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 Number: T 744/90 - 3.3.3

Application No.: 84 114 870.3

Publication No.: 0 144 983

Title of invention: Process for preparing polyacrylonitrile articles having high tensile strength and modulus

Classification: D01F 6/18

D E C I S I O N
of 18 December 1991

Proprietor of the patent: STAMICARBON B.V.

Opponent: 1. Bayer AG
2. Hoechst AG

Headword:

EPC Article 56

Keyword: "Inventive step (confirmed) - Hindsight analysis"

Headnote

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Beschwerdekammern | Boards of Appeal | Chambres de recours

Case Number : T 744/90 - 3.3.3

D E C I S I O N
of the Technical Board of Appeal 3.3.3
of 18 December 1991

Appellant : (Proprietor of the patent)
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Respondent 01 : (Opponent 01)
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Decision under appeal :
Decision of Opposition Division of the European Patent Office dated 29 May 1990, issued in writing on 16 July 1990, revoking European patent No. 0 144 983 pursuant to Article 102(1) EPC.

Composition of the Board :

Chairman : F. Antony
Members : C. Gérardin
R. Schulte

Summary of Facts and Submissions

I. The mention of the grant of the patent No. 0 144 983 in respect of European patent application No. 84 114 870.3 filed on 6 December 1984 and claiming the priority of 10 December 1983 from an earlier application in the Netherlands, was published on 28 October 1987 on the basis of 13 claims, the two independent claims reading as follows:

Claim 1: "Process for preparing articles having a high tensile strength and a high modulus by converting a solution of polyacrylonitrile into a solvent containing article, cooling the articles formed and, stretching them at increased temperature, this process being characterised in that
a. to a 0.5-15% (wt) solution of polyacrylonitrile or a copolymer of acrylonitrile with at most 15% (wt) comonomer with a weight-average molecular weight of 0.3 x 10^6 to 10 x 10^6 in a solvent suitable for that purpose a bivalent metal compound is added in a quantity of 0.01 - 0.2 mole per mole acrylonitrile units;
b. the solution thus obtained is converted at increased temperature into a solvent containing article;
c. this article is cooled to below the gelation temperature;
d. the gel article thus formed is largely freed of solvent and metal compound;
e. the article thus obtained is stretched at a temperature above the glass transition point of polyacrylonitrile and below the decomposition temperature of polyacrylonitrile."

Claim 13: "Filaments of polyacrylonitrile or a copolymer of acrylonitrile with at most 15% (wt) comonomer with a weight-average molecular weight of at least 0.3 x 10^6 to

10×10^6 having a tensile strength above 1.2 GPa and a modulus above 16 GPa."

Claims 2 to 12 are dependent process claims directed to preferred embodiments of the main claim.

II. On 25 July 1988 Opponent 1 filed a Notice of Opposition against the grant of the patent on the ground that the subject-matter of Claim 13 did not meet the requirements of novelty and inventive step under Article 100(a) EPC.

On 27 July 1990 Opponent 2 lodged an opposition to the granted patent and requested revocation thereof on the ground of lack of inventive step under Article 100(a) EPC of both the process according to Claim 1 and the filaments according to Claim 13.

These objections, which were emphasised and elaborated during oral proceedings, were based, on the one hand, on documents relevant to the issues of novelty and inventive step of the filaments according to Claim 13, and, on the other hand, essentially on the following documents

(7) US-A-4 344 308

(10) GB-A-692 462

as to the issue of inventive step of the process according to Claim 1.

III. By decision of 29 May 1990, issued in writing on 16 July 1990, the Opposition Division revoked the patent on the ground of lack of inventive step of the subject-matter of Claim 13 as granted, as well as proposed to be amended according to either of two auxiliary requests. By contrast, the process according to Claim 1, whose novelty had not been questioned, involved an inventive step, since the incorporation of bivalent metal compounds as mentioned

in document (10) could not be transposed to the process disclosed in document (7) regarded as the closest state of the art.

IV. The Patentee (Appellant) thereafter filed a Notice of Appeal against that decision on 15 September 1990 and paid the prescribed fee at the same time. Together with the Statement of Grounds of Appeal filed on 16 November 1990 the Appellant proposed various amendments to the claims as granted, to be considered as first and second auxiliary requests, the latter being limited to the process Claims 1 to 12 as granted.

During oral proceedings held on 18 December 1991, after an extensive discussion on the importance of the various parameters mentioned in the documents relied upon by the Opponents (Respondents) for the issues of novelty and inventive step of the filaments according to Claim 13, the Board informed the parties that it took the same view as the Opposition Division regarding the non-inventiveness of that subject-matter. Thereafter the Appellant decided to pursue the appeal procedure on the sole basis of the process Claims 1 to 12 as granted.

V. In favour of the patentability of these claims the Appellant argued that the documents relied upon by the Respondents did not suggest the general process features which would enable the skilled man to make articles having both a high tensile strength and a high modulus. More specifically, the process according to document (7), although theoretically suitable for the preparation of polyacrylonitrile (PAN) filaments, would not lead to the desired properties. As to document (10), it dealt basically with the technical difficulties occurring when highly viscous polymer solutions were spun; the mere fact that these difficulties could be overcome by the

incorporation of certain metal salts did not constitute an incentive for the skilled man to do the same for an entirely different purpose.

In his arguments, the Appellant also made reference to a document not mentioned before, i.e.

(12) "The Influence of Coagulation Variables on the Structure and Physical Properties of an Acrylic Fiber" by J.P. Knudsen, Textile Research Journal 33(1963), 13.

VI. The Respondents did not agree with this restrictive interpretation of documents (7) and (10). In their view, although the examples of document (7) only illustrated the production of polyolefin filaments, it was nevertheless specified that the spinning process described therein could suitably be carried out from PAN solutions as well. Further, the teaching of document (10) was relevant in the present case, since a reduction in viscosity would necessarily result in improved spinnability.
The Respondents additionally relied on two newly cited documents,

(11) Thesis of L. Dobrecov
(13) JP-A-54/160 820.

VII. The Appellant requested that the decision under appeal be set aside and that the patent be maintained on the basis of Claims 1 to 12 as granted.

The Respondents requested that the appeal be dismissed.

Reasons for the Decision

1. The appeal complies with Articles 106 to 108 and Rule 64 EPC and is, therefore, admissible.

2. The wording of the claims does not give rise to any objections under Article 123 EPC, since the present set of claims corresponds exactly to Claims 1 to 12 as originally filed and granted.

3. The Board has examined the late-filed documents (11) to (13) in order to determine their relevance, namely their evidential weight compared with that of the documents filed in time, and has found that none of them was sufficiently relevant to be taken into consideration. These documents are therefore disregarded hereinbelow pursuant to Article 114(2) EPC.

4. The patent in suit concerns a process for preparing PAN articles having high tensile strength and modulus. A similar process is described in document (7), which the Board, like the Opposition Division, regards as the closest state of the art. That citation discloses a four-step process for making polymer filaments, comprising (a) spinning a solution of a polymer, ranging from 1 to 5% by weight of polymer to solvent, through a spinning aperture to form a filament, (b) cooling said filament to below the solution temperature of the polymer without promoting the elimination of the solvent, (c) bringing said filament to a temperature between the swelling point of the polymer in the solvent and the melting point of the polymer, and (d) stretching the resulting filament (Claim 1). Tensile strength and modulus of the filaments can be controlled to a large extent by varying the stretching ratio in step (d), since both are about proportional to the stretching ratio applied (column 3, lines 41 to 53; Figures 2 and 3);

the values which can be obtained by the above process for these two parameters are said to be considerably higher for any given material than for filaments made by any of the usual dry spinning processes (column 2, lines 50 to 55). Although any material that can be dry spun to filaments may be used in principle (column 4, lines 4 to 14), the level achieved for the above parameters leaves to be desired in the case of PAN.

In the light of this shortcoming the problem underlying the patent in suit may thus be seen in providing a process for the preparation of PAN filaments which exhibit higher tensile strength and higher modulus.

According to the patent in suit this problem is to be solved by the addition of 0.01 to 0.2 mole, per mole acrylonitrile units, of a bivalent metal compound to the solution of PAN, the latter having a weight-average molecular weight of 0.3 to 10 x 10^6, prior to step (a).

In view of the experimental data in Table I of the patent in suit, which show the beneficial influence of such addition of a bivalent metal salt on both tensile strength and modulus of the resulting PAN filaments (compare Reference Examples I to VI with Comparative Example A), the importance of the valency of the metal of the salt added (compare Reference Example VI with Comparative Example B), and the criticality of the weight-average molecular weight of PAN (compare Reference Examples I to VI with Example XII), the Board is satisfied that the above-defined technical problem has been effectively solved.

5. After examination of the documents relied upon by the Respondents the Board has come to the conclusion that this technical teaching is not disclosed in any of them and

that the subject-matter of the patent in suit is, therefore, novel. Since the issue of novelty of the process has not been raised by the Respondents at any stage, it is not necessary to consider this matter in detail.

6. It still remains to be decided whether the subject-matter of the patent in suit as defined in Claim 1 involves an inventive step with regard to the teaching of the documents relied upon by the Respondents.

As argued by the Respondents, the incorporation of metal salts into solutions of PAN in organic solvents to be spun to filaments is known in the art. Document (10) describes such solutions containing 0.,1 to 5% based on the weight of the organic solvent of a metal salt selected from calcium chloride, lithium bromide, sodium thiocyanate and magnesium bromide (Claim 1). These solutions are said to have a relatively low viscosity and increased fluidity or mobility as compared to the viscosity and mobility of the solutions normally obtained by intimately mixing the fibre-forming PAN with the organic solvent (page 1, lines 13 to 69). This in turn permits the steps of washing, drying and heat-stretching the fibres to be performed continuously at high speed (page 2, lines 10 to 23).

In the Board's view, however, it is not clear how the beneficial effect on viscosity and the further advantages resulting therefrom mentioned in document (10) could be related to the improvement of the mechanical properties of PAN filaments; more specifically, the Respondents failed to demonstrate how improved spinnability achieved by the incorporation of metal salts into PAN solutions could be an incentive for the skilled man to do the same in order to improve tensile strength and modulus according to the

above-defined technical problem. In reality, the authors of that citation have not identified the parameters which are essential for the improvement of these two properties and go beyond the mere incorporation of metal salts. In particular, nothing in document (10) would suggest that the valency of the metal and the weight-average molecular weight of PAN could be critical features, as the Appellant demonstrated convincingly (see point 4 above, paragraph 4), since no difference is shown between the four suitable salts which are derived from metals of different valency; nor is there any reference at all to any particular range of weight-average molecular weight being preferred.

A further point to be considered is the fact that the rather complicated process envisaged in document (10), at least as it is illustrated in Example VI thereof, bears little resemblance to the four-step process described in document (7). There would thus be no incentive for the skilled man having the existing problem in mind, to consider a specific feature in isolation, i.e. the incorporation of metal salts, from document (10); to modify it appropriately by selecting bivalent metal salts; and to combine it with the general process according to document (7). In the Board's view, such a combination would not be made without knowledge of the teaching of the patent in suit.

For these various reasons, the Board regards the subject-matter according to Claim 1 of the patent in suit as unobvious with regard to the teaching of documents (7) and (10); since the other documents relied upon by the Respondents are even less relevant in respect of the improvement of tensile strength and modulus, an inventive step can be acknowledged.

7. Claim 1 being allowable, the same applies to dependent Claims 2 to 12, which are directed to preferred embodiments of the subject-matter of Claim 1 and whose inventiveness is supported by that of the main claim.

Order

For these reasons, it is decided that:

1. The decision under appeal is set aside.

2. The case is remitted to the first instance with the order to maintain the patent on the basis of Claims 1 to 12 as granted.

The Registrar: The Chairman:

E. Görgmaier F. Antony