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Title of invention:

CHROMIUM CATALYSED ETHYLENE COPOLYMER POWDER

Patent Proprietor:

Ineos Sales (UK) Limited

Opponent:

The Dow Chemical Company

Relevant legal provisions:

EPC Art. 56

Keyword:

Inventive step - (no)



Beschwerdekammern Boards of Appeal Chambres de recours

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Case Number: T 2210/19 - 3.3.03

DECISION
of Technical Board of Appeal 3.3.03
of 13 January 2021

Appellant: The Dow Chemical Company

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Decision under appeal: Interlocutory decision of the Opposition

Division of the European Patent Office posted on

26 July 2019 concerning maintenance of the European Patent No. 2635613 in amended form.

Composition of the Board:

C. Brandt

- 1 - T 2210/19

Summary of Facts and Submissions

- The appeal by the opponent lies from the decision of the opposition division posted on 26 July 2019 concerning maintenance of European patent No. 2 635 613 in amended form on the basis of the 3rd auxiliary request filed with letter of 5 April 2019.
- II. Claim 1 as granted read as follows:
 - "1. Process for producing a chromium catalysed ethylene copolymer powder by polymerising the corresponding monomers in the presence of an activated supported chromium oxide based catalyst, wherein the chromium catalysed ethylene copolymer powder is **characterised by** a particle fragmentation coefficient ("m") equal or superior to 0.29, preferably 0.30, more preferably 0.31 wherein the particle fragmentation coefficient ("m") of the copolymer powder is defined by
 - m = Ln (APS particle/ APS catalyst) / Ln (Productivity)
 wherein
 - "Ln" is the "natural logarithm",
 - "APS particle" is the median particle size (D50) of the polymer powder,
 - "APS catalyst" is the median particle size (D50) of the catalyst, said D50's being measured according to ISO 13320:2009, and
 - "Productivity" is the number of grams of polymer per gram of catalyst, and
 - wherein the process for the activation of said supported chromium oxide based catalyst is performed in a fluidised bed activation reactor which comprises a catalyst bed being fluidised by a fluidisation gas, said activation including treatment at temperatures

- 2 - T 2210/19

above 500°C, characterised in that the fluidisation velocity (Vf1) of the fluidisation gas is initially maintained below 6.5 centimetres per second (cm/sec), preferably below 6 cm/sec, more preferably below 5.5 cm/sec, even more preferably below 5 cm/sec until the temperature inside the activation reactor reaches at least 200°C, and said fluidisation gas is then brought to a value (Vf2) which is at least 1 cm/sec higher than Vf1, preferably at least 1.5 cm/sec higher than Vf1."

III. The decision of the opposition division was based *inter*alia on the following documents:

D3: WO-A-2009/108174

D5: WO-A-2007/015927

D6: Experimental Report signed by John H. Moorhouse on 5 September 2017

D7: Max P. McDaniel, Advances in Catalysis, Volume 53, Chapter 3, "A Review of the Phillips Supported Chromium Catalyst and its Commercial Use for Ethylene Polymerization", 2010, pages 138-139; 146-149; 364-371; 565-577 and 586-587.

D8: Applied Catalysis, A: General, 335 (2008) M.P. McDaniel et al, pages 252-261.

D9: Applied Catalysis, A: General, 335 (2008) M.P. McDaniel et al, pages 180-186.

D10: Decision of the opposition division dated 10 November 2017 revoking patent EP-B-2635612

D11: US-A-2010/0069585 D12: WO-A-03/033550

IV. The decision of the opposition division was based on the claims as granted as the main request, on the $1^{\rm st}$ and $2^{\rm nd}$ auxiliary requests filed with letter of 5 March 2018 and on the $3^{\rm rd}$ auxiliary requests filed with letter of 5 April 2019.

- 3 - T 2210/19

Claim 1 of the 1st auxiliary request corresponded to claim 1 of the main request to which "and further wherein the fluidisation gas used during the consecutive stage of the activation process is an inert gas followed by an oxidising gas." was added at the end of the claim.

Claim 1 of the $2^{\rm nd}$ auxiliary request corresponded to claim 1 of the $1^{\rm st}$ auxiliary request to which "the consecutive stage being the fluidisation stage performed at a higher fluidisation velocity" was added within the addition to the $1^{\rm st}$ auxiliary request.

Claim 1 of the 3rd auxiliary request read:

- "1. Process for producing a chromium catalysed ethylene copolymer powder by polymerising the corresponding monomers in the presence of an activated supported chromium oxide based catalyst, said process comprising:
 - a) activating a supported chromium oxide based catalyst, wherein the process for the activation of said supported chromium oxide based catalyst is performed in a fluidised bed activation reactor which comprises a catalyst bed being fluidised by a fluidisation gas, said activation including treatment at temperatures above 500°C, characterised in that the fluidisation velocity (Vf1) of the fluidisation gas is initially maintained below 6.5 centimetres per second (cm/sec), preferably below 6 cm/sec, more preferably below 5.5 cm/sec, even more preferably below 5 cm/sec until the temperature inside the activation reactor reaches at least 200°C, and said fluidisation gas is then brought to a value (Vf2)

- 4 - T 2210/19

which is at least 1 cm/sec higher than Vf1, preferably at least 1.5 cm/sec higher than Vf1, and b) polymerising the corresponding monomers in the presence of the activated supported chromium oxide based catalyst;

wherein the chromium catalysed ethylene copolymer powder is characterised by a particle fragmentation coefficient ("m") equal or superior to 0.29, preferably 0.30, more preferably 0.31 wherein the particle fragmentation coefficient ("m") of the copolymer powder is defined by

m = Ln (APS particle/ APS catalyst) / Ln (Productivity)
wherein

"Ln" is the "natural logarithm",

"APS particle" is the median particle size (D50) of the polymer powder,

"APS catalyst" is the median particle size (D50) of the catalyst, said D50's being measured according to ISO 13320:2009, and

"Productivity" is the number of grams of polymer per gram of catalyst."

- V. The contested decision, as far as it is relevant to the present appeal, can be summarized as follows:
 - D10-D12 were late filed documents that were not admitted into the proceedings.
 - The requirement of sufficiency of disclosure was met by the main request.
 - D6 showed that D3-D5 disclosed processes for producing a chromium catalysed polyethylene powder having particle fragmentation coefficients according to claim 1 of the main request. The activation process of the catalyst was not a

- 5 - T 2210/19

feature of the polymerisation process of claim 1 of the main request and the product-by-process feature was not limiting as there was no evidence that it led to a different catalyst. Claim 1 of the main request therefore lacked novelty over D3-D5. That conclusion also applied to claim 1 of the $1^{\rm st}$ and $2^{\rm nd}$ auxiliary requests.

- The process of claim 1 of the 3rd auxiliary request involved a step of activation that was characterized by the fluidisation gas velocity and the temperature of the gas flow. These parameters of the activation step were not disclosed in D3-D5. Claim 1 of the 3rd auxiliary request was therefore novel over D3-D5.
- D3-D5 could all reasonably be seen as closest prior art documents since they concerned the preparation of chromium catalysed ethylene polymer powders. Example 20 of D3 in particular disclosed a chromium based catalyst on a silica support that was activated according to a process disclosed in the description of D3. D6 showed that the copolymers of the examples of D3-D5 had a particle fragmentation coefficient above 0.29. The velocity profile of the fluidisation gas was not disclosed in D3 and therefore was the distinguishing feature of claim 1 of the 3rd auxiliary request over D3.
- There was no evidence that the powders described in the patent in suit had improved properties, such as ESCR, creep, melt index and catalyst activity as compared to the compositions of D3. The problem solved over D3 was to provide an alternative process of preparation of an ethylene copolymer with a high fragmentation coefficient with an

- 6 - T 2210/19

activated chromium oxide catalyst.

- D7 taught that activation at high temperatures should be carried out at a high air flow rate and that the air velocity could be varied during activation. D7 however did not teach that the flow of the activation gas should be increased by at least 1 cm/sec above a temperature of 200°C. The prior art did also not give a motivation that would have led the skilled person to claim 1 of the 3rd auxiliary request. In particular, there was no pointer in D7 as to how a change in the gas velocity at different temperatures during the activation process would change one or more of the parameters that were relevant to the process of the 3rd auxiliary request. Also, D8 did not teach a change in the gas flow dependant on the temperature nor its influence on the particle fragmentation coefficient or its underlying parameters. Claim 1 involved therefore an inventive step.
- VI. The opponent (appellant) lodged an appeal against that decision.
- VII. With the rejoinder to the statement setting out the grounds of appeal the patent proprietor (respondent) filed a main request which corresponded to the set of claims maintained by the opposition division ($3^{\rm rd}$ auxiliary request) as well as a $1^{\rm st}$ and a $2^{\rm nd}$ auxiliary requests.

Claim 1 of the 1^{st} auxiliary request corresponded to claim 1 of the main request amended to recite that the fluidisation gas used during the consecutive stage of the activation process was an inert gas followed by an oxidising gas. In the 2^{nd} auxiliary request, claim 1

- 7 - T 2210/19

was further amended by adding that the consecutive stage was the fluidisation stage performed at a higher fluidisation velocity.

- VIII. The parties were summoned to oral proceedings. Issues to be discussed at the oral proceedings were then specified by the Board in a communication dated 17 June 2020.
- IX. With letter of 23 September 2020 the respondent filed new auxiliary requests (2nd and 3rd auxiliary request corresponded to claim 1 of the 1st auxiliary request in which it was further specified that the step in which the fluidisation velocity was brought to a value of Vf2 was a consecutive stage to the step in which the fluidisation gas was maintained at a fluidisation velocity Vf1. The 3rd auxiliary request corresponded to the 2nd auxiliary request filed with the reply to the statement of grounds of appeal.
- X. Oral proceedings were held on 13 January 2021, the parties being connected remotely by videoconference.
- XI. The appellant's arguments, insofar as relevant to the present decision, may be summarised as follows:

Main request

Inventive step over D3

- Example 20 of D3 represented the closest prior art. The process disclosed in that example did not disclose the fragmentation coefficient of the polymer produced but that parameter was calculated in D6 on the basis of the information contained in

- 8 - T 2210/19

D3. Claim 1 of the main request therefore differed from the process of example 20 of D3 only in the fluidisation velocity profile of the activation step including Vf1 and Vf2 as defined in claim 1 of the main request.

- The single example and the two comparative examples of the patent in suit did not give enough information to conclude that the fluidisation velocity profile had any effect on the fragmentation coefficient. In particular, the processes disclosed in the examples of the patent in suit differed from one another so significantly that no comparison of these examples could show an effect resulting from the fluidisation velocity profile. D6 also showed that regardless of whether the required fluidisation velocity profile was used or not, a fragmentation coefficient of at least 0.29 was obtained.
- The problem was thus to provide an alternative process for the preparation of an ethylene copolymer with a fragmentation coefficient equal to or greater than 0.29 with an activated chromium oxide catalyst.
- Since D3, D4 and D5 already produced an ethylene copolymer having a particle fragmentation coefficient of at least 0.29 and used a supported chromium oxide catalyst activated in a fluidised bed activation reactor at a temperature above 500°C, the solution to the objective technical problem was simply to provide an appropriate fluidisation velocity profile for the activation step. This was well within the ability of one skilled in the art, and was merely a matter of

routine experimentation.

- One skilled in the art was also taught that a fluidisation velocities of below 6.5 cm/s at low temperatures and at least 1 cm/s higher than this velocity at higher temperatures was routine. This could be seen, for example, in D7, which gave the background of chromium catalyst activation procedures. In particular its section 20.2 summarized different solutions for commercial catalyst activation procedures using a fluidized bed of catalyst containing 1 wt% Cr(III) on silica including heating at a linear ramp up to 788°C, and then holding at 788°C for 5 hours. It was shown therewith that at a higher fluidization velocity of 0.8 L/min compared to 0.4 L/min, the conversion to the Cr(VI) was higher. Also, section 20.4 and its Figure 253 disclosed how most air velocity data were in the range of about 3 to about 7.5 m/min corresponding to 5 to 12.5 cm/sec (which included the value $6.4~\mathrm{cm/s}$ that was close to $6.5~\mathrm{cm/s}$ defining claim 1 of the main request). This range of air velocities overlapped substantially with the range taught in the patent in suit in absolute terms for the Vf2 in paragraph 22. This velocity was employed in D7 up to 800°C so it was also valid for the process according to claim 1 of the main request. Section 20.5 of D7 taught in addition that the fluidisation velocity profile could be varied during the catalyst activation. Claim 1 of the main request therefore lacked an inventive step over D3.

1st auxiliary request

The modification performed in the 1st auxiliary request did not constitute any difference with

- 10 - T 2210/19

regard to the prior art. The catalyst used in the process according to example 20 of D3 was already produced in a fluidisation gas being nitrogen followed by an oxidizing gas. Also, the term "consecutive stage" used in claim 1 of the 1st auxiliary request was unclear and did not constitute a clear distinction with respect to D3. In particular section 12.4 of D7 disclosed a commercial activation in which nitrogen was followed by air. That commercial practice from D7 corresponded exactly to the profile used in the examples of the patent in suit (Table 1). The profile used in the patent in suit was also disclosed in section 3.8 of D8. Claim 1 of the 1st auxiliary request lacked therefore an inventive step.

2nd and 3rd auxiliary requests

- Claim 1 of the 2^{nd} and 3^{rd} auxiliary requests did not differ substantially from claim 1 of the 1^{st} auxiliary request. The arguments provided for the 1^{st} auxiliary request also applied to the 2^{nd} and 3^{rd} auxiliary requests.
- XII. The respondent's arguments, insofar as relevant to the decision, may be summarised as follows:

Main request

Inventive step over D3

- D3 represented the closest prior art. Claim 1 of the main request differed from example 20 of D3 in the selection of a specific fluidisation velocity profile in the activation step that was not

- 11 - T 2210/19

addressed in D3.

- Therefore the objective problem was to provide a reliable and consistent method for the production of a chromium catalysed ethylene copolymer powder with a low fragmentation, i.e. a high fragmentation coefficient. The problem was solved by selecting a process characterised by the step of activation of the chromium catalyst with a fluidisation velocity coefficient profile as claimed in claim 1.
- Starting from example 20 of D3, the skilled person could arrive at the subject matter of claim 1 of the main request only with hindsight as the skilled person would have had to choose to change the fluidisation velocity profile and select specific velocities which were not suggested in the prior art.
- D3 in particular addressed the modification of chromium based catalyst by contacting it with a reducing agent under specific conditions of feed rate, agitation and addition time during the reaction of the reducing agent in order to obtain a catalyst with an adjustable flow index response after activation of the catalyst.
- With regard to the activation of the catalyst as such, the only teaching provided in D3 was that any given catalyst was to be activated under specific conditions. These conditions were the temperature, holding time at a set temperature and type of gas used. No reference was made in D3 to the fluidisation velocity profile of the fluidisation step. Also, D3 did not suggest any relation between the flow index response of the catalyst that was

- 12 - T 2210/19

the object of D3 and the activation of the catalyst or the chosen fluidisation velocity profile during the catalyst activation. D3 did not motivate the skilled person to alter the catalyst or activation procedure. Moreover, a small change in the parameters defining the activation could affect the fragmentation coefficient to such an extent that it would be outside the range defined in claim 1 of the main request, as shown in examples 20-23 of D3 in combination with D6. In fact, D3 and D6 showed that a skilled person considering raising the fragmentation coefficient would decrease the addition time of the diethylaluminum ethoxide (DEAlE) during reduction and would adjust the temperature of the process, as shown in examples 20-23 and Table 2b of D3. There was thus no motivation in D3 to adopt a fluidisation velocity profile as defined in claim 1 of the main request.

D7 and in particular its section 12.4 which concerned the commercial activation of chromium oxide catalysts did not suggest varying the fluidisation gas velocity during activation of the catalyst. In fact, the fluidisation velocity was normally constant during the activation process. The whole of section 20 of D7 was also not relevant to D3 since it addressed the issues relating to the presence of vapour during activation of the catalyst solely in air and at high temperatures of up to about 800°C. Furthermore section 20.4 indicated that the commercial activations disclosed therein were carried out at constant fluidisation velocity (Figures 252 and 253). The passage in the third paragraph on page 576 in section 20.5 mentioning the possibility of varying the velocity related only to the use of a bent ramp which was

- 13 - T 2210/19

not the standard commercial procedure. That passage was not relevant to D3 because the examples of D3 used a linear ramp and not a bent ramp. Also, section 20.5 concerned predictions relating to the bent ramp model and did not correspond to a process that had been commercially operated. Furthermore, the use of a bent ramp was intended to address issues caused by vapour on the catalyst activated at 800°C. These issues were not relevant to example 20 of D3 in which heating was performed in nitrogen first to draw out water from the catalyst and then activation was performed at 600°C, a temperature at which there were no issues with vapour (first paragraph of page 573 of D7).

- The teaching of D8 was similar to that of D7.

 Therein it was indicated that in the commercial activation of chromium oxide catalysts the fluidisation velocity was constant at 6.5 cm/s (page 255). The passage on page 258 of D8 did not suggest a variation of the fluidisation gas velocity as it pointed at the maximum possible air flow when activation was carried out at 800°C. In that regard, D8 did not contain a pointer towards the solution provided in the patent in suit.
- As laid out in the decision of the opposition division the prior art should contain a motivation for change even when an alternative solution was considered. There should thus be a pointer to the influence of the activation gas flow profile on the fragmentation coefficient of the polymer to be obtained. As there was no pointer anywhere in D7 or D8 how a change in the gas velocity at different temperatures during the activation process would change one or more of the parameters influencing

the fragmentation coefficient, D7 and D8 did not contain a motivation to modify D3 so as to arrive at the process of claim 1 of the main request. Claim 1 of the main request therefore involved an inventive step.

1st auxiliary request

Claim 1 of the 1st auxiliary request required i) the change of the fluidisation velocity, ii) the use of a high temperature and iii) the use of an oxidizing gas. That combination was not disclosed nor suggested in the prior art. In particular the use of a specific fluidisation velocity profile after which the inert gas was replaced by an oxidizing gas in the consecutive stage of the activation process allowed water to be drawn out from the catalyst. That alone was not rendered obvious by the prior art and in particular not by D7 which disclosed the increase of gas velocity when air was used at high temperatures. In particular there was no evidence that sensitivity to moisture was at all a consideration in D3 and thus there was no reason to apply the combination of measures found in separate parts of D7 and D8 to the activation process used in example 20 of D3. Claim 1 of the 1st auxiliary request involved therefore an inventive step.

2^{nd} and 3^{rd} auxiliary requests

Claim 1 of the 2nd and 3rd auxiliary requests contained clarifying language relating to the consecutive stages of the activation process and they did not differ substantially from claim 1 of the 1st auxiliary request. The arguments provided - 15 - T 2210/19

for the 1^{st} auxiliary request also applied to the 2^{nd} and 3^{rd} auxiliary requests.

- XIII. The appellant requested that the decision under appeal be set aside and that the patent be revoked. It was also requested to admit documents D10-D12 into the proceedings and that the 2nd auxiliary request filed with letter dated 23 September 2020 not be admitted into the proceedings.
- XIV. The respondent requested that the appeal be dismissed (main request) or that the patent be maintained on the basis of the 1st auxiliary request as filed with the reply to the statement setting out the grounds of appeal, or the 2nd auxiliary or 3rd auxiliary requests filed with letter dated 23 September 2020. It was also requested not to admit documents D10-D12 into the proceedings.

Reasons for the Decision

Main request

- 1. Inventive step
- 1.1 Claim 1 of the main request generally pertains to a process for producing a chromium catalysed ethylene copolymer powder by polymerising the corresponding monomers in the presence of an activated supported chromium oxide based catalyst, the process comprising the step of activation of the catalyst. Claim 1 of the main request corresponds to claim 1 of the 3rd auxiliary request found to be inventive over D3 in the contested decision. D3 and in particular its example 20 was chosen as the closest prior art with respect to operative claim 1 in the contested decision. That

- 16 - T 2210/19

choice was not disputed in appeal. D3, as the patent in suit, concerns olefin polymerization catalysts and more specifically chromium based catalysts and methods of use of chromium based catalysts for the production of polyolefins (D3, paragraph 2; patent in suit, paragraph 1). The Board does not see a reason to deviate from D3 as the closest prior art.

- 1.2 Example 20 of D3 was seen as particularly relevant as starting point for the assessment of inventive step of operative claim 1. Example 20 of D3 discloses the use of a DEALE-reduced chromium oxide based catalyst supported on 957HS silica (paragraphs 97, 105 and 111) to produce an ethylene/hexene copolymer (Table 2a on page 40). The catalyst is said to be prepared in accordance with Example 7 (Table 2a). The activation conditions of that catalyst are given in paragraph 97, in particular the catalyst was charged into a fluidised bed heating vessel, heated under dry nitrogen up to 325°C and held at that temperature for 2 hours after which the nitrogen stream was replaced by a stream of dry air and the catalyst heated slowly up to 600°C where it was activated for 6 hours. The activated catalyst was then cooled with dry air to about 300°C and further cooled to room temperature with dry nitrogen. It is apparent from that description that the silica supported DEALE-reduced chromium oxide catalyst used in example 20 was activated in a fluidised bed activation reactor and that it was submitted to a temperature of above 500°C, as required by operative claim 1.
- 1.3 D3 does not disclose the particle fragmentation coefficient of the copolymer produced. However, it was not in dispute that the particle fragmentation coefficient, which is defined by a formula given in

- 17 - T 2210/19

operative claim 1 and in paragraph 6 of the patent in suit, could be calculated from the information provided in the examples of D3. In this context the experimental report D6, and in particular its Table 3, shows that the fragmentation coefficient calculated for the copolymer produced from the process according to example 20 of D3 is 0.360, i.e. it is in the range defined in operative claim 1 (at least 0.29). Also that conclusion was not in dispute between the parties in appeal.

- 1.4 The fluidisation gas velocity profile is not given in D3 nor is derivable from the information contained in that document. The fluidisation gas velocity profile defined in operative claim 1 requires that the fluidisation velocity (Vf1) of the fluidisation gas is initially maintained below 6.5 cm/s, until the temperature inside the activation reactor reaches at least 200°C, and said fluidisation gas is then brought to a value (Vf2) which is at least 1 cm/s higher than Vf1. That fluidisation velocity profile constitutes therefore the distinguishing feature over the process of example 20 of D3.
- 1.5 The opposition division found in the contested decision that there was no evidence on file that the polymer powders obtained in the example of the patent in suit had improved properties, such as ESCR, creep, melt index and catalyst activity as compared to the closest prior art D3 and also with respect to the comparative examples of the patent in suit. The opposition division considered that due to the differences in the processes disclosed in the examples of the patent in suit and in example 20 of D3, it could not be concluded that the fluidisation gas velocity profile according to operative claim 1 resulted in any effect over the

- 18 - T 2210/19

process of the closest prior art. The opposition division concluded therefrom that the problem over D3 was "to provide an alternative process of preparation of an ethylene copolymer with a high fragmentation coefficient with an activated chromium oxide catalyst" (section 3.2, last paragraph on page 12). By contrast, the respondent contended at the oral proceedings before the Board that the problem over D3 was the provision of a reliable and consistent method for the production of a chromium catalysed ethylene copolymer powder with a low fragmentation i.e. a high fragmentation coefficient. This, it was argued, would be achieved by the adoption of the claimed fluidisation gas velocity profile. The respondent based their formulation of the problem on the example and the two comparative examples of the patent in suit.

- 1.6 According to the case law of the boards of appeal, alleged advantages to which the patent proprietor merely refers, without offering sufficient evidence to support the comparison with the closest prior art, cannot be taken into consideration in determining the problem underlying the invention and therefore in assessing inventive step (Case Law of the Boards of Appeal, 9th Edition, July 2019, I.D.4.2).
- 1.7 The patent in suit contains a single example according to operative claim 1. In that example the polymer obtained from the polymerization process had a fragmentation coefficient of 0.315. It is however clear from the description of example 1 of the patent in suit in paragraph 53 that apart from being a silica supported chromium oxide catalyst, the catalyst used in that example differs in multiple instances from that used in example 20 of D3 such as in the type of silica support (957HS in D3, MS-3050 in example 1 of the

- 19 - T 2210/19

patent in suit), different particle sizes (40 µm in paragraph 97 in D3, 90 µm in paragraph 54 in example 1 of the patent in suit) and surface areas $(300 \text{ m}^2/\text{g})$ in paragraph 97 in D3, 470 m^2/g in paragraph 54 in example 1 of the patent in suit). The appellant made plausible that these differences would have an impact on the process and on the polymer produced. In particular, since the particle size of the catalyst is involved in the formula defining the fragmentation coefficient of the polymer produced, it is immediately clear that the different particle sizes of the catalysts used in the example of the patent in suit and in example 20 of D3 will affect the fragmentation coefficient of the resulting polymer. In that regard, any effect resulting from the use of the catalyst disclosed in the example of the patent in suit on the fragmentation coefficient of the produced polymer cannot be attributed to the distinguishing feature of claim 1 of the main request over D3.

1.8 It can thus not be concluded from the example of the patent in suit that the process of claim 1 of the main request leads to olefin polymers with improved properties or to a particularly reliable and consistent method for the production of a chromium catalysed ethylene copolymer powder with a low fragmentation i.e. a high fragmentation coefficient. The comparative examples 1 and 2 of the patent in suit do not support a different conclusion either since the catalysts used in the examples of the patent in suit are different commercial products (PQC35105 in example 1; PQC24340 in comparative example 1; EP350X in comparative example 2) and have morphologies that differ from the catalyst according to example 1 and also involve different supports (aluminium modified vs. titanium modified supports) and different chromium loadings as apparent

- 20 - T 2210/19

from paragraphs 60-68 of the patent in suit. Moreover, the conditions applied during the following olefin polymerisation were also different (polymerisation temperatures, partial pressures, proportions of hexene copolymer, bed levels, fluidisation velocities and production rates) as is apparent from the example of the patent in suit, paragraph 59 and comparative examples 1 and 2, paragraphs 66 and 73. The achievement of a given fragmentation coefficient cannot therefore be attributed to the use of a specific fluidisation profile.

- 1.9 The Board concludes therefrom that the data contained in the patent in suit do not support the formulation of problem as the provision of a reliable and consistent method for the production of a chromium catalysed ethylene copolymer powders with a low fragmentation i.e. a high fragmentation coefficient.
- 1.10 Since there are no data on file that can show that the fluidisation velocity profile according to claim 1 of the main request results in any improvement over the process disclosed in example 20 of D3, the problem can only be defined as the provision of a further process for producing a chromium catalysed ethylene copolymer powder, in accordance with the problem that was proposed in the preliminary opinion of the Board (section 10.10).
- 1.11 The skilled person, looking for a further process, would consider variations of the process already known from the closest prior art, taking into account the common general knowledge in the field and the knowledge made available in the prior art. That includes variations of any of the parameters of the process that can be expected to be suitable to carry out alternative

- 21 - T 2210/19

processes without exercising any inventive activity. As the problem is simply the provision of a further process, no further motivation is needed by the skilled person to perform the modified process.

- 1.11.1 The formulation of the activation step a) in claim 1 of the main request is open ("comprising") and allows a change of the fluidisation gas from dry nitrogen to dry air, as done in D3. The question with respect to obviousness in the present case is whether, in the activation process of the silica supported chromium oxide catalyst used in example 20 of D3, the selection of a gas velocity of below 6.5 cm/s until the temperature inside the activation reactor reaches at least 200°C, followed by an increase of said fluidisation gas velocity at any point after that temperature was reached by at least 1 cm/s in order to merely provide a further process, could be seen as involving an inventive step.
- 1.11.2 The activation of chromium catalysts is discussed broadly in paragraphs 51-53 of D3, where it is taught that when a fluidized bed is used, the passage of a stream of dry air or oxygen through the supported chromium-based catalyst during the activation aids in the displacement of any water from the support and converts, at least partially, chromium species to Cr⁺⁶. There is however no mention of the gas velocity during activation in D3 and thus no teaching that the gas velocity would be limited in any way as long as it makes technical sense in the context of the activation.
- 1.11.3 D7, a textbook on catalysis containing a section on supported chromium catalysts used in ethylene polymerization processes (section 3, A Review of the Phillips Supported Chromium Catalyst and Its Commercial

- 22 - T 2210/19

Use for Ethylene Polymerization, pages v and vi of the index), provides some insights as to which types of fluidisation gas velocities were known to be used in activation processes of supported chromium catalysts. In particular, section 20.4 of D7 relates to the mathematical modeling of an activation process and mentions an activation example in which the supported chromium catalyst was brought to a temperature of up to 800°C under an air flow having a velocity of 6.4 cm/s (page 573, second paragraph and Figure 252). The model shows that the use of a gas velocity close to the value defined in operative claim 1 (6.5 cm/s) at a temperature that is above 500°C was known in the art. Also, gas velocity values in the broader range of about 5-12.5 cm/s, but still encompassing the threshold of 6.5 cm/s defined in operative claim 1, are disclosed for other commercial teste activations on Figure 253 (middle part relating to air velocity) on page 574 of D7. Also, D8, which is an academic publication relating to the same type of supported chromium catalyst activation mentions fluidized bed gas velocities of 1.5 to 10 cm/s as typical for commercial activation processes (page 254, second column, second paragraph). D7 and D8 show therefore that fluidized bed gas velocities below and above the threshold of 6.5 cm/s set out in claim 1 of the main request were already common in the art for the same type of activations.

1.11.4 While the activations disclosed in section 20.4 of D7 and in the second column on page 254 and page 255 of D8 for which the fluidisation gas velocity are given were performed at high temperatures of up to 800°C and more, as underlined by the respondent, there is nothing in D7 and D8 that limits the fluidisation gas velocities used during activation to any range of temperatures. Indeed, D8 (Section 3.2 and Figure 3 on page 254) and D7

- 23 - T 2210/19

(Figure 9 page 147; last paragraph of page 364 and section 20.3) teach that temperatures of as low as 400°C could be successfully used during activation and that the moisture released during activation was not detrimental to the stability of chromium VI in the catalyst up to a temperature of about 600°C (first paragraph on page 573 of D7). That teaching is compatible with the closest prior art D3 in which an activation temperature of 600°C was used in the activation of the catalyst involved in example 20. Also, the range of temperatures that can be used in the activation according to D3 largely overlaps with the one known from D7 and D8 since any temperature from about 300°C up to the temperature at which substantial sintering of the support takes place can be used (paragraph 51 of D3), which in paragraph 52 of D3 may be up to about 900°C. There is no teaching in D3, D7 or D8 that would lead away from the broad range of gas velocities of 1.5-10 cm/s at any activation temperature chosen between 400°C and 800°C in the cited prior art.

- 1.11.5 The choice of a fluidisation gas velocity in the range of 1.5-10 cm/s at an activation temperature of 600°C, would therefore be in view of the teachings of D7 or D8, a common measure that a skilled person could take to merely provide a further activation process that ultimately would provide a further process for producing a chromium catalysed ethylene copolymer powder.
- 1.11.6 The additional increase in velocity by 1 cm/s after the temperature reaches 200°C as defined in operative claim 1 was also not shown to provide any effect so that an increase of the gas velocity is seen as an arbitrary measure that the skilled person could have selected as well. In that regard, the measure is also generally

known in the context of the activation of the catalyst, as suggested by D7 (page 576, $3^{\rm rd}$ paragraph) and D8 (page 258, column 1, $2^{\rm nd}$ paragraph). Increasing the velocity at any point during activation, even by 1 cm/s, is thus not seen as an inventive measure. Claim 1 of operative claim 1 is thus found to lack an inventive step.

1.11.7 The fact that the catalyst in D3 is treated, after its activation and prior to its use in the polymerization of olefins, with a reducing agent in order to tailor its flow index response (paragraph 17; claim 1 of D3) does not change the conclusion reached by the Board on obviousness. The treatment of the supported chromium catalyst with a reducing agent such as diethylaluminum ethoxide (DEALE) as it is disclosed in D3 and which is said to have an influence on the molecular weight of the polyolefin produced (paragraphs 26-28) has not been shown to have a negative impact on the fragmentation coefficient of that polymer. Examples 20, 21 and 24-28 according to D3 that are also reported in Table 3 of D6 in particular show that a supported chromium catalyst activated under a gas velocity profile (#2) according to claim 1 of the main request and treated with a reducing agent (Tables 2A and 2B of D3) all display a fragmentation coefficient above 0.29 as required in claim 1 of the main request. In that regard, the fact that examples 22 and 23 of D3 are not reported in D6 alongside the other examples of D3 (examples 20,21 and 24-28) is as such not relevant to the question of obviousness since the process according to example 20 of D3, which is the starting point for the assessment of inventive step already leads to a fragmentation coefficient of the produced polymer of 0.360, a value that is within the range of operative claim 1 (equal or superior to 0.29). Finally, the process of claim 1 of

- 25 - T 2210/19

the main request, because of its open formulation ("comprising") does not exclude a further step after the activation step a) and the polymerization step b) in which the activated catalyst is treated with a reducing agent in the same manner as in D3.

1.12 It follows that starting from example 20 of D3 the selection, in the activation step of the supported chromium catalyst, of a fluidisation gas velocity of below 6.5 cm/sec until the temperature inside the activation reactor reaches at least 200°C, followed by an increase of said fluidisation gas velocity at any point after that temperature was reached by at least 1 cm/sec is an obvious measure when aiming at merely providing a further process for producing a chromium catalysed ethylene copolymer powder. Therefore the process of claim 1 of the main request does not involve an inventive step.

1st auxiliary request

- 2. Inventive step
- 2.1 Example 20 of D3, which was accepted by the parties as the closest prior art for the main request, remains the most relevant starting point for the assessment of inventive step of claim 1 of the 1st auxiliary request.
- 2.2 Claim 1 of the 1st auxiliary request differs from claim 1 of the main request in that, in the activation step of the supported chromium oxide based catalyst, the fluidisation gas used during the consecutive stage of the activation process is an inert gas followed by an oxidising gas.

T 2210/19

2.3 The respondent argued that there was no disclosure in the prior art of a process containing the three stages relating to the specific fluidisation velocity profile involving the increase of the gas velocity as defined in claim 1 of the main request and additionally using first an inert gas followed by oxidising gas after the temperature in the activation reactor reached at least 200°C. However, the activation of the supported chromium catalyst used in example 20 of D3 is disclosed in paragraph 97. According to that process, porous silica support containing 2.5 weight percent chromium acetate is charged to a fluidized bed heating vessel and heated slowly at a rate of about 50°C per hour under dry nitrogen up to 325°C and held at that temperature for about 2 hours, after which the nitrogen stream was replaced with a stream of dry air and the catalyst composition was heated slowly at a rate of about 50°C per hour to 600°C where it was activated for about 6 hours. It is apparent from that passage that the activation involved the use of an inert gas (dry nitrogen), followed by an oxidising gas (dry air) after the temperature in the activation reactor reached 325°C (therefore at least 200°C).

- 26 -

- 2.4 It follows that operative claim 1 differs from example 20 of D3 only in the fluidisation velocity profile that was already part of the definition of claim 1 of the main request.
- 2.5 The added feature in claim 1 of the 1st auxiliary request was not shown to result in any further effect by comparison to the main request. The patent in suit and in particular paragraphs 17 and 20, which concern the fluidisation gas used in the activation process, do not disclose any effect associated with the use of an inert gas followed by an oxidising gas in the

- 27 - T 2210/19

fluidisation velocity profile according to operative claim 1. In view of the absence of any effect resulting from the feature added in operative claim 1, the problem remains, as for the main request, the provision of a further process.

2.6 In that regard, since claim 1 of the main request was found to lack an inventive step in view of D3 in combination with D7, the same conclusion is also reached in case of claim 1 of the 1st auxiliary request for the same reasons as for claim 1 of the main request. Claim 1 of the 1st auxiliary request lacks therefore an inventive step.

 2^{nd} and 3^{rd} auxiliary requests

- 3. Inventive step
- Claim 1 according to the 2^{nd} and 3^{rd} auxiliary requests 3.1 differs from claim 1 of the 1st auxiliary request only in the use of additional language meant to clarify the order of stages relating to the different gas velocities in the activation step. In particular, the language used in claim 1 according to the 2nd and 3rd auxiliary requests underlines that the stage at which the fluidisation gas velocity is at the value Vf1 and the one with an increase in gas velocity to a value (Vf2) which is at least 1 cm/s higher than Vf1 are consecutive stages of the activation. That however was not in dispute between the parties at the oral proceedings before the Board. Also, it was already understood that the stages at which the velocities are defined as being Vf1 and Vf2 in claim 1 of the main request are consecutive stages of the activation process. In that regard, the reasoning of inventive step applying to claim 1 of the 1st auxiliary request

- 28 - T 2210/19

equally applies to claim 1 according to the 2^{nd} and 3^{rd} auxiliary requests. Indeed the parties did not provide any separate argument on inventive step for these requests.

- 3.2 It follows that claim 1 according to the 2nd and 3rd auxiliary requests lacks an inventive step over D3 as the closest prior art. In view of this the issue of admittance of the 2nd auxiliary request into the proceedings becomes irrelevant, as acknowledged by the appellant.
- 3.3 As all the requests of the respondent do not meet the requirements of Article 56 EPC, the appeal is to be dismissed and there is no need for the Board to decide on any other issue.

Order

For these reasons it is decided that:

- 1. The decision under appeal is set aside.
- 2. The patent is revoked.

The Registrar:

The Chairman:



B. ter Heijden

D. Semino

Decision electronically authenticated