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**Datasheet for the decision
of 25 February 2009**

Case Number: T 1707/06 - 3.3.06

Application Number: 98964176.6

Publication Number: 1042408

IPC: C09C 1/36

Language of the proceedings: EN

Title of invention:

Process for producing coated TiO₂ pigment using cooxidation to provide hydrous oxide coatings

Patentee:

E.I. DU PONT DE NEMOURS AND COMPANY

Opponent:

KRONOS INTERNATIONAL, INC.

Headword:

Coated TiO₂ particles/E.I. DU PONT DE NEMOURS

Relevant legal provisions:

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Relevant legal provisions (EPC 1973):

EPC Art. 56

Keyword:

"Inventive step (yes)"

"Burden of proof"

Decisions cited:

T 0939/92

Catchword:

-



Case Number: T 1707/06 - 3.3.06

D E C I S I O N
of the Technical Board of Appeal 3.3.06
of 25 February 2009

Appellant: KRONOS INTERNATIONAL, INC.
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D-51307 Leverkusen (DE)

Respondent: E.I. DU PONT DE NEMOURS AND COMPANY
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Decision under appeal: Decision of the Opposition Division of the
European Patent Office posted 13 September 2006
rejecting the opposition filed against European
patent No. 1042408 pursuant to Article 102(2)
EPC 1973.

Composition of the Board:

Chairman: P.-P. Bracke
Members: E. Bendl
A. Pignatelli

Summary of Facts and Submissions

- I. This appeal is against the decision of the Opposition Division to reject the opposition against the European patent 1 042 408.
- II. Claim 1 as granted, the only independent claim out of a set of eleven claims reads as follows:
- "1. A process for producing a titanium dioxide pigment, comprising the steps of:
- a) reacting titanium tetrachloride in the vapor phase with an aluminum halide and an oxygen-containing gas in a reactor at a temperature in the range of 900°C to 1600°C to provide a gaseous suspension comprising TiO₂ particles;
- b) contacting the gaseous suspension with at least two oxide precursors, wherein the first oxide precursor is a silicon halide and the second oxide precursor is selected from the group consisting of oxide precursors of boron, phosphorus, magnesium, niobium, germanium, and mixtures thereof; and
- c) cooling the gaseous suspension to provide a pigment comprising TiO₂ particles having a coating comprising silica and a second oxide wherein the second oxide is selected from the group consisting of oxides of boron, phosphorus, magnesium, niobium, germanium, and mixtures thereof."
- III. With the letter dated 07.11.06 the Opponent (Appellant) filed an appeal against the decision of the Opposition Division. In the grounds for appeal and in his letter

of 29.10.07 he provided a reasoning why the patent-in-suit allegedly does not meet the requirement of Article 56 EPC. Documents B1-B6 were submitted.

IV. The Proprietor of the patent (Respondent) replied with the letters dated 01.06.07 and 23.01.09. He submitted four auxiliary requests and comparative example E1.

V. During appeal procedure inter alia the following documents were cited:

D2 = WO-A-96/36441

D5 = EP-B-0245984

D6 = EP-B-0791036

D7 = US-A-4214913

D8 = US-A-3060001

B1 = phase diagram $\text{SiO}_2\text{-B}_2\text{O}_3$

B2 = phase diagram $\text{SiO}_2\text{-P}_2\text{O}_5$

B3 = phase diagram $\text{SiO}_2\text{-MgO}$

B4 = phase diagram $\text{SiO}_2\text{-Nb}_2\text{O}_5$

B5 = phase diagram $\text{SiO}_2\text{-GeO}_2$

B6 = US-A-5562764

E1 = comparative example

VI. Appellant's main arguments were as follows:

- The combination of D2 with either D5 or D6 leads to the subject-matter as claimed. D7 and D8 give additional information concerning the use of boron and phosphorous compounds.

- Documents B1-B5 show that in the temperature range 900-1600°C the mixtures of silicon oxide and a second

oxide are only partly liquid. Eutectic temperatures are higher than the lower temperature limit given.

- Improved durability has merely been shown for the combination of SiO_2 and B_2O_3 . According to T 939/92 (OJ EPO 1996, 309) the problem can only be regarded as being solved, if all compounds claimed show the desired effect.

- E1 cannot be used as a suitable comparative example, because the compositions comprising PCl_3 contain a higher absolute amount of coating material than the compositions without PCl_3 .

VII. The Appellant requested that the decision of the Opposition Division be set aside and the patent be revoked in its entirety.

VIII. The Respondent requested that the appeal be dismissed as a main request or that the patent be maintained on the basis of one of the auxiliary requests I-IV, filed with the letter of 23.01.09.

Reasons for the Decision

Main request

1. Novelty

The Board comes to the finding that none of the cited prior art documents discloses all parameters of the claimed process. This was not contested any more by the Appellant.

2. Inventive step

According to the problem-solution-approach, which is used by the Boards of Appeal of the European Patent Office in order to decide on the question of inventive step, it has to be determined which technical problem the object of a patent objectively solves vis-à-vis the closest prior art document. It also has to be determined whether or not the solution proposed to overcome this problem is obvious in the light of the available prior art disclosures.

2.1 It has not been contested that D2 represents the closest state of the art.

According to page 2, second paragraph of D2, it is the aim of this disclosure to provide a simple and reliable process for coating TiO_2 particles in the gas phase to give coatings with low surface areas, low oil absorption and uniform thickness. In a preferred embodiment the addition of a volatile aluminium-containing precursor to the titanium-containing precursor is recommended (page 3, third paragraph).

2.2 The problems to be solved by the claimed process are defined in paragraphs 4 and 10 of the patent-in-suit as the provision of a process which overcomes problems associated with conventional wet treatment methods for producing rutile TiO_2 pigments with a dense uniform coating, durability and gloss. Furthermore, according to paragraphs 40 and 41 of the patent-in-suit the effect achieved by the addition of a second oxide precursor is a more uniform deposition/improved durability of the coated TiO_2 pigments.

Thus, the problem to be solved with regard to D2 according to the patent-in-suit is to provide a process for producing TiO₂ pigments showing such improved deposition/durability.

- 2.3 The proposed solution to this problem is defined in Claim 1.

The difference between D2 and the subject-matter of Claim 1 of the patent-in-suit is the addition of a **second** oxide precursor being selected from boron, phosphorous, magnesium, niobium and germanium, as well as their mixtures.

- 2.4 The question whether the problem has actually been solved has to be answered with regard to the individual second oxide precursors mentioned in Claim 1:

- (i) boron oxide precursors
- (ii) phosphorous oxide precursors
- (iii) oxide precursors of magnesium, niobium, germanium and mixtures of any of the compounds cited in (i)-(ii)

- 2.4.1 (i) Use of boron oxide precursors

The Appellant has confirmed during the oral proceedings as well as in the letter of 05.01.07, page 7, last paragraph, page 8, third paragraph and in the letter of 29.10.07, page 1, second paragraph, page 2 second paragraph and page 3, second paragraph, that improved durability and a homogeneous coating of the TiO₂ particles have been demonstrated, when using silicon halide/boron oxide precursor combinations. Thus, the

Appellant concedes that the desired effect has been achieved and that the problem described above has been solved for the said combination.

2.4.2 (ii) Use of phosphorous oxide precursors

With the letter dated 23.01.09 the Respondent has submitted comparative tests, named document E1, to demonstrate improved effects with regard to particle coating/improved durability by using silicon halide/phosphorous oxide precursors.

The Appellant argued that the comparative tests cannot demonstrate any effect achieved, because by adding PCl_3 to $\text{AlCl}_3/\text{SiCl}_4$ the total amount of coating material on each particle would be increased compared to the mere coating with $\text{AlCl}_3/\text{SiCl}_4$. He argued, that the percentage of coated area could consequently not be used to demonstrate that improved effects have been achieved. However, no proof that the total amount of oxide precursors influences the percentage of completely coated particles in the tests described in E1 has been submitted by the Appellant.

The Board is not convinced that merely the total amount of coating material is responsible for the effect achieved. Example C-2 of E1 describes higher amounts of coating materials than Example B-2. However, the percentage of particles completely coated is lower in Example C-2, compared to Example B-2. Thus, Appellant's argument is not considered to be sufficiently substantiated.

- 2.4.3 (iii) Oxide precursors of magnesium, niobium, germanium and mixtures of any of the compounds (i)-(iii)

In analogy to the reasoning given above no proof or evidence has been filed by the Appellant that oxide combinations containing the remaining oxide precursors or any combination of silicon halide with a mixture of oxide precursors as claimed do not show the desired effect.

- 2.4.4 Summarizing it is to be stated that the Appellant has failed to show that **any** of the claimed combinations of oxide precursors (i)-(iii) do not achieve the desired effect. Since he has the burden of proof, the problem of the patent-in-suit is to be regarded as being solved by the subject-matter of Claim 1.

- 2.4.5 This finding is not in contrast to the decision T 939/92:

T 939/92 concerns an appeal against the decision of the Examining Division to refuse a patent application relating to a Markush formula for substituted triazole sulphonamides allegedly possessing herbicide activity.

One of the Appellant's (Applicant's) arguments was, that the prior art disclosures of substituted triazole sulphonamides could not be taken into consideration, because even minor structural modifications of the molecule would lead to unpredictable biological effects.

On the other hand the claimed structure formula in T 939/92 covered literally thousands of possible

compounds. Tests showing the herbicidal activity were not submitted for **all** the compounds encompassed. Thus, applying the same logic as to the prior art disclosures, **not all** of the compounds claimed could be expected to show herbicidal activity. Consequently, given the prior art disclosures available to the Examining Division and to the Board, the skilled person would have severe doubts whether the problem has been solved by **all** compounds claimed.

The situation of the present patent-in-suit is quite different from the situation described in T 939/92 for several reasons:

T 939/92 was based on a decision of the **Examining Division**, whereas in the present patent-in-suit a decision of the **Opposition Division** is under dispute. In examination phase, in case of reasoned doubts by the Examining Division, it is up to the Applicant to demonstrate the alleged effects. In opposition the burden of proof shifts in general to the party making the allegation, i.e. usually to the Opponent. Consequently, in T 939/92 it was the Applicant's obligation to file evidence for the allegations made (see paragraph 2.6.1), whereas in the present case the burden of proof is on the Opponent (now Appellant).

In the present case, neither in opposition phase nor during appeal procedure the Opponent/Appellant provided convincing evidence that an effect is **not** obtained with all second oxide precursors according to Claim 1.

Furthermore, in the patent-in-suit, in contrast to T 939/92, only a very **limited** number of possible oxide

precursors, namely boron, phosphorous, magnesium, niobium, germanium has been claimed in the present patent-in-suit and the arguments and prior art disclosures presented do not give rise to serious doubts whether the described effects have been achieved.

Given the reasoning above, the tenor of T 939/92 is not applicable to the present case.

- 2.5 Finally, it has to be answered whether the solution proposed by Claim 1 is obvious taking into account the prior art disclosures presented in the course of the appeal proceedings.

Document D2 discloses on page 2, lines 17-33 a gas phase process for coating TiO_2 pigments with at least one volatile metal-containing coating precursor. As coating precursors silicon, aluminium and zircon compounds are mentioned on page 3, lines 16/17. There is neither any teaching of coating compounds other than those three compounds nor about the effect of improving durability of the particles.

D5 refers to a wet process of coating TiO_2 particles. First an aqueous slurry of rutile TiO_2 particles is heated, then a silicate and B_2O_3 containing solution is added, by lowering the pH the coating is deposited on the TiO_2 particles and the product is cured (see page 2, lines 45-52). D5 does not refer to a gas phase reaction.

According to D6 TiO₂ particles may be prepared in the vapour phase, but the coating is done in an aqueous slurry (paragraphs 8 and 9).

D7 aims at obtaining higher rutile TiO₂ at any given level of AlCl₃ (col.1, lines 28-33). The process does not use silicon halides.

The object of D8 is the reduction of the acidity of oxides. TiO₂ particles are reacted with boron chloride. However, the use of a second oxide precursor, let alone an effect associated therewith has not been mentioned.

Documents B1-B5 show the phase diagrams of SiO₂ with oxides of other oxide precursors claimed. Appellant's argumentation about the electron configuration of the compounds or the explanation that an eutectic for each combination can only be formed at certain temperatures and that therefore this effect cannot be used to explain the effect achieved has been counter-argued by the Respondent by saying that the phase diagrams only concern equilibrium conditions and on the particles super-cooled liquids are formed. Both parties' arguments concern possible theoretical explanations without any proof or substantiation and have therefore to be disregarded.

With respect to B6 similar considerations as with regard to D2 are of relevance, the use of a second oxide precursor or the effects achieved have not been disclosed therein.

Thus, none of the documents cited above refers to the claimed combination of oxide precursors or mentions the effect caused by using two of the precursors.

Combinations of D2 with either D5 or D6 would also not lead to the claimed subject-matter, since the skilled person had no incentive to use the teaching of either D5 or D6 in combination with the gas phase reactions of D2. The Appellant is misled arguing that it was already known from D2 that oxides can also be used in the gas phase and that he would consequently search for other oxide precursors. Since there was no hint in D2 about increased durability of pigments with an oxide precursor combination, the skilled person would not have had any motive to search for alternative combinations in documents referring to wet processes, i.e. referring to a different kind of process.

Also the combination of D2 with either of D7 or D8 would not lead to the claimed process, since both documents have entirely different purposes, i.e. the increase of the rutile content and the reduction of acidity.

Finally, the combination of D2 with either of D5/D6 and either of D7/D8 is even less obvious, because the skilled person would not have any incentive to combine documents with different purposes **and** relating to different methods (gas phase reactions versus wet processes).

Thus, none of the documents cited nor their combinations would lead the person skilled in the art towards the claimed subject-matter.

The requirement of Article 56 EPC 1973 is consequently met by the subject-matter of the main request.

3. The other requirements for patentability were not put into question. The decision of the Opposition Division is therefore to be maintained and the appeal to be dismissed under Article 111(1) EPC.

Order

For these reasons it is decided that:

The appeal is dismissed.

The Registrar:

The Chairman:

G. Rauh

P.-P. Bracke