BESCHWERDEKAMMERN DES EUROPÄISCHEN PATENTAMTS

BOARDS OF APPEAL OF THE EUROPEAN PATENT OFFICE

CHAMBRES DE RECOURS DE L'OFFICE EUROPEEN DES BREVETS

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File No.: T 0446/90 - 3.3.2

Application No.: 79 104 321.9

Publication No.: 0 010 777

Classification: C04B 20/00

Title of invention: Shaped article and composite material and method for producing same

DECISION
of 14 July 1993

Applicant:

Proprietor of the patent: Densit a/s

Opponent:
01) Höganäs AB,
02) Sika AG, vormals Kaspar Winkler & Co.
03) K/S Noco Tech A/S
04) AB Betongindustri
05) S.A. Lafarge Réfractaires monolithiques
06) Eternit-Werke Ludwig Hatschek AG

Headword: Concrete/DENSIT

EPC: Art. 84, 54, 56

Keyword: "Definition of a process step by the result to be achieved" - "Result defined with reference to a theoretical model" - "Result can be verified by tests stated in the description and carried out on the final product" - "Novelty (yes)" - "Inventive step (yes)" - "Non-obvious improvement"

Headnote
Catchwords

Europäisches Patentamt | **European Patent Office** | **Office européen des brevets**

Beschwerdekammern | Boards of Appeal | Chambres de recours

Case Number: T 0446/90 - 3.3.2

DECISION
of the Technical Board of Appeal 3.3.2
of 14 July 1993

Appellant: (Proprietor of the patent)	Densit a/s Soelystvej 18 DK - 9220 Aalborg Oest (DK)
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Decision under appeal:

Decision of the Opposition Division of the European Patent Office dated 28 March 1990 revoking European patent No. 0 010 777 pursuant to Article 102(1) EPC.

Composition of the Board:

Chairman: A.J. Nuss
Members: M.M. Eberhard
J.C.M. de Preter

Summary of Facts and Submissions

I. European patent No. 0 010 777 based on application No. 79 104 321.9 was granted on the basis of 116 claims.

II. The six Respondents (Opponents) filed Notices of Opposition against the European patent. Of the documents cited during the opposition procedure only the following remain relevant in the present appeal:

(1) US-A-4 111 711
(2) CH-A-574 880
(3) "FLYDEBETON"; B.Hylén and H.H.Bache; Publication 2:1978; Dansk Betonforening.
(3a) English Translation of pages 29 to 33 of doc. (3)
(4) JP-A-49-125428 [English Translation]
(5) DE-A-2 730 943
(6) DE-A-2 219 478
(8) DE-A-2 708 004

III. The Opposition Division revoked the patent on the grounds that the shaped article as claimed in the amended Claim 1 of 12 January 1990 lacked novelty over the disclosure of either (1) or (2). It considered that the conditions "densely packed" and "homogeneously arranged" were not suitable to distinguish a new product from the shaped articles disclosed in (1) or (2), the homogeneity criterium being arbitrary. Moreover, even if novelty were acknowledged over (1) and (2), the shaped article would lack an inventive step over (3) in combination with the disclosure of (1). In the light of the teaching of (1), it was obvious to a skilled person, who wanted to improve the compressive strength of the concrete known from (3), to fill the voids between the cement particles with ultra fine silica by carefully mixing the latter in the cement composition as indicated in (1).

IV. The Appellant lodged an appeal against this decision and filed several sets of amended claims together with the statement of grounds of appeal. Further sets of claims were submitted in reply to a communication of the Board as well as during the oral proceedings. In the appeal statement, the Appellant relied *inter alia* upon the following documents:

(7) "SUPERPLASTICIZERS IN CONCRETE", Vol. 1, pages 49 to 85, Proceedings of an International Symposium in Ottawa, Canada held on 29-31 May 1978
(9) Superplasticizing Admixtures in Concrete, Cement and Concrete Association, June 1978, pages 4,5,8

V. Oral proceedings were held on 14 October 1992 and 3 June 1993. Respondents 01, 03, 04 and 05 did not attend these proceedings although duly summoned thereto. At the beginning of the last oral proceedings the Appellant submitted two sets of amended claims as main and auxiliary requests. Claim 1 of the main request reads as follows:

"1. A process for preparing a shaped article, the process comprising combining
A) inorganic solid silica dust particles of a size of from 50 A to 0.5 μm, and
B) solid particles having a size of 0.5 - 100 μm and being at least one order of magnitude larger than the respective particles stated under A), at least 20% by weight of the particles B being Portland cement particles,
the amount of particles A being 5-50 by volume of the total volume of particles A+B,

water in a weight ratio between water and particles A+B of 0.12-0.30 to fill the voids between particles A and B,

and a concrete superplasticiser, the amount of superplasticiser dry matter being in the range of 1-4% by weight, calculated on the total weight of the Portland cement and the silica dust, this amount of concrete superplasticiser being sufficient to impart to the composite material a fluid to plastic consistency in a low stress field of less than 5 kg/cm^2, preferably less than 100 g/cm^2,

and fibers, selected from the group consisting of metal fibers, including steel fibers, mineral fibers, including glass fibers, asbestos fibers and high temperature fibers, carbon fibers, and organic fibers, including plastic fibers, by mechanically mixing at least the above ingredients A, B, water and superplasticiser, optionally together with additional bodies which are different from the particles B and which have at least one dimension which is at least one order of magnitude larger than the particles A, e.g. inorganic bodies of compact shape such as sand or stone, until a viscous to plastic mass comprising the particles A and B and optionally other particles has been obtained, whereby the particles B become densely packed, the dense packing being substantially a packing corresponding to the one obtainable by gentle mechanical influence on a system of geometrically equally shaped large particles in which locking surface forces do not have any significant effect, with homogeneously arranged particles A in the voids between particles B,

and thereafter if the said fibers were not present during said mechanical mixing, combining the resulting mass with such fibers and, if desired, with additional bodies which are different from the particles B and which have at least one dimension which is at least one order of magnitude larger than the particles A, e.g.

inorganic bodies of compact shape such as sand or stone, by mechanical means to obtain the desired distribution of such additional bodies, and finally casting the resulting mass in the desired shape in a stress field, optionally with incorporation of additional bodies which are different from the particles B and which have at least one dimension which is at least one order of magnitude larger than the particles A, and allowing the cast mass to cure to produce a shaped article comprising a coherent matrix."

VI. In connection with the main request the Appellant put forward *inter alia* the following arguments:

Claim 1 fulfils the requirements of Article 123(2) EPC. It is abundantly clear from the application as originally filed that additional bodies may be incorporated at any stage of the process, that the term "additional bodies" is understood to include both smaller bodies (such as sandstone and fine fibres) and larger bodies (such as steel bars) and that the determination of the stage at which a particular type of additional bodies is to be added is a simple matter for a person skilled in the art on the basis of information in the application as well as common general knowledge in the art. Furthermore, Example 3 discloses the addition of the fibres and of other additional bodies at two different stages of the process. The presence of the word "curing" in Example 3, page 28, line 34, of the patent in suit (page 56, line 5 of the original application) makes no technical sense and represents an obvious error. Deletion of this word is requested pursuant to Rule 88 EPC.

As regards clarity, there is no need to define a specific group of superplasticizers in Claim 1, the definition of a superplasticiser and the differences between the latter and a normal plasticizer being well known to the skilled person as shown by document (9).

The subject-matter of Claim 1 is novel with respect to document (8) since the latter discloses general classes of detergents but does not mention the use of superplasticizers. Moreover, in the working example on page 55, the proportion of the ligninsulfonate detergent is very small (0.05%).

Document (4) represents the closest prior art. The compressive strength data reported in the tests 1 to 12 of (4) show that a homogeneous distribution of ultra fine silica particles between densely packed larger cement particles has not been obtained. It can be derived from these tests that the maximum compressive strength for each of the three series of tests is obtained with concretes containing less than 10% by volume ultra fine silica based on the total volume of the cement and silica, whereas with silica amounts greater than 10% the strength decreases dramatically. In view of this falling off, it is clear that the inventors of (4) failed to achieve the dense packing. In contrast thereto, the compressive strength of the concretes according to the present invention increases with increasing amounts of silica dust up to a silica content of about 30% by volume (cf. Example 5). Furthermore, document (4) is based on a chemical approach whereas the invention relies upon how physically to achieve a dense packing during the production process. Accordingly, the known production process is different from that of the patent in suit. In particular, the process of the invention involves the use of large amounts of superplasticizers and a mixing step which may be drastically prolonged compared to the traditional mixing. The difference in the mixing step is expressed in Claim 1 by a functional limitation. Mixing should be carried out such that homogenity and dense packing as defined in Claim 1 are achieved. Although it is not possible to test immediately after the mixing step whether this condition is fulfilled, the achievement of the dense packing can be ascertained by testing the final product. The properties of the final product, in particular the higher compressive strength despite the use of large amounts of ultra fine silica and the fact that the rupture passes mainly through the aggregate particles, indicate that a dense packing was obtained in the mixing step.

As a consequence of the very dense micro-structure obtained by the claimed process, the matrix has at least one order of magnitude better mechanical locking effect for short fibres and the reinforced shaped article exhibits a much better toughness. By adding fibres to a matrix produced according to (4) the skilled person would expect only an average increase in toughness. The better anchoring of the fibres makes it possible to use shorter fibres than usual to obtain the same reinforcing effect.

Document (3) does not concern a binary system of cement and ultra fine silica but a cement-cement system in which the fine cement particles do not have the fineness indicated in Claim 1 of the main request. This document does not deal with the specific problem of arranging ultra fine particles in the voids between the larger cement particles. Document (6) indeed teaches the use of large amounts of superplasticiser, however as (4) and (6) concern two totally unrelated systems, the skilled person would not combine their teaching.

VII. The Respondents' arguments in connection with the main request filed on 3 June 1993 may be summarised as follows:

Claim 1 does not fulfil the requirements of Articles 123(2) and (3) EPC. It is not disclosed in the original documents that the fibres on the one hand and the other additional bodies on the other hand may be added at different stages of the process. Example 3, series 4, cannot form a basis for such a general disclosure since it lies outside the scope of Claim 1, the steel fibres being poured into the mixing vessel after curing of the mortar. It is also questionable whether the insertion of the term "at least" in Claim 1 after "by mechanically mixing" is supported by the original application.

The definition of a dense packing given in Claim 1 does not meet the requirements of Article 84 since it is not clear what is meant by the terms "gentle mechanical influence", "significant effect", "substantially a packing corresponding ...". The patent in suit contains no additional information in this respect nor a measuring method of the homogeneous dense packing to be achieved during the mixing step. As dense packing is the aim of any skilled person in the cement industry, the corresponding functional feature in Claim 1 cannot form a basis for an inventive step. It appears from the description that the invention does not lie in the dense packing as such but that it is based on the addition of an extremely high amount of superplasticiser to the cement mixture and on a specific mixing procedure leading to said dense packing. In the process of the patent in suit chemical reactions between the components also occur so that it is not possible to distinguish between a dense packing achieved by a physical effect and one resulting from a chemical effect. Furthermore, the patent in suit neither contains a definition of the term superplasticiser nor distinguishes between dispersing agents in general and a superplasticiser. It is common general knowledge that any dispersing agent for cement is either a plasticiser or a superplasticiser depending on its absolute dispersing activity. When used in high amounts, plasticisers have the same activity as superplasticisers.

Document (8), in particular Claims 1, 5, 8, 18 and 22 as well as pages 25, 36, 39, 45, 53, 55, and Example 6, discloses the five components used in the process of Claim 1 . Furthermore, it emphasizes that a homogeneous distribution of very fine particles around coarser particles is advantageous and that a low water to cement ratio leads to products of higher rigidity. The dispersing agents described in (8) actually comprise products which are known to have the activity of concrete superplasticizers, for example protein lignosulfonate. Therefore, the subject-matter of Claim 1 lacks novelty.

As regards inventive step, it is agreed that document (4) represents the closest prior art. In particular the three series of tests reported there clearly show an increasing strength of the material with increasing superplasticiser contents. The fact that these tests were performed with lower amounts of superplasticiser than in Example 5 of the patent in suit or that starting materials containing a high amount of calcium sulphate were cured in an autoclave might explain the lower compressive strength values reported in (4).

Document (6) mentions all the constituents used in the claimed process except the ultra fine silica dust. It teaches the use of extremely high amounts of superplasticiser, e.g. 0.5% to 5% with respect to the weight of cement, the water to cement ratio being correspondingly low, i.e. 0.24. Moreover, the advantages achieved by the addition of fibres are explained there in detail. Not only (6) but also documents (2) and (7) disclose the use of high amounts of superplasticisers. It derives from (7) that these high amounts are added in order to achieve a high flowability and therefore a dense packing.

Furthermore, in the light of the teaching of document (3), the Appellant's approach with respect to obtaining a dense packing can not be regarded as a new way of producing concrete. It is recommended in the first part of this document to use concrete superplasticisers in view of their advantages, namely the lower water need at high fluidity, the prevention of bleeding and the low vibration forces. In the second part it is further stated on page 11 that the particle shape and particle size distribution in cement alone would - apparently physically - allow a very dense packing corresponding to a water-cement ratio of 0.17. According to page 15 one of the two means to increase packing density is the use of surface-active components to eliminate cohesive forces. On page 25, it is said that slow mixing with additional high frequency vibration, or compaction using high-frequency vibration would be particularly useful for making superplasticised concrete. Very low water-cement ratios of 0.3 - 0.2 are mentioned in the last paragraph. Finally, this document teaches at page 30, that for strength reasons it is desirable to use fine grain cement the merits of which cannot usually be fully exploited due to its high water need. However, if superplasticised, even extremely fine grain cement pastes can be brought to fluidity and packing at very low water-cement ratios of about 0.2. Naturally, the superplasticiser amount will have to be adjusted to the greater surface, meaning a higher amount of superplasticiser.

In view of the teaching of these documents, the achievement of a dense packing and the use of high amounts of a superplasticiser to obtain a matrix with a high compressive strength as well as the addition of fibres to improve toughness lacks an inventive step.

According to Respondent 02 (letter dated 7 January 1993), the addition of fibres during the manufacture of a shaped article was familiar to the skilled person before the priority date. Therefore, adding fibres to known compositions which already contain ultra fine silica particles, Portland cement, a water reducing agent and water does not involve an inventive step. Such compositions are disclosed in the documents (1), (2) and (4) which emphasise the advantage of using the ultra fine silica in combination with the other components.

VIII. The Appellant requested that the decision under appeal be set aside and that the patent be maintained on the basis of the main request or alternatively on the basis of the auxiliary request both submitted at the oral proceedings of 3 June 1993. Correction of an obvious mistake at page 28, line 34, of the patent in suit was also requested.

The Respondents requested that the appeal be dismissed.

Reasons for the Decision

1. The appeal is admissible.

2. As regards the request for correction in Example 3 of the patent in suit (page 28, line 34), the Board observes that, for a correction to be allowable under Rule 88, it must be established that an error is present

and that the correction is obvious in the sense set out in Rule 88, i.e. "in the sense that it is immediately evident that nothing else would have been intended than what is offered as the correction". Both conditions are met in the present case. On the one hand, pouring the steel fibres into the mixing vessel "after curing the final mixing" and then continuing the mixing for an additional 5 minutes as indicated in lines 34 to 36 makes no technical sense and is manifestly erroneous and, on the other hand, the deletion of the word "curing" is the only possible correction the skilled person would immediately think about, taking into account the subsequent process steps, namely further mixing followed by casting in moulds on a vibrating table and curing for 24 hours in the moulds (see lines 35 to 37). Therefore, the request for correction is allowable and consequently the corrected version of the patent and of the corresponding passage in the original application (cf. page 56, line 5) is taken as the basis for the decision of the Board.

3. There are no formal objections under Article 123(2) and (3) to the amended claims of the main request. The subject-matter of Claim 1 is supported by the process Claim 41 as originally filed, the original Claims 1, 17, 18, 20, 39 and 40 and the original description page 2, lines 23 to 26; page 6, lines 20 to 33; page 8, lines 25 to 31; page 15, lines 26 to 28; page 18, lines 23 to 25, page 31, lines 2 to 6, page 32, lines 19 to 26; page 36, line 5; Example 2. In particular, the addition of fibres at a process step other than the step at which the additional bodies are incorporated is directly and unambiguously derivable from the original description page 38, lines 10 to 19 in connection with page 40, lines 5 to 11, and Example 3, series 4. Furthermore, the application as originally filed is not limited to mechanically mixing only the silica dust particles, the

particles B, water and a superplasticiser. The possible presence of other substances is supported by the original Claims 41 and 1 and the description page 30, lines 29 to 31. Therefore, the expression "by mechanically mixing *at least* the above ingredients ...." is also in agreement with the requirements of Article 123(2). The dependent Claims 2, 3, 4 to 7, 8, 9, 10, 11, 12 to 15, 16 to 21 of the main request find a support in Claims 14, 16, 15, 37 to 40, 8, 18, 17, 8, 22 to 24, 42 to 48 as originally filed.

Moreover, the process Claim 1 of the main request clearly does not broaden the scope of protection of the granted process Claim 46.

4. *Clarity of Claim 1*

4.1 As regards the alleged lack of definition of a superplasticiser, the Board cannot follow the Respondents' arguments since document (9), which represents the common general knowledge about superplasticisers, shows that the person skilled in the art not only knows the definition of a superplasticiser but is also aware of the differences between a superplasticiser and a normal plasticiser in particular as regards their activity in concrete, their chemical structure and their dosage (cf. pages 3 and 4). Therefore, there is no need for a further reference to a specific class or group of chemical components in Claim 1 to meet the requirements of Article 84.

4.2 The mixing step is defined in Claim 1 by the result to be achieved, namely obtaining a dense packing of particles B. According to Claim 1 "a dense packing is substantially a packing corresponding to the one obtainable by gentle mechanical influence on a system of geometrically equally shaped large particles in which locking surface forces do not have any significant effect". The packing which is referred to in this definition represents in fact a theoretical, idealized scientific model about the packing of particle systems without surface forces, as explained in the description of the patent in suit with reference to textbooks relating to particulate technology and particulate mechanics (cf. page 6, lines 1 to 23). It derives from this passage that such models are well known as well as the influence of the mechanical compaction method which is discussed in lines 14 to 19. Therefore, the terms "gentle mechanical influence" are considered to be understandable in view of the explanations in the description. Furthermore, the word "substantially" used in the context of a definition with reference to an idealized model is also understandable since such a model, per definition, can in practice never be totally achieved. For practical applications and to verify to what extent the model has been reached, it is possible to determine the properties or characteristics of the system at a later stage if the density of the packing cannot be observed or measured directly at the end of the mixing step. It has not been disputed that, in the present case, conclusions about the density of the packing at the end of the mixing step can be drawn for example on the basis of scanning electron microscopy photographs of the rupture faces of the cured fibre-reinforced product at very high degrees of magnification as indicated in the patent in suit (cf. page 8, lines 37 to 45; page 26, Example 2, line 65 and Figure 5). Furthermore, density measurements on the cured products also indicate whether or not these products are densely packed as pointed out at page 29, lines 42 to 43; therefore, these measurements also permit the density of the packing of several samples to be estimated and compared at the end of the mixing if the subsequent process steps (in particular the compaction) are the same.

It follows from the above that the reference to a theoretical model of a particle system in Claim 1 can be understood by the skilled person on the basis of the further information given in the patent in suit and that at least one indirect method suitable to test the density of the packing is disclosed in the description. Under these circumstances the definition of the mixing conditions by the result to be achieved is considered to meet the requirements of clarity. In this respect the Board notes that the incorporation in Claim 1 of the mixing conditions described in the examples, in particular the mixing time, would unduly limit the scope of protection since it is clear that the prolonged mixing times used to obtain the dense packing depend upon parameters such as the kind of mixing device, the speed of mixing, the amount of superplasticiser and the amount of silica dust.

5. *Novelty*

Respondent 02 has argued that document (8) renders the subject-matter of Claim 1 not new. The process according to (8) comprises mechanically mixing 100 parts by weight of a hydraulic binder, 2 to 100 parts of a fine powder having a specific surface of 1-75 m^2/g, 10-45 parts of water and optionally other components, i.e. 0-1.6 parts by weight of a dispersion agent and 0-1000 parts by weight of coarse aggregates. Fibres may be added to the mortar (cf. Claims 1, 2, 8, 18). The binder can be dewatered gypsum, cement or a mixture thereof. However, the compositions comprising normal cement (i.e. Portland cement) do not contain a dispersing agent and their fine powder has a grain size <0.8µm, i.e. greater than the upper limit stated in Claim 1 as pointed out by Respondent 02 (cf. pages 39 and 40 of this document). The composition disclosed on page 55 comprises sodium hexametaphosphate as a dispersing agent and a "protein lignosulfonate detergent", however the binder is **α**-hemidydrate and the amounts of these components are very low. i.e. 0.15 and 0.05 wt% with respect to the weight of **α**-hemihydrate respectively. It is not stated in (8) whether or not this dispersing agent and this detergent are superplasticisers. As it is general knowledge that lignosulfonate compounds may be either normal plasticisers or superplasticisers depending upon whether or not and how they have been modified it cannot be concluded in the absence of further information in this respect that the protein lignosulfonate detergent used in (8) is a superplasticiser. The total amount of this detergent and Na-hexametaphosphate in the composition is anyway more than 6 fold smaller than the lower limit stated in Claim 1 of the main request for the superplasticiser content. As regards the mixing step, it derives from page 39, lines 4 to 6, and page 36, last paragraph, that the bulk prisms containing Portland cement were prepared by mixing the starting components with water for only about 45 seconds. This can be regarded as the usual procedure used in the concrete industry as confirmed by the statement at page 33, last paragraph. It is not directly and unambiguously derivable from (8) that mixing with the water is performed until a structure as defined in Claim 1 is achieved. It follows from the above that (8) does not disclose the combination of features recited in Claim 1.

Since novelty with respect to the other cited documents is no longer in dispute, it is not necessary to consider this matter any further. Therefore the process of Claim 1 according to the main request is novel.

6. *Inventive step*

Although document (4) does not concern a fibre reinforced concrete it was regarded by the parties as representing the closest prior art. The Board can follow this approach since the process of (4) is closer to the process for manufacturing the matrix of the shaped article according to the patent in suit than the methods disclosed in the documents concerning fibre-reinforced concretes.

6.1 Document (4) discloses a method of manufacturing high strength concretes, wherein a cement composition comprising a pozzolanic material, cement and calcium sulphate is moulded by a usual method and cured at a high temperature optionally under pressure (cf. Claim 2). According to tests 1 to 12 different portions of Portland cement, silica flour, anhydrite, water, fine and coarse aggregates having physical properties comparable to those of the corresponding materials referred to in the patent-in-suit, were charged into a mixer, dry-mixed for 30 seconds and thereafter mixed for 5 minutes adding water containing dissolved "Mighty-150", i.e. a highly condensed sulfonated naphthalene formaldehyde condensate in the form of a 42 wt% aqueous solution well known as a concrete superplasticiser (cf. document (7), pages 49 and 50). After moulding, the concrete mixture was heated to 140 °C by water vapour at a rate of 40 °C/3hrs and maintained at this temperature for 10 hours. Thereafter the temperature was lowered at a rate of 40°C/3hrs and the moulded concrete was cured in water for 14 days. The concrete mixtures of Examples 7 and 8 comprising 600 kg cement and 40 or 80 kg silica dust per m^3 wet concrete and those of Examples 11 and 12 with 700 kg cement/m^3 wet concrete and 40 or 80 kg silica dust are particularly relevant. The calculation of the corresponding compositions expressed on the same basis as in the patent in suit leads to superplasticiser contents of 0.79%, 1.30%, 0.91% and 1.51% (% dry matter by weight of cement plus silica), water/cement plus silica ratios of 0.281, 0.265, 0.236 and 0.264 and silica dust contents of 8.1%, 14.9%, 7.0% and 13.1% (% by volume) for tests No. 7, 8, 11 and 12, respectively (cf. the conversion Table submitted by the Appellant on 14 October 1992). The corresponding cured products exhibit compressive strengths of 93.3, 83.2, 100.8 and 88.8 MPa respectively. These calculated proportions of components fall within the ranges stated in Claim 1 except for the superplasticiser contents of Examples 7 and 11 which are lower than 1 wt%. However, as the cured products of these two examples exhibit the highest compressive strength, they are considered as more relevant than Examples 8 and 12 despite their lower superplasticiser contents.

6.2 In the light of this closest prior art, the technical problem underlying the patent in suit can be seen in providing a process for preparing a shaped article which apart from being tougher comprises a matrix with an improved compressive strength.

It is proposed to solve this problem by the combination of features defined in Claim 1, in particular by adding fibres to the starting materials or to the viscous mass resulting from the mixing step, by using an amount of superplasticiser dry matter in the range of 1% to 4% by weight calculated on the total weight of Portland cement plus silica dust and by mechanically mixing the starting materials until a viscous to plastic mass is obtained, whereby the solid particles B including the Portland cement become densely packed as defined in Claim 1 with homogeneously arranged particles of silica dust in the voids between the particles B.

6.3 Having regard to the statement on page 8, lines 33 to 38, of the patent in suit, to Example 2 and to Figures 3 and 4, there is no reason for the Board to doubt that the addition of fibres to the starting materials or to the viscous mass resulting from the mixing step leads to a tougher shaped article. This was not disputed by the parties.

As regards the compressive strength, Example 5 of the patent in suit shows that a composition comprising 530 kg cement and 42 kg silica dust per m^3 of wet concrete (i.e. 10 Vol.% silica with respect to the total volume of silica plus cement) exhibits a compressive strength of 109.6 MPa after curing the test specimen in water at 20°C for 28 days. With a composition comprising cement and silica amounts of about 471 kg/m^3 and 84 kg/m^3 respectively (i.e. 20 Vol.% silica dust) the compressive strength after curing is 118.5 MPa. When comparing these compressive strength values with those reported in (4) for the test specimens Nos. 7, 8, 11 and 12, it clearly appears that the compressive strength of the matrix prepared according to the claimed process is significantly higher than that of these known products, which contain comparable aggregates, despite the lower cement content in the matrix of Example 5 according to the patent in suit.

It is true, as stressed by the Respondents, that the curing conditions used in Example 5 of the patent in suit and in document (4) are not the same and that the curing conditions have an influence on the compressive strength. However, it is stated in the patent in suit that extremely good quality can be obtained by curing at approximately 20°C or 80°C as well as 200°C (autoclave), (i.e. low temperature curing, heat curing and autoclave treatment) and that heat curing seems probably to be the most promising technique for the material of the present invention (cf. page 22, lines 6 to 12). Furthermore this statement is illustrated by Example 1 in which test specimens prepared from a concrete mixture containing 400 kg cement and 133 kg silica dust per m^3 and the same aggregates as in Example 5 were cured under different conditions. The specimens cured in water at 20°C for 14 days and 28 days exhibit a lower compressive strength (115.9 and 124.6 MPa) than those water-cured at 80°C for 20 hours or autoclave for about 96 hours at 214°C/20at whose compressive strengths are 128 MPa and 140 MPa respectively. It clearly derives therefrom that the curing conditions used in Example 5 are not the most favourable ones and that heat curing or heat curing in autoclave would certainly not lead to values lower than those reported in Table IV for Example 5.

It follows from the above that, although the test specimens of Example 5 include a lower amount of Portland cement than the tests Nos. 7, 8, 11 and 12 of (4) and have been cured under conditions which are less favourable as regards the compressive strength, they nevertheless exhibit a higher compressive strength.

Respondent 02 has further argued that the lower compressive strength in (4) might result from the lower amounts of superplasticiser than in Example 5 of the patent in suit or from autoclave curing of materials containing a high amount of calcium sulphate.

In this respect it should be noted that according to (4) an increase of the superplasticiser amount from 0.79 wt% to 1.30 wt% in tests 7 and 8 or from 0.91 wt% to 1.51 wt% in tests 11 and 12 results in a decrease of the compressive strength. As the solution defined in Claim 1 requires not only different conditions for the mixing step but also a higher superplasticiser content than in the tests 7 and 11 which exhibit the highest compressive strength and moreover the addition of calcium sulphate is not a mandatory feature of the claimed solution, it appears that the differences pointed out by Respondent 02 are in fact reflected by corresponding features in Claim 1. Under these circumstances, it is credible that the features recited in Claim 1 lead to a matrix with an improved compressive strength. Therefore, in the absence of evidence to the contrary, it is plausible that the technical problem stated above has been solved by the process defined in Claim 1.

7. Table 5 of document (4) and the corresponding calculations of the Appellant show that by increasing the superplasticiser amount up to 0.79 wt% (% dry matter by weight of cement plus silica) in the series of tests 5 to 7 or up to 0.91% in the series of tests 9 to 11 while simultaneously increasing the amounts of silica dust and anhydrite, the compressive strength of the cured products is **improved**. However, with superplasticiser amounts of 1.30 wt% or 1.51 wt% as in tests 8 and 12 a drastic decrease of the compressive strength is obtained. In view of this teaching the skilled person faced with the problem defined above would consider slightly increasing the superplasticiser content of test 7 (or test 11) to contents intermediate between those of tests 7 and 8 (or tests 11 and 12) in order to check whether or not the increase in compressive strength has already reached its maximum in test 7 (or test 11). Doing so, he would arrive at superplasticiser amounts of about 1 wt% or slightly above 1%, i.e. amounts which lie within the claimed range.

According to (4) the voids in the concrete or in the cement paste are filled up by the cement gel (tobermorite or its similars) or ettringite formed by the reaction of the pozzolanic-calcium sulphate material with calcium hydrate, and the free amount of water can be decreased remarkably. This leads to cured products having both a high compressive strength and a good freezing and thawing durability (cf. page 3, last paragraph, page 4, 1st and 2nd paragraphs). This explanation, although based on a chemical approach, stresses the importance of filling up the voids in the cement paste and in the concrete. However, this teaching does not hint at the possibility of improving the compressive strength by changing the mixing conditions, i.e. by performing the mixing of the starting components, superplasticiser and water until the cement particles become densely packed in the sense defined in Claim 1 with homogeneously arranged silica particles in the voids between the cement particles. This feature, which, in combination with the higher amount of superplasticiser, contributes to the improvement of compressive strength, can also not be inferred from the mixing conditions described at page 7 of (4) since it is only mentioned there that the water containing "Mighty-150" was mixed for 5 minutes with the dry powder mixture.

7.1 Document (3) discloses a theoretical review as to how the attracting forces between the cement particles lock them together and affect their packing and how this locking effect can be overcome. The study is focused in particular on the physical/mechanical mode of action of superplasticisers in improving the flow behaviour and the compacting of fresh cement paste or concrete.

As emphasised by Respondent 06, document (3) teaches the advantages brought about by the use of superplasticisers, namely the better workability, the improved flow properties, the lower water demand and the reduced risk of bleeding. It is further stated at page 11 that the shape of the cement particles and their size distribution make it possible to produce a dense packing with a volume concentration of about 0.65 corresponding to a water-cement ratio of 0.17. This is shown in Figure 1. However, according to page 15 (1st and 2nd paragraphs) there are two different ways of increasing the packing density, namely by mechanical compacting or by adding surface-active substances which eliminate the forces locking the particle together, and the results of Figure 1 were obtained by *mechanical compacting*. At page 25, document (3) teaches, in the context of the flow behaviour of superplasticised cement pastes, that slow mixing with additional high-frequency vibration and compaction using high-frequency vibration would seem to be suitable for mixing and compacting superplasticised concretes having a high dilatancy. Moreover, in view of the third paragraph of page 30 also relied upon by Respondent 06, the skilled person is aware of the fact that the use of fine grained cement is desirable for strength reasons and that with the presence of superplasticisers even extremely fine grained cement pastes may be made to flow and pack at a very low water content (W/C about 0.2). Of course, the amount of superplasticisers must be adjusted to the larger surface area of the finer cements.

The Board observes that the whole teaching reported above and relied upon by Respondent 06 does not relate to binary particle systems, let alone to a binary system comprising ultra fine silica dust as in the patent in suit. No information about the flow behaviour of such a binary system can be derived from the cited passages, nor whether or not a dense packing of the cement particles might still be achieved in the presence of ultra fine silica particles. Furthermore, the teaching of (3) about the adjustment of the superplasticiser amount to the cement fineness would be of no assistance to the skilled person faced with the problem of improving the compressive strength of the binary system according to (4) since the latter already contains relatively high amounts of superplasticisers and it can be expected in view of tests 8 and 12 already discussed above that a further increase of the superplasticiser amount would cause a further decrease of the strength.

However, a short reference to a binary particle system can be found at page 33 of (3) (see Fig. 18). This figure shows the density of a vibropressed dry special cement consisting of a binary mixture of a coarse cement with a particle size above 40 µm and a fine cement with particles smaller than 2 µm. The samples were compressed at five different levels of oscillating pressure (see translation of the key to Fig. 18 in document (3a)). It can be deduced from the graphs that the density increases with increasing oscillating pressures during compaction. The graphs also disclose the most appropriate proportion of the fine cement in the cement mixture at the different compaction pressures. However, as on the one hand, the increase of density is attributed to the increase of pressure during the **compacting step** and, on the other hand, there is no further information as to how the density of a binary system might be otherwise improved, this teaching cannot point towards the claimed solution which involves performing the **mixing step** as defined in Claim 1.

7.2 Document (8), which was already discussed in detail above, points out the importance of the changes in the physical structure caused by the presence of the fine powder (cf. page 18, second full paragraph). It is indicated at page 39, Example 2, that all the mortar products, including those with cement or hydraulic oxides as binder, exhibit higher strength values when the binder is enveloped in the fine powder. The Respondents have given no reasons as to why this structure would suggest performing the mixing step until a dense packing of the coarse particles B with homogeneously arranged silica particles in the voids between the particles B as defined in Claim 1. Taking into consideration that, on the one hand, the structure disclosed in (8) differs from the structure which has to be achieved at the end of the mixing step and, on the other hand, the mixing of the water with the powder mixture is performed there for a usual mixing time of 45 seconds and in the absence of superplasticiser, the Board does not see how this teaching could hint at the claimed solution.

7.3 Document (5) concerns the preparation of concretes having a high corrosion resistance. The process involves the use of starting components, i.e. cement, silica and superplasticiser, in amounts which appear to fall within the ranges stated in Claim 1 (cf. page 16, lines 27 to 30, page 17 and Claim 7).

However the water/(cement plus silica)ratios calculated from the data reported in the table of page 12 lie within the range of 0.39 to 1.03 wt%, i.e. clearly above the upper limit of 0.30 stated in Claim 1. According to page 7, lines 1 to 10, the fine reactive silica is uniformly distributed in the concrete mixture in order to cause a complete reaction with the calcium hydroxide which is formed during the cement hydration and then the concrete is cast in the usual way. Document (5) further teaches that the maximum corrosion resistance is achieved when this reaction is complete and that the increase of the concrete strength points to the formation of tobermorite (cf. page 15, lines 9 to 19). This teaching suggests neither the criticality of the mixing step nor that the latter must be performed until the particular structure defined in Claim 1 is achieved in order to improve the compressive strength of the products of (4).

7.4 Document (2) relates to a cement composition comprising Portland cement and ultra fine silica dust in the same proportions as those stated in Claim 1 assuming that the density of these products is similar to that mentioned in the patent in suit. The upper amount of plasticiser is 2 wt% with respect to the mixture of cement plus silica (cf. Claims 1 and 2). Even if it were assumed that the amino-s-triazine resin used in Example 1 is a superplasticiser and not a normal plasticiser, the teaching of document (2) would not give the skilled person an incentive to perform the mixing step of document (4) in a different way to the one disclosed since (2) is silent about the process of preparation of the shaped products and does not even mention the water to cement ratio.

7.5 Document (1) is directed to cementitious compositions consisting of 10-30 wt% of cement particles (constituent I), 14-56 wt% of ultra fine particles having a particle size between 100 A° and 0.1 µm, i.e. ultra fine silica (constituent II) and 14-56 wt% of an inert filler with a particle size between 1 and 100 µm (constituent III) (cf. Claim 1). The ultra fine silica acts to fill a considerable portion of the voids existing between the particles of the inert filler, i.e. between the particles of a "mineral matter insensitive to hydration" (cf. col. 2, lines 16 to 21 and col. 3, lines 11 to 14). However, document (1) is silent as to the density of packing of the non-inert component, in particular of the Portland cement particles, and as to the arrangement of the ultra fine silica particles with respect to the cement particles at the end of the mixing step. The process from preparing concrete using the cementitious composition of (1) involves carefully mixing **in the usual fashion** said composition with water, aggregates and optionally a dispersing agent such as sodium tripolyphosphate, the amount of which (if present) being from 0.01 to 0.05% by weight with respect to the weight of concrete (cf. col. 4, lines 1 to 4; col. 5, lines 16 to 21 and 44 to 47; col. 6, lines 4 to 5 and 60 to 61; Claim 5). Accordingly, the process of (1) does not involve the use of a high amount of superplasticiser nor does it require special measures during the mixing step of the components with water, this mixing being performed "in the usual fashion". Moreover, although some examples using an aluminous cement or an aluminous slag as constituent I yield concretes having a very high compressive strength, i.e. about 147 MPa after 8 days (Example 6) or about 118 MPa after 20 days (Example 3), the sole example in which the constituent I is Portland cement leads to a concrete with a compressive strength of only about 50 MPa after a curing time of 15 days and stoving at 110°C. It follows from the preceding that document (1) does not contain any information which would encourage the skilled person to perform the mixing step of the process according to (4) in the manner defined in Claim 1 in order to improve the compressive strength of the shaped article.

7.6 The remaining documents, i.e. documents (6) and (7) do not relate to binary particle systems comprising ultra fine silica particles and are already for this reason less relevant than the preceding documents. Document (7) discloses the mechanism of dispersing cement particles by the "Mighty" superplasticisers and the use of Mighty as a chemical means to achieve high strength concrete (over 100 MPa). According to page 56 Mighty can safely be mixed in the concrete at levels as high as 1.0% and makes it possible to achieve water reduction of 33% (cf. page 56, 1st paragraph and page 67, Table 1). Table 2 on page 68 shows that the values of compressive strength increase progressively with decreasing water-cement ratios and increasing Mighty contents, thus attaining the maximum strength at 0.25 W/C ratio and 1.24 wt% Mighty (dry matter) (cf. page 57, last paragraph and page 68, Table 2). According to page 55 (penultimate paragraph) Mighty is effective in reducing or eliminating silica in a method involving the addition of 30 to 40 wt% of silica powder by weight of cement into the mix and autoclave curing for much easier formation of tobermorite. However, this teaching cannot be helpful to the skilled person faced with the problem of improving the compressive strength of the shaped bodies according to (4) since the process of this document already makes use of the Mighty superplasticiser and it derives from Tables 6 and 7 that higher amounts of Mighty than in tests 4, 8 or 12 would impair the compressive strength. Moreover, as document (7) does not disclose the behaviour or the properties of a binary system comprising ultra fine silica particles nor draws the attention to the criticality of the mixing step for such binary systems or to the necessity of achieving a particular particle arrangement at the end of the mixing step, this document, even in combination with the other documents analysed above, does not point towards the claimed solution.

Document (6) teaches that the compressive strength of concrete prepared from a composition comprising cement and a superplasticiser can be improved if a cement having a tricalcium aluminate (C_3A) content of less than 1 wt% is used instead of a cement with the usual C_3A content of about 5 wt%. The amount of the superplasticiser lies within 0.5 to 5% with respect to the weight of cement, preferably 2 wt%, the W/C ratio being then of 0.24 (cf. page 2, 2nd and 3rd paragraphs; page 3, last paragraph; page 4, 2nd paragraph; page 5, 3rd paragraph; pages 21 and 22, Example III and Table III;

Claims 1, 3, 4, 6, 11 and 12). Moreover, (6) discloses the incorporation of short fibres into the cement mixture and the resulting improvement of strength. However, as (6) does not relate to binary particle systems comprising ultra fine silica particles, the preceding findings as regards document (7) apply analogously to the combination of documents (6) and (4).

7.7 It follows from what precedes that it was not obvious to the skilled person in view of the cited prior art to perform the mixing of the starting materials with water and the high amount of superplasticiser until the particular particle structure as defined in Claim 1 had been achieved in order to improve the compressive strength. Therefore, the subject-matter of Claim 1 is considered to meet the requirement of inventive step as set out in Article 52(1) and 56. Under these circumstances, it can remain open whether the addition of fibres to the starting materials (or to the viscous mass) in order to improve the toughness (see points 6.2 and 6.3 above) also involves an inventive step or not.

8. The dependent Claims 2 to 21 which relate to preferred embodiments of Claim 1 derive their patentability from that of Claim 1.

9. In view of the above, there is no need to deal with the Appellant's auxiliary request.

Order

For these reasons, it is decided that:

1. The decision under appeal is set aside.

2. The case is remitted to the Opposition Division with the order to maintain the European patent on the basis of Claims 1 to 22 of the main request submitted during the oral proceeding of 3 June 1993 with a description to be adapted, the word "curing" being deleted at page 28, line 34 of the patent.

The Registrar: The Chairman:

Martorana

P. Martorana A.J. Nuss

M. Eh.