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Datasheet for the decision of 26 March 2013

Case Number: T 0406/11 - 3.3.09

Application Number: 02739571.4

Publication Number: 1401924

IPC: C08J 3/20, C08K 9/00, C08J 3/22, C08K 13/06

Language of the proceedings: EN

Title of invention:
Organic/inorganic nanocomposites obtained by extrusion

Patent Proprietor:
Nelson, Gordon L.
Yang, Feng

Opponent:
Evonik Degussa GmbH

Headword:
-

Relevant legal provisions:
EPC Art. 54, 56

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

Decisions cited:
-

Catchword:
-

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Beschwerdekammern | Boards of Appeal | Chambres de recours

Case Number: T 0406/11 - 3.3.09

D E C I S I O N
of the Technical Board of Appeal 3.3.09
of 26 March 2013

Appellant: (Opponent)
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Decision under appeal: **Decision of the Opposition Division of the European Patent Office posted 2 December 2010 rejecting the opposition filed against European patent No. 1401924 pursuant to Article 101(2) EPC.**

Composition of the Board:

Chairman: W. Sieber
Members: N. Perakis
F. Blumer

Summary of Facts and Submissions

I. Mention of the grant of European patent No. 1 401 924 in the name of Nelson, Gordon L., et al, was published on 9 July 2008 (Bulletin 2008/28). The patent was granted with 18 claims, independent claim 1 reading as follows:

"1. A method for producing an organic-inorganic nanocomposite, comprising extrusion, in a single-screw extruder, of a polymer resin with a concentrate of an organic polymer and a non-clay inorganic additive, in the form of particles less than 100 nm in size, whose surface is covered with organic functional groups which improve compatibility between the additive and the organic polymer."

II. An opposition was filed by Evonik Degussa GmbH requesting revocation of the patent in its entirety on the grounds that the claimed subject-matter was neither novel nor inventive (Article 100(a) EPC).

The opponent filed *inter alia* the following documents:

D1: Technical Information TI 1206 "Antiblocking Agents", Degussa AG; issued in 1998 (see the notice at the bottom of page 6: 03/1998);
D2: Product Information "AERODISP® G 1220(G 320)", Degussa AG (notice of publication: September 2006);
D3: English Abstract of RU 2 109 772 C1;
D4: DE 40 41 042 A1;
D6: "Study of Modified Silicas by Inverse Gas Chromatography. Part I: Influence of Chain Length on Grafting Ratio", H. Balard et al,

Chromatographia, 25(8), August 2008, pp 707-711 (letter of 22 September 2010); and

D7: CAS Abstracts of JP 63280763 A and SU 1305621 A1 (letter of 14 October 2010).

III. By its decision announced orally on 16 November 2010 and issued in writing on 2 December 2010, the opposition division rejected the opposition because it considered that none of the grounds for opposition raised by the opponent prejudiced the maintenance of the patent as granted.

Regarding novelty, the opposition division held that:

- D1 did not directly and unambiguously disclose a single-screw extruder but a variety of processing machines, e.g. extruders, calenders, planetary mixers, which required the skilled reader to make two selections in order to arrive at the claimed single screw extruder, and
- D1 did not disclose particles covered with functional groups which improved the compatibility between the additive and the organic polymer.

Regarding inventive step, the opposition division held that:

- D4, which disclosed a process comprising mixing polyester resin with ultrafine pyrogenic silica particles in a twin-screw extruder, should be considered to represent the closest state of the art;
- the technical problem was seen in the provision of a further method for producing organic-inorganic nanocomposites;

– the skilled person starting from D4 and aiming at a further method for producing nanocomposites would not find in D4 or any other prior-art document the hint to change the configuration of the extruder.

IV. On 3 February 2011 the opponent (hereinafter: the appellant) filed an appeal against the decision of the opposition division and paid the appeal fee on the same day. The statement setting out the grounds of appeal was filed on 26 March 2011. The appellant reiterated the objections raised before the opposition division and requested that the decision of the opposition division be set aside and the patent be revoked in its entirety. In support of its argument regarding lack of novelty the appellant submitted the following further documents:

D8a: http://de.wikipedia.org/wiki/Extruder; and
D8b: http://de.wikipedia.org/wiki/Extruderschnecke.

V. By letter dated 8 August 2011, the patent proprietor (hereinafter: the respondent) filed observations on the appeal and requested that the appeal be dismissed.

VI. By letter dated 13 March 2013, the appellant withdrew its request for oral proceedings and announced that it would not be represented at the oral proceedings scheduled for 26 March 2013.

VII. Oral proceedings were held before the board on 26 March 2013 in the absence of the appellant. During the oral proceedings the respondent filed a new main request (sole request) and a description adapted thereto.

Claim 1 of the main request reads as follows (amendment over claim 1 as granted in bold):

"1. A method for producing an organic-inorganic nanocomposite, comprising extrusion, in a single-screw extruder, of a polymer resin with a concentrate of an organic polymer and a non-clay inorganic additive, in the form of particles less than 100 nm in size, whose surface is **chemically modified** with organic functional groups which improve compatibility between the additive and the organic polymer."

VIII. The relevant arguments put forward by the appellant in its written submissions may be summarised as follows:

– The subject-matter of claim 1 as granted lacked novelty in view of D1. D1 disclosed the mixing of polymers with nanoparticles of silica (particle size 40 nm) and related therefore to a method for producing organic-inorganic nanocomposites. One example was Aerosil® Dispersion G320, which was, as shown by D2, a dispersion of silica nanoparticles in ethylene glycol. Since silica particles were highly adsorptive, ethylene glycol would adhere to the particles, i.e. cover the surface of the particles. The particles and the polymer formed a master batch, which was extruded with further polymer. For a person skilled in the art the term "extruder" was to be understood to mean a single-screw extruder, as could be seen from D8a and D8b. The ethylene glycol adsorbed onto the surface of the silica provided the organic functional groups

which improved the compatibility between the inorganic additive and the organic polymer.

– The subject-matter of claim 1 as granted lacked an inventive step in view of D1 or D4. D4 disclosed the manufacture of polyester fibres starting from a master batch made by mixing pyrogenic silica Aerosil® 300 with a particle size between 5-15 nm with a polyester resin. The mixture was extruded and the extruder given as an example was a double-screw extruder. Inorganic particles whose surface was covered with functional groups were not explicitly disclosed but were implicit in view of the technical background knowledge of the skilled person (e.g. D1 and D2). Thus the replacement of the double-screw extruder by a single-screw extruder, which was a standard apparatus, was an obvious choice for the manufacture of organic-inorganic nanocomposites.

– The subject-matter of claim 1 as granted also lacked an inventive step in view of D3, which disclosed the manufacture of organic-inorganic nanocomposites. The missing (non-explicit) features, such as the inorganic particle size and the organic cover of the inorganic particles, belonged to the technical background knowledge of the skilled person (reference was made to D1, D2 and D4).

IX. The relevant arguments put forward by the respondent in its written submissions and during the oral proceedings may be summarised as follows:

– The amendment to claim 1 of the main request fulfilled the requirements of Articles 123(2) and (3) EPC. Paragraph [0044] of the application as filed (International publication WO 02/096982 A1) disclosed that the surface of the inorganic particles was chemically modified. Furthermore, "chemical modification" was narrower in scope than "covering the particles".

– The subject-matter of claim 1 of the main request was novel over D1, which disclosed neither a single-screw extruder nor the chemical modification of the surface of the inorganic particles.

– The subject-matter of claim 1 of the main request involved an inventive step. D1 and D4 were irrelevant for the production of nanocomposites with improved compatibility between the additive and the organic polymer and should not be considered to represent the closest prior art. In fact, the prior art cited in the patent in suit and the problem to be solved in view of this prior art had to be seen in the provision of a further method for producing nanocomposites. The skilled person would not find any hint in the state of the art to chemically modify the surface of the inorganic particles in order to improve their compatibility with the organic polymer.

X. The appellant requested in writing that the decision under appeal be set aside and that European patent No. 1 401 924 be revoked.

XI. The respondent requested that the decision under appeal be set aside and that the patent be maintained on the basis of the following documents:

- claims 1 to 17 filed as the main request during the oral proceedings before the board;
- description
  - pages numbered 2, 4, 6-12 and 30 of the patent specification,
  - pages 3, 5 filed during the oral proceedings before the board;
- figures 1 to 14 of the patent specification.

Reasons for the Decision

1. The appeal is admissible.

2. Amendments under Articles 123(2) and (3) EPC

2.1 The subject-matter of claim 1 of the main request (sole request) differs from claim 1 as granted in that the surface of the particles of the non-clay inorganic additive have to be "chemically modified" with organic functional groups which improve the compatibility between the additive and the organic polymer.

This amendment is supported by paragraph [0044] of the application as filed, which discloses:

"In order to improve compatibility between the additive and the polymer matrix, ***the additive's surface is modified****. A surface modifier* ***will chemically react*** *with the functional groups on the additive's surface,*

producing functional groups that have similar physical properties as the base polymer(s) that will be used." (emphasis added)

Consequently, claim 1 satisfies the requirements of Article 123(2) EPC.

2.2 The term "chemically modified" now used in claim 1 is narrower in scope than the term "covered" in claim 1 as granted. A surface modifier will chemically react with the functional groups on the additive surface, producing functional groups that have similar properties to those of the base polymers that will be used (in order to improve compatibility between the additive and the base polymer). By contrast, the term "covered" can be interpreted to be much broader. Consequently the requirements of Article 123(3) EPC are also fulfilled.

3. Novelty

3.1 The appellant's novelty objection concerning the subject-matter of granted claim 1 was based on the disclosure of D1. This document discloses the extrusion of a mixture of polyester and the Aerosil® Dispersion G320 (page 4, tables 2 and 3), the latter containing silica particles of Aerosil® dispersed in ethylene glycol (D2: page 1, left-hand column), the particles having a mean particle size of 40 nm (page 3, table).

3.2 Nevertheless, claim 1 of the main request contains the limitation that the particle surface of the non-clay inorganic additive is chemically modified. This establishes an unambiguous difference over D1. As set

out above, a surface modifier will chemically react with the functional groups on the additive surface, whereas the ethylene glycol in the dispersion of D1 is at most reversibly adsorbed on to the surface of silica particles. This does not meet the criterion of chemical modification. Indeed it is explicitly stated in D1, table on page 3, footnote (7), that the silica particles have not been subjected to surface treatment.

Apart from that, D1 does not disclose a single-screw extruder. It is completely unfounded to say that the term extruder is generally short for single-screw extruder. There are many different types of extruder, and a single-screw is but one example. Neither D8a nor D8b nor any other document shows that the extruder is to be understood to mean a single-screw extruder.

3.3 On the basis of these differences the board concludes that the method of claim 1 of the main request is novel over the cited prior art.

4. Inventive step

4.1 The closest prior art

4.1.1 None of the documents cited by the appellant concerns a method for producing organic-inorganic nanocomposites, let alone for improving compatibility between the additive particles and polymer matrix to enhance the mechanical properties and the thermal stability of the nanocomposite.

4.1.2 D1 discloses antiblocking agents which comprise silica particles dispersed in ethylene glycol. These

antiblocking agents are used to minimize adhesion, which is particularly strong when two very smooth surfaces are in contact (page 1, left-hand column, lines 1-5). This is quite the opposite of the aim of particles in a nanocomposite, which reinforce the polymer. Thus, D1 is irrelevant to the manufacture of organic-inorganic nanocomposites with improved compatibility between the additive and the organic polymer.

4.1.3 D4 (claim 1) is also irrelevant since it relates to a method for the manufacture of polyester fibres with an increased production rate. For the extrusion step a two-screw extruder is given as an exemple (page 3, lines 26-35). Although nanoparticles of pyrogenic silica are used as part of the master batch in D4, their surface is not disclosed to have been chemically modified. The inorganic particles are merely used to slow down crystallization and thereby increase the production rate.

4.1.4 Under the present circumstances, the board considers that the methods disclosed in the patent specification (see paragraphs [0004] to [0013]) represent the closest state of the art since they relate to the production of organic-inorganic nanocomposites. Apart from the sol-gel process, *in-situ* polymerization, and solution blending, the extrusion of polymers and additives is also mentioned. However, the extrusion approach suffers from agglomeration of the inorganic phase.

4.2 The technical problem

4.2.1 Thus, in the light of the closest prior art cited in the patent in suit, the technical problem can be seen in the provision of a method for producing organic-inorganic nanocomposites in large quantities which overcomes the drawbacks of the state of the art, in particular agglomeration of the inorganic phase.

4.2.2 As a solution to this problem the patent proposes an extrusion method employing a non-clay inorganic additive, whose surface has been chemically modified, in a single-screw extruder.

4.2.3 The board is satisfied that the above stated problem is solved. The patent specification contains sufficient technical evidence that the method of claim 1 allows the manufacture of organic-inorganic nanocomposites in a simple and effective manner, whereby the problem of agglomeration is overcome, as can be seen from the physical properties of the obtained nanocomposites (tables 1 to 6).

4.3 The question of obviousness

4.3.1 The question which remains to be answered is whether the skilled person starting from the method of the prior art identified above would find in the available prior art any hint towards the claimed method for producing organic-inorganic nanocomposites.

4.3.2 As stated above, D1 and D4 do not disclose the chemical modification of the surface of the inorganic particles and consequently do not contain the necessary pointer

towards the claimed method. Such a hint can not be found in any of the other cited prior-art documents either. D3, an abstract of a Russian patent application, does not disclose the chemical modification of the surface of the zinc oxide particles. D6, a scientific study of polyethylene oxide grafted onto silica, has no relation to the preparation of organic-inorganic nanocomposites. Lastly, the Chemical Abstracts of D7 concern either a glycol-grafted filler for abrasion resistant films or grafted silica particles as pigments for the conductive coating of an electrographic paper. They are therefore irrelevant to a method for producing nanocomposites.

4.4 The board thus concludes that the subject-matter of claim 1 of the main request is not obvious from the available prior art and thus fulfils the requirements of Article 56 EPC.

5. In summary, the subject-matter of claim 1 of the main request is allowable.

6. Dependent claims 2 to 17, which correspond to specific embodiments of the method of claim 1, are *mutatis mutandis* allowable.

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 patent as amended in the following version:

   - claims 1 to 17 filed as the main request during the oral proceedings before the board;
   - description
     - pages numbered 2, 4, 6-12 and 30 of the patent specification,
     - pages 3, 5 filed during the oral proceedings before the board;
   - figures 1 to 14 of the patent specification.

The Registrar: The Chairman:

M. Cañueto Carbajo W. Sieber