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Veröffentlichung im Amtsblatt	Ja/Nein
Publication in the Official Journal	Yes/No
Publication au Journal Officiel	Oui/Non

Aktenzeichen / Case Number / N° du recours : T 176/89 - 3.3.3

Anmeldenummer / Filing No / N° de la demande : 83 307 928.8

Veröffentlichungs-Nr. / Publication No / N° de la publication :

Bezeichnung der Erfindung: Process for producing stretched articles of ultrahigh-molecular-weight polyethylene
Title of invention:
Titre de l'invention :

Klassifikation / Classification / Classement : D01F 6/46

ENTSCHEIDUNG / DECISION

vom / of / du 27 June 1990

Anmelder / Applicant / Demandeur :

Patentinhaber / Proprietor of the patent / Titulaire du brevet : Mitsui Petrochemical Industries, Ltd.

Einsprechender / Opponent / Opposant : DSM Research B.V.

Stichwort / Headword / Référence : UHMV polyethylene/MITSUI

EPÜ / EPC / CBE Art. 56

Schlagwort / Keyword / Mot clé : "Inventive step - confirmed" - "Combination of conflicting citations"

Leitsatz / Headnote / Sommaire

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Case Number : T 176/89 - 3.3.3

D E C I S I O N
of the Technical Board of Appeal 3.3.3
of 27 June 1990

Appellant : (Proprietor of the patent)
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Decision under appeal :
Decision of the Opposition Division of the European Patent Office dated 22 November 1988, issued 11 January 1989 revoking European patent No. 00 115 192 pursuant to Article 102(1) EPC.

Composition of the Board :

Chairman : F. Antony
Members : R. Lunzer
J. Stephens-Ofner

Summary of Facts and Submissions

I. European patent No. 115 192 was granted on 21 May 1986 on the basis of application No. 83 307 928.8 filed on 23 December 1983, having priority dates of 28 December 1982 and 7 April 1983 derived from Japanese Applications Nos. 227447/82 and 59976/83.

II. On 18 February 1987 an opposition was lodged by the Respondent on the ground of Article 100(a) EPC, alleging lack of novelty (Article 54 EPC), and/or lack of inventive step (Article 56 EPC). The Opponent referred in its statement of grounds to nine documents, of which the following in particular were referred to in the Appeal proceedings:

(1) GB-A-2 042 414
(2) GB-A-2 051 667
(6) Applied Polymer Symposia 6 (1967) pages 109 to 149
(7) JP-A-37-9765
(9) US-A-4 130 618.

III. By its decision given orally on 22 November 1988 and issued in writing on 11 January 1989, the Opposition Division revoked the patent, holding that the alleged invention was lacking in any inventive step having regard to the teachings of documents (1) and (9) above.

IV. An appeal against this decision was lodged on 10 March 1989, the appeal fee was paid on the same day, and the Grounds of Appeal were filed on 16 May 1989. The Appellant sought the reversal of the decision of the Opposition Division, and introduced main and auxiliary requests directed to claims which were further limited in an attempt to overcome previous objections. Claim 1 of

the main request as amended on 25 May 1990 was in the following terms:

"A process for producing a stretched filament of ultrahigh-molecular-weight polyethylene, which comprises

(1) melt-kneading a mixture composed of

(A) 15 to 80 parts by weight, per 100 parts by weight of components (A) and (B) combined, of ultrahigh-molecular-weight polyethylene having an inherent viscosity (η), determined at 135°C in decalin, of at least 5 dl/g and

(B) 85 to 20 parts by weight, per 100 parts by weight of the components (A) and (B) combined, of a paraffinic wax having a melting point, determined by the DSC method, of 40 to 120°C and a weight average molecular weight (Mw), determined by the GPC method, of at least 230 but less than 2,000 in a screw extruder while maintaining the temperature of the mixture at 180 to 280°C,

(2) melt-extruding the molten mixture through a spinneret type die kept at a temperature of 180 to 300°C to form a spun filament, a draft being applied to the spun filament at a draft ratio of not less than 2,

(3) cooling the resulting drafted filament to solidify it, and

(4) subjecting the solidified filament to a stretching treatment at a temperature of 60 to 140°C at a stretch ratio of at least about 3:1."

V. In the course of its written and oral submissions, the Appellant contended that the demand for such a product as

a strong rope made of ultra-high molecular weight polyethylene had long been known to the industry, and that whereas prior processes had been disclosed which enabled high tensile strength and modulus of elasticity to be attained, they could not be operated economically, and on a mass production scale, because they were only suited to ram extrusion. In contrast, the alleged invention disclosed a method which overcame all the problems which had until now prevented the commercial exploitation of ultra-high molecular weight polyethylene as a filament for widespread use in commerce, such as in rope making.

VI. The Respondent contended that the prior art, including documents (1) and (2), showed the way ultra-high molecular weight polyethylene filaments of very high tensile strength and modulus of elasticity could be produced, using a very dilute solution of the polymer in decalin. The alternative of using wax as a solvent was also known, such as from document (9). It was common knowledge to use drafting, when seeking to improve mechanical properties, but to confine its use to situations where there was a high concentration of polymer, such as when using a wax solvent, but not when using a very dilute solution, such as was proposed in (1) and (2). Accordingly, a skilled worker, if seeking to use the process of these prior disclosures, but modified by the substitution of wax for decalin as the solvent so as to be able to make use of a screw extruder, would as a matter of course make use also of the well known step of drafting.

VII. Oral proceedings were held on 27 June 1990. The Appellant requested that the decision under appeal be set aside, and that the patent be maintained on the basis of the amendments submitted on 25 May 1990 in its main or its auxiliary requests. The Respondent requested that the appeal should be dismissed.

Reasons for the Decision

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

2. Admissibility of amendments

2.1 While Claims 2 to 5 stand as granted, Claim 1 has been amended in the following respects:

(i) whereas the Claim as granted related to an "article", this is now limited to a (spun) "filament";

(ii) it is now specified that the die is of the "spinneret type";

(iii) the previously optional feature of drafting (cf. step (4) of both the original and the granted version of Claim 1) is now made essential, and a minimum draft ratio of not less than 2 is specified.

2.2 With regard to the above proposed amendments, in the view of the Board:

(i) the restriction of Claim to a "spun filament" is merely a limitation to what is in fact disclosed in Examples 1 to 6 inclusive of the specification, both as published and as filed;

(ii) the use of a spinneret with an orifice diameter of 1 mm is disclosed in each of the aforesaid Examples;

(iii) the application of a draft ratio of not less than 2 is disclosed in the application as filed at page 14 lines 8 to 11 (corresponding to page 5 lines 46 to 47 of the specification as published).

2.3 Accordingly, these limitations to the scope of the Claims are an allowable amendment in accordance with the provisions of paragraphs (2) and (3) of Article 123 EPC.

3. Background to the invention

3.1 Ultra-high molecular weight polyethylene has long been known as a useful plastic material. It has a high melt viscosity, which makes it very difficult to extrude in conventional equipment. Document (7) is a patent specification published in 1962, the application having been made in 1959. It relates to proposals for spinning ultra-high molecular weight polyethylene by including in it an additive having a boiling point higher than the melting point of the ethylene polymer. It includes a number of examples of possible additives, which may be included at a rate of 20% to 150% in proportion to the polymer. This document shows that the problem of finding a method of making useful filaments of ultra-high molecular weight polyethylene is one which has confronted the industry for a great many years prior to the priority date of the patent in suit.

3.2 Although a number of documents have been brought into these proceedings which relate to the desirability of making filaments of ultra-high molecular weight polyethylene, against the background of its known intractability and the resultant difficulties of extrusion, there is no evidence that the problem had ever been solved to the extent of giving rise to any commercially useful process.

4. The invention

4.1 The specification contains at page 2 line 23 to page 3 line 11 a discussion of the known advantages of ultra-high molecular weight polyethylene in terms of its superior impact strength, abrasion resistance, chemical resistance, and tensile strength. Although it has found wide application in engineering plastics, the difficulties of extrusion had thus far prevented its use as a filament. Furthermore, although it had been proposed to improve its flowability, such as by the inclusion of liquid solvents including decalin, such solvents acted as lubricants in screw extruders, preventing them from working, and giving rise to flammability risks. Thus, when using decalin, extrusion had to be performed in a ram extruder, which is non-continuous, and therefore economically disadvantageous.

4.2 At page 2 line 61 to page 3 line 23 reference is made to a prior Japanese specification which refers to the use of wax to improve flowability of ultra-high molecular weight polyethylene, but not in the context of drawn films or filaments. The alleged invention, as now contained in the proposed amended Claim 1, resides essentially in the extrusion from a screw extruder of a mixture of ultra-high molecular weight polyethylene with paraffinic wax, mixed in the roughly equal proportions as specified in the Claim, drafting prior to solidification, cooling to solidify, and stretching at a temperature between 60 to 140°C.

5. Closest prior art

5.1 Although there is a significant difference between their respective teachings, the Board regards documents (1) and

(2) read in conjunction with each other as constituting the closest prior art. They are exceptionally read together because they belong to the same patentee (in fact an affiliated company of the present Respondent), were invented by substantially the same inventors, and apparently relate to the same series of investigations.

5.2 Document (1) teaches the production of ultra-high molecular weight polyethylene filaments, having high tensile strength and modulus of elasticity, by dissolving the polyethylene in a large excess of a volatile solvent and then solution spinning using a ram extruder. Although at page 2 line 63 it is taught that in dry spinning processes as usually applied, 5 to 30% by weight of polymer may be included in the solvent, and that such concentrations can be used in accordance with its proposals, in fact at line 68 a preference is expressed for a concentration of 1 to 5%, while in all three examples a solution at 2% concentration is used.

5.3 The most important teachings of this document are the following:

"Surprisingly, the process according to the invention can produce filaments that are considerably stronger than filaments made by any of the usual dry spinning processes that is to say, filaments of considerably higher tensile strength and modulus." (Page 2 lines 2 to 8)

5.4 It continues at line 14 to identify what is the kernel of its proposals in these terms:

"The process according to the invention differs from the usual dry spinning process in that a filament containing an appreciable amount of solvent for the spinnable material is stretched at a temperature at which the

spinnable material will at least swell in the solvent, with removal of the solvent, whereas in the usually applied spinning processes solvent free filaments are subjected to stretching."

5.5 At page 2 lines 26 to 30 it deals with the nature of the solvent, and teaches that it must be so volatile that it is not difficult to evaporate the solvent from the filament. At page 2 lines 34 to 46 it is explained that the small proportion of polymer in a much larger proportion of volatile solvent gives rise to a gel consistency, and this is emphasized in Example 1 at page 3 lines 37 and 38, which indicate that the filament was gel-like and contained about 98% of solvent.

5.6 Fig. 1, which schematically illustrates apparatus working in accordance with that invention, shows a ram extruder, and there is no suggestion anywhere in that document that it could be replaced with a screw extruder.

5.7 To summarize, in the Board's view document (1) teaches that ultra-high molecular weight polyethylene filaments having high tensile strength and modulus of elasticity can be attained provided certain conditions are satisfied, notably, by solution spinning using a very large excess of a volatile solvent compared with the polymer, deliberately retaining the solvent after cooling so that a gel is formed, and ensuring that the solvent is present to a large extent in the drawing step.

5.8 Document (2) indicates that its inventors were two out of the team of three who were responsible for document (1). Although at page 1 line 87 to page 2 line 13 there is an extensive discussion of the many attempts which had been made prior to its priority date (June 1980) to solve the problems associated with making strong filaments out of

ultra-high molecular weight polyethylene, no mention at all is made of the earlier application, document (1), filed by the same company only four months previously.

5.9 That fact becomes less surprising when it is observed that in a very material respect its teaching is exactly the opposite of document (1). At page 2 lines 70 to 79 it teaches that all or part of the solvent may be evaporated prior to drawing, while Figures 1 and 2 seem to show that, contrary to the teaching of document (1), it hardly matters whether the solvent is present to a significant extent or not during drawing.

5.10 It comments at page 4 lines 1 to 3 that "somewhat" higher draw ratios can be achieved in drawing solvent-containing filaments. Better properties are shown to have been achieved when solution spinning polymers of relatively high molecular weights. The Examples also illustrate the use of a range of concentrations of polymer in solvent from 1% to 8%, the solvent of choice always being decalin.

5.11 At page 3 lines 48 to 54 it teaches as follows:

"Surprisingly, it has been found that filaments with a greater modulus and strength can be produced by the present method than by melt spinning of the same polymer, drawing conditions being as far as possible the same, e.g. with the same drawing temperature and drawing rate."

The immediately following lines refer to the benefits which result from using the larger spinning apertures associated with the spinning of dilute solutions.

5.12 To summarize, the skilled reader, taking document (2) in conjunction with document (1), may reasonably conclude

that (1) was in error in its emphasis on the need to have a large proportion of solvent present during the drawing step, although it is preferable for obtaining the best results. He could not fail to notice the emphasis placed on solution spinning from dilute solutions, as contrasted to melt spinning, as being essential to the attainment of the desired good mechanical properties.

6. The problem

Documents (1) and (2) teach the use of solution spinning as the way of making ultra-high molecular weight polyethylene filaments having high tensile strength and modulus of elasticity. The drawback of the process disclosed is that it can only work with a ram extruder, for reasons which are explained in the patent in suit. Therefore, against this background the objective problem can be seen as the need to overcome the very serious drawback of having to make use of ram extrusion, while producing filaments having mechanical properties which are of the same order as those shown by these citations.

7. Solution and its effectiveness

7.1 The solution to the above problem proposed by the patent in suit essentially involves two steps which differentiate it from the teachings of documents (1) and (2). First, there is the adoption of melt spinning from wax as a solvent, which is used in much smaller proportions than the solutions in decalin proposed in the cited documents. Secondly, there is the drafting step, which does not figure at all in these citations. In Table 9 on page 12 of the patent in suit the best result given is the achievement of a modulus of 50.6 GPa and a tensile strength of 2.43 GPa at a stretch ratio of 9.2. This compares favourably, insofar as the data can be regarded

as comparable, with some of the figures given in document (1). In Table 1 it shows that with a molecular weight of 1.5×10^6, and a stretch ratio of 11, the modulus is 23.9 GPa, and the tensile strength 1.32 GPa.

7.2 Although at the oral proceedings considerable attention was directed to comparisons of mechanical performance data, the Board is not satisfied that it has been shown that the tensile strength and modulus of elasticity of filaments made in accordance with the alleged invention is necessarily either better or worse than the properties which can be attained in accordance with these citations. The figures in relation to tensile strength and modulus of elasticity produced by the Appellant satisfy the Board that the claimed process is capable of producing a useful product, while the Respondent has not produced any evidence to show that the mechanical properties of filaments made by melt spinning using wax as a solvent, as proposed in the present alleged invention, are significantly inferior to the properties of filaments made by solution spinning in accordance with documents (1) and (2).

7.3 It is not disputed by the Respondent that the process in accordance with the alleged invention is capable of operation in a screw extruder, whereas that in accordance with this prior art is not. Accordingly, the issue of whether or not the process according to the invention is capable of producing filaments which are mechanically superior to those of these citations is left open by the Board, because it does not matter. The Board is satisfied that the process according to the invention affords a credible advantage, because it is capable of continuous operation, whereas ram extrusion disclosed in documents (1) and (2) is not. In this sense the existing technical problem has been solved.

8. Novelty

Having reviewed all the cited documents, the Board is satisfied that none of them discloses a process for producing a stretched article of ultra-high molecular weight polyethylene having all the features defined in Claim 1. As novelty was not seriously contested on appeal, the Board accepts that the subject matter of Claim 1 is novel within the meaning of Article 54 EPC.

9. Inventiveness

9.1 The issue of inventiveness turns on whether a skilled person, having as his starting point the disclosure of documents (1) and (2), and confronted with the problem of overcoming the obvious disadvantage of those disclosures, i.e. the necessity to use a ram extruder, would have been led by the application of other knowledge in the art to replace solution spinning from a very dilute solution of polymer in a volatile solvent such as decalin with melt spinning in a modest proportion of a paraffinic wax, and at the same time to employ a drafting step as an essential part of the process.

9.2 As for the drafting step, the Board does not regard this as a significant distinction. In the light of present day knowledge in relation to the making of high strength filaments, the Board takes the view that the skilled worker would, as a matter of course, experiment with various take-up speeds, including such speeds as would introduce a measure of drafting, in order to ascertain whether or not some drafting before drawing is beneficial. Equally, the Board finds it difficult to see any significance in the lower limit of a draft ratio of not less than 2, because this is a modest lower limit, and

one which seems likely to be exceeded if deliberate drafting is introduced at all. However, in view of what follows, this distinction need not be relied on in support of inventiveness.

9.3 Regarding the combined teachings of documents (1) and (2), the Board can see no reason why a skilled worker, possessed of ordinary skill and knowledge in the art, and seeking to overcome the problem formulated above, would have any reason to turn away from solution spinning from a large volume of volatile solvent in favour of melt spinning from a much smaller proportion of wax. The drawback of solution spinning, with its inherent limitation to the use of uneconomic ram extrusion, must have been evident to any worker in the art reading documents (1) and (2). If it had been obvious that high molecular weight polyethylene could readily be spun from a roughly equal proportion of wax, it is surprising that that simple solution to the problem of spinning this intractable polymer does not seem to have occurred to anyone since the publication of document (7) in 1962, nor does it seem to have occurred to the authors of documents (1) and (2). Therefore, in the Board's view, the objection of lack of inventiveness in the light of the teachings of these two documents is not established.

9.4 Turning next to document (9), this relates essentially to mouldings or extrusions which can be made by dissolving ultra-high molecular weight polyethylene in a roughly equal amount of paraffin wax. Although no specific extruder is identified, the Board is satisfied that the commonly used kind of screw extruder is referred to when extrusion is mentioned.

9.5 At column 4 lines 61 to 68 possible uses for its compositions are indicated. These include articles such as

bottles, toys, cable jacketing, and also films or tapes which may be oriented. Example IV Part B is directed to what is described as being a highly biaxially oriented film. Reference is also made at column 5 lines 10 to 12 to the possibility of making fibrillated fibres out of uniaxially oriented film, but it is not exemplified. Apart from this brief reference, there is no mention whatever of the manufacture of extruded fibres or filaments, nor is there any indication of the strength of the fibres which might be obtained from fibrillated uniaxially oriented film.

9.6 Considering the issue of inventiveness against the background of the combination of the teachings of documents (1) and (2), together with document (9), attention has first to be directed to the teaching of document (2) at page 3 lines 48 to 54 already quoted at paragraph 5.11 above. This is to the effect that melt spinning is not compatible with the achievement of optimum mechanical properties. In the view of the Board, the skilled worker would take that teaching seriously, and would be unlikely to abandon the solution spinning taught so emphatically by documents (1) and (2) in favour of the melt spinning of document (9).

9.7 Some reliance was also placed by the Respondent on document (6). This discloses at page 130 Experiment No. 24 the extrusion of 5% of high molecular weight polyethylene in a 50:50 mixture of naphthalene and paraffin wax, with a negative draw down or draft ratio of 92.5%. This Experiment involves the use of a polymer to solvent concentration which is well below that claimed in Claim 1 of the patent in suit, and the negative draft ratio is a further pointer away from the alleged invention. The Board does not regard this document, either alone, or in combination with any other, as leading towards the alleged

invention. Accordingly, the Board is not satisfied that the objection of lack of inventive step has been proved.

10. The decision under appeal

10.1 In the decision under appeal, the Opposition Division apparently identified document (1) as being the closest prior art, teaching a way of making strong fibres out of high density polyethylene, but making use of a ram extruder. It then correctly identified the problem (on the basis of Claim 1 as it was then) as being to find a process for producing stretched articles such as stretched filaments, films or the like of ultra high molecular weight polyethylene by using the industrially advantageous screw extruder.

10.2 However, it is to be noted that document (1) teaches that the desired properties were only attainable if the volatile solvent were present during spinning. Thus, in the view of the Board, it is illogical to combine the teachings of documents (1) and (9), (1) teaching the use of a volatile solvent, and (9) teaching the use of a wax solvent, when in fact (1) taught that its desired results were attainable if, and only if, the essential part of its teaching were adhered to. In fact, the teachings of two documents ought rarely, if ever, to be combined, when it is apparent that their teachings are mutually conflicting.

10.3 Furthermore, although document (2) was cited with the Grounds of Opposition, it was not referred to by the Opposition Division. That would not matter if it had been irrelevant to the matters in issue. However, instead of being irrelevant, document (2) contains two teachings which are significant to the evaluation of the existence of any inventive step. First, although filed by the

authors of document (1) only four months after the filing of document (1), it casts serious doubt on the validity of the teachings of the earlier document. Secondly, it is to be observed that in document (2) at page 3 lines 48 to 54 there is a teaching to the effect that filaments with a greater modulus and strength could be produced by its method, i.e. the use of a large proportion of volatile solvent, than by melt spinning the same polymer.

10.4 This explicit teaching would discourage a skilled worker, seeking to produce strong filaments, from turning from the solution spinning of document (1), to the melt spinning of document (9). Before rejecting any patent as lacking in inventive step, all the documents in the case ought to be read carefully, to ensure that the decision is not based on a selection involving too much hindsight. It is important not to overlook pointers in the direction opposite to the alleged invention, particularly if they are the kind of pointers would have been taken seriously by any skilled worker in the art who did not start from actual knowledge of the invention.

11. Conclusion

For the reasons given above, the Board is satisfied that Claim 1 as proposed to be amended covers an invention which is non-obvious, and therefore patentable having regard to Article 56 EPC. The remaining claims are all dependent on Claim 1, and therefore derive their patentability from Claim 1.

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 with its claims as set out in the main request of 25 May 1990, with consequential amendments to the description to be submitted by the Appellant.

The Registrar: The Chairman:

M. Beer F. Antony