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DECISION of 25 July 2002

Case Number: T 1059/99 - 3.3.3

Application Number: 92870114.3

Publication Number: 0580930

IPC: C08F 10/02

Language of the proceedings: EN

Title of invention:

Process for producing polyethylene having a broad molecular weight distribution

Patentee:

ATOFINA Research S.A.

Opponent:

Basell Polyolefine GmbH

Headword:

Relevant legal provisions:

EPC Art. 56

Keyword:

"Inventive step - problem and solution"

Decisions cited:

T 0119/82, T 0246/91, T 0495/91

Catchword:



Europäisches Patentamt

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Beschwerdekammern

Boards of Appeal

Chambres de recours

Case Number: T 1059/99 - 3.3.3

DECISION of the Technical Board of Appeal 3.3.3 of 25 July 2002

Appellant: (Opponent) Basell Polyolefine GmbH

Brühler Strasse 60

D-50389 Wesseling

Representative:

Hoffmann, Peter Basell Polyolefine GmbH

Patentstelle Gebäude E 413

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D-65926 Frankfurt am Main (DE)

Respondent:

ATOFINA Research S.A.

Zone Industrielle C (Proprietor of the patent)

B-7181 Seneffe (Feluy)

Representative:

Decision under appeal:

Decision of the Opposition Division of the European Patent Office posted 29 September 1999

rejecting the opposition filed against European patent No. 0 580 930 pursuant to

Article 102(2) EPC.

Composition of the Board:

Chairman:

Members:

R. Young W. Sieber

J. De Preter

## Summary of Facts and Submissions

I. The mention of the grant of European patent
No. 0 580 930, with 3 claims, in respect of European
patent application no. 92870114.3, filed on
31 July 1992 was published on 20 November 1996
(Bulletin 1996/47). Claim 1 read as follows:

"Process for co-polymerizing ethylene with at most 20 mole % of one or more other alpha-alkene of 3 to 10 carbon atoms, in two liquid full loop reactors in series at a temperature of 50 to 120°C, preferably 60 to 110°C under an absolute pressure of 1 to 100 bar in the presence of a catalyst consisting of a transition metal component (component A) which is the reaction product of an organomagnesium compound with a titanium compound, an organoaluminium compound (component B), and optionally one or more electron donor, the average molecular mass being regulated with hydrogen, which process comprises carrying out the polymerization such that the introduction of the co-monomer is carried out essentially in the first reactor and that the hydrogen concentration is at a very low concentration in the first reactor to form ethylene polymers having HLMI between 0.01 and 5g/10', and that a very high hydrogen pressure is maintained in the second reactor to form ethylene polymers having a HLMI higher than 5q/10'."

Claims 2 and 3 were dependent claims directed to elaborations of the process according to Claim 1.

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- II. A Notice of Opposition was filed on 20 August 1997, on the grounds of Article 100(a) EPC (lack of inventive step). The opposition was supported inter alia by the following documents:
  - D1 EP-A-0 057 420
  - D2 DE-B-17 20 611
  - D5 EP-B-0 302 242.
- III. By a decision which was dispatched on 29 September 1999, the opposition was rejected, because the subject-matter of the granted claims of the patent in suit involved an inventive step.

According to the decision under appeal, the objective problem to be solved over the teachings of D2, which was considered to be the closest state of the art, was to provide a process that led to an ethylene/ $C_3$ - $C_{10}$ - $\alpha$ olefin block copolymer that had an improved combination of Environmental Stress-Crack-Resistance (ESCR) and Flexural Modulus when compared with an ethylene/ $C_3$ - $C_{10}$ - $\alpha$ olefin block copolymer prepared according to the twostep process of D2. It was held that the examples in the patent in suit would illustrate the improved combination of ESCR and Flexural Modulus. None of the other documents suggested that replacing the catalyst of D2 with the catalyst used in Claim 1 of the patent in suit in combination with the use of two serial loop reactors had a positive effect on the ESCR and/or Flexural Modulus.

IV. On 25 November 1999, a Notice of Appeal against the above decision was filed, the prescribed fee being paid on the same day.

> In the Statement of Grounds of Appeal, filed on 8 February 2000, the appellant opponent argued in substance as follows:

(a) The formulation of the objective problem in the decision under appeal, based on an improvement of the polymers obtained according to the process of the patent in suit, was wrong because these polymers were identical with the polymers of D2.

Furthermore, the comparative data, relied upon by the opposition division for formulating the objective problem, did not represent the closest state of the art. It was the clear teaching of D2 that, if polymers with high ESCR were intended, a polymer with a high molecular weight and containing a comonomer had to be produced in the first step, whereas a polymer with a low molecular weight and not containing a comonomer had to be produced in the second step (column 6, lines 22 to 33). Apart from that, the ESCR properties were not even mentioned in the patent in suit.

- (b) Thus, the objective problem of the patent in suit could only be seen in carrying out the polymerisation of D2 in a different reactor.
- (c) The use of loop reactors was, however, known from D1, including the advantages associated therewith. Furthermore, the specific catalyst required in the patent in suit was only a further development of the catalyst of D1 and already known from D5. At the filing date of the patent in suit, a person skilled in the art would have inevitably used the more advanced catalyst of D5.
- V. With a submission received on 21 August 2000, the respondent proprietor rejected the allegation that the same polymers as in the patent in suit had already been obtained in D2 as unsubstantiated because no real comparison of the relevant properties of the polymers had been made. Furthermore, the respondent questioned

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the conversion of the figures of the reduced specific viscosity, the parameter used in D2 to indicate the molecular weight of the polymers produced in the first and second step, into figures for high load melt index (HLMI), ie the parameter used in the patent in suit to indicate the molecular weight. The burden was on the appellant to demonstrate the conformity of the polymers.

- VI. In a communication accompanying a summons to oral proceedings and dispatched on 8 March 2002, the salient issue was identified by the board as being whether the process of Claim 1 of the patent in suit yielded an improved product (as argued by the respondent) or whether the claimed process was a mere alternative to the process of the closest state of the art not providing a different product (as argued by the appellant)
- VII. Following the issue of the communication, both parties submitted experimental data prior to the oral proceedings.
- VIII. Oral proceedings were held on 25 July 2002. In the course of the oral proceedings the discussion centred on the issue of the technical effects achieved by the process of Claim 1 of the patent in suit with the appellant opponent still arguing that the claimed process was a mere alternative to the process of the closest state of the art not providing a different product. The additionally filed comparative data would demonstrate that the use of a loop reactor instead of a stirred tank reactor had no influence on the final product. The respondent proprietor, on the other hand, relied on his own comparative data which showed that the process of the patent in suit led indeed to an improved product.

IX. The appellant opponent requested that the decision under appeal be set aside, and the patent in suit be revoked.

The respondent proprietor requested that the appeal be dismissed.

## Reasons for the Decision

- 1. The appeal is admissible.
- 2. Admissibility of late-filed evidence

Both parties filed experimental data prior to oral proceedings and to this extent must be regarded as not submitted in due time under Article 114(2) EPC. However, these experiments were filed in response to a question raised by the board in the communication annexed to the summons to oral proceedings. Furthermore, neither of the parties raised an objection with regard to the admissibility of each other's late filed experimental data. Consequently, the board decided to admit these data into the proceedings (Article 114(1) EPC).

3. Inventive step

In the present case, only inventive step is at issue.

- 3.1 The patent in suit; the closest state of the art
- 3.1.1 The patent in suit is concerned in general terms with a process for producing ethylene polymers having a broad molecular weight distribution, where an ethylene/ $\alpha$ -olefin copolymer having a high molecular weight is produced in a first polymerization loop

reactor, and an ethylene polymer with a low molecular weight is produced in a second loop reactor. The catalyst used in this process consists of a transition metal component (component A) which is the reaction product of an organomagnesium compound with a titanium compound and an organoaluminium compound (component B). The products obtained according to this process have good processability, good physical properties and diverse applicability (page 2, lines 53 to 54 of the patent specification). Furthermore, it is stated on page 4, line 33 that the resulting bimodal ethylene copolymers have a better compromise between ESCR and Flexural Modulus than the monomodal resin of Comparative Example 7.

The production of polyethylene or copolymers of 3.1.2 ethylene and higher  $\alpha$ -olefins having a broad molecular weight distribution in a two-step polymerization process is known from D2. This process comprises two reaction steps of a suspension or gaseous phase polymerization, in which a mixed catalyst of a trivalent chlorine-containing titanium compound and an aluminium trialkyl or a reaction product of aluminium trialkyls or aluminium alkyl hydrides with diolefins is used, the hydrogen content of the gas zone being low in one step and high in the other step (Claim 1). The process can be carried out continuously or discontinuously, whereby the continuous method is carried out in two reactors arranged in series, by operating in the various polymerization vessels (column 7, lines 15 to 20).

In a specific embodiment, the process of D2 can provide polymers having an extremely broad molecular weight distribution, in which the higher molecular weight polymer chains also have a higher degree of branching, when, in the one step in which the hydrogen content is low, a mixture of ethylene and  $\alpha$ -olefins is

polymerized and, in the other step in which the hydrogen content is high, either ethylene alone or a mixture of ethylene and  $\alpha$ -olefins having a lower  $\alpha$ -olefin content is polymerized. Owing to their special structure (extremely broad molecular weight distribution and higher degree of branching of the high molecular weight portion), the polymers are especially suitable for producing bottles, pipes, cables and sheets or films having a high stress crack resistance (column 6, lines 5 to 42). The stress crack resistance parameter reported in D2 is apparently identical with the ESCR parameter indicated in the patent in suit, because both parameters are measured according to the same test, ie the Bell-test.

3.1.3 Therefore, a process as described in column 6, lines 5 to 42 of D2 and exemplified in Examples 1 and 2, is considered by the board, in line with both parties, to represent the closest prior art. Such a process yields an ethylene polymer with an extremely broad molecular weight distribution, the polymer portion produced in the first step having a higher degree of branching, ie higher  $\alpha$ -olefin comonomer content, and a high molecular weight.

The process claimed in Claim 1 of the patent in suit differs from this closest prior art in using two liquid full loop reactors in series and a different Ziegler catalyst. Furthermore, the molecular weight of the polymers is indicated in the patent in suit with reference to the HLMI whereas D2 uses the reduced specific viscosity. However, the use of a different parameter for the same physical property is not a distinguishing feature, provided the conformity of the various parameters is given.

- 3.2 The objective technical problem
- An objective definition of the technical problem to be 3.2.1. solved should normally start from the technical problem that is described in the patent in suit. Only if it turns out that an incorrect state of the art was used to define the technical problem or the technical problem disclosed has in fact not been solved, can an inquiry be made as to which other technical problem objectively existed (see T 246/91 of 14 September 1993, point 4.4 of the Reasons for the Decision; T 495/91 of 20 July 1993, point 4.2 of the Reasons for the Decision; neither published in OJ EPO). In the present case the closest prior art was apparently not available at the time of filing but came into notice only during the opposition procedure. Thus, an inquiry has to be made as to which technical problem objectively existed.
- 3.2.2 It is clear from the above analysis that the processes of both the patent in suit and the closest prior art provide polymers with good ESCR properties. Thus, the salient point in the present case is whether the process of Claim 1 of the patent in suit yields an improved product (as argued by the respondent) or is a mere alternative to the process of the closest state of the art not providing a different product (as argued by the appellant). The outcome of this issue is important because in this step of the problemsolution approach the technical effect(s), if any, that the patent in suit provides over the closest prior art are taken into account when formulating the objective technical problem. In this connection, the effect of a process manifests itself in the result, ie in the product in chemical cases (T 119/82, OJ EPO 1984, 217).

- 3.2.3 A valid comparison of the polymers obtained by a process according to the patent in suit on the one hand and by a process according to the closest prior art on the other hand presupposes that the polymers are indeed comparable. In other words, polymers with identical or at least nearly identical macroscopic properties have to be compared. Thus, (i) the ethylene- $\alpha$ -olefin copolymers produced in the first step should have a similar  $\alpha$ -olefin comonomer content and HLMI, (ii) the products from the second reactor should be similar with regard to HLMI, density and molecular weight distribution, and (iii) the ratio of high to low molecular weight portion should be comparable.
- 3.2.3.1 It is immediately evident that Comparative Example 7 in the patent in suit does not represent the closest state of the art because this example was carried out in a single reactor under reaction conditions yielding a monomodal resin, ie no low molecular weight part was produced. Thus, this example cannot serve as a suitable basis to establish any relevant effects achieved by the patent in suit over the closest prior art.
- 3.2.3.2 The appellant relied upon Example 1 of D2 for the comparison between the closest prior art and the patent in suit. The polymer of this Example comprises a highly branched, high molecular weight portion (first step) and a low molecular weight portion (second step) and has a stress crack resistance of 220 (Table). Even if one would concede in favour of the appellant, that the reduced specific viscosities for the polymers obtained in the first and second reaction step corresponded to the respective HLMI values required in Claim 1 of the patent in suit, nevertheless, a "counterpart" polymer for a valid comparison with Example 1 of D2 is missing. Neither

the examples in the patent in suit describe a polymer having similar macroscopic characteristics to those of Example 1 of D2 nor did the appellant produce such a comparable "counterpart" polymer. The further experimental data submitted by the appellant prior to the oral proceedings are not suitable because these experiments were run in "monomodal" configuration in a one step process using a single loop and a single stirred tank reactor, respectively, yielding an ethylene homopolymer only. The results of this experiment cannot be used to draw any conclusion in a different reaction, ie a two step reaction where two (co)polymer portions having different molecular weight are formed. On the other hand, without a valid comparison, the data from Example 1 of D2 alone cannot support appellant's argumentation that the process according to the patent in suit employing two loop reactors provides polymers which are identical with the prior art.

3.2.3.3 The respondent proprietor submitted comparative tests where the polymer of Example 2 of the patent in suit has been compared with a polymer produced in a two step process in a stirred tank reactor. This "counterpart" polymer has identical, or nearly identical, macroscopic characteristics to the polymer of Example 2 of the patent in suit: (i) the ethylene- $\alpha$ -olefin copolymer produced in the first step has a similar  $\alpha$ -olefin comonomer content (320 g/cm<sup>3</sup> versus 300 g/cm $^3$ ) and a similar HLMI (0.25 versus 0.22), (ii) the product of the second reactor has a similar HLMI (16.5 versus 17.1), density (0.950 in both cases) and molecular weight distribution (11.1 versus 12.1), and (iii) the ratio of high to low molecular weight portion is comparable (+/- 40 wt.-% in both cases). Thus, the "counterpart" polymer provided by the respondent indeed provides a suitable basis for a fair comparison between the closest prior art and Example 2

of the patent in suit. The result of this comparison is that polymers with equivalent composition, density and HLMI but being produced in different reaction vessels have distinctively different ESCR values: the polymer produced in the two loop reactors (according to the patent in suit) shows an ESCR value of greater than 300 whereas the polymer produced in the stirred tank reactor (according to the closest prior art) has an ESCR value of 74 with 50% of the samples broken.

- 3.2.3.4 The criticism of the appellant that the additional experiment of the respondent produced a different polymer that is not comparable with Example 2 of the patent in suit is not justified. In fact, the above listed macroscopic characteristics demonstrate quite the opposite. The fact that different reaction conditions have been employed in the loop and the stirred tank reactor is not in itself an indication that different products were obtained. Due to the different reaction phases present in the two types of polymerization reactor, one has slightly to modify the process conditions in order to obtain similar products.
- 3.2.3.5 It should also be mentioned that the comparative test of the respondent goes even one step further than the closest prior art because in the comparative test the same catalyst as required in the patent in suit has been used and not the catalyst as described in the closest prior art. Thus, the comparative test submitted by the respondent lies closer to the patent in suit than the closest state of the art and a possible technical effect arising from the use of a

different catalyst has not been taken into account. But even this variant of the closest prior art shows convincingly that the ESCR properties for a similar polymer are significantly improved due to the use of two loop reactors instead of a two step process in a stirred tank reactor.

- 3.2.4 Based on the result of these comparative data the objective technical problem of the patent in suit can therefore be seen in providing a process which improves the ESCR properties of an ethylene copolymer having a broad molecular weight distribution.
- 3.2.5 Appellant's objection in the written proceedings, but which was not pursued in the oral proceedings, namely that the ESCR properties were not mentioned in the patent in suit and therefore could not be used as a basis for the formulation of the objective technical problem is unfounded. The ESCR properties are not only derivable from the application as originally filed, they are even explicitly mentioned on page 4, line 33 of the patent in suit (page 8, lines 20 to 21 of the application as originally filed) and in Table 1.
- 3.2.6 From the above discussion of the comparative data filed by the respondent it is clearly evident that the objective technical problem is solved by the features of Claim 1 of the patent in suit.
- 3.3 Inventive Step
- 3.3.1 It remains to be decided if the proposed solution, ie the use of two liquid full loop reactors in a two step polymerization process in combination with a specific catalyst, is obvious from the prior art.

- 3.3.2 In D2 itself, there is no suggestion as to how the ESCR properties might further be improved, let alone a hint to the combination of two loop reactors with a modified catalyst as a more promising variant of the disclosed process.
- D1 discloses a process for producing an ethylene 3.3.3 polymer or ethylene-α-olefin copolymer having a broad molecular weight distribution, good melt flowability, and desirable uniformity with very little incompatible or heterogeneous particle content (page 1, lines 1 to 6). The polymerization is carried out by using a catalyst system comprising a high-activity supportedtype Ziegler catalyst component and an organometallic compound component and hydrogen as molecular weight regulator in two or more reaction zones connected in series and kept full with the liquid phase to substantially exclude the gas phase (Claim 1). For the polymerization, any of the loop-type, tube-type, and vessel-type reactors may be used. Of these reactors, the loop-type is most suitable for the operation in each stage of the reaction (page 14, lines 13 to 16). Although D1 favours loop-type reactors in a multi step ethylene polymerization process, there is no indication in that document that the use of a loop reactor would improve the ESCR properties of the resulting polymer, in particular the ESCR properties of a polymer having the characteristics required in Claim 1 of the patent in suit.
- 3.3.4 D5 relates to a process for the preparation of a polyolefin with an extremely wide molecular mass distribution by a two-stage polymerization of ethylene with 0 to 10% by weight, based on the total amount of monomers, of an  $\alpha$ -olefin, in suspension or in the gas phase, in the presence of a mixed catalyst which meets the requirements of Claim 1 of the patent in suit and hydrogen as molecular weight regulator. Polyolefins

with different average molecular masses are formed in both reaction stages whereby the comonomer, if present, is preferably introduced in the stage in which the polymer with the higher molecular mass is formed (page 4, lines 17 to 36). D5 does not, however, indicate a preference to introduce the comonomer in the first step of the process. In fact, in all examples employing a comonomer, the comonomer is introduced in the second step, contrary to the requirements of Claim 1 of the patent in suit. In addition, D5 does not mention the use of loop reactors but uses in all examples stirred tank reactors. Thus, D5 cannot provide any hint how the process of the closest prior art has to be amended in order to solve the objective technical problem.

- 3.3.5 The mere fact that D1 favours the use of loop reactors and D5 discloses the required catalyst is not sufficient to come to the conclusion that it was obvious to modify the process of the closest prior art, ie D2, accordingly. Neither D1 nor D5 suggest that the use of loop reactors and/or the specific catalyst would improve the ESCR properties of the resulting polymer. Hence, any combination of D2 with D1 and D5 would be based on hindsight.
- In summary, the documents cited by the appellant cannot render the claimed subject-matter obvious. The subject-matter of Claim 1 of the patent in suit, and, by the same token, of dependent Claims 2 and 3, involves an inventive step within the meaning of Article 56 EPC.

## Order

## For these reasons it is decided that:

The appeal is dismissed.

The Registrar:

E. Görgmaie

The Chairman:

R. Young

