

Publication in the Official Journal ~~Yes~~ / No

File Number: T 675/89 - 3.5.2

Application No.: 82 100 091.6

Publication No.: 0 056 257

Title of invention: Method for production of metal magnetic particles

Classification: H01F 1/06

D E C I S I O N
of 13 March 1991

Proprietor of the patent: Hitachi Maxell Ltd.

Opponent: BASF Aktiengesellschaft, Ludwigshafen

Headword:

EPC Art. 56, 111(1) and 114

Keyword: "Inventive step (denied)" - "Remittal to first instance (no)"

Headnote



Case Number : T 675/89 - 3.5.2

D E C I S I O N
of the Technical Board of Appeal 3.5.2
of 13 March 1991

Appellant : BASF Aktiengesellschaft, Ludwigshafen
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Representative : Dr. E. Sobotta
BASF

Respondent : Hitachi Maxell Ltd.
(Proprietor of the patent) No. 1-1-88, Ushitora
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Representative : Jönsson, H.-P. Dr.
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Decision under appeal : Decision of the Opposition Division of the
European Patent Office dated 30 August 1989
rejecting the opposition filed against European
patent No. 0 056 257 pursuant to Article 102(2)
EPC.

Composition of the Board :

Chairman : E. Persson
Members : A. Hagenbucher
J. van Voorthuizen

Summary of Facts and Submissions

- I. European patent No. 0 056 257 was granted with 8 claims, Claims 2-8 being dependent on Claim 1.
- II. The Appellant filed a notice of opposition requesting revocation of the patent on the ground of lack of inventive step.

The following prior art documents were cited in support of the opposition:

- D1: US-A-4 133 677
- D2: JP-A-52-122 213 (corresponds to the non-pre-published US-A-4 262 037)
- D3: JP-A-52-134 858 and translation
- D4: Ullmanns Encyklopädie der technischen Chemie, Vol. 1, 1951, pages 731, 735 and 736
- D5: W. Schnitker and H. Rau: IEEE Transactions on Magnetics, Vol. MAG-16, No. 1, January 1980, pages 14-16
- D6: DE-A-1 583 167
- D7: US-A-2 936 286
- D8: DE-A-2 907 255
- D9: DE-A-2 553 635

- III. The Opposition Division rejected the opposition.
- IV. The Appellant lodged an appeal citing the following additional documents:

- D10: DE-A-2 731 845
- D11: DE-C-1 907 691

V. In order primarily to allow a proper interpretation of the term "doping", which is used in this specific prior art in an ambiguous way (cf. D5, D14 and D15), a communication was issued by the Board introducing into the proceedings the following further documents by virtue of Article 114(1) EPC:

D12: G. Bate in D.J. Craik, "Magnetic Oxides", New York, 1975, pages 689, 697-728, 739

D13: US-A-3 748 119 (referred to in the Encyclopedia of Chemical Technology by Kirk Othmer as an example for a practical process of reduction with hydrogen of iron oxides)

D14: IEEE Transactions MAG-9, 1973, pages 191-194 (referred to in D5)

D15: IEEE MAG-5, 1969, pages 317-320 (referred to in D14).

VI. In response to this communication, the Respondent filed, *inter alia*, a new set of claims by letter of 10 January 1991 of which Claim 1 reads as follows:

"1. A method for the production of acicular metal magnetic particles comprising predominantly iron and having a coercive force of not less than 1290 oersted, which comprises the steps of coating the surface of metal compound particles containing predominantly acicular iron oxyhydroxide or iron oxides with an aluminium compound and a silicon compound, pelletizing the thus coated particles into uniformly moulded pellet form by compression of the particles, and reducing the pellets with heating under reducing atmosphere in a stationary reduction furnace."

VII. Oral proceedings were held on 13 March 1991.

VIII. The arguments of the Appellant can be summarised as follows:

For the production of acicular metal magnetic particles comprising predominantly iron D3 taught coating the surface of metal compound particles containing predominantly acicular iron oxyhydroxide or iron oxides with an aluminium compound and a silicon compound. After that a dry filter cake of such treated particles was measured into a combustion boat and reduced in a reduction device under reducing atmosphere. The term "dry filter cake" made it clear that no "fluidised bed reactor" but a "fixed bed reactor", i.e. a stationary reduction furnace was meant.

Regarding reduction, it was generally known to granulate or pelletize particles in order to permit the supply of a large amount of hydrogen gas at high speed and to achieve thereby a reduction reaction uniformly within a short period of time and without sintering.

For a large-scale synthesis D13 (examples 6 and 7) therefore taught pelletizing of magnetic particles coated with a silicon and a further metal compound (bismuth).

It followed from D3 that in the production method of D13 bismuth could be replaced by aluminium.

In connection with the claimed subject-matter the magnetic particles were only coated in order to stabilise the particle shape during reduction. No further effect especially concerning the alleged scattering of external forces during pelletizing was disclosed in the patent documents. Coating known from D13 by means of a bismuth

salt and a silicon compound had an analogous effect as coating by means of a silicon compound and an aluminium compound as known from D3.

With respect to the claimed coercive forces of not less than 1290 oersted and the smaller coercive forces disclosed in D3 (especially example 1) and D13, attention was drawn to the fact that the resulting coercive forces depended on many parameters, such as thickness of coating, starting materials (e.g. the basic FeOH), duration and temperature of reduction process, size of particles etc. Hence, it was difficult to draw conclusions from the obtained coercive force without knowing all the other parameters or where several of these parameters had been varied simultaneously. Figures 10A and 10B of D1 showed even higher coercive forces for particles coated with only a silicon compound. The smaller coercive force achieved according to D13 was possibly due to a shorter reduction time.

IX. The Respondent essentially argued as follows:

The claimed method led to a new product which was already clear from the indication that the coercive force was not less than 1290 oersted. Neither the solution described in D3 nor that of D13 achieved such a high coercive force.

There was a difference between granulation as suggested in D5 and the claimed pelletizing. This became clear from the result of the comparative experiment filed on the 11 January 1991.

Moreover, whereas the prior art acknowledged in the description of the patent in suit and also that of D5 used a fluidized bed reactor, the claimed subject-matter required a fixed bed reactor which was meant by the term "stationary reduction furnace".

It had been found that coating helped also in the production of pellets because external forces exerted upon the particles in the step of pelletizing were scattered by the coating materials. The coating compounds served moreover as binding agents. Neither D3 nor D13 mentioned such effects of coating.

Hence, further binding agents as suggested in D4 for briquetting were not necessary. The high coercive forces achieved by the invention were only due to the claimed combination of features, namely coating with specific compounds, pelletizing and reduction in a stationary reduction furnace.

The term "uniformly moulded" in Claim 1 was chosen in order to distinguish the pellets from granules and allowed a certain range of size (cf. Claim 2).

X. The Appellant requested that the patent be revoked.

The Respondent requested that the patent be maintained in amended form on the basis of Claims 1-8 filed with the above letter of 10 January 1991 subject to the deletion of Claim 2 and a consequential renumbering of Claims 3-8. Auxiliarily he requested that the case be remitted to the first instance for further prosecution.

Reasons for the Decision

1. The appeal is admissible.
2. Novelty

The subject-matter of Claim 1 is novel, since none of the cited prior art documents discloses a method for the

production of acicular metal magnetic particles which comprises all the features specified in Claim 1. In this context, it is particularly to be noted that D3 does not mention pelletising and D13 does not show coating by means of an aluminium compound and a silicon compound.

3. Inventive step

3.1 In the Board's view, the closest prior art is represented by D3 (cf. page 8 and example 1 on pages 13 to 15 of the translation).

D3 (cf. page 8 and example 1) concerns a method for the production of acicular magnetic particles which comprises the steps of coating the surface of metal compound particles containing predominantly acicular iron oxyhydroxide or iron oxides with an aluminium compound and a silicon compound.

Subsequently a dry filter cake of such treated particles is reduced in a reduction device by heating under reducing atmosphere. The term "dry filter cake" makes it clear that in contrast to a fluidization reduction furnace mentioned in the introduction of the patent in suit and in D5, a "fixed bed reactor" is used according to D3. During the oral proceedings, the Respondent pointed out that the term "stationary reduction furnace" meant a fixed bed reactor.

However, D3 appears to deal mainly with the production of small amounts of magnetic particles (cf. the examples) and is silent about how to proceed when it is desired to scale up the disclosed method to a large-scale synthesis where for the reduction a large amount of hydrogen gas needs to be supplied at a high speed and the reduction reaction should be uniformly achieved within a short period of time

avoiding undesirable sintering of the particles. It would have been clear to the person skilled in the art that the use of large filter cakes may give rise to difficulties in this respect.

- 3.2 In the light of D3, the problem underlying the present invention is therefore to be seen in adapting the method of D3 so that it allows the obtention of high quality acicular metal magnetic particles at high speed in a large-scale industrial production (cf. the Respondent's letter dated 27 June 1990, page 7, 4th paragraph; page 8, last paragraph).
- 3.3 Recognising this problem does not require any inventive activity, since it is the normal aim of the skilled person to try to scale up known manufacturing methods so as to obtain larger quantities with good quality at optimum process speed.
- 3.4 A person skilled in the art who was confronted with the problem set out in paragraph 3.2 above must be assumed to consult a standard handbook for chemical processes such as the Encyclopaedia of Chemical Technology by Kirk Othmer which refers in this context to D13 as describing an example of a practical process for reducing iron oxides with hydrogen.

In connection with the large-scale synthesis example 6, D13 recommends pelletizing of magnetic particles already "doped" with a silicon compound and a bismuth compound.

From the volume ratio of the coating compounds with respect to iron mentioned in D13, from D14 (which states on page 191, left column, last paragraph, that dopants for preserving the needle shape can be present either built into the lattice or onto the surface) and from D15 (in

particular page 319, right column, last paragraph, which mentions absorption of tin salt by soaking the iron oxide particles in a solution of a tin salt) it must be concluded that the term "doping" in D13 is to be understood as meaning surface coating.

Since D13 recommends for a large-scale synthesis pelletizing of particles coated with a silicon compound and a bismuth compound, it was clear that a person skilled in the art must have expected pelletizing to be equally useful for a large-scale production with particles coated according to D3 with a silicon compound and an aluminium compound, because the purpose of both composite coatings is to reduce sintering effects during reduction.

If, as alleged by the Respondent, the coating materials may in addition to their shape-preserving purpose have side effects for the pelletizing process, then such effects must be equally expected for the coating materials disclosed in D13 and in D3 for the reasons just given.

As far as the difference between granulation and pelletizing is concerned, the Board agrees that the term 'pellets' can be understood as implying a uniform size and shape.

Pellets may be produced by some compression method (cf. Brockhaus Encyklopädie, 1972, Vol. XIV, page 346).

Regarding the Respondent's argument with respect to the claimed coercive force of being not less than 1290 oersted, attention is drawn to the fact that (as indicated during the oral proceedings) the lower limit 1290 oersted is only disclosed in connection with example 3 (coating with sylicic acid hydrate but not with an additional aluminium compound as now claimed). The achieved coercive

force depends on many parameters such as thickness of coating, choice of magnetic starting material, temperature and duration of reduction process, size of particles etc. so that it cannot be concluded that coercive forces of 1290 oersted or higher, such as those obtained under the exactly defined conditions of examples 5 and 6, are only due to the claimed combination of very generally worded method steps, irrespective of the values of the different parameters. A person skilled in the art knows the influence of the various parameters, however, and may adapt them without using inventive skill in order to obtain the desired coercive force, if necessary by carrying out such experiments as are conventional in this art.

3.5 To summarise, the Board concludes that the skilled person being confronted with the problem of carrying out the method of D3 on a large scale would arrive in an obvious manner at the method defined in the present Claim 1 by using pelletizing as proposed in D13. Thus, the subject-matter of Claim 1 does not involve an inventive step and, consequently, the patent cannot be maintained in amended form on the basis of this claim, as requested by the Respondent.

4. As regards the Respondent's auxiliary request, made for the first time at the oral proceedings, that the case be remitted to the first instance for further prosecution, it appeared that this request was based on the assumption that it would be possible for the Respondent to obtain protection for a more limited subject-matter than that covered by present Claim 1 by introducing technical features contained in the dependent claims, especially concerning preliminary heat treatment before the reduction step.

4.1 In this respect it is first to be noted that the only specific proposal for amendment of the claims was that according to the main request which, as set out above, could not lead to maintenance of the patent in amended form.

Obviously, in such a situation a remittal to the first instance for further prosecution of the case can only be considered as justified, if, prima facie, there seems to be a reasonable chance that such further prosecution could involve substantial new aspects deserving consideration by two instances of jurisdiction. However, in the Board's view this does not apply to the present case having regard in particular to the fact that the preliminary hearing referred to by the Respondent is well known in the art of preparing acicular metal magnetic particles (cf. D7, D8, D9).

4.2 It will be clear from these considerations that the circumstances of the present case are not similar to those of the case T 273/84 referred to by the Respondent.

Hence the Respondent's auxiliary request has to be rejected.

5. In these circumstances, revocation of the patent in suit must be ordered.

Order

For these reasons, it is decided that:

1. The decision under appeal is set aside.
2. European patent No. 56 257 is revoked.

The Registrar:

The Chairman:

M. Kiehl

E. Persson