 55-085461 is cited as a prior art in D10, the latter does neither make any reference to the additives disclosed in this Japanese publication, nor teaches that said additives (in particular alumina) may be a substitute for the carbide additive used in D10. The board can thus not accept the argument that D10 discloses or suggests the presence of alumina as an additive in the graphite-based refractory disclosed therein.

3.3 As argued by the respondents and also disclosed in the patent in suit at paragraph [0008], the carbon refractories produced according to the method of D10 have the disadvantage that they present high thermal conductivity and bending strength in the direction parallel to alignment of particles but not in the perpendicular direction. Furthermore, according to

paragraph [0015] of the patent in suit, if the particle size of the artificial raw graphite material exceeds 1 mm, the artificial graphite grains exposed on the machined surface of the blast furnace refractory are dissolved preferably, leading to the progress of pit-like erosion.

The patent in suit proposes to solve this problem by the carbon refractory according to claim 1, which differs from that of D10 in particular by the presence of an artificial coarse-grain aggregate obtainable by the method and the formulation as defined in claim 1.

3.4 Whether the incorporation into a carbon refractory of such an artificial coarse-grain aggregate solves the problem stated above is in the board's view evidenced as follows.

The examples of the patent in suit represent the three separate embodiments defined in claim 1 in the following respects. Examples 1 and 2 illustrate formulations of an artificial coarse-grain aggregate in which graphite is in the form of flake graphite; Examples 3 and 4 correspond to an artificial coarse-grain aggregate in which graphite is in the form of artificial graphite with a particle size of 1 mm or less and Example 5 illustrates the third embodiment of claim 1 according to which the graphite of the artificial coarse-grain aggregate is in the form of flake graphite and of artificial graphite with a particle size of 1 mm or less.

The results given in Tables 2 and 4 of the patent in suit show that all the samples containing an artificial coarse-grain aggregate as claimed equally have a "smooth" surface after exposure to molten iron in comparison with the surface of the sample of comparison Example 4, on which "large pits" were observed. Comparison Example 4 was made using as the coarse grains fraction an artificial graphite with a grain size of 1-5 mm, instead of an artificial coarse-grain aggregate having the same size. Examples 3 and 4 show that the substitution of artificial graphite coarse grains as exemplified in comparison Example 4 by artificial coarse-grain aggregates as defined in claim 1, wherein graphite is in the form of an artificial graphite having a particle size of 1 mm or less, has a beneficial effect on pit-like erosion, as evidenced by the disappearance of the large pits observed in comparison Example 4. Since all the samples of carbon refractory made according to the invention have such a "smooth" surface condition, it is credible for the three embodiments covered by the claims that erosion, in particular pit-like erosion resulting from the presence of artificial graphite grains with a particle size > 1 mm, has been reduced by use of the claimed artificial coarse-grain aggregate.

Examples 1, 2 and 5 (which use an artificial coarse-grain aggregate containing flake graphite) provide evidence when compared with comparison Example 2 (which contains flake graphite but no artificial coarse-grain aggregate according to the invention) that the anisotropy in the properties (orientation problems) due to the presence of flake graphite is substantially reduced by substitution of the latter by the claimed

artificial coarse-grain aggregates containing the graphite in the form of flake graphite; see in this respect the improvement of the perpendicular bending strength and of the perpendicular thermal conductivity. As a consequence, the second aspect of the problem is thus equally solved for the three embodiments covered by the claimed carbon refractory.

3.5 The appellant contested - by means of the test report filed with the grounds of appeal - that the effects produced by the artificial coarse-grain aggregate as claimed would occur over the whole breadth of the claims, arguing in particular, that in view of the large size of carbon refractories for blast furnace (up to 600 x 700 x 2500 mm; see [0012] of the patent in suit), no effect would take place with low amounts of artificial coarse-grain aggregate in the refractory. This was reflected e.g. by the copies of photographs of samples A/11 and B/15 which showed that with respectively 10 grains and 400 g of the artificial coarse-grain aggregate in 40 kg of the solids mixture, no improvement in erosion resistance by molten iron would be observed when compared with the reference sample A/3, which contained no artificial coarse-grain aggregate.

In the board's view, although some erosion differences between the samples may be identified on the copies of the photographs, their poor quality makes it difficult without having seen the samples to objectively assess and draw conclusions as to whether the erosion resistance to molten iron was improved or not, in particular the pit-like erosion. As regards pit-like erosion, the appellant argued at the oral proceedings

that the dark spots on the copies correspond to pits due to the dissolution of coarse grains from the surface of the samples. The board observes that although some dark spots may be identified on the copies, it is questionable whether they can be attributed to erosion pits. The poor quality of the copies in any case does not allow a quantitative assessment of the pit-like erosion. The board further observes in this context that the appellant gave a quantitative evaluation of the erosion expressed as a weight loss for sample A3 (which does not contain any artificial coarse-grain aggregate) when left in contact with molten iron for respectively 30 minutes, 1 hour and 2 hours; however no such comparative data were provided for the other samples which contained the artificial coarse-grain aggregate. In the absence of such quantitative evaluation, in particular for samples A11 and B15, and only on the basis of a copy of poor quality of the photographs, the board cannot conclude that no improvement at all was achieved with low amounts of artificial coarse-grain aggregate in the composition.

The respondents argued at the oral proceedings that when only one or some grains of the artificial coarse-grain aggregate were present in the carbon refractory, the improvement as regards erosion was achieved in the area around the grain(s), i.e. in the neighbourhood of the grains. The appellant itself confirmed at the oral proceedings that it did not dispute the achievement of an improvement at the microscopic scale, i.e. in the area neighbouring each of the artificial coarse-grain aggregates. What was contested was the fact that an erosion improvement was also obtained at the

macroscopic scale with very low amounts of artificial coarse-grain aggregate in the composition, such as in sample A11 (containing 10 grains of artificial coarse-grain aggregate in 40 kg of raw materials) or sample B15 (containing 1 wt. % of artificial coarse grain aggregate). However, as pointed out above, no quantitative evaluation was provided which would show in a convincing way that no improvement at all was obtained with only very low amounts of the said aggregate. Therefore the appellant's arguments concerning the test report cannot change the finding in point 3.4 above that the technical problem has actually been solved by the claimed carbon refractory.

Since the board is not convinced by the above evidence that an effect may not be obtained already with very small amounts of the artificial coarse-grain aggregate claimed, there is no need to comment on the decisions cited by the appellant, in particular T 939/92 (OJ 1996, 309), which in any case concerns a situation different from the present case. The board nevertheless points out that in the present case the technical contribution of the patent in suit to the art is the substitution of prior art coarse graphite grains by an artificial coarse-grain aggregate as defined in claim 1, i.e. the artificial coarse grain aggregate itself and not the amount thereof in the refractory (see also the considerations on inventive step hereinafter). Therefore a further limitation of the claims to the amount of artificial coarse-grain aggregate used in the examples would be an undue limitation of the scope of protection of the patent in suit.

3.6 The appellant's argument that claim 1 would not involve an inventive step because the skilled person would combine the teaching of D9 with the content of D10 and thus arrive at the subject-matter claimed also did not convince the board for the following reasons.

D9 (claim 1; column 1, line 65 to column 2, line 4) concerns a process for manufacturing a carbon-containing refractory. In the embodiment described at column 4, lines 20-42, an intermediate carbon-containing product which, for the sake of argumentation may be called "artificial coarse-grain aggregate", is obtained by mixing refractory raw materials, additives, humidity, liquid hydrocarbons and a chemical binder. A plastic mass, which may also contain graphite, was made from said mixture, let harden, separated into pieces and classified into different sizes. The board notes that these last two steps may be assimilated to crushing and screening operations, respectively. However, that a baking step is implicit from the passage at column 4, lines 20-32 cannot be accepted by the board, because the German word "aushärten" is not equivalent to "baking". The artificial coarse-grain aggregate obtained by the above screening operation is then, in admixture with plastic materials and humidity, molded and dried and the resulting green body is burnt in a reducing atmosphere to get said carbon-based refractory.

The appellant argued that it did not matter that the purpose of the artificial coarse-grain aggregate in D9 was to prevent segregation problems occurring with high carbon contents in the refractory whereas the patent in suit was concerned with the problem of orientation of

graphite, because the reduction in orientation would be automatically achieved during the crushing operation in D9, thereby creating a one-way street situation. The board cannot follow this argument because D9 neither discloses nor suggests any of the specific graphite varieties, in particular flake graphite, defined in claim 1 of the patent in suit. D9 being thus silent as to the kind of graphite used, it can obviously not suggest that the crushing operation would reduce the orientation of flake graphite and no one-way street situation can thus be created. In fact, the purpose of an artificial coarse-grain aggregate in D9 is the prevention of segregation problems occurring with high carbon contents in the refractory, i.e. a problem different from that addressed in the patent in suit, which is concerned with either orientation of flake graphite or erosion by molten iron. The board is finally also not convinced that D9 relates to carbon refractory of the kind disclosed in D10 or in the patent in suit, because as can be inferred from column 1, line 65 to column 3, line 2 of D9, this document concerns refractory products which not only contain carbon but also comprise refractory materials other than carbon.

Accordingly, since in D9 the incorporation of an artificial coarse-grain aggregate in the preparation process of the refractory is recommended for solving a different problem existing in a different type of refractory and since D9 contains no information suggesting that such an artificial coarse-grain aggregate might solve the problem stated above for carbon refractories, the skilled person would have no incentive to apply the teaching of D9 to the carbon

refractory of D10 with the expectation to solve the said technical problem. Furthermore the combination of the teaching of D9 with the content of D10 would not lead to the claimed subject-matter as none of these documents discloses an artificial coarse-grain aggregate containing alumina particles.

Although D1 may suggest the use of alumina as an additive for improving *inter alia* erosion resistance of a graphite-based refractory article (column 5, lines 39-42), this document does not disclose the use of an artificial coarse-grain aggregate of the type defined in claim 1. D1 can thus alone or in combination with the teaching of D10 and D9 not lead in an obvious way to the subject-matter presently claimed.

3.7 The remaining documents cited during the opposition proceedings were not relied upon by the appellant at the appeal stage. In the board's judgment they contain no further information which would point towards the claimed solution of the problem stated above.

3.8 In view of the arguments developed in items 3.1 to 3.7, the board considers that the subject-matter of claim 1 is not obvious to a person skilled in the art and therefore its subject-matter involves an inventive step (Article 56 EPC).

3.9 The considerations indicated above in connection with the inventive step of the claimed carbon refractory apply analogously to the method for manufacturing said refractory according to claim 2, which method also involves the use of the specific artificial coarse-grain aggregate as defined in claim 1. Therefore the

subject-matter of this claim also meets the requirements of inventive step.

Order

For these reasons it is decided that:

The appeal is dismissed.

The Registrar: The Chairman:

A. Wallrodt M. Eberhard