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D E C I S I O N
of 15 November 2000

Case Number: T 0279/98 - 3.3.3

Application Number: 91307217.9

Publication Number: 0471497

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Language of the proceedings: EN

Title of invention:
Gas phase polymerisation

Patentee:
BP Chemicals Limited, et al

Opponent:
Union Carbide Corporation
BASF Aktiengesellschaft, Ludwigshafen

Headword:

-

Relevant legal provisions:
EPC Art. 54, 56

Keyword:
"Novelty - implicit disclosure (no) - combination of disclosures (no)"
"Inventive step - non-obvious combination of known features"

Decisions cited:
T 0378/94 - T 0153/85

Catchword:

-



Case Number: T 0279/98 - 3.3.3

D E C I S I O N
of the Technical Board of Appeal 3.3.3
of 15 November 2000

Appellant: BP Chemicals Limited
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Respondent 2: BASF Aktiengesellschaft, Ludwigshafen
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Decision under appeal: Decision of the Opposition Division of the
European Patent Office posted 26 January 1998
revoking European patent No. 0 471 497 pursuant
to Article 102(1) EPC.

Composition of the Board:

Chairman: C. Gérardin
Members: B. ter Laan
A. Lindqvist

Summary of Facts and Submissions

- I. Mention of the grant of European patent No. 0 471 497 in respect of European patent application No. 91 307 217.9, filed on 6 August 1991, claiming priority from an earlier application in France (9010281 of 7 August 1990), was published on 8 June 1994 (Bulletin 94/23) on the basis of twelve claims, Claim 1 reading:

"A process for stopping an olefin polymerisation which is carried out in a low pressure gas phase reactor, which process comprises introducing an effective quantity of deactivating agent into the reactor characterised in that the olefin polymerisation is carried out using a chromium oxide catalyst and in that the deactivating agent is selected from oxygen, ammonia, water and carbon monoxide and is introduced over a relatively short period of time."

Claims 2 to 12 referred to preferred embodiments of the process according to Claim 1.

- II. On 3 March 1995 and 6 March 1995 two Notices of Opposition against the granted patent were filed, in which the revocation of the patent in its entirety was requested on the grounds set out in Articles 100(a) EPC (both Opponents) and 100(b) EPC (Opponent II).

The oppositions were, *inter alia*, supported by the following documents:

- D1 Yu.I.Ermakov et al., "Transfer process during polymerization of ethylene on a chromium catalyst" *Kinetika i Kataliz*, 10 (1969), pp. 411-419,

- D3 US-A-4 234 716,
- D4 EP-A-0 359 444,
- D5 US-A-4 547 555,
- D6 US-A-4 003 712,
- D7 US-A-4 326 048,
- D8 Journal of Catalysis, Academic Press,
11 (1968), pp.263-266,
- D9 Crystalline Olefin Polymers (Part I),
XX (1965), Interscience Publishers and
- D13 J.C.W.Chen "Coordination polymerisation",
Academic Press Inc. (1975), filed after the
expiry of the opposition period, but
admitted into the proceedings.

III. By a decision taken on 13 January 1998 and issued in writing on 26 January 1998 the Opposition Division revoked the patent. That decision was based on a set of claims of which Claim 1 was filed on 26 October 1995 and Claims 2 to 12 remained as granted. Claim 1 read:

"A process for stopping an olefin polymerisation which is carried out in a low pressure gas phase reactor, which process comprises introducing an effective quantity of deactivating agent into the reactor characterised in that the olefin polymerisation is carried out using a chromium oxide catalyst and in that the deactivating agent is selected from oxygen, ammonia, water and carbon monoxide and is introduced into the reactor in less than 5 minutes."

The Opposition Division held that the requirements of Articles 123(2) and (3), 83 and 54 were complied with, but that the claimed subject-matter lacked an inventive step (Article 56 EPC). D5, which was regarded as the closest prior art document, disclosed a method for

terminating a gas phase fluid bed or stirred bed olefin polymerisation in an emergency situation, which comprised the use of a transition metal-based catalyst system and injecting the kill gas (carbon monoxide) within 10 to 30 seconds. Although D5 did not specifically mention chromium oxide catalysts, the use of that catalyst was deemed obvious in the light of D3, D1 and the late filed D13, which described carbon monoxide as a deactivating agent for chromium oxide catalysts in gas phase polymerization. Therefore, the use of a chromium oxide catalyst in gas phase olefin polymerisation as well deactivating that catalyst with carbon monoxide was obvious.

IV. On 24 March 1998 the Appellant (Proprietor) lodged an appeal against the above decision and paid the prescribed fee simultaneously. With the Statement of Grounds of Appeal, which was filed on 2 June 1998, the Appellant submitted two new sets of claims as main request (eleven claims) and auxiliary request (ten claims). During the oral proceedings held on 15 November 2000, those requests were replaced by a set of ten claims as the sole request, Claim 1 reading:

"A process for stopping an olefin polymerisation which is carried out in a low pressure gas phase reactor allowing the polymerisation reaction to be restarted quickly without having to empty the polymerisation reactor, which process comprises introducing an effective quantity of deactivating agent into the reactor characterised in that the olefin polymerisation is carried out using a chromium oxide catalyst and in that the deactivating agent is selected from oxygen, or water and is introduced into the reactor in less than 5 minutes."

Claims 2 to 10 refer to preferred embodiments of the process according to Claim 1.

V. The arguments of the Appellant can be summarized as follows:

(a) As regards novelty, that ground had not been raised in the appeal proceedings before and should therefore not be admitted. In substance, none of the documents disclosed the present combination of features. D5 mentioned Ziegler-Natta catalysts, not chromium oxide catalysts. Combining D5 with other documents was not permitted for judging novelty, but even if one would combine the disclosure of D5 with one or more other documents to which it referred, like D6 or D7, the chromium containing catalysts mentioned in those documents could not be regarded as chromium oxide catalysts. Therefore, the claimed subject-matter was novel.

(b) Regarding inventive step, the problem to be solved was to provide a rapid polymerisation stopping system that could be restarted quickly, without cumbersome technical measures. The closest document, D5, taught the rapid stopping of a gas phase fluidised bed polymerisation process catalysed by transition metal compounds, in practice Ziegler catalysts, with carbon monoxide, carbon dioxide or oxygen containing gases. It contained therefore a general list of possible kill gases for use with Ziegler catalysts with no information about possible systems based on chromium oxide catalysts.

D3 was directed to the separation of polyolefins

produced with a transition metal compound and an alkylaluminium compound, by means of adding carbon monoxide to the reaction mixture at any time between the polymerisation and the end of the separation. Therefore, D3 did not teach to stop the polymerisation process in the reactor, nor did it contain any indication that the process of D5 would be suitable for use with a chromium oxide catalyst. D3 was not combinable with D5, but even if one would combine those two documents, this would not lead to the claimed subject-matter.

None of the other documents, whether taken alone or in combination, rendered the claimed subject-matter obvious. D8 in particular did not refer to a catalyst system in polymerisation conditions, nor to gas phase polymerisation, and could therefore not be combined with D5. None of the other documents on file contained any teaching about the rapid stopping of a chromium oxide catalyst gas phase polymerisation process with the possibility to restart it quickly and easily. Hence the claimed subject-matter was inventive.

VI. In reply to the Statement of Grounds of Appeal, Respondent 1 filed a new citation which had not been mentioned in the proceedings before. The Respondents' arguments can be summarized as follows:

- (a) In view of the new claims, the objections under Article 100(b) were not maintained, nor were any objections raised pursuant to Articles 123(2), 123(3) and 84 EPC.
- (b) Although D5 referred primarily to Ziegler

catalysts, its broad disclosure was not restricted to that type of catalyst. D5 expressly referred to D7 and D6, which used chromium halide and chromate catalysts, respectively. The latter contained chromium oxygen bonds and hence fell under the definition of "chromium oxide". It could also be supported on silica. Both compounds were capable of being converted to chromium oxide, which was how the expression "chromium oxide" should be interpreted. Since D5 incorporated the disclosure of both D6 and D7 by reference, it disclosed all the features of the claimed subject-matter which therefore was not novel.

- (c) Starting from D5, the problem to be solved was to provide a process for rapid stopping of a low pressure gas phase olefin polymerisation using a chromium oxide catalyst, allowing to restart the process quickly. D5 referred to a broad class of catalysts, including chromium oxide catalysts, the actual use of which was clearly implied by the reference to other catalyst systems than Ziegler-Natta. It also mentioned reversible kill gases. Therefore, the skilled person would, on the basis of D5 alone, have considered the combination of features now being claimed with a view to solving the above-indicated problem.

D3 taught the stopping of olefin polymerisation reactions using chromium oxide catalysts in a very short time by adding carbon monoxide under conditions which allowed a quick restart of the process. It referred to a post-polymerisation situation, but it showed that chromium oxide could be used in gas phase polymerisation and be

efficiently poisoned and killed. Hence, D3 provided the features missing from D5.

Also, according to D8, water and oxygen were potent, reversible inhibitors of chromium oxide catalysts. D1 and D13 also disclosed oxygen and carbon monoxide as chromium oxide catalyst inhibitors. Therefore, the use of oxygen or water as reversible catalyst poisons or killers was known, so that the claimed subject-matter was not inventive.

VII. The Appellant requested that the decision under appeal be set aside and the patent be maintained on the basis of Claims 1 to 10 submitted during oral proceedings as its sole request.

The Respondents requested that the appeal be dismissed.

Reasons for the Decision

1. The appeal is admissible.

Procedural matters

2. Regarding the new citation provided by Respondent 1 for the first time in the appeal proceedings, the Board invited the representative to justify the relevance of that evidence in the light of the Reasons for the Decision given by the first instance and the arguments put forward so far in writing. Since it appeared that the late document was not more relevant than the ones already on file, it was not admitted to the proceedings (Article 114(2) EPC).

The wording of the claims

3. The Respondents did not object to the amended claims under Articles 123(2), 123(3) and 84 EPC and the Board concurs with that view for the following reasons.

3.1 Claim 1 differs from Claim 1 as originally filed in that

(a) the possibility of quickly restarting the polymerisation has been added, the basis for which can be found on page 2, lines 9 to 13, and page 3, lines 13 to 15, as originally filed. Although this amendment had not been necessary in view of any objection under Article 100 EPC, none of the parties objected to its introduction in Claim 1.

(b) the kill gas has been specified as being oxygen or water. Those gases were present in original Claim 1 as two out of four options.

(c) the "relatively short period of time" of original Claim 1 during which the kill gas is introduced into the reactor has been specified as being less than 5 minutes. This is based upon original Claim 10.

Therefore, Article 123(2) EPC is complied with.

3.2 Amendment (a) above has no bearing on the scope of the claim since it merely serves to clarify the aim of the invention and amendments (b) and (c) are restrictions of previously disclosed possibilities, so that the requirements of Article 123(3) are satisfied.

- 3.3 The amendments to the claims do not introduce any unclarities. Accordingly, the requirements of Article 84 EPC are complied with.

Novelty

4. The Appellant challenged the admissibility of the novelty objection since until then it had not been an issue during the appeal proceedings. However, novelty had been an issue before the first instance (see the opposition letters filed on 3 March and 6 March 1995), so that it cannot be considered as a new ground for opposition. Accordingly, novelty was discussed.
5. The only document cited against novelty was D5.
- 5.1 D5 describes a method for terminating a gas phase fluid bed or stirred bed olefin polymerisation reaction during a power failure, said olefin polymerisation reaction taking place in the presence of a transition metal-based catalyst system in a reactor having a gas recycle line means equipped with a compressor, said compressor producing a coast-down flow of recycle gas for at least one minute during the power failure, said method comprising injecting within 10 to 30 seconds after said power failure an amount of kill gas into said recycle line mains sufficient to terminate the olefin polymerisation reaction, whereby said kill gas is carried into the reaction medium by said coast-down flow of the recycle gas during said power failure (Claim 1).

The kill gas can be any gas which is capable of poisoning the olefin polymerisation catalyst to the

extent of terminating the polymerisation. Preferred is a reversible poison, such as carbon monoxide or carbon dioxide or their mixtures, since these make it possible to reactivate poisoned catalysts simply by purging the reactor (column 3, lines 53 to 65). The time to kill the catalyst is largely established by the time it takes to disperse the kill gas through the system: a time of less than one minute is mentioned (column 7, lines 37 to 42).

The olefin polymerization catalysts capable of being poisoned include systems based on transition metal compounds, preferably of the Ziegler-type, which are characterized as chemical complexes derived from a transition halide, e.g. $TiCl_4$ and a metal hydride or metal alkyl, e.g. aluminium alkyl (column 3, line 66 to column 4, line 6).

The process of D5 is particularly suitable for use in fluidized bed reaction systems such as described in e.g. D6 and also in other gas-phase systems such as stirred-bed type gas phase reactors (column 4, lines 7 to 15).

5.2 D5 neither mentions chromium oxide catalysts, nor does it refer to water or oxygen as reversible catalyst poisons. Hence the combination of chromium catalyst and oxygen or water as the kill gas, as now required, is not disclosed.

5.3 One of the Respondents' lines of reasoning as regards novelty was based on the opinion that a chromium catalyst and water or oxygen as reversible kill gases were encompassed by the possibilities mentioned by D5, and were therefore included in its disclosure. However,

it is standard jurisprudence of the Boards of Appeal that such a situation is not suitable to destroy novelty. In order to establish a lack of novelty, the claimed subject-matter should be clearly and unambiguously disclosed. Even if that subject-matter would fall within the scope of the prior art document, that does not mean that it has actually been disclosed; any generic concept does not automatically prejudice the novelty of specific embodiments (see Decision T 378/94, 19 March 1996; not published in OJ EPO).

Therefore, the Board cannot follow the Respondents' arguments and, accordingly, the claimed subject-matter is novel over D5 taken alone.

5.4 A second line of reasoning was based upon a combination of D5 with D6 or D7. In particular, the Respondents maintained that the latter documents disclosed the use of chromium oxide catalysts, so that the disclosure of D5 also included that specific olefin polymerisation catalyst.

5.4.1 According to standard jurisprudence of the Boards of Appeal, novelty is assessed by comparing the claimed subject-matter with one document only. It is only in very special cases that a combination with another document is permitted (see Decision T 153/85, OJ EPO 1988, 1, Reasons 4.2).

5.4.2 D6 is mentioned several times in D5: e.g. in column 1, lines 20 and 56, column 4, lines 9 and 22, column 6, lines 11 and 22. These references all concern either the fluidized bed reactor as a type of reactor suitable for the polymerisation according to D5 or the process parameters (temperature, pressure), so that any

incorporation of the matter of D6 into D5 would be restricted to that information and could not serve to imply the use of a chromium catalyst. However, the above-mentioned passages do not indicate the obligatory use of a fluidized bed reactor but merely describe it as a suitable possibility, so that the disclosure of D6, even only in respect of the reactor, cannot be regarded as an integral part of the teaching of D5. Therefore, a combination of D6 with D5 for the assessment of novelty is not permitted.

5.4.3 The reference to D7 (column 1, lines 11 and 17) relates to the injection of carbon oxides and the site of injecting the gas into the reaction. Therefore, also any incorporation of D7 would not refer to the catalyst to be used in the process according to D5. Like the reference to D6, no obligatory use of the system of D7 in the process of D5 is described, so that a combination of these two documents is not permitted either.

5.4.4 Even if, for the sake of argument, one would incorporate the whole disclosure of D6 and/or D7 in D5, such a combination would still not result in the claimed subject-matter since neither D6 nor D7 disclose chromium oxide catalysts. D7, like D5, refers to transition metal compounds, in particular $TiCl_3$, in combination with an aluminium alkyl, hence to Ziegler catalysts, whereas D6 mentions silyl chromate catalysts containing hydrocarbyl groups. No calcination is mentioned. Since calcination would result in removing the hydrocarbyl groups, it also cannot be assumed to have been implicitly present in the disclosure of D6. Therefore, neither of D6 or D7 refers to chromium oxide catalysts, so that these documents do not provide that

feature for a combination with the process of D5.

5.4.5 For these reasons, the Board sees no reason to incorporate the disclosure of either of D6 or D7 in the disclosure of D5.

5.5 In view of the above, the subject-matter of Claim 1 is novel.

Inventive step

6. The patent in suit concerns a gas phase polymerisation.

6.1 Such a polymerisation process is, as mentioned above (point 5.1), disclosed in D5, which the Board, like the Opposition Division and the parties, regards as the closest state of the art. Although the system of D5 is said to enable a rapid termination of a polymerisation based on a Ziegler catalyst and a preference for the use of reversible catalyst poisons as the killing agent is mentioned, chromium oxide catalysts are not referred to, nor is there any indication which kill gases would be reversible for that type of catalyst. Therefore, the technical problem underlying the patent in suit can be seen in providing a system for rapidly stopping an olefin polymerisation based on a chromium oxide catalyst which would enable a quick restart of the polymerisation, in line with the definition of the object of the invention according to the patent specification (column 1, lines 37 to 42 and column 25 to 27).

6.2 According to the patent in suit that problem is to be solved by the use of oxygen or water as the killing agent, as defined in Claim 1.

- 6.3 The examples in the patent show that the above-defined problem is effectively solved. In particular, it has been shown that chromium oxide catalysed polymerisations can be stopped within 10 minutes by injecting a mixture of oxygen and nitrogen and be restarted without having to drain the reactor bed or even without purging the reactor (Examples 1 to 3).
7. It remains to be decided whether the subject-matter is obvious having regard to the documents on file.
- 7.1 The general teaching of D5 pertains to the rapid termination of Ziegler-catalyst based polymerisation processes in case of an emergency, such as a power failure, by introducing carbon monoxide, carbon dioxide or a mixture of the two within 10 to 30 minutes after the occurrence of the power failure. A preference for reversible catalyst poisons such as carbon oxides is indicated (column 3, lines 56 to 60), whereas the use of irreversible catalyst poisons, such as oxygen or air, are less recommended. Since the teaching of D5 concerns Ziegler catalyst-based reactions, it does not contain any suggestion about which killing agents are reversible for chromium oxide catalysts. In fact, by its clear statement that oxygen is an irreversible kill gas, it teaches away from its use. Therefore, D5 by itself cannot render the claimed subject-matter obvious.
- 7.2 The Respondents combined the teaching of D5 with D3, which describes a process for the separation of a polyolefin, manufactured by low pressure polymerisation of at least one alpha-olefin monomer with the aid of a catalyst, from the reaction mixture resulting from the

polymerization and containing the polyolefin, unreacted monomer and the catalyst, comprising: adding carbon monoxide to the reaction mixture (Claim 1). Although chromium oxide is mentioned as a possible catalyst, the process described in D3 is especially suitable for separating polyolefins which are manufactured with the aid of catalysts comprising a transition metal compound and an organo-aluminum compound, especially chlorine-containing titanium compounds (column 3, lines 29 to 43). Therefore, D3 refers to a different stage of the process, that is, the separation of the polymer outside the reactor, after pressure release, so that the reversibility of the system under polymerisation conditions is not relevant and accordingly it does not contain any teaching in that respect. Also, like D5, it contains no indication about the use of water or oxygen to kill chromium oxide-based polymerisation processes. Therefore, it does not provide the features missing from D5, so that a combination of D3 with D5 does not lead to the claimed subject-matter.

7.3 The other combinations with the documents on file envisaged by the Respondents do not lead to the claimed subject-matter either.

7.3.1 D8 describes the inhibiting effects of water and oxygen on the activity of silica-alumina-chromia catalysts (page 263, header). Although the effect of water and oxygen was said to be reversible, it is also clearly stated that evacuation at 300°C restores the original activity of the catalyst activity completely, at 200°C activity is restored to only 60% of its original value and evacuation at 100°C did not restore any activity at all (page 263, Results, second paragraph). The concept of reversibility of inhibition is therefore evidently

different from the one referred to in the patent in suit and no conclusions can be drawn as regards the behaviour and reversibility of chromium oxide/oxygen or water systems under actual reaction conditions. In fact, in view of the difficulty of reversing the inhibiting effect of oxygen, the skilled person would not be inclined to use oxygen as the kill gas if he aimed at a quick restart of the reaction. Therefore, also a combination of D8 with D5 would not render the claimed-subject matter obvious.

7.3.2 In D1 the influence of several gases on the rate of gas phase ethylene polymerisation by a chromium oxide catalyst is studied. The effective transfer and inhibition constants for those gases, which are present as impurities, is calculated (page 333, first paragraph) in laboratory scale experiments (pages 335 and 336: Experimental). Although the inhibiting effect of oxygen-containing and other substances on chromium oxide catalysts is referred to (page 1, second paragraph), there is no teaching about rapidly stopping and quickly restarting the polymerisation system, which are requirements that are mainly relevant for full scale production facilities.

7.3.3 As can be seen from point 4.4.4 above, the teaching of D6 and D7 does not pertain to chromium oxide catalysts. D6 does not concern any rapid stopping of polymerisation reactions. D7 does mention termination of a Ziegler catalyst based polymerization reaction by injecting carbon dioxide and the importance of a quick restart (Claim 1; column 3, lines 44 to 47), but no indication is given of how to obtain such a result for a chromium oxide-based polymerisation. As a consequence, neither D6 nor D7 provide the features

lacking in D5.

7.3.4 In D13 the interaction of various quenching agents with the active centres of solid catalysts is studied (page 94, 2.2). Amongst other combinations, a $\text{CrO}_3/\text{SiO}_2$ catalyst with C^{14}O is shown (page 94, Table 1). There is no information about quickly restarting the polymerisation reaction after its complete termination, or about the use of water or oxygen as reversible killing agents for chromium oxide catalysts under gas phase reaction conditions.

7.3.5 D4 describes the addition of very small amounts of activity retarders such as, amongst many others, oxygen and water. Its general teaching is directed to continuing polymerisation at a high level, so that a skilled person would not take this document into account when looking for means to stop a reaction completely and nevertheless maintaining the possibility of a quick restart.

7.3.6 D9 only mentions that water, oxygen and many other compounds are poisons for chromium oxide catalysts (page 360, first full paragraph).

7.4 In view of the above, it can be concluded that although it is known that chromium oxide catalysts can be used for olefin polymerisation, on the one hand, and that water and oxygen are poisons for chromium oxide catalysts, on the other hand, there is no teaching that the use of the latter two compounds would not only enable the skilled person to terminate a chromium oxide-based olefin polymerisation reaction rapidly, but also to restart it quickly without having to empty the reactor.

8. For those reasons, the Board concludes that the subject-matter of present Claim 1 cannot be derived from the documents relied upon by the Respondents, whether taken alone or in combination, and, therefore, it involves an inventive step.
9. As Claim 1 is allowable and Claims 2 to 10 relate to further embodiments of the process according to Claim 1, their patentability is supported by that of Claim 1.

Order

For these reasons it is decided that:

1. The decision under appeal set aside.
2. The case is remitted to the Opposition Division with the order to maintain the patent on the basis of Claims 1 to 10 submitted during oral proceedings, after any consequential amendment of the description.

The Registrar:

The Chairman:

E. Görgmaier

C. Gérardin