Datasheet for the decision of 29 January 2009

Case Number: T 0347/07 - 3.2.05
Application Number: 95305665.2
Publication Number: 0700769
IPC: B29C 47/00
Language of the proceedings: EN

Title of invention:
Process for modifying a polyethylene in an extruder

Patentees:
INEOS EUROPE LIMITED, et al

Opponents:
Basell Polyolefine GmbH
Union Carbide Corporation
Exxon Chemical Patents, Inc.

Headword:

Relevant legal provisions:
EPC Art. 54

Relevant legal provisions (EPC 1973):

Keyword:
"Novelty (yes)"

Decisions cited:

Catchword:
Case Number: T 0347/07 - 3.2.05

DECISION
of the Technical Board of Appeal 3.2.05
of 29 January 2009

Appellants: INEOS EUROPE LIMITED
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Composition of the Board:
Chairman: W. Zellhuber
Members: P. Michel
M. J. Vogel
Summary of Facts and Submissions

I. The appellants (patent proprietors) lodged an appeal against the decision of the Opposition Division revoking European Patent No. 0 700 769.

II. The patent in suit was revoked by the Opposition Division on the ground of a lack of novelty.

III. Oral proceedings were held before the Board of Appeal on 29 January 2009.

The appellants requested that the decision under appeal be set aside and that the patent in suit be maintained on the basis of claims 1 to 13 of the patent in suit as granted, as an auxiliary measure, they request that the patent in suit be maintained on the basis of either claims 1 to 13 or claims 1 to 12 filed on 24 December 2008 as first and second auxiliary requests, respectively.

Respondents I, II and III (opponents 01, 02 and 03) requested that the appeal be dismissed.

Respondent II further requested that, in case the Board of Appeal finds that the claims of the patent in suit are novel, the case be remitted to the first instance for consideration of inventive step, or that an extended time limit should be set for the parties to file submissions relating to the issue of inventive step.

Respondent III further requested that, in case the Board of Appeal finds that the claims of the patent in
suit are novel, the case not be remitted to the first instance.

IV. The following documents are referred to in the present decision:

E1: EP-A-0 121 740
E16: "Effect of polydispersity on the thermomechanical degradation of HDPE polymerized using a chromium catalyst", Harlin et al, Polymer Degradation and Stability, 39, 1993, pages 29 to 34
E33: First Declaration by Mr Poloso, dated 3 November 2006
E35: Experimental Report of Mr Neubauer

V. Claim 1 of the main request of the appellant reads as follows:

"1. Process for improving the bubble stability of a linear polyethylene converted into film by blown extrusion, the polyethylene having a density of 0.900 to 0.970, preferably from 0.932 to 0.965 g/cm³, a molecular mass distribution such that the ratio of the weight-average molecular mass, Mw, to that based on the number-average, Mn, is from 8 to 40, preferably from 9 to 30, and a value of the loss tangent measured by dynamic rheometry at 190°C at a frequency of 1.5 x 10⁻² radians per second, ranging from 1.5 to 3, preferably from 1.6 to 2.5, the process being characterized in that, before its complete melting in an extruder, the
polyethylene is brought into contact with oxygen or a
gas mixture containing oxygen, and the polyethylene
thus brought into contact is treated thermomechanically
in the molten state in the extruder supplying a
specific mechanical energy of 0.15 to 0.5, preferably
from 0.17 to 0.35 kWh per kilogram of polyethylene, the
preliminary bringing into contact and the
thermomechanical treatment being combined so that the
treatment is conducted to its completion when the value
of the loss tangent of the polyethylene has lost from
15 to 70%, preferably from 20 to 65% of its initial
value before treatment and bringing into contact."

VI. The appellants argued substantially as follows in the
written and oral procedure:

It is not possible to determine the specific mechanical
energy supplied in document E1, or to determine that it
falls within the range specified in claim 1. Energy may
be applied as both thermal and mechanical energy. Thus,
in Tables 3 and 4 of the patent in suit and at page 481
of document E9, experiments are performed in which
differing inputs of mechanical energy result in the
same melt temperature.

Polyethylene can have a value of the loss tangent,
measured by dynamic rheometry at 190°C at a frequency
of $1.5 \times 10^{-2}$ radians per second, ranging from 0.5 to 11.
Figure 7 of document E9, which shows a Ziegler HDPE,
indicates that values of 10 or higher may be obtained.

It is thus not inevitable that a value ranging from 1.5
to 3 would be chosen. This range is removed from the
examples of document E1 and is chosen in order to
select a polyethylene whose bubble stability can be improved (see paragraph [0013] of the patent in suit).

Similarly, the specified change in loss tangent also constitutes a selection. The experiments set out in document E35 are carried out on polymers selected to have an initial loss tangent falling within the range specified in claim 1. Even so, two of the runs of Table 3 result in a reduction in loss tangent outside the specified range.

In addition, other factors not specified in document E1 affect the change in loss tangent, for example, the choice of extruder, as indicated at page 33 of document E16, and whether or not an anti-oxidant is used.

The same arguments also apply with respect to the disclosure of document E16.

The subject-matter of claim 1 according to the main request is thus new.

VII. The respondents argued substantially as follows in the written and oral procedure:

Documents E1 and E16 disclose concrete examples which fall within the scope of claim 1, specifically Test Nos. 3, 6, 8 and 11 of document E1 and Sample C of document E16, carried out in the presence of air at low, medium and high specific mechanical energies.

The range of specific mechanical energy as specified in claim 1 is a broad range and covers all of the mechanical energies which would need to be supplied to
achieve the desired temperature. Whilst it may be possible to supply or remove energy by heating or cooling, the person skilled in the art would not take the uneconomic approach of introducing excess mechanical energy which must be removed by cooling. Further, as stated in the second declaration of Mr Poloso attached to the submission of 17 December 2008, it would be practically impossible to run an extruder outside the specified range of specific mechanical energy.

The process of document E1 results in long chain branching and a consequent reduction in loss tangent. As shown in document E35, the reduction in loss tangent, which occurs as a result of oxygen tailoring at elevated temperature, is largely dependant upon temperature and oxygen concentration, whilst extruder design is not relevant to any significant extent. A reduction in loss tangent within the range specified in claim 1 would thus follow from use of the process of document E1, which teaches that little or no anti-oxidant should be used.

Document E33 shows that a commercially available resin treated in accordance with the teaching of document E1 undergoes a reduction in loss tangent within the claimed range. Document E35 (Table 4) shows that a reduction in loss tangent of between 24 and 30% is obtained under the conditions specified in document E16.

The subject-matter of claim 1 of the main request is thus not new having regard to the disclosure of each of documents E1 and E16.
Reasons for the Decision

Main Request

1. Novelty

1.1 Document E1

Document E1 relates to a process for treatment of a high density polyethylene prepared by means of a Ziegler catalyst so as to produce a resin suitable for blow moulding (page 1, lines 2 to 6). Whilst the requirements for such a resin are similar to those for a resin intended for producing a film, for example good flowability, they are not identical.

Table 1 at page 12 of document E1 shows tests carried out with polyethylene having a density of 0.953 and 0.954 g/cm³, and a molecular mass distribution such that the ratio of the weight-average molecular mass, Mw, to that based on the number-average, Mn, from 8.4 to 8.6. In addition, during extrusion, the polyethylene is brought into contact with air or a gas mixture containing oxygen.

It was only disputed between the parties whether or not the process disclosed in document E1 exhibits the following features:

(i) a specific mechanical energy of 0.15 to 0.5 kWh per kilogram of polyethylene is supplied to the molten polyethylene in the extruder;
(ii) the initial value of the loss tangent of the polyethylene measured by dynamic rheometry at 190°C at a frequency of $1.5 \times 10^{-2}$ radians per second, is from 1.5 to 3; and

(iii) the thermomechanical treatment is conducted to its completion when the value of the loss tangent of the polyethylene has lost from 15 to 70% of its initial value.

1.1.1 Specific Mechanical Energy

Claim 1 of the patent in suit specifies that the thermomechanical treatment of the polyethylene in the molten state in the extruder supplies a specific mechanical energy of 0.15 to 0.5 kWh per kilogram of polyethylene.

The patent in suit itself refers to a specific mechanical energy of 0.180 kWh per kilogram of polyethylene as being relatively low (Table 2 and page 7, line 44) and to a specific mechanical energy of 0.27 kWh per kilogram of polyethylene as being relatively high (Table 3 and page 8, line 27). In addition, even the comparative examples of the patent in suit use levels falling within the specified range. Finally, documents E9 (page 481, 2nd paragraph, right hand column) and E16 (page 30, 1st paragraph, right hand column) refer to low, medium and high levels of specific mechanical energy which fall within the specified range.

The Board is thus of the opinion that the person skilled in the art, when carrying out the process of
document E1, would inevitably use a specific mechanical energy falling within the range specified in claim 1.

1.1.2 Initial Loss Tangent

Figure 7 of document E9 shows $G'/G''$, which is the inverse of loss tangent, as a function of shear rate. Extrapolation indicates that, at a shear rate of $1.5 \times 10^{-2}$ radians per second, the HDPE has a value of $G'/G''$ less than 0.2, so that the loss tangent is greater than 5. This demonstrates the existence of polyethylenes having a loss tangent outside the specified range of 1.5 to 3.

Document E1 does not give any indication that the starting material for the disclosed process, including those exemplified as Test Nos. 3, 6, 8 and 11 of the Table at page 12, should have any particular value of loss tangent.

As stated in the patent in suit at paragraph [0013], it is polyethylenes having a loss tangent falling within the specified range which are capable of having the defect of bubble instability corrected by the process of the invention. As noted above, document E1 is not concerned with polyethylenes suitable for the production of films, so that there is no suggestion in document E1 that the selection of a loss tangent in the specified range could have any such effect.

Documents E33 and E35 show experiments carried out on polyethylenes falling within the specified range. There is, however, no indication that such polyethylenes
would have been selected without knowledge of the patent in suit.

The selection of a polyethylene having a loss tangent of from 1.5 to 3, thus constitutes a selection from known polyethylenes.

1.1.3 Reduction of Loss Tangent

Runs 1-3 and 1-4, shown in Table 3 of document E35, show a reduction of loss tangent of 71.9 and 74.3% respectively. Each of these runs was carried out at a temperature above 230°C in the presence of air, and are thus in accordance with the teaching of document E1. It is noted that these runs were carried out in the presence of air, and thus a high amount of oxygen, and without an anti-oxidant. This is, however, in accordance with the teaching of document E1, which teaches the use of either no anti-oxidant, or as small amount of anti-oxidant as possible (see page 4, lines 25 and 26, and page 10, lines 10 to 17).

It is thus not inevitable, when following the teaching of document E1, that the reduction of loss tangent during thermomechanical treatment is from 15 to 70%.

1.1.4 Thus, document E1 does not disclose a process in which the polyethylene has an initial loss tangent measured by dynamic rheometry at 190°C at a frequency of 1.5 x 10^{-2} radians per second, ranging from 1.5 to 3 and in which the reduction in loss tangent during treatment is from 15 to 70%.
1.2 Document E16

Document E16 relates to a study concerning the influence of polydispersity on thermomechanical degradation of high density polyethylene (page 29, left hand column, second paragraph). As far as the features of claim 1 of the patent in suit are concerned, the disclosure of document E16 is similar to that of document E1, but, in addition, suggests the use of levels of specific mechanical energy falling within the range specified in claim 1 (see page 30, right hand column, second paragraph). The document further mentions the use of high density polyethylene for the manufacture of blown films (page 29, right hand column, first paragraph).

However, there is no hint in document E16 that the starting material for the extrusion process should be selected so as to have a particular value of loss tangent. The subject-matter of claim 1 is thus new in view of the disclosure of document E16 at least for the reason given in paragraph 1.1.2 above).

2. The Opposition Division has not had the opportunity of assessing the issue of inventive step. Therefore, in order to enable this issue to be examined by two instances, the Board exercises its discretion under Article 111(1) EPC to remit the case to the Opposition Division for further prosecution.
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 for further prosecution.

The Registrar:    The Chairman:

D. Meyfarth     W. Zellhuber