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D E C I S I O N
of 9 February 2004

Case Number: T 0819/99 - 3.2.7

Application Number: 92916122.2

Publication Number: 0596947

IPC: C23C 22/36

Language of the proceedings: EN

Title of invention:

Zinc phosphate conversion coating composition and process

Patentee:

HENKEL CORPORATION

Opponent:

Chemische Werke Kluthe GmbH & Co.

Headword:

-

Relevant legal provisions:

EPC Art. 56

Keyword:

"Inventive step (yes)"

Decisions cited:

-

Catchword:

-



Case Number: T 0819/99 - 3.2.7

D E C I S I O N
of the Technical Board of Appeal 3.2.7
of 9 February 2004

Appellant: Chemische Werke Kluthe GmbH & Co.
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Decision under appeal: Decision of the Opposition Division of the
European Patent Office posted 1 July 1999
rejecting the opposition filed against European
patent No. 0596947 pursuant to Article 102(2)
EPC.

Composition of the Board:

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

Summary of Facts and Submissions

I. The appellant (opponent) filed an appeal against the decision of the opposition division rejecting the opposition against European patent No. 0 596 947.

Opposition was filed against the patent as a whole based on the ground of opposition according to Article 100(a) EPC (lack of inventive step).

The opposition division rejected the opposition in the light of the following prior art documents:

E1: EP-B- 0 106 459

E2: Machu W. "Die Phosphatierung", 1950, Verlag Chemie Weinheim/Bgstr., pp. 157-158

E3: EP-A-0 315 059.

II. Requests

(i) The appellant requested that the decision under appeal be set aside and that the patent be revoked. A request for oral proceedings was withdrawn.

(ii) The respondent (patentee) requested that the appeal be dismissed (main request), that the decision under appeal be set aside and the patent be maintained with claims 1 to 9 or 1 to 8 filed with letter dated 21 February 2000 (first and second auxiliary request). As a further auxiliary request oral proceedings were requested.

(iii) Claim 1 of the patent in suit reads as follows:

" A concentrate composition for use in formulating an aqueous coating solution for phosphatizing metal substrates, said concentrate composition being an aqueous solution and consisting essentially of water, acid, hydroxylamine sulfate, zinc ions, nickel ions, manganese ions and phosphate ions, and optionally also one of both of fluoride (including complex fluoride) ions and nitrate ions, in amounts such that (i) the weight ratio of zinc ions to phosphate ions is from 1:10 - 25, (ii) the weight ratio of zinc ions to the sum of manganese and nickel ions is from 1:0.5 - 1.5, (iii) the weight ratio of manganese ions to nickel ions is from 1:0.5 - 1.5, and (iv) dilution of 48 g of the concentrate composition with water to form 1 liter of total aqueous coating solution will produce an aqueous coating solution having a total acidity of about 15 to 25 points and a free acidity of about 0.5 to 1.0 points that being free of Fe(II) and consists essentially of

- (A) 0.5 to 2.5% by weight of phosphate ions;
- (B) 0.05 to 0.2% by weight of zinc ions;
- (C) 0.02 to 0.15% by weight of nickel ions;
- (D) 0.02 to 0.15% by weight of manganese ions; and
- (E) 0.1 to 0.25% by weight of hydroxylamine sulfate; and, optionally, one or both of:
 - (F) up to 0.2% by weight of nitrate ions; and
 - (G) up to 0.15% of total fluoride ions."

(iv) Claim 6 of the patent in suit reads as follows:

" An aqueous coating solution for phosphatizing metal substrates, said aqueous coating solution consisting essentially of water, acid, hydroxylamine sulfate, zinc ions, nickel ions, manganese ions and phosphate ions, and optionally also one of both of fluoride (including complex fluoride) ions and nitrate ions, in amounts such that (i) the weight ratio of zinc ions to phosphate ions is from 1:10 - 25, (ii) the weight ratio of zinc ions to the sum of manganese and nickel ions is from 1:0.5 - 1.5, (iii) the weight ratio of manganese ions to nickel ions is from 1:(0.5 - 1.5, and (iv) the aqueous coating solution having a total acidity of about 15 to 25 points and a free acidity of about 0.5 to 1.5 points being free of Fe(II) and consists essentially of

- (A) 0.5 to 2.5% by weight of phosphate ions;
- (B) 0.05 to 0.2% by weight of zinc ions;
- (C) 0.02 to 0.15% by weight of nickel ions;
- (D) 0.02 to 0.15% by weight of manganese ions; and
- (E) 0.1 to 0.25% by weight of hydroxylamine sulfate; and, optionally, one or both of:
 - (F) up to 0.2% by weight of nitrate ions; and
 - (G) up to 0.15% of total fluoride ions."

The subject-matter of claim 6 thus differs from the one according to claim 1 in that, without reference to a concentrate composition for use in formulating an aqueous coating solution, it directly defines an aqueous coating solution.

(v) Claim 12 of the patent in suit reads as follows:

"A process for phosphatizing a metal surface, said process comprising treating the metal surface with an aqueous coating solution as defined in any one of claims 6 to 11."

III. The arguments of the appellant may be summarised as follows:

- (i) Document E1 constitutes the closest prior art. According to this document an aqueous coating solution for phosphatising metal substrates can comprise nitrite ion or nitrite ion and chlorate ion as phosphating accelerator.
- (ii) Concerning concentrate compositions within document E1 it is stated that, if a concentrate composition is used to formulate the aqueous solution, it is not advisable to add any phosphating accelerator to the concentrate since the accelerators tend to decompose and cause other problems. Thus a clear recommendation is given to use accelerators other than nitrite in case a concentrate composition is to be used.
- (iii) The person skilled in the art looking for an accelerator other than nitrite for a concentrate composition would have been led by document E2 to use hydroxylamine, since according to this document hydroxylamine is the most effective one of the accelerators referred to in this document. The amount of hydroxylamine sulfate comprised in

the concentrate composition according to claim 1 of the patent in suit is obvious in view of the optimal dose referred to in document E2. Thus the subject-matter of claim 1 is obvious in view of documents E1 and E2.

- (iv) Document E3 comprises in addition to the components of the coating solution according to document E1 the use of hydroxylamine as an accelerator. According to this document the quantity in which hydroxylamine is present influences the type of the resulting coating in that it is stated that, when present in sufficient quantities, the resulting coating is altered from platelet to columnar and/or nodular structure. Thus document E3 clearly suggests that the concentration of hydroxylamine should be set according to the result to be obtained.

- (v) Document E3 does not refer to a concentrate composition for use in formulating an aqueous coating solution but to a possible use of replenishers containing a hydroxylamine agent. The combination of compatible components in a concentrate composition comes within the technical knowledge, in particular considering claim 39 of document E3, according to which a concentrate composition is possible, which comprises the main components referred to in claim 1 of the patent in suit in the quantities defined in the patent in suit.

- (vi) According to the only embodiment given in document E3 the aqueous solution from which the

phosphatising is started contains no hydroxylamine, which is added thereafter, and before test panels were processed the bath was aged. In the interpretation of the opposition division, that the addition of hydroxylamine is to be avoided at the beginning if such an aging step is not to be accounted for, it has been overlooked that the aging, which consists in the panels treated first being discarded for testing purposes, is merely a usual measure for ensuring that the bath is in equilibrium and test results are meaningful.

(vii) "Example V" of document E3 shows that on aluminum and galvanized surfaces, and thus surfaces which according to the patent in suit are preferred ones, corresponding to the patent in suit, coatings with platelet morphology can be obtained. Thus, contrary to the opinion expressed in the decision under appeal, document E3 indicates that deviating from these conditions, which according to this document lead to the predominantly nodular and/or columnar crystalline coating aimed for, leads to the platelet crystalline morphology aimed for according to the patent in suit.

(viii) Considering Table I of document E3 it needs to be recognised that the incomplete coating for the platelet morphology is easily recognised as resulting from the short reaction time. It is obvious that an increase of the reaction time leads to a complete coating in these cases also, such that this table cannot hinder the person

skilled in the art from using hydroxylamine to produce coatings with platelet morphology as indicated in document E1.

- (ix) Considering the argument of the decision under appeal according to which the components and their concentrations are decisive for the success of coating solutions of the kind concerned, it needs to be taken into account that within the patent in suit a wide variation of the concentrations is permitted. Considering such broad claims with little experimental proof it is obvious that from the teaching of document E3 it can be expected, that alteration of the accelerator into hydroxylamine does not necessarily change the platelet morphology, leads to acceptable coatings and allows for the only disadvantage of the approach according to this document, namely the use of nitrite as accelerator, to be eliminated.

IV. The arguments of the respondent may be summarised as follows:

- (i) Document E1 constituting the closest prior art corresponds to document US-A-4838957, which is acknowledged in the patent in suit. Starting from the coating solution of document E1, the problem underlying the patent in suit is to reduce environmental impact and safety concerns associated with nitrite. At the same time, as indicated in the patent in suit, the advantages of the use of manganese and nickel should be retained without changing the platelet- or

needle-like crystalline morphology of the resulting coating.

- (ii) The statement in document E1 that it is not advisable to add any phosphating accelerator to the concentrate, since the accelerators tend to decompose and cause other problems, is clear and suggests that no accelerator should be used within concentrates. This statement thus cannot be understood as suggesting the use of accelerators other than the ones disclosed in this document in connection with coating solutions.
- (iii) Document E2 does not suggest selecting hydroxylamine as accelerator from the variety of accelerators disclosed in this document in order to replace nitrite as an accelerator in a phosphating solution such as the one according to document E1.
- (iv) A combined consideration of documents E1 and E2 must take into account that in document E1 it is stated that, in case phosphating concentrates are used, no accelerator should be employed at all and furthermore that a concentrate is not referred to in document E2.
- (v) Document E3 corresponds to US-A-4865653 acknowledged in the patent in suit. According to this document, hydroxylamine sulfate is employed to alter the morphology of a coating into a predominantly columnar and/or nodular one. Document E3 thus would not have been considered

in an attempt to solve the problem underlying the patent in suit, which concerns the provision of an accelerator under the condition that the coating retains its platelet- or needle-like crystalline morphology.

Reasons for the decision

1. It remains undisputed that, as correctly stated in the decision under appeal, the claims are novel (Article 54 EPC).
2. *Inventive step*
 - 2.1 Closest prior art

Document E1 is undisputedly considered as closest prior art. It discloses aqueous coating solutions for phosphatising metal substrates, the components and the concentrations given therefor partially overlapping with the ranges defined in the claims of the patent in suit. The values for the weights of the individual components given for the concentrate relate to g/l, whereas corresponding values according to the patent in suit relate to weight %, such that contrary to an argument of the appellant these values cannot be compared directly.

Concerning the use of an accelerator, document E1 discloses with respect to aqueous coating solutions the use of nitrite ions with or without being combined with chlorite ion, nitrate ion, or hydrogen peroxide (claims 1, 9). With respect to concentrate compositions

this document discloses that "it is not advisable to add any phosphatizing accelerator to the concentrate, since the accelerators tend to decompose and cause other problems" (page 5, lines 35, 36).

The subject-matter of claim 1 thus differs from the concentrate composition according to document E1 in that an accelerator is provided for the concentrate composition defined by claim 1 and that as accelerator hydroxylamine sulfate is used.

Concerning the coating obtained according to document E1 reference is made to an acidic aqueous phosphate solution and a process which can give an excellent phosphate film on a metal (cf. e.g. page 2, lines 58 to 63); the morphology of the film is not defined.

2.2 Problem underlying the invention

With respect to document E1 the technical problem underlying the patent in suit consists in providing a concentrate composition, an aqueous solution and a process for phosphatising metal substrates, retaining the advantages of the use of manganese and nickel, such that polycrystalline coatings can be obtained without undesirable accelerators, such as nitrite, being necessary and without changing the platelet- or needle-like crystalline morphology (page 2, lines 50 to 53).

2.3 Solution

According to claim 1 this problem is solved with a concentrate composition for use in formulating an aqueous coating solution for phosphatising metal

substrates as defined in this claim, the concentrate composition comprising as an accelerator 0.1 to 0.25% by weight of hydroxylamine sulfate.

2.4 Obviousness

Due to the statement in document E1 (cf. section 2.1 above) that it is not advisable to add any phosphatising accelerator to the concentrate, since accelerators tend to decompose and cause other problems (page 5, lines 33 to 36), and due to the fact that within document E1 as accelerators for aqueous solutions for phosphatising metal substrates nitrite is referred to (cf. claim 1; page 3, lines 60 to 63), document E1 does not give an indication leading to the concentrate composition according to claim 1.

The argument of the appellant that the above-mentioned statement in document E1, according to which a concentrate composition should not comprise an accelerator, relates only to the use of nitrite ions as phosphatising accelerator, thus giving a clear indication for the use of a different type of accelerator to the one mentioned, cannot be accepted. The statement concerned clearly advises "not to add any phosphatizing accelerator to the concentrate", without any reference to a particular type of accelerator. Consequently this statement cannot be understood as being limited to a particular type of accelerator, which in document E1 is not referred to in context with a concentrate composition, but instead in context with aqueous coating solutions.

Thus, as indicated in the decision under appeal, according to document E1 it is not advisable to add any phosphatising accelerator to the concentrate. Thus, starting from this document and in an attempt to solve the problem underlying the patent in suit with a concentrate composition, consideration of documents E2 and E3, according to which hydroxylamine can be employed as an accelerator, cannot lead to the concentrate composition according to claim 1.

For the sake of completeness it should be indicated that this holds all the more true since, as referred to in the decision under appeal, neither document E2 nor E3 relates to a concentrate composition.

Document E2 is a chapter of a technical textbook concerning organic compositions as phosphatising accelerators, within which a variety of organic accelerators, including hydroxylamine, are referred to. These organic accelerators can be used in combination with common accelerators such as nitrate, nitrites, other oxydizing agents and copper, etc. (cf. page 157, paragraph 2).

Document E3 discloses an aqueous solution used in a metal finishing process (cf. claim 1) or in a replenishing composition (cf. claim 39). As correctly pointed out in the decision under appeal, a replenishing composition normally does not comprise all of the particular components of a coating solution. Irrespective of whether or not a replenishing composition comprises the main components of the corresponding aqueous solution, due to its different composition and kind of use, it cannot be considered as

being a concentrate composition for use in formulating an aqueous coating solution.

Finally, according to document E3, hydroxylamine is used to cause the aqueous solution to produce a coating, the morphology being predominantly columnar and/or nodular as opposed to platelet crystalline morphology (cf. e.g. claim 1; page 4, lines 26 to 28), which is desired according to the patent in suit.

3. *Claim 6*

3.1 The subject-matter of claim 6 differs from the one according to claim 1 in that it directly defines an aqueous coating solution, which essentially corresponds to the total aqueous solution of claim 1, and not a concentrate composition for use in formulating an aqueous coating solution.

3.2 It is uncontested that document E1 constitutes the closest prior art with respect to the subject-matter of claim 1.

3.3 With respect to the aqueous coating solution according to document E1 the problem indicated in section 2.2. above still applies, since the problem underlying the patent in suit (page 2, line 50, to page 3, line 5) also relates to an aqueous solution for phosphatising metal substrates, irrespective of whether or not this aqueous solution is formulated using a concentrate composition.

3.4 According to claim 6, this problem is solved with an aqueous coating solution for phosphatising metal

substrates as defined in this claim, the concentrate composition comprising as an accelerator 0.1 to 0.25% by weight of hydroxylamine sulfate.

- 3.5 Since in document E1 nitrite ions are referred to as the phosphating accelerator (cf. claim 1), and since no indication is given concerning replacement of this accelerator, this document cannot be considered as leading in the direction of the solution according to claim 6. Since hydroxylamine sulfate is not referred to in document E1 at all, an indication could have been given even less with respect to a replacement of the accelerator used according to document E1 by hydroxylamine sulfate.

Document E2 is a chapter of a technical textbook which is concerned with organic compositions such as phosphating accelerators. Within this chapter a variety of organic accelerators, including hydroxylamine, are referred to (cf. page 157, paragraphs 1 and 2). It is indicated that these organic compositions can be used together with common accelerators such as nitrate, nitrites, other oxydizing agents and copper, etc. (cf. page 157, paragraph 2).

While document E2 is concerned with suitable accelerators, the composition of an aqueous coating solution, within which such an accelerator is to be used, is not further defined.

Since, according to document E1 and document E2, nitrite can be employed as an accelerator, a combined consideration of these documents does not suggest replacing nitrite as an accelerator in the aqueous

coating solution according to document E1, and even less replacing nitrite as an accelerator by hydroxylamine sulfate.

Thus, as correctly indicated in the decision under appeal, consideration of both documents E1 and E2 in combination cannot lead to the use of hydroxylamine sulfate as the accelerator in an aqueous coating solution having the composition defined by claim 6.

Document E3 refers to distinct types of crystal morphologies, namely platelet, columnar or nodular ones, wherein columnar and/or nodular coatings are considered to be the generally preferred ones (page 2, lines 27 to 35).

According to document E3 a problem encountered by employing known coating baths and coating processes with zinc concentrations at relatively low levels consists in the form of the coating rapidly changing to the less desirable platelet morphology, if the zinc level increases in the bath, e.g. due to dissolution of zinc from galvanized parts or due to process control problems (page 2, lines 36 to 2).

To avoid this disadvantage according to document E3 it has been found that where predominantly columnar and/or nodular coatings are desired, the inclusion of a hydroxylamine accelerator in a zinc phosphate solution enhances the process and broadens the range of zinc content at which the desired coating is obtained (claim 1; page 3, lines 48 to 52; page 4, lines 26 to 28).

Although, as recognised in the decision under appeal, examples of document E3 relate to coatings having a platelet morphology (cf. Table I of page 6, lines 4 to 20; Example V of page 7, lines 4 to 10), these cannot be considered, as indicated in the decision under appeal, as leading to the provision of hydroxylamine as accelerator in the aqueous coating solution according to claim 1, the reason being that the compositions of the aqueous coating solutions for which a platelet morphology is obtained are not completely defined. To the extent that they are defined they do not lead to the subject-matter of claim 6 being derivable in an obvious manner from the combination of documents E1 and E3, as indicated in the decision under appeal.

It is evident that the above reasoning holds true irrespective of whether or not it is considered obvious in view of the results in Table I, according to which the coating is incomplete in the case of platelet morphology being obtained, that a complete coating with platelet morphology can be obtained with the aqueous coating solution according to document E1 by increasing the reaction time and the speed of reaction.

The above reasoning also holds true irrespective of whether or not it is considered obvious that, as alleged by the appellant, hydroxylamine, which according to Example 1 of document E3 is added after the bath was aged (page 5, lines 36 to 53), can be added before such aging takes place.

In the context of the disclosure of the aqueous coating solution according to document E3, for the sake of completeness it should be pointed out that, contrary to

an allegation by the appellant, the limits of the ranges of the individual components defined for the aqueous coating solution in claim 6 and the manner in which this claim is supported by the description are of no concern with respect to the assessment of inventive step, since the width and the limits of these ranges do not result in particular ones of the compositions of the aqueous coating solution defined by claim 6 being made obvious by the available prior art.

Consequently, as stated in the decision under appeal, combined consideration of documents E1 and E3 cannot be considered as leading to the subject-matter of claim 6.

4. For reasons corresponding to the ones given with respect to claim 6, the subject-matter of claim 12, being directed to a process for phosphatising a metal surface, the process comprising treatment of the metal surface with an aqueous coating solution as defined in any one of claims 6 to 11, is not suggested by a combined consideration of documents E1 to E3.

As indicated in the decision under appeal, the subject-matters of independent claims 1, 6 and 12, and with them of dependent claims 2 to 5, 7 to 11 and 13 to 15 of the patent in suit thus involve an inventive step (Article 56 EPC).

Order

For these reasons it is decided that:

The appeal is dismissed.

The Registrar:

The Chairman:

D. Spigarelli

A. Burkhart