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**D E C I S I O N
of 31 May 1994**

Case Number: T 0152/91 - 3.3.2

Application Number: 86900244.4

Publication Number: 0215123

IPC: C04B 7/147

Language of the proceedings: EN

Title of invention:

Hydraulic hardening material and method of manufacturing the same

Applicant:

Nippon Kokan Kabushiki Kaisha

Opponent:

-

Headword:

Finer slag/NIPPON

Relevant legal norms:

EPC Art. 56

Keyword:

"Inventive step (no) - obvious to try"

Decisions cited:

-

Headnote/Catchword:

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Case Number: T 0152/91 - 3.3.2

DECISION
of the Technical Board of Appeal 3.3.2
of 31 May 1994

Appellant: Nippon Kokan kabushiki Kaisha
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Decision under appeal: Decision of the Examining Division of the
European Patent Office dated 26 June 1990, posted
on 14 September 1990 refusing European patent
application No. 86 900 244.4 pursuant to
Article 97(1) EPC.

Composition of the Board:

Chairman: P. A. M. Lançon
Members: M. M. Eberhard
E. M. C. Holtz

Summary of Facts and Submissions

- I. European patent application No. 86 900 244.4 was refused by a decision of the Examining Division. The decision was based upon the sets of amended claims filed on 18 October 1988 (main request) and on 26 June 1990 (auxiliary request).
- II. The grounds for the refusal were that the subject-matter of the process and product claims lacks an inventive step. The following documents were cited in the examining procedure either by the Examining Division or by the Appellant:
- (1) EP-A-021 681
 - (2) FR-A-1 335 026
 - (3) Zement-Kalk-Gips, 1980, vol. 33, Sept., No. 9, pages 452-455
 - (4) FR-A-930 852
 - (5) Ratgeber für Zement Ingenieure, page 161, 1982
 - (6) 8th International Congress on the Chemistry of Cement, Rio de Janeiro, 22-27 September 1986, Theme 3, vol. IV, pages 239-244
 - (7) NKK Giho, No. 115, 1986, pages 97-105.

According to the decision, the technical problem to be solved was to improve the hydraulic hardening properties of blastfurnace slag, in particular the bending and compressive strength of cementitious compositions containing 60% wt ordinary Portland cement and 40% wt blastfurnace slag. The Examining Division took the view that the solution according to Claim 1 of the main request, namely classifying the milled slag to smaller particle sizes than in document (3), such that a Blaine surface of 6000 to 12000 cm²/g is obtained, did not involve an inventive step. It belonged to the general

knowledge of the skilled person that the strength of cured cementitious compositions increased when the particle size of the cement particles was reduced as illustrated by document (1) in connection with the flexural strength. In the Examining Division's view, although the Blaine values of the classified cement particles were not disclosed in (1), they would doubtlessly fall within the range of 6000 to 12000 cm²/g stated in Claim 1. According to the figures of the present application, a classified cement powder with a mean particle size of about 5 µm has a Blaine specific surface of 7000 cm²/g.

It was further held that the chemical composition and the hydraulic properties of blastfurnace slag were not so different from those of ordinary Portland cement that the teaching of document (1) could not be applied to blastfurnace slag. Blastfurnace slag was much more closely related to ordinary Portland cement than calcium sulphate hemihydrate cement. Thus, if the same effect was observed for both cements, the skilled person would certainly have expected the same effect for blastfurnace slag. Moreover not only (1) but also document (2) disclosed the use of a classifier in order to reduce the particle size of a cement.

The slag cement of Claim 7 was also considered as lacking an inventive step since blastfurnace slag was nearly exclusively used in the art in combination with Portland cement as shown by documents (3), (4) and (5). The independent Claims 1, 6 and 9 of the auxiliary request also did not meet the requirement of inventive step since a cut size of 10 µm was already disclosed in document (1).

III. The Appellant lodged an appeal against this decision and made reference to the six following documents in the appeal statement:

- (8) "Fundamental knowledge of cement and concrete", Kashima Shuppan-Kai (1976), pages 58 to 60,
- (9) 41st Cement Technology Meeting, 1987, pages 34 to 35,
- (10) Proceedings of the International Symposium, Rilem, 1990, pages 440 to 448,
- (11) Material and Process, Vol. 1, 1988, page 993,
- (12) Proceedings of 2nd World Congress Particle Technology, 1990, Kyoto,
- (13) NKK News, vol. 29, No. 8, 1989.

In a communication of the Board pursuant to Article 11(2) RPBI the Appellant was informed that it was questionable whether Claims 8, 9 and 13 of the main request and Claims 7, 8, 11 and 12 of the auxiliary request fulfil the requirements of Article 123(2) EPC. The Appellant's attention was also drawn to an inconsistency between Claim 1 and Claims 4 and 5 of the main request.

IV. Oral proceedings were held on 31 May 1994. At these proceedings the Appellant submitted three amended sets of claims as main request, first auxiliary request and second auxiliary request. Claim 1 of the main request reads as follows:

"A method of manufacturing a hydraulic hardening material, comprising the following steps:

- milling vitreous blast furnace slag and
- classifying the milled slag to provide slag powder having a Blaine specific surface area of 6000 to 12000 cm²/g."

Claim 1 of the first auxiliary request differs therefrom by the incorporation of the terms "at a cut size of 5 to 10 µm" after the terms "milled slag" in the classifying step.

Claim 1 of the second auxiliary request recites all the features of Claim 1 of the first auxiliary request plus the following subsequent mixing step: "thereafter mixing the slag powder in an amount of 40 parts by weight and 60 parts by weight of ordinary Portland cement".

- V. The Appellant's arguments put forward in the appeal statement and at the oral proceedings in connection with inventiveness can be summarised as follows:

The technical problem underlying the invention was to provide a hydraulic hardening material having an improved strength at early and later stages. The product of document (3), which has been obtained by a milling step and a subsequent auxiliary classifying step, exhibits a Blaine surface area of 3800 cm²/g. It can be derived from document (6), in particular from figure 2, that an improvement of the compressive strength is achieved with the blastfurnace slag of the invention over the product of document (3).

Document (1) is silent as to the importance of the Blaine surface area of the cement and does not mention the use of a blastfurnace slag as hydraulic material. The improvement of strength is related to the size of the particles, not to the Blaine surface area of the

cement. Contrary to the Examining Division's assumption, the reduction of the particle size of the cement particles does not automatically give outstanding products, but rather brings about considerable difficulties, for example, reduction in the flowability of the concrete or mortar, higher milling costs, complications in the drying step. This is described, for example, in document (8). Document (1) cannot make the invention obvious when combined with (3) since it does not teach the importance of the Blaine surface area.

Certainly, blastfurnace slag is closer to the ordinary Portland cement than calcium sulphate hemihydrate cement as far as the chemical composition alone is concerned. However, the ordinary Portland cement is a polycrystalline material consisting of several kinds of crystals such as tricalcium silicate, β -dicalcium silicate and tricalcium aluminate, whereas blastfurnace slag is of different nature, namely it is glassy and amorphous. Moreover, the behaviour of slag during the size reduction and its hydraulic reaction mechanism have not yet been clarified theoretically in many aspects. In view of the technical knowledge at the priority date, it is not reasonable to state that the effect produced by the crystalline calcium sulphate hemihydrate or the ordinary Portland cement can be expected practically in the same manner in the glassy blastfurnace slag. There seems to be nothing in the cited documents which could support such an opinion. In this connection, reference is made to document (9) in which it is explained that if the pulverised cement composition is classified, segregation of the components takes place. As blastfurnace slag is formed of a single substance which cannot be compared with the other materials, its size reduction and classification cannot be handled on the same level as those of ordinary cement. Size reduction

may be usual to increase the strength of cement where the particle size is close to the ordinary range, for example 3000 to 4000 cm²/g of Blaine specific surface area. However, the particle size of blastfurnace slag used in the present invention differs considerably from this ordinary range. Therefore, the characteristics of the blastfurnace slag according to the invention cannot be expected.

Document (2) is directed to producing cement, wherein cut size material is obtained having a Blaine surface area of 7000 to 8000 cm²/g by classifying milled cement. The technique of classifying is applied to the cement mixture itself, but not to any blastfurnace slag. The characteristics of the present invention cannot be expected from the technique as disclosed in (2).

The slag having a high fineness tends to be used in very special fields rather than in the conventional cement-concrete technology: see documents (10) to (13). From document (11) it is clear that the strength of the slag is **not** necessarily increased when the fineness of the slag increases.

- VI. The Appellant requested that the decision under appeal be set aside and that a patent be granted on the basis of the main request, alternatively, one of the two auxiliary requests, submitted in the oral proceedings.

Reasons for the Decision

1. The appeal is admissible.
2. The three amended sets of claims submitted at the oral proceedings meet the requirements of Article 123(2) EPC.

In particular the "cut size of 5 to 10 μm " is supported by the application as originally filed, page 3, lines 29 to 30, and the mixing of 40 parts by weight of classified slag powder with 60 parts by weight of ordinary Portland cement finds a support at page 6, lines 7 to 8.

Main request

3. The Appellant argued at the oral proceedings that in the alternative where gypsum is added to the vitreous blastfurnace slag before milling (cf. Claims 4 and 5) the Blaine specific surface area of 6000 to 12000 m^2/g of the classified powder is that of the slag powder alone and not of the mixture. In view of these arguments the Board is satisfied that the features recited in Claims 4 and 5 are in agreement with those of Claim 1. Therefore, these claims fulfil the requirements of Article 84.
4. The subject-matter of Claim 1 is new with respect to the cited prior art. This was also acknowledged by the Examining Division.
5. In the Board's opinion document (3) represents the closest prior art. This document discloses a slag cement grinding plant using the closed circuit grinding technique. The slag cement is prepared by milling blastfurnace slag, clinker and gypsum in a mill and thereafter classifying the mixture in a cyclone air separator. During experimentation with the cyclone separator the conditions were controlled such that the final product exhibits a Blaine surface area of 3800 cm^2/g (cf. page 452, summary, figures 1, 2, 4 and corresponding description thereof; page 454, point 6; page 455, left-hand column).

Starting from this prior art, the technical problem underlying the application can be seen in providing a process of manufacturing a hydraulic hardening material having an improved strength at early and later stages, as indicated by the Appellant.

According to Claim 1 it is proposed that this problem be solved by milling vitreous blastfurnace blast and classifying the milled slag to provide a slag powder having a Blaine specific surface area of 6000 to 12000 cm²/g. Thus, the claimed solution mainly differs from the process of document (3) by the classifying step being carried out in such conditions that a finer slag powder, i.e. a powder with a Blaine surface area of 6000 to 12000 cm²/g, is obtained.

Document (6), which is a post-publication from the four inventors of the present patent application, relates to the properties of very fine blastfurnace slag prepared by a process comprising a classification step of the blastfurnace slag identical to the claimed process. The results reported in this document, in particular the curves of figure 2 at page 242, show that the Portland blastfurnace cements containing 40 wt% of blastfurnace slag which has been classified to provide slag powders with a Blaine surface area of 6000, 8000 and 12000 cm²/g respectively, exhibit an improved compressive strength at 3 days, 7 days and 28 days over a Portland blastfurnace cement containing the same amount of a classified slag powder with a Blaine surface area of about 4000 cm²/g. Having regard to these comparative examples, the Board is satisfied that the problem stated above has been plausibly solved by the claimed process.

6. It remains to be considered whether or not the claimed solution was obvious to the skilled person in view of the available prior art.
- 6.1 Document (3) itself discloses classifying the blastfurnace cement such that the final product has a Blaine surface area of 3800 cm²/g. It is not suggested in this document that by classifying the blastfurnace slag at a cut size which provides a slag with a higher Blaine surface area of 6000 to 12000 cm²/g, the strength of the blastfurnace cement containing this finer slag might be improved.
- 6.2 Document (1) relates to a hydraulic cement composition and addresses the problem of improving the strength of cementitious products produced therefrom, in particular their flexural strength. According to this document the properties of such products are improved where the hydraulic cement in the composition has a weight average mean particle size of less than 20 µm, preferably in the range of from 2 µm to 15 µm, i.e. a mean particle size lower than that conventionally used. This reduced mean particle size is produced by particle size classification techniques, e.g. by air classification or by sieving a commercially available cement. The latter may be first ground and then classified (c.f. page 1, line 16 to page 2, line 15; page 3, lines 15 to 20; page 4, lines 1 to 2 and 19 to 27; Claims 1 and 3). In example 1 it is shown that a classified Portland cement with a mean particle size of 5 µm which has been prepared by classifying ordinary Portland cement at a cut size of 10 µm in an air classifier, leads to a cured product having a flexural strength which is more than twice the strength obtained with an ordinary Portland cement classified to a mean particle size of 25 µm under otherwise identical conditions. Examples 2 and 3 were

carried out with a classified calcium aluminate cement having a mean particle size of 8 μm and with a classified calcium sulphate hemihydrate cement having a mean particle size of 3 μm respectively in place of classified Portland cement. The products obtained with these classified cements exhibit flexural strengths which are greatly improved over those of the products prepared from the corresponding unclassified cements. Thus, it can be inferred from document (1) that by classifying a commercially available hydraulic cement at a cut size of for example 10 μm in order to reduce its mean particle size to the desired range of 2 μm to 15 μm (optionally after first grinding the cement), the strength of the products resulting from this classified cement, in particular the flexural strength, is improved, and that this improvement is achieved with different kinds of hydraulic cements such as Portland cement, calcium aluminate cement, calcium sulphate hemihydrate cement.

Furthermore, document (2), which also deals with the problem of improving the strength of cements, discloses a cement which leads to mortars and concretes having improved flexural, tensile and compressive strengths at 2, 7, 28 and 90 days. This cement is characterised by a higher fineness than usual, i.e. a Blaine specific surface area of 7000 to 8000 cm^2/g . This fineness may be achieved either by grinding the clinker in the conventional industrial ball mill or by classifying a cement of usual fineness in a cyclone separator (cf. page 2, left-hand column, lines 29 to 38; right-hand column, lines 20 to 29, summary points 1 and 2).

In view of documents (1) and (2) the skilled person faced with the problem of improving the strength at early and later stages of the blastfurnace cement of

document (3) would have contemplated applying the teaching of these documents to blastfurnace cement or to blastfurnace slag. Thus, he would have considered classifying the blastfurnace slag at a particular cut size (for example 10 μm) in order to reduce its mean particle size to the range stated in (1) or to increase its fineness measured by the Blaine permeability according to the teaching of (2). Doing so, the skilled person would have arrived at the claimed process. In this context it should be noted that document (1) is indeed silent about the Blaine specific surface area of the classified cement as pointed out by the Appellant. However, it was not contested by the Appellant that the classified cement having a mean particle size of 5 μm , which was produced by classifying a commercially available ordinary Portland cement at a cut size of 10 μm as disclosed in (1), exhibits a Blaine surface area lying within the range of 6000 to 12000 cm^2/g . Furthermore, the classification step is defined in document (2) with reference to the fineness of the cement measured by the Blaine permeability. Finally, it is well known to determine the fineness of cements either by the Blaine permeability method (used in (2)) or by sieve analysis or air elutriation (used in (1) to determine the mean particle size: see page 4, lines 31 to 34). In these circumstances, no inventive step can be seen in the fact that the classification step is defined in Claim 1 with reference to the Blaine specific surface area.

- 6.3 The Appellant argued that the skilled person would not have applied the teaching of documents (1) and (2) to the vitreous blastfurnace slag since ordinary Portland cement and blastfurnace slag are very different from each other (see point V above) and the effect produced by the crystalline calcium sulphate hemihydrate or the

ordinary Portland cement could not be expected in the same manner with the glassy blastfurnace slag. These arguments do not convince the Board that the skilled person faced with the problem stated above would not have been encouraged to classify the blastfurnace slag at a specific cut size to reduce its mean particle size or to increase its fineness for the following reasons:

According to document (1) the improvement of strength is obtained not only with products made from a classified Portland cement but also with products prepared from a classified calcium hemihydrate cement, i.e. a cement which is very **far removed** from Portland cement as regards the **composition, the components of the cement, the hydraulic reaction mechanism and the segregation** during classification, if any. Furthermore, the teaching of document (1) is not limited to the exemplified cements but also covers other hydraulic cements. Thus, it derives from page 4, second paragraph, that the invention disclosed in (1) is applicable to any material which sets and hardens in the presence of water. It cannot be inferred therefrom that the improvement of strength is achieved mainly with material for which segregation of the components occurs during classification. In these circumstances, it is the Board's opinion that the skilled person would have tried to classify the blastfurnace slag to a finer slag powder with the expectation that this classification step could also lead to an improvement of strength.

- 6.4 The Appellant's arguments that the improvement of strength is related to the size of the particles in document (1) and that a reduction of the particle size of the cement particles brings about considerable difficulties as mentioned in document (8) and does not automatically give outstanding products, cannot change

the preceding findings. It is indeed well known and also suggested in document (8) that an optimum limit of fineness exists and that beyond this limit, i.e. with "unduly fine" particles, it is necessary to use a larger amount of water per unit volume of the concrete. However, this problem is dealt with in document (1) at page 3, lines 24 to 34. According to this passage with progressive decrease in the weight average mean particle size of the hydraulic cement the water-containing cement composition becomes progressively more difficult to process, as it becomes progressively less fluid, for a given proportion of water to hydraulic cement, especially where the composition contains a low proportion of water. To overcome these difficulties it is recommended that the mean particle size of the cement be of at least 1 μm and the more preferred range is 2 μm to 15 μm . Therefore, as the said difficulties have been taken into consideration and avoided in document (1), they would not deter the skilled person from applying the teaching of documents (1) and (2) to vitreous blastfurnace slag.

6.5 The additional documents (10) to (13) cited by the Appellant at the appeal stage were all published after the priority date. They illustrate the possible uses of the classified blastfurnace having a high fineness in very special fields rather than in the conventional cement-concrete technology. These documents contain no information which could reverse the preceding findings as regards inventive step.

7. *First auxiliary request: inventive step*

Claim 1 of this request differs from Claim 1 of the main request only by the fact that "a cut size of 5 to 10 μm " is mentioned in the classification step.

As document (1) discloses the reduction of the mean particle size of the cement by classification in an air classifier at a cut size of 10 μm in example 1, the skilled person would first of all try to classify the blastfurnace slag at the cut size mentioned in the examples when applying the teaching of (1) and (2) to the slag. Therefore the reasons stated above in connection with the obviousness of the process according to the main request apply analogously to Claim 1 of the first auxiliary request.

8. *Second auxiliary request: inventive step*

Claim 1 of this request recites the additional step of mixing the classified slag powder in an amount of 40 parts by weight and 60 parts by weight of ordinary Portland cement.

It is well known to mix blastfurnace slag and Portland cement to manufacture slag cement. The slag cement of document (3) contains blastfurnace slag in an amount of 57%, i.e. an amount higher than stated in Claim 1. However, it belonged to the general knowledge of the skilled person before the priority date that blastfurnace cement contains about 36 to 85% blastfurnace slag and 15-64% Portland cement clinker (cf. document (5), page 161). Therefore, the amounts of slag and Portland cement recited in Claim 1 lie within the **known ranges** normally used for manufacturing slag cement. Assuming, to the Appellant's benefit, that the amounts of classified slag and Portland cement stated in Claim 1 lead to optimal strengths at early and later stages, the choice of these amounts cannot, however, be regarded as involving an inventive step. It is obvious to the skilled person who aims at achieving the optimal improvement of strength to mix the classified slag with

the known amounts of Portland cement normally used to prepare the conventional slag cement and to determine by routine experimentation the appropriate relative amounts of classified slag and cement which lead to the optimal improvement of strength.

9. It results from the above that the processes according to Claim 1 of the main request, Claim 1 of the first auxiliary request and Claim 1 of the second auxiliary request do not involve an inventive step. Therefore these claims do not meet the requirements of Articles 52(1) and 56 EPC and none of the requests can be granted.

Order

For these reasons it is decided that:

The appeal is dismissed.

The Registrar:

The Chairman:

P. Martorana

P. A. M. Lançon