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т 253/85

Anmeldenummer / Filing No / No de la demande : 80 200 516.5

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Bezeichnung der Erfindung: Process for the manufacture of fibres from

Title of invention:

poly-p-phenylene terephthalamide

Titre de l'invention:

Klassifikation / Classification / Classement : D 01 F 6/60

entscheidung / Decision vom/of/du 10 February 1987

Anmelder / Applicant / Demandeur :

Patentinhaber / Proprietor of the patent /

Akzo (Respondent)

Titulaire du brevet :

du Pont (Appellant)

Einsprechender / Opponent / Opposant :

"Dry jet-wet spinning/AKZO"

Stichwort / Headword / Référence :

Articles 52, 54(2), 56 and 114 EPC

EPÜ / EPC / CBE

"Inventive step"

Kennwort / Keyword / Mot clé : "NOV

"Novelty test - Testing for non-identity

through properties"

"Late submission of documents - Both parties

relying on reference"

Leitsatz / Headnote / Sommaire

In inter partes cases, a new reference filed by the Appellant (Opponent) with his Statement of Grounds and accepted by the Board, may be fully considered by the Board of Appeal without remitting to the first instance, if the Respondent (Patentee) not only refrains from raising any objections to the document being considered but also argues that the same document is in his own favour. A further condition for such full consideration of the reference by the Board itself is that it does not introduce any new aspect making it proper to remit the case to the Opposition Division in order to avoid bypassing one level of jurisdiction (cf. T 273/84 "Silico-aluminate", OJ EPO 10/1986, page 346, in particular point 6 of the reasons for that decision).

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Case Number : T 253 /85



DECISION
of the Technical Board of Appeal 3.3.2
of 10 February 1987

Appellant: (Opponent)

E.I. du Pont de Nemours and Company Wilmington, Delaware 19898

USA

Representative :

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Representative :

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Decision under appeal:

Decision of Opposition Division of the European Patent Office dated 25 April 1985 (posted on 5 August 1985), rejecting the opposition filed against European patent No. 21 484 pursuant to Article 102(2) EPC.

Composition of the Board :

Chairman : P. Lançon
Member : G. Szabo

Member : E. Persson

Summary of Facts and Submissions

- I. European patent No. 21 484 was granted with 11 claims in response to the European patent application No. 80 200 516.5 filed on 3 June 1980 claiming the priority of the earlier application of 8 June 1979. Claim 1 is worded as follows:
- 1. A process for the manufacture of fibres from a polyamide containing more than 95 mole % of poly-p-phenylene terephthalamide, by spinning a spinning mass having a temperature of 20°-120°C and consisting of a mixture of concentrated sulphuric acid having a strength of at least 96% by weight and, calculated on the weight of the mixture, at least 15% of polyamide having an inherent viscosity of at least 2,5, the spinning mass being downwardly extruded into a coagulation bath using the dry jet-wet spinning technique, characterized in that the spinning mass is prepared by the successive steps of cooling the concentrated sulphuric acid to below its solidifying point, combining the sulphuric acid thus cooled and the polyamide and intermixing the two substances into a solid state mixture and heating the resulting solid state mixture to spinning temperature.
 - II. The Appellant (Opponent) filed an opposition against the grant of the patent on 22 September 1983 on grounds of lack of novelty and/or of inventive step. The following documents were, inter alia, cited in support of the opposition:
 - (1) DE-A-2 219 703
 - (2) JP-A-50 51 552
 - (4) US-A-3 817 941
 - (5) US-A-4 018 735
 - (6) DE-B-1 810 426, and

- (7) JP-A-49 78 163, representing the priority document of reference (5).
- III. The Opposition Division rejected the opposition in a decision notified on 5 August 1985. According to the decision the main claim was novel since none of the cited documents had disclosed all the features of the process. The closest state of the art according to (1) taught the admixture of poly-p-phenylene terephthalamide (PPDT) with sulphuric acid at elevated temperatures to provide a spinnable homogeneous mixture. None of the documents suggested to freeze the sulphuric acid before admixture. Although comparative examples in (2), (5) and (7) suggested the mixing of the two components at O°C, there was no hint that this implied a pre-freezing of sulphuric acid. The skilled practitioner would have rather combined the same with PPDT at room temperature and cooled the same down to control the developed heat to avoid loss of quality. The cited examples rather showed that a spinnable mass had only been obtained after heating to 90°C or above. Thus nothing in the prior art would have led the skilled person to the use of the characterising condition of the invention.
 - IV. The Appellant filed an appeal against this decision on 7 October 1985 with the payment of the appropriate fee and submitted a Statement of Grounds on 11 October 1985. At the same time the Appellant filed a new reference, JP-B-18612/71 with a certified translation (13). An oral hearing was held on 10 February 1987.
 - V. The Appellant submitted during the proceedings and at the oral hearing substantially the following arguments:

- (a) The risk of decomposition in consequence of exposure and prolonged stirring clearly called for mild conditions and lowering the temperature. It was not only that there had been no prejudice against going below the freezing point of sulphuric acid but the cited documents, in particular the comparative examples of (2), (5) and (7) showed that it was already quite normal to operate at 0°C.
- (b) Although comparative Example 1 of (5) only showed a low concentration (2.0 g of PPDT with 80g sulphuric acid) this must have been clearly a typing error frequently happening when Japanese applications were translated in to English. The inadvertent shift of the decimal point could also be seen with Example 2 (col. 7, line 11) showing a ratio of 2.2g/78g when compared to the corresponding Example 1 in the priority document (7), indicating correctly 7.8g for the acid, i.e. a 22% concentration. In the absence of the same example in the priority application, the only reasonable conclusion for comparative Example 1 was to take the 10 times higher value since otherwise the mixture would have been uniform at 40°C. The fact that this was not so and that "its flowability became superior" at 90°C was indicative that the earlier mixing at 0°C was as effective as the pre-freezing according to the patent at the same temperature. No technical achievement distinguishing the claimed process from the state of the art was recognisable.
- (c) As further proof that the use of ice/water bath was perfectly natural in these circumstances at the admixing stage, documents (4) and (6) were cited (i.e. col. 23, lines 12-20, respectively). In both cases PPDT

was mixed with sulphuric acid under such cooling conditions, without running into difficulties or disadvantageous results.

(d) In view of the above, there could have been no problem in applying the teaching of document (13) using frozen solvents to that of (1), in order to carry out the process under conditions which helped to avoid damage due to heat. This reference was relevant to fibres manufactured by the "wet or dry process" and suggested that as a first step the polymer material should be mixed with a frozen solvent. The disclosure generalised the idea to any solvent and mentioned m-phenylenediamine-isophthalic acid as a polymer. The subjectmatter of the present patent was an obvious application of, and a non-inventive selection from, the possibilities suggested in this citation.

VI. The Respondent (Patentee) argued substantially as follows:

- (a) Whilst the concentration of PPDT in sulphuric acid might have been subject to a typing error in the comparative Example 1 of (5), the skilled person was in no position to know what the correct value should be in the circumstances. There was no corresponding Example in the priority document. A notional correction was therefore impossible. The other cited processes in (4) and (6) employed concentrations no more than 12% and could not therefore be indicative of the problems arising substantially above that value.
- (b) If anything, comparative Examples 1 in (2) and (5) as well as comparative Example 2 of (7) showed that lack of uniformity, i.e. heterogeneity and unsatisfactory consistency characterised these systems representing a 20% concentration of PPDT after cooling to 0°C. In all

these instances heating to 130°C was necessary to achieve spinnability. This was also evidence to the effect that no pre-freezing of sulphuric acid could have been involved and that exactly this critical measure provided spinnability at 80°C.

- (c) Newly cited document (13), if anything, supported the submission that the omission of PPDT and sulphuric acid from the document was for a good reason, i.e. in view of the prevailing trend it was not expected at all to be applicable to systems which use neither wet nor dry spinning but the special wet-dry spinning representing different conditions.
- (d) None of the citations would enable the skilled person to predict the advantageous possibility made available by the invention, including the simplified preparation of the spinning mass.
- VII. The Appellant requests that the decision under appeal be set aside and the patent be revoked. The Respondent requests that the appeal be dismissed and the patent be maintained.

Reasons for the Decision

- 1. The appeal complies with Articles 106 to 108 and Rule 64 EPC and is, therefore, admissible.
- The Board has considered whether reference (13), filed by the Appellant for the first time in the appeal proceedings, should not be disregarded under Article 114(2) EPC as having not been submitted in due time. However, it is to be

noted that the Respondent has not only refrained from raising any objections to this reference being considered in the course of the appeal proceedings but even argues that the same document is in his own favour.

Furthermore, reference (13) does not introduce any new aspect but is mainly to be regarded as a complement to references already considered by the Opposition Division. Thus, there is no need for remitting the case to the Opposition Division in order to avoid bypassing one level of jurisdiction (cf. T 273/84 "silico-aluminate", OJ EPO 10/1986, page 346, in particular point 6 of the reasons for the decision).

In these circumstances the Board is prepared not only to admit document (13) in view of its relevance into the proceedings but also to fully consider it in the appeal stage.

The claimed subject-matter relates to the spinning of a 3. mass of PPDT in sulphuric acid at elevated temperatures. According to the closest state of the art (cf. (1), Example 1) the components are admixed at room temperature and then heated to 95°C for spinning at 100°C. It was recognised that the prolonged homogenisation of the system resulted in a partial decomposition of the polymer and loss in tenacity, particularly at high concentrations of PPDT. This represented a technical problem which is resolved according to the patent in dispute by mixing first solidified sulphuric acid with the polymer at substantially reduced temperatures and then heating the mixture gradually to a spinning temperature reduced to 80°C. According to the examples of the patent (e.g. Example I), the heat sensitivity index of the fibres was reduced when compared to cases in which the sulphuric acid had been admixed at room temperature (Example II) (cf. col. 17, lines 5, 35 and

- 65). The solution of the technical problem, i.e. the improvement of quality and the elimination of the necessity to raise the temperature to 100°C or above, has therefore been achieved with spinning masses possessing the desired minimum of 15% concentration.
- Although there was no dispute about novelty at the oral proceedings before the Opposition Division, the Appellant raised the issue again with reference to the comparative examples in (2), (5) and (7) and argued that cooling to 0°C must mean the use of frozen sulphuric acid. The Board, however, agrees with the Respondent that the 130°C processing temperature in these cases and the unsatisfactory quality of the mixture rendering the dopes unsuitable for spinning at substantially lower temperatures must lead to the conclusion that none of the advantages of admixing with pre-frozen sulphuric acid were apparent and that the disclosures could not therefore be taken as anticipating the subject-matter of the present patent.
- As far as the confusing comparative Example 1 of (5) is 5. concerned, the low concentration of PPDT prima facie suggests that this embodiment is apparently outside the scope of the claims of the present patent. Notwithstanding the fact that contradictions within the Example might point to a higher concentration, there is no way to establish the correct value with any degree of certainty on that basis or by reliance on common general knowledge. Apart from shifting the decimal point, some other value could be equally correct. The former approach would bring the concentration very close to the concentration corrected on the basis of priority document in the following Example, in spite of the substantial differences in the relevant descriptions of the properties of such systems. Thus no acceptable correct value is available, which renders Example 1 unsuitable for doubting the validity of the claims in the present case.

- 6. For the inventive step it is relevant that the dry jet-wet spinning of PPDT in sulphuric acid has been an established technique for a considerable time. Yet none of the other references cited use temperatures anywhere near to freezing for admixing before heating up the solution to elevated temperatures, although the risk of sulphonation of the polymer or of shearing the chain was well known. It is again characteristic that the dopes are all the time maintained at around 100°C.
- The above picture is not contradicted but rather 7. supported by references (2), (5) and (7) in which the effect of cooling of the mixture is demonstrated. If anything, the result suggests that even under such conditions the dope with a high concentration, i.e. above 15%, only acquires a proper consistency around 130°C. If the established technique gave no hint whatsoever that any freezing of the sulphuric acid before admixture would be useful, the latter group of references only confirm that even cooling en route down to 0°C would not change the situation, let alone improve the results significantly. This emerges in spite of the fact that it was known that the tenacity of the fibres worsens whenever the processing temperature is increased. Thus the absence of prejudice against mixing with frozen sulphuric acid may only mean that the skilled person had no objections on theoretical grounds against such a step, i.e. he could have acted in such manner, but there is no good reason to suggest that he would have done so in the expectation of some gain or advantage (cf. T 2/83 "Simethicone Tablet", OJ EPO, 6/1984, 265, 270 and Headnote II).

- The same applies to document (13) suggesting the 8. possibility of using frozen solvents mixed with polymers. It is not irrelevant in the circumstances that neither PPDT nor sulphuric acid is mentioned in spite of the fact that this pair was already in use industrially. That a similar meta-analogue was mentioned and not PPDT with the parastructure could also represent differences in the conditions for admixing. For instance, the latter being more straight in structure should mean additional rigidity and different viscosity, which might render this less predictable in its behaviour. This and the possible special circumstances of the dry jet-wet technique, in distinction to the pure dry or wet processes expressly mentioned in the citation, are indeed raising doubts against the generalisation of the document to the situation under the present patent.
- 9. Whilst no prejudice could be firmly established by the Respondent it is sufficient to show an established trend in the art (T 2/81 "Methylenebis(phenylisocyanate)" OJ EPO 10/1982, 394, 401 and Headnote I) contrary to the suggested further use of the idea in the field of the present invention, in order to dismiss the proposal that it was "obvious to try" the prefreezing step. Part of the trend was that moving towards a lower temperature itself caused no apparent improvement, in spite of alleged expectations. The Appellant has not demonstrated that the effect of the claimed process was derivable from the state of the art with any degree of probability. Accordingly the subjectmatter of Claim 1 is not obvious and it involves an inventive step.

Order

For these reasons it is decided that:

The appeal is rejected.

The Registrar

The Chairman

F.Klein

P.Lançon