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D E C I S I O N
of 14 July 2003

Case Number: T 1101/02 - 3.2.7

Application Number: 96942834.1

Publication Number: 0866886

IPC: C23C 22/12

Language of the proceedings: EN

Title of invention:

Zinc phosphate conversion coating compositions and process

Applicant:

PPG Industries Ohio, Inc.

Opponent:

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Headword:

-

Relevant legal provisions:

EPC Art. 56

Keyword:

"Inventive step - (yes)"

Decisions cited:

-

Catchword:

-



Case Number: T 1101/02 - 3.2.7

D E C I S I O N
of the Technical Board of Appeal 3.2.7
of 14 July 2003

Appellant:

PPG Industries Ohio, Inc.
3800 West 143rd Street
Cleveland
OH 44111 (US)

Representative:

Sternagel, Fleischer, Godemeyer & Partner
Patentanwälte
Braunsberger Feld 29
D-51429 Bergisch Gladbach (DE)

Decision under appeal:

Decision of the Examining Division of the
European Patent Office posted 20 March 2002
refusing European application No. 96942834.1
pursuant to Article 97(1) EPC.

Composition of the Board:

Chairman: A. Burkhart
Members: H. E. Hahn
C. Holtz

Summary of Facts and Submissions

- I. The appellant (applicant) lodged an appeal against the decision of the Examining Division to refuse the European patent application No. 96 942 834.1.
- II. The Examining Division held that the subject-matter of the method of claim 1 lacked an inventive step.
- III. The following documents of the available prior art are considered to be most relevant for the appeal:
- D1: US-A-4 865 653
- D3: US-A-2 487 137
- D4: Kirk-Othmer, Encyclopedia of Chemical Technology, second edition, vol. 11, (1996), pages 496-497
- D5: Ullmanns Encyklopädie der technischen Chemie, vol. 13 (1977), page 169
- IV. The appellant requested that the decision under appeal be set aside and a patent be granted on the basis of claims 1 to 22 filed on 2 July 2003 with letter of 30 June 2003.
- V. The independent claims 1, 14 and 21 under consideration read as follows:
- "1. A process for forming a zinc phosphate coating having a coating weight of at least 1612 mg/m² (150 mg/ft²) on an aluminum substrate, comprising contacting said aluminum substrate with an aqueous

acidic zinc phosphate conversion coating bath
containing:

- (a) from 0.4 to 2.5 g/l zinc ion;
- (b) from 5 to 26 g/l phosphate ion;
- (c) from 0.5 to 1.0 g/l of fluoride ion measured as F^- ;
- (d) from 4 to 400 mg/l ferrous ion; and
- (e) from 0.01 to 2 g/l ammonium ion,

wherein the source of fluoride ion is selected from the group consisting of water-soluble bifluorides, mixtures of bifluorides, and combinations of bifluorides with monofluoride and/or complex fluoride ions."

"14. An aqueous zinc phosphate conversion coating concentrate which comprises:

- (a) from 10 to 60 g/l zinc ion;
- (b) from 125 to 500 g/l phosphate ion;
- (c) from 2 to 40 g/l fluoride ion measured as F^- ;
- (d) from 0.1 to 10 g/l ferrous ion; and
- (e) from 0.2 to 50 g/l ammonium ion,

wherein the source of fluoride ion is selected from the group consisting of water-soluble bifluorides, mixtures of bifluorides, and combinations of bifluorides with monofluoride and/or complex fluoride ions."

"21. Use of the concentrate of any of claims 14 to 20 to form an aqueous acidic zinc phosphate conversion coating bath by dilution with water in a weight ratio (concentrate : water) 1:10 to 1:100."

Reasons for the Decision

1. *Original disclosure - Article 123(2) EPC*

The claims 1 to 22 of the sole request are based on the original claims 1 to 11, 13 to 19 and 21 to 22 in combination with description page 3, line 8 to 26; page 5, lines 20 to 22; page 7, line 13 to page 8, line 1; page 9, lines 8 to 22; page 12, table A; and page 14, lines 27 to 28 of the application as originally filed.

Hence the requirements of Article 123(2) EPC are met for the claims 1 to 22.

2. *Novelty*

The Board concurs with the view of the Examining Division that the subject-matter of the claims 1, 14 and 21 is novel with respect to the available documents, particularly with respect to documents D1 and D3.

3. *Inventive step*

3.1 Closest prior art

The closest prior art is represented by document D1. Document D1 discloses a zinc phosphate coating process for ferrous substrates but mentions also aluminium substrates (cf. column 2, lines 22 to 25; column 6, lines 13 to 19). With regard to the zinc level document D1 allows levels up to 2.0 g/l, but for safety reasons in controlling the process zinc levels of 0.45 g/l to

1.1 g/l are preferred (cf. column 4, lines 16 to 22). Sufficient quantities of hydroxylamine as accelerator are required to alter the morphology of the coating from platelet to columnar and/or nodular over a broad range of zinc concentrations (cf. column 3, lines 62 to 68; column 4, lines 29 to 63); preferably 0.5 to 50 g/l of the hydroxylamine (sulfate) salt are used. The bath comprises optionally 0.01 to 0.5 g/l ferrous ions (cf. column 5, lines 5 to 13) and optionally simple or complex fluoride ions (cf. claim 13).

- 3.2 The approach of the Examining Division concerning a conversion of hydroxylamine into ammonium ions of the phosphatizing bath according to document D1 is not supported by the available state of the art.

The Board concurs with the appellant's view that the hydroxylamine is stable in acidic solution which is confirmed by the documents D4 and D5.

Even if it would be assumed that the hydroxylamine during its use as an accelerator in the phosphatizing bath can be, or will be, consumed (i.e. it could be oxidized), the extent thereof is unknown. Proof or evidence that 100% of the hydroxylamine will be converted into ammonium is missing. Any concentration of such ammonium ions produced by the reaction of the phosphatizing bath and the metallic substrate would be dependent upon the amount of the substrate material phosphatized which is also not known from document D1. Document D1 is absolutely silent in this respect. Thus, document D1 cannot support any calculation of an ammonium ion concentration based on the hydroxylamine sulfate concentrations.

Additionally, the statement in the originally filed present application concerning the possible use of hydroxylamine salts as source of ammonium ions does not imply that the hydroxylamine is converted in the acidic phosphatizing bath or concentrate. It could be converted at different pH conditions in a separate replenisher solution. The said passage in the present application is absolutely silent with respect to the exact conditions therefor. In any case the said passage in the present application does not belong to the relevant prior art in the meaning of Article 54(2) EPC.

- 3.3 Furthermore, the Board concurs with the appellant that only example V of document D1 concerns the treatment of an aluminium substrate but it results in a platelet morphology which does not solve the problem underlying the present application. The bath of example V contains 0.5 g/l Ni^{2+} , 0.47 g/l Zn^{2+} , 13.3 g/l PO_4^{3-} , 1.4 g/l F^- and 2.3 g/l hydroxylamine sulfate and produced on cold rolled steel a coating containing mostly nodular and a few columnar crystals (cf. column 7, example V).

Hence, the bath did not comprise any deliberate addition of ferrous ions and it is also not clear whether or not the aluminium substrate was treated in the same bath as the mentioned cold rolled steel. If it was not the same bath then the bath did not contain any ferrous ions at all since aluminium substrates when phosphatized cannot produce iron ions through the pickling action of the bath. Even if it would have been the same bath after the treatment of the cold rolled steel substrate the skilled person does not know the iron content thereof, let alone the content of any

ferrous ions. Although the optional addition of ferrous ions according to document D1 is stated to be advantageous for increasing the zinc level range which will produce the desired morphology (cf. column 5, lines 5 to 20) there exists only one single example (i.e. example IV) wherein ferrous ions were added to the bath. According to examples I, II, III and V the desired morphologies were obtained on steel substrates without any ferrous ion addition but with additions of fluoride ion (cf. examples II, IV and V) and also without additions of fluoride ions (cf. examples I and III). Hence the skilled person would conclude that ferrous ions are not essential and the morphology is only caused by the amount of zinc ions and hydroxyl amine sulfate.

3.4 Therefore, the difference between the subject-matter of claim 1 and the disclosure of D1 is that the phosphatizing solution according to claim 1 comprises as essential components 0.5 to 1.0 g/l of fluoride ion measured as F^- selected from the group consisting of water-soluble bifluorides, mixtures of bifluorides, and combinations of bifluorides with monofluoride and/or complex fluoride ions; and from 4 to 400 mg/l ferrous ions; and from 0.01 to 2 g/l ammonium ions.

3.5 Problem to be solved

The problem to be solved is considered to be the provision of a zinc phosphate coating composition and process for forming a zinc phosphate coating having an appropriate coating weight and more complete coating coverage on aluminium substrates (see page 2, lines 1 to 4 of the description).

3.6 Solution to the problem

The problem is solved by the phosphatizing method as defined in claim 1, the concentrate as defined in claim 14 and the use of the concentrate as defined in claim 21. The use of the claimed method and the claimed concentrates results in a columnar or nodular morphology of the coating and thus a complete coating coverage on an aluminium substrate (compare the examples of the present application).

3.7 The Board considers that the subject-matter of the independent claims 1, 14 and 21 is not obvious to the person skilled in the art for the following reasons:

The Board concurs with the view of the appellant that documents D1 and D3 concern totally different phosphatizing technologies (they have different zinc levels, use different accelerators, have different ferrous ion contents, use different total acid and free acid ratios, etc.) so that the skilled person would not combine the teachings of these two documents. And even if he were to do so, which is most unlikely to be the case, he would neither derive the process of claim 1 nor the concentrate of claim 14.

Furthermore, the skilled person has no reason to deliberately add ammonium ions to the bath of document D1 at all, let alone to obtain a specific concentration range, since the skilled person does not know that the addition of ammonium ions would influence the coating morphology. Both documents D1 and D3 are totally silent in this respect.

Additionally, the skilled person has no conclusive reason to amend the fluoride ion concentration of example V of document D1 since he cannot expect any specific effect, let alone when taking account of the teaching of document D3. As already mentioned the skilled person would not combine the teachings of documents D1 and D3 due to the totally different technologies. But even if he were to do so the fluoride concentration of 1.4 g/l of said example V is well within the fluorine range of 0.15 to 8 g/l of document D3 so that the skilled person would have no reason to amend the fluoride concentration. Said fluoride concentration of 1.4 g/l is, however, outside the range of 0.5 to 1.0 g/l according to claim 1 of the sole request.

Thus when starting from example V of document D1 the skilled person would have to add ferrous ions in a specific amount, to add ammonium ions in a specific amount, to reduce the fluoride ion concentration from 1.4 g/l to 1.0 g/l and to replace the simple or complex fluoride ions according to document D1 by water soluble bifluorides or mixtures thereof with monofluoride and/or complex fluorides. However, the skilled person has no reason or incentive to do so, particularly when taking account of the technical problem to be solved (compare point 3.5 above) and taking account of the teachings of documents D1 and D3 and the common general knowledge as proven by the documents D4 and D5.

Hence the subject-matter of claim 1 is not derivable from documents D1 and D3, neither when taken alone nor in combination. Similarly, the subject-matter of

claims 14 and 21 is not derivable therefrom either, let alone in an obvious manner.

3.8 The subject-matter of the independent claims 1, 14 and 21 thus involves an inventive step within the meaning of Article 56 EPC.

3.9 The same applies to the subject-matter of the dependent claims 2 to 13, 15 to 20 and 22 which define further preferred embodiments of the process for forming a zinc phosphate coating, the aqueous zinc phosphate conversion coating concentrate and the use thereof according to the claims 1, 14 and 21, respectively.

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 grant a patent in the following version:

Claims: 1 to 22 as filed on 2 July 2003 with letter of 30 June 2003

Description: Pages: 1, 1A, 2 to 9, and 11 to 22 as filed on 4 July 2002 with letter of 2 July 2002
Page: 10 as filed on 17 June 2003 with letter of 16 June 2003

The Registrar:

The Chairman:

D. Spigarelli

A. Burkhardt