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D E C I S I O N
of 26 February 1998

Case Number: T 0281/93 - 3.3.5

Application Number: 87302947.4

Publication Number: 0240367

IPC: C09C 1/64

Language of the proceedings: EN

Title of invention:
Aluminium pigment composition

Patentee:
TOYO ALUMINIUM KABUSHIKI KAISHA

Opponent:
Silberline Manufacturing Co., Inc.

Headword:
Aluminium pigment composition/TOYO ALUMINIUM

Relevant legal provisions:
EPC Art. 123(3), 123(2), 83, 54, 56

Keyword:
"Amendments - allowed"
"Novelty and inventive step - yes"
"Sufficiency of disclosure - yes"

Decisions cited:
-

Catchword:

Europäisches Patentamt | European Patent Office | Office européen des brevets

Beschwerdekammern | Boards of Appeal | Chambres de recours

Case Number: T 0281/93 - 3.3.5

D E C I S I O N
of the Technical Board of Appeal 3.3.5
of 26 February 1998

Appellant: (Proprietor of the patent)
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Decision under appeal: **Decision of the Opposition Division of the European Patent Office posted 22 January 1993 revoking European patent No. 0 240 367 pursuant to Article 102(1) EPC.**

Composition of the Board:

Chairman: G. J. Wassenaar
Members: G. Dischinger-Hoeppler
J. H. van Moer

Summary of Facts and Submissions

I. This appeal is against the decision of the Opposition Division to revoke European patent No. 0 240 367 corresponding to application No. 87 302 947.4. The decision under appeal was based on claims which were amended during the opposition proceedings.

II. The Opposition Division held that the amended Claims 1 and 16 extended the content of the patent as granted (Article 123(3) EPC). They further held that the claimed subject-matter was insufficiently disclosed (Article 83 EPC) due to an inconsistent teaching concerning the content of phosphorus in the pigment composition.

III. In the contested decision, the following documents, inter alia, were cited:

D1: US-A-4 350 535, and
D4: US-A-3 411 923.

IV. In his statement of the grounds of appeal, the Appellant (Proprietor) contested the arguments on which the Opposition Division had based its decision. With a letter dated 26 January 1998, he filed new claims according to a main and first auxiliary request. During oral proceedings held on 26 February 1998 he further filed new claims according to a second and third auxiliary request.

Claim 1 of the main request reads as follows:

"1. An aluminium pigment composition comprising

(i) aluminium flakes;

(ii) an inorganic oxo acid of phosphorus in an amount such that from 500 to 10 000 ppm calculated as phosphorus based on the weight of the aluminium forms a coating on the aluminium flakes;

(iii) an aliphatic compound having from 8 to 40 carbon atoms and having at least one functional group within the molecule, the aliphatic compound being present in an amount of from 0.3 to 5 parts by weight per 100 parts by weight of the aluminium;

(iv) water in an amount of from 0.05 to 10 parts by weight per 100 parts by weight of the aluminium;

(v) a water-compatible alcohol in an amount of from 5 to 100 parts by weight per 100 parts by weight of the aluminium; and

(vi) a hydrocarbon oil in an amount of from 1 to 70 parts by weight per 100 parts by weight of the aluminium."

Claims 1-14 of the first auxiliary request are formulated in terms of a product-by-process, Claim 1 reading:

"An aluminium pigment composition obtainable by a process which comprises mixing together:

(i) aluminium flakes;

(ii) an inorganic oxo acid of phosphorus in an amount of from 500 to 10 000 ppm calculated as phosphorus based on the weight of the aluminium;

(iii) an aliphatic compound having from 8 to 40 carbon atoms and having at least one functional group within

the molecule, the aliphatic compound being present in an amount of from 0.3 to 5 parts by weight per 100 parts by weight of the aluminium;

(iv) water in an amount of from 0.05 to 10 parts by weight per 100 parts by weight of the aluminium;

(v) a water-compatible alcohol in an amount of from 5 to 100 parts by weight per 100 parts by weight of the aluminium; and

(vi) a hydrocarbon oil in an amount of from 1 to 70 parts by weight per 100 parts by weight of the aluminium."

The first auxiliary request further contained a process Claim 15 reading:

"A process for the preparation of an aluminium pigment composition, which process comprises mixing together: ... ", the rest being identical to Claim 1 of the first auxiliary request.

V. Concerning Article 123 EPC, the Appellant presented the following arguments:

(a) A person skilled in the art would interpret "derived from an inorganic oxo acid" as meaning "consisting of the oxo acid itself" and readily realise that said oxo acid was present in chemisorbed form. This was further evident from the FT-IR spectra, filed with the letter dated 9 February 1998. The term "derived from" as defined in original Claim 16 included, therefore, a coating "of" the inorganic oxo acid as claimed in the main request.

(b) The subject-matter of independent Claims 1 and 15 according to the first auxiliary request was derivable from granted Claim 17 and from the fact that all phosphorus supplied from the inorganic oxo acid must be bound to the large surface of the aluminium flakes.

Concerning the objection under Article 100(b) EPC, the Appellant submitted that an invention was sufficiently disclosed if at least one way was given which enabled a person skilled in the art to carry out the invention. Contrary to the decision under appeal, there was no requirement in the EPC for there to be a consistent technical teaching in order to meet the requirements of sufficiency of disclosure, so long as there was no lack of clarity. In the patent in suit there was ample disclosure as to how the phosphorus content can be measured and the invention put into practice.

With respect to novelty and inventive step, the Appellant submitted that it was known that a mixture containing phosphoric acid and alcohol would not simply react to produce phosphoric acid ester.

VI. The Respondent raised objections as to the admissibility of the new claims under Article 123 EPC. He argued essentially as follows:

(a) It was originally not disclosed that the coating might consist of the oxo acid itself as was now claimed in the main request, nor was this feature present in the claims as granted.

(b) Concerning the first auxiliary request, it was submitted that there was no basis for admixing the inorganic oxo acid in an amount as now claimed, either in the application as originally filed or

in the claims as granted. Moreover, it did not follow from the application as originally filed that the phosphorus present in the composition was solely derived from the phosphoric acid.

Objections were also raised under Article 100(b) EPC. The Respondent argued that, in the present case, the artisan was confronted with different possibilities, which did not, however, present the same effects. In addition, it was impossible for someone skilled in the art to determine exactly the amount of oxo acid to be added or the amount of phosphorus present in the composition.

As to the question of novelty and inventive step, the Respondent referred to D1 which disclosed a mixture containing aluminium flakes, organic phosphoric acid ester, water, alcohol and a lubricant. According to D4, phosphoric acid would react with alcohol to give phosphoric acid ester in equilibrium with the starting mixture. Hence, no difference could be seen between the claimed mixture and that of D1. Moreover, the addition of phosphoric acid to the aluminium flakes was known from

D9: US-A-2 858 230 (cited in the patent).

VII. The Appellant requested that the decision under appeal be set aside and that the patent be maintained on the basis of either the main or first auxiliary request filed with the letter of 26 January 1998, or second or third auxiliary request submitted during the oral proceedings. He further declared that these requests superseded all previous requests.

The Respondent requested that the appeal be dismissed.

Reasons for the Decision

1. The appeal is admissible.

2. *Main Request*

According to Claim 1 of the main request, the aluminium flakes have a coating of an inorganic oxo acid of phosphorus (Claim 1, part (ii)).

The application as originally filed does not disclose the exact composition of the coating. It is merely indicated that the coating contains "phosphorus supplied from the oxo acid" (Claim 1 and page 4, lines 5/6) or that it "is derived" from the oxo acid (Claim 16). However, in the Board's judgment, these terms denote a product of the oxo acid which actually differs from the oxo acid itself, because under the conditions mentioned in the examples, a chemical reaction between the oxo acid and the aluminium flakes is likely to take place. These examples, in particular the method for determining the phosphorus content on the aluminium flakes (page 10, lines 11-23), show that a phosphorus content remains after washing the samples with a mixture of water and isopropanol. This indicates that the phosphorus containing compound remaining on the flakes is not the oxo acid itself, because any unreacted oxo acid of phosphorus would be removed. Even if, as alleged by the Appellant, the chemical reaction involved was a chemisorption, so that the oxo acid did not wash off because it was chemisorbed on the surface of the aluminium flakes, a modification of the oxo acid must have taken place insofar as the produced coating is insoluble in the above mentioned solvents and, hence, objectively distinguishable from the unreacted or unmodified oxo acid of phosphorus.

The FT-IR spectra of ortho-phosphoric acid and of an aluminium foil treated with ortho-phosphoric acid filed by the Appellant, show some similarities, but in fact more differences in that there is only one comparable peak (wave number 1150 cm-1 or 1190 cm-1, respectively), whereas the main peak of ortho-phosphoric acid having a wave number of about 1010 cm-1 is totally missing in the FT-IR-spectrum of the treated aluminium foil. Therefore, the FT-IR spectra do not prove that any oxo acid remained unchanged in the coating. Moreover, even if the IR-spectra would have supported the Appellant's allegation that the coating consisted of phosphoric acid, any such information was not included in the disclosure of the application as originally filed and was, therefore, irrelevant for allowing the amendments under Article 123(2) EPC.

Consequently, the Board considers that the application as originally filed does not disclose that the oxo acid itself is present in the form of a coating. Contrary to Article 123(2) EPC, the subject-matter of Claims 1 and 15 of the main request, therefore, extends beyond the content of the application as originally filed.

3. *First auxiliary request*

3.1 The only feature objected to in respect of Article 123 EPC was the indication in Claims 1 and 15 that an inorganic oxo acid of phosphorus is admixed 'in an amount of from 500 to 10 000 ppm calculated as phosphorus based on the weight of the aluminium'.

3.1.1 The Respondent argued that the only origin for admixing the inorganic oxo acid was process Claim 17 as originally filed, according to which the oxo acid is admixed in an amount of 500 to 10 000 ppm based on the weight of the aluminium which was clearly in

contradiction to the amount of oxo acid to be added now claimed. Whilst it was admitted that this amount was insufficient to provide the phosphorus content in the composition required in original Claim 1, there was no contradiction between original Claims 1 and 17, because the oxo acid was not the only possible source of phosphorus in the composition. As mentioned on page 6, lines 11 to 14, of the application as filed and as shown in examples 10-12 and 15 and in comparative example 3, the composition may also contain an organic phosphate ester as component (iii) which binds to the surface of the aluminium flakes (see page 17 of the application as originally filed).

The Board agrees with the Respondent insofar as component (iii) represents a further possible source of phosphorus. The claims as originally filed and the present claims do not in fact exclude further sources of phosphorus. With respect to the specified amount of phosphorus, however, original Claim 1 clearly requires that it **is supplied** from an inorganic oxo acid of phosphorus. This implies that the oxo acid is added in an amount to provide the specified amount of phosphorus.

This fact is also clear from examples 1-9, 13, 14 and 16 which are all prepared with an inorganic oxo acid of phosphorus as the only source of phosphorus, hence in the absence of any organic phosphate ester, but contain more than 500 ppm of phosphorus based on the weight of the aluminium in a composition comprising components (i) to (vi). The phosphorus content indicated in table 1 is determined on a dry basis, ie without any solvents present and after washing with water/isopropanol and ether/acetone. It ranges, for the above examples from 530 to 9000 ppm, ie well distributed within the range of original Claim 1. It

follows therefrom that said amount of phosphorus is present on the aluminium flakes and that the oxo acid must have been added at least in an amount corresponding thereto. This is in line with the statement on page 4, lines 3-6 and 11-13, of the contested patent, where it is specified that phosphorus which is supplied from the inorganic oxo acid, is present in a thin and dense coating on the aluminium flakes and that this coating contains phosphorus in an amount ranging from 500 to 10 000 ppm based on the weight of the aluminium. Comparative example 2 shows that a phosphorus content of 430 ppm supplied by orthophosphoric acid is unsatisfactory. The fact that additional phosphorus, supplied from component (iii), may also be present, does not affect the unambiguous original teaching that the specified amount of phosphorus **is supplied** from the inorganic oxo acid, so that the amount of inorganic oxo acid added to the mixture should be calculated as phosphorus. The amount of inorganic oxo acid of phosphorus in original Claim 17 should therefore read as the amount calculated as phosphorus. If the Respondent's position were correct and the amount of orthophosphoric acid according to original Claim 17 could be 500 ppm of orthophosphoric acid taken as such, the amount of orthophosphoric acid calculated as phosphorus would be substantially lower. Such a lower amount would not only be contrary to the statement on page 4 of the original description that "if the phosphor content is less than 500 ppm the larger volume of hydrogen gas evolves" but would also make said comparative example an example according to the invention. Thus Claim 1 of the first auxiliary request fulfils the requirements of Article 123(2) EPC. Since the process as claimed in Claim 15 is identical to the process as defined in Claim 1, the same must apply to Claim 15 of the first auxiliary request.

3.1.2 For the reasons given above, it is held that the protection conferred by granted Claim 1 comprises the admixture of oxo acid in the amount as stated. The amended Claims 1 and 15, therefore, also meet the requirements of Article 123(3) EPC.

3.2 Concerning Article 100(b) EPC, the Respondent argued that the required amount of phosphorus on the aluminium flakes according to the description is contrary to the amount of oxo acid added according to present Claim 1. The product obtained according to the patent in suit comprised aluminium flakes having a coating containing phosphorus in an amount of 500 to 10 000 ppm based on the weight of the aluminium. Comparative example 3 showed that not all phosphorus present in the mixture was bound to the flakes so that a process for manufacturing such coated flakes required the addition of an excess of the phosphorus containing compound. Hence, product Claim 1 and process Claim 15 were inconsistent insofar as, contrary to what is claimed, the amount of oxo acid of phosphorus to be added to the composition and that contained in the product obtained must be different. This reasoning cannot, however, be accepted by the Board, because the embodiment of comparative example 3 is made without any inorganic oxo acid of phosphorus at all. Instead, the only source of phosphorus offered therein is an organic phosphate ester which must indeed be added in a large surplus in order to obtain a small amount of phosphorus bound to the flakes. From these findings it cannot, without further proof, be inferred that the oxo acid must also be added in excess in order to obtain a definite amount of phosphorus bound to the aluminium flakes. More important, however, is the fact that nothing in the patent in suit actually indicates that it is necessary to add the oxo acid in excess in order to obtain said amount on the flakes. The Board, therefore, accepts, as

submitted by the Appellant, that the oxo acid in essence completely reacts with or binds to the surface of the aluminium flakes.

The Respondent further objected that it was not possible for the skilled person to realise whether he was working within the forbidden area of the present claims, because he was unable to determine in the composition the amount of phosphorus which is solely derived from the oxo acid. This objection was, however, not substantiated by evidence. It is credible, as put forward by the Appellant, that there exist analytical methods which enable the skilled person to determine the origin of the phosphorus contained in the composition.

Hence, the Board has no doubt that the skilled person can prepare the claimed mixture and thus obtain the claimed product.

The requirements of Article 100(b) are therefore met.

3.3 The Respondent based his novelty objection on D1. This document discloses a metal pigment composition obtained by mixing a filter cake produced by milling the metal together with a lubricant, preferably in an organic solvent, adding an organic phosphoric ester in an amount of 0.1 to 20% wt. based on the weight of the metal flakes, and dispersing the product in a pasting medium including inter alia water or alcohol. The amount of pasting medium is from 5 to 50% by weight based on the weight of the metal flakes (see column 3, lines 53-59, column 4, lines 21-38, and column 4, line 67 to column 5, line 12). In examples 1 to 3 the metal is aluminium, the organic solvent is mineral spirit, and the pasting medium is water. For example stearyl acid phosphate is added in an amount of 3 parts

per 100 parts of a filter press cake consisting essentially of aluminium and containing stearyl amine as milling agent (see example 2, sample 18).

With reference to D4 (see column 2, lines 38-50, and column 3, lines 49-60), which discloses the possible formation of phosphate esters by reacting phosphoric acid with a mixture of alcohols, the Respondent has argued that compounds (ii) (phosphoric acid) and (iii) or (v) (alcohol) of the claimed composition would also form organic phosphoric esters, at least in equilibrium with the reactants. The addition of phosphoric acid and alcohol was, therefore, identical to the addition of phosphoric esters as disclosed in D1.

The Board cannot accept this argument, because the mixing conditions are quite different. According to D1 and D4 no aluminium is present during the formation of the organic phosphoric ester. In D1 the organic phosphoric ester is first formed and then mixed with the aluminium flakes and other ingredients (see examples). Nothing in D1 indicates that the aluminium flakes are contacted with a mixture containing phosphoric acid and alcohol in addition to the organic phosphoric ester. D4 does not even mention aluminium at all, but merely describes the formation of the ester and its use in complexed form as an additive in various technical fields (see also example 29 and column 13, lines 55-70). By contrast, in the present patent, aluminium and alcohol or another organic reactant are, as a matter of principle, in competition with each other in any reaction with phosphoric acid. It is, therefore, credible that the reaction between aluminium and phosphoric acid is preferred, so that no acid at all remains available for any reaction with alcohol. It is also credible that under the given reaction conditions, such reaction products do not simply

further react with compounds (iii) and/or (v) in a manner to provide the same product as is present in the prior art. The evidence provided by the Respondent is therefore insufficient to derive the claimed subject-matter directly and unambiguously from the disclosure of D1. Moreover, D1 does not disclose that water and alcohol are simultaneously present and that water is present in the amount as now claimed.

The other documents cited during the opposition and appeal proceedings, but not further relied on by the Respondent during the oral proceedings, do not provide a technical teaching suitable to challenge the novelty of the claimed subject-matter according to the first auxiliary request. Since this was not disputed by the Respondent during the oral proceedings, there is no need to give further details.

The subject-matter of the independent claims is, therefore, new.

3.4 The Board agrees with the parties that D1 represents the closest prior art. Like the patent in suit D1 is concerned with the problem of gas generation, dispersion stability and the coating appearance of water based aluminium pigment compositions (see in particular column 6, line 13 to column 8, line 8 and column 11, remarks to Table 1). The patent in suit makes reference to such prior art by mentioning conventional anti-corrosive coatings which are formed on aluminium flakes by adding organic phosphate ester (see specification, page 2, lines 13-15).

3.4.1 The technical problem mentioned in the patent in suit in view of such prior art consists in providing an aluminium pigment composition which is further improved with respect to the evolution of hydrogen gas in

aqueous medium, the colour tone and the prevention of aggregation during storage (see specification, page 2, lines 15-18 and page 3, lines 1-4).

According to the patent in suit it is proposed that this problem be solved by providing a composition as set out in Claim 1. This composition differs from that of D1 essentially in that it is obtainable by admixing in addition to or instead of an organic phosphoric ester an inorganic oxo acid of phosphorus in an amount of from 500 to 10 000 ppm calculated as phosphorus based on the weight of the aluminium, and water in an amount of only 0.05 to 10 parts by weight per 100 parts by weight of the aluminium together with a water-compatible alcohol.

Whilst the known compositions do not evolve gas in aqueous medium and do not change colour or form aggregates during storage under definite test conditions (eg gas evolution is measured after storage at a temperature of 50°C for 20 hours; see in D1, column 6, line 13 to column 8, line 8 and Tables 1-3), it is shown in comparative example 3 of the patent in suit (cf. in D1, sample 18 of example 2) that such compositions are not satisfactory under more severe test conditions such as storage at 50°C for one month (see specification page 5, line 43 to page 5, line 16). By contrast, examples 1-16 show that the claimed composition provides products which are superior with respect to all three aspects, ie gas evolution, colour difference and aggregation amount. The Board is therefore convinced that the above mentioned problem has actually been solved by the products according to the claimed composition.

3.4.2 D1 does not contain any information apt to encourage someone skilled in the art to modify the known composition in the claimed manner, in particular to add an inorganic oxo acid of phosphorus in addition to or instead of the organic phosphoric ester in order to obtain the above mentioned improvements.

3.4.3 D9 referred to by the Respondent and cited in the contested patent (see page 2, line 21) is the only document on file which mentions the contacting of the aluminium flakes with phosphoric acid. It discloses a process for making a pigment composition wherein aluminium metal is subjected to an attrition process for producing aluminium flakes, in the presence of a suitable amount of lubricant (eg stearic acid), water and phosphoric acid or a salt thereof, preferably an ammonium salt, in an amount of at least 0.7% based on the weight of the aluminium (see examples and column 1, line 67 to column 2, line 18). As is shown in the examples, the amount of lubricant is well within the claimed ranges, whereas the amount of water is considerably higher (see eg examples 1 and 5). Moreover, the composition of D9 does not include a water-compatible alcohol or a hydrocarbon oil.

It is said in D9 that the pigment composition is relatively stable and does not undergo substantial deterioration over long periods when used as a paint (see column 1, lines 62-66 and column 3, lines 16-26). According to column 3, lines 26-35, however, the pigments in some instances steadily give off small volumes of gas for extended periods after manufacture and thereafter may absorb gas from the atmosphere. Whilst it is said that these phenomena have not hampered commercial use or resulted in any gross degradation of the pigment quality, someone skilled in the art would still realise that the pigments of D9

are, at least to some extent, subject to instability and degradation. He would, therefore, not consider that the teaching of D9 was suitable to provide a solution to the above problem.

Even if, for some reason, the skilled man would nevertheless have expected that benefits could be attained, any combination of D1 and D9 would not result in the subject-matter as presently claimed, because the amounts of water recommended in both D9 and D1 are considerably higher. Moreover, even if D1 already mentions the addition of alcohol instead of water, there was no incentive for someone skilled in the art to admix it in addition to water in an amount as defined in present Claim 1.

4. The other documents cited during the appeal and opposition proceedings do not contain suitable information which could lead to the claimed subject-matter. Since the Respondent did not rely on these documents in the inventive step argumentation against present Claims 1 and 15, it is not necessary to give reasons for this finding.

It follows, therefore, from the above that the aluminium pigment composition of Claim 1 of the first auxiliary request as well as its process for preparation according to Claim 15 involve an inventive step (Article 56 EPC) and fulfil the requirements of Article 52(1) EPC.

5. This conclusion also extends to dependent Claims 2-14 which relate to particular embodiments of the subject-matter of Claim 1. Since the claims of the first auxiliary request are allowable, it is not necessary to consider the Appellant's further (second and third) auxiliary requests.

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 with the order to maintain a patent with the following claims and a description to be adapted:

   - Claims 1 to 15 (first auxiliary request).

The Registrar:

A. Townend

The Chairman:

G. Wassenaar