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**Datasheet for the decision
of 27 May 2010**

Case Number: T 0886/07 - 3.2.07

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

Patent Proprietor:

PPG Industries Ohio, Inc.

Opponents:

Opponent I: Henkel AG & Co. KGaA

Opponent II: CHEMETALL GmbH

Headword:

-

Relevant legal provisions:

EPC Art. 83, 56

Relevant legal provisions (EPC 1973):

-

Keyword:

"Sufficiency of disclosure (yes)"

"Inventive step (yes)"

Decisions cited:

-

Catchword:

-



Case Number: T 0886/07 - 3.2.07

DECISION
of the Technical Board of Appeal 3.2.07
of 27 May 2010

Appellant: CHEMETALL GmbH
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Representative: -

Respondent: PPG Industries Ohio, Inc.
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Other Party: Henkel AG & Co. KGaA
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Representative: Endres, Helmut
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Decision under appeal: **Decision of the Opposition Division of the
European Patent Office posted 28 March 2007
rejecting the opposition filed against European
patent No. 0866886 pursuant to Article 102(2)
EPC.**

Composition of the Board:

Chairman: P. O'Reilly
Members: H. Hahn
E. Dufrasne

Summary of Facts and Submissions

- I. The appellant (opponent II) lodged an appeal against the decision of the Opposition Division to reject the two oppositions against the European patent EP-B-0 866 886.

The Opposition Division did not admit a new ground of opposition raised under Article 123(2) EPC as *prima facie* not relevant but admitted the late filed documents D13 (DE-A-36 31 759) and D14 (DE-A-28 18 426) into the proceedings as *prima facie* highly pertinent in accordance with Article 114(2) EPC. It further held that the patent in suit sufficiently discloses the invention so that the person skilled in the art is enabled to carry out the subject-matter of claims 1, 14 and 18 as granted, and thus that the patent meets the requirements of Article 100(b) EPC. The Opposition Division further held that the priority was validly claimed so that the intermediate document D10 (WO-A-96/16204) is only relevant for novelty while D9 (WO-A-97/30192) is not relevant at all. The Opposition Division held that the subject-matter of claims 1 and 14 is novel over the prior art, particularly with respect to documents D1 (EP-A-0 315 059), D7 (US-A-5 328 526), D8 (EP-A-0 659 906) and D13 (with respect to claim 1) and documents D5 (US-A-5 238 506), D6 (US-A-5 399 208), D9, D10, D11 (DE-A-39 27 613) and D12 (US-A-4 330 345) (with respect to claims 14 to 22). It further held that the subject-matter of claim 1 as granted involved an inventive step in view of D13 and D1 (or D8 and D1, or D5 and D1) while the subject-matter of claim 14 involved an inventive step in view of D5 and D14, or D12 and D14.

- II. The appellant requested in the written proceedings that the decision under appeal be set aside and that the patent be revoked. In case the Board should intend to confirm the impugned decision oral proceedings were requested.
- III. The respondent (patent proprietor) requested in the written proceedings that the appeal be dismissed. As an auxiliary request oral proceedings were requested.
- IV. The party of the appeal proceedings as of right (opponent I; hereinafter referred to as "party as of right") did not make any substantive submissions or requests during the written proceedings.
- V. Independent claims 1, 14 and 21 of the patent as granted under consideration in the decision under appeal read as follows:

"1. A process for forming a zinc phosphate coating having a coating weight of at least about 1612 milligrams/m² (150 milligrams/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 iron;
- (c) from 0.5 to 1.0 g/l of fluoride ion measured as F⁻;
- (d) from 4 to 400 mg/l ferrous iron; 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 bifluoride 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 about 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 bifluoride 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."

VI. With a communication dated 21 January 2010 and annexed to the summons to oral proceedings the Board presented its preliminary opinion with respect to claims 1 to 22 underlying the impugned decision.

Amongst others the Board remarked that the compositions and conditions used for making comparative examples appeared to be **not** identical with those specified in the patent in suit in the context of the examples of the patent in suit and that this also applied to the comparative example with respect to example V of D1. Thus it appeared that the appellant failed to prove that the person skilled in the art cannot execute the claimed invention.

With respect to the issue of inventive step the Board remarked amongst others that D1 appeared to represent the closest prior art for process claim 1 which appeared to be distinguished over it in that it further requires ammonium ions to be present in the phosphate bath. For concentrate claim 14 either of documents D5 or D12 - both do not explicitly disclose ferrous ions in their concentrates - could be taken as closest prior art.

Thus it would be discussed whether or not the subject-matter of claims 1, 14 and 21 is rendered obvious by the available prior art documents.

With respect to the appellant's arguments concerning D12/D14 and the use of a stainless steel equipment for the making and storage of the phosphate concentrate it was remarked that no evidence had been submitted in this context which would prove a particular ferrous ion content in such concentrates. Furthermore, it was remarked that stainless steel, similarly as steel with high Ni-contents, generally cannot be phosphatised so that the amount of metal that can be dissolved from such stainless steel (which is normally selected such that any corrosion during the intended use thereof is minimized) should be small and is additionally not known, let alone whether any dissolved iron is actually present as ferrous ion.

VII. With letter dated 26 April 2010 the appellant submitted further arguments as well as fresh evidence, partly taking account of the Board's comments in the summons.

Additionally it raised for the first time in the appeal proceedings an objection under Article 123(2) EPC.

- VIII. No further written submissions were filed by the respondent or by the party as of right.
- IX. Oral Proceedings before the Board were held on 27 May 2010. After the start the appellant withdrew its objection under Article 123(2) EPC. First of all the issue of sufficiency of disclosure was discussed. Thereafter inventive step in respect of the subject-matter of independent claims 1 and 14 was discussed.
- (a) The appellant and the party as of right both requested that the decision under appeal be set aside and that the patent be revoked.
 - (b) The respondent requested that the appeal be dismissed, i.e. that the patent be maintained as granted.

At the end of the oral proceedings the Board announced its decision.

- X. The appellant argued essentially as follows:
The person skilled in the art does not understand the term "fluoride ion measured as F⁻" as used in claims 1 and 14. The equilibrium diagrams based on the NMR-measurements (see Annexes 4(1) to 4(3) of the appellant's letter dated 26 April 2010) show that in order to provide a fluoride ion concentration as required by claims 1 and 14 and within the pH range of 2.5 to 5.5 according to the patent in suit (see paragraph [0012]), wherein the fluoride content is

strongly dependent upon the pH, it would not be possible to come to such a high total fluoride content for the concentrate of claim 14 which has a pH of about 0-2 a total fluorine content of about 1200 g/L would be necessary to provide 2-40 g/L F⁻. On the other hand if the content of 2-40 g/L F⁻ were to be added to the phosphating composition then after some minutes, due to said equilibrium, it will no longer be present as F⁻. Thus it appears that the total fluorine content should have been defined in claims 1 and 14. The total fluorine content is only shown in table III. The patent in suit discloses no specific measurement of said F⁻ content (see patent, paragraph [0024]). Said equilibria diagrams additionally show that the total fluorine content does not correspond to the fluoride content. Normally the fluoride content is measured by a potentiometric method but in the absence of further indications in the patent no correct measurement at the pH values of the concentrate is possible. The concentrate either has to be diluted or a buffer has to be added. Furthermore, there is a difference between complex and simple fluorides or bifluorides. This fluoride feature not only causes a clarity problem as argued by the respondent but also a problem as to whether or not someone works in the forbidden area of the claims. Furthermore, in the USA such a statement which totally changes the meaning of a feature would not be accepted.

There is a lack of disclosure concerning the type of the crystals to be produced and the main properties of the phosphate coating such as the corrosion resistance and paint adhesion. Likewise no descriptions of the cleaner (Chemkleen[®]) and the conditioner used in the

examples are given in the patent. It is additionally unclear whether that Chemkleen^R now available has the same composition as that in 1995. The conversion from weight% to g/l given for the examples A-L in the tables I, III to V results in different calculation factors for each of the specified components. It makes a difference of more than 20% if one uses in these calculations a density of 1.0 or 1.29 g/ml for the phosphating bath. Likewise it is not known which water content the nickel nitrate should have, which type of aluminium alloy can be treated, and at which temperature it should be worked. This influence has been shown with the first comparative tests. There is no example which reveals all parameters (coating weight, morphology, etc.). The phosphating bath is a living system due to said many equilibria and you have to consider the actual situation and not the starting point of the bath after mixing the components.

The comparative tests were made to the best knowledge of the appellant though there remain doubts as to whether or not they are identical to what the inventor did in 1995. The respondent admitted that the free acidity of the solutions made from the concentrate according to the examples has to be adjusted to the desired pH value (see patent, paragraph [0012]) in order to be suitable for a phosphating treatment. Cleaning of the aluminium substrates for a longer period likewise results in a clean surface.

The invention aims to solve different objectives (see paragraph [0006]), but only that of the coating weight and of producing columnar or nodular crystals actually seems to be solved. Starting from D1 as the closest

prior art the objective problem would be the provision of a phosphate coating comprising columnar or nodular crystals on an aluminium substrate while not using an accelerator. The throughput test shows an enrichment of ferrous and ammonium ions in the phosphating bath after coating of 50 sheets falling within the concentration ranges of claim 1, while the equilibria of the fluoride shows that always there will be a bifluoride present in the phosphating solution. Furthermore, NH_4F is one of the most interesting fluoride compounds which produces bifluoride in the solution. Nitrogen compounds are always comprised in the phosphating solution so that it needs only some hours or days to get into the claimed ammonium ion range taking account of the oxidising potential of the bath while the ferrous ions are automatically obtained from the treated steel sheets. It cannot be seen which compound generates an effect. D3, D8 and D13 do not show any advantages of these ions. The phosphate coatings of the examples according to D13 were suitable for electro-dip coating. There is also no synergistic or bonus effect apparent when filling the missing gaps based on said documents. There exists also no long-felt need and no prejudice or specific problems that had to be overcome. Examples G and J show either only the crystal structure or only the coating weight. The replenisher mentioned according to comparative tests 3 contained mainly zinc and phosphate in order to maintain the concentrations of these ions constant during the coating steps.

Likewise the concentrate of claim 14 lacks an inventive step since the ferrous ion content is unavoidable due to the use of steel or iron containing equipment during the manufacturing or transport of said concentrate.

Thus the subject-matter of the claim is derivable either from D5 and the common general knowledge or a combination of D5 and D14 or of D12 and D14.

XI. The party as of right argued at the oral proceedings essentially as follows:

Claims 1 and 14 specify that the amount of "F⁻" has to be measured but the patent is silent as to which method is to be used and not all of the methods give the same results. Table III discloses the addition of 2.27 weight% NH₄FHF corresponding to 18 g/l F but does not specify the actual F⁻ concentration and is silent as to whether or not it has been measured. Simply mixing the components does not result in a bath or concentrate according to claims 1 and 14, respectively, since the fluoride concentration has to be the "fluoride ion measured as F⁻" (see patent, paragraph [0017]). Since no method is disclosed it is impossible to make these compositions. No evidence is at hand which would prove that different methods produce different results. However, it is clear that different results are obtained in the case that the bath as such is measured or that a sample thereof is mixed with a buffer solution.

D5 represents the closest prior art for concentrate claim 14. The concentrate A1 of example 1 differs from the concentrate of claim 14 in the missing ferrous ions. The objective of D5 is to phosphate steel, aluminium and zinc-coated metal parts and it discloses that the phosphophyllite coating of the prior art is not applicable for substrates not containing iron (see column 1, lines 56 to 61; column 2, lines 6 to 9 and

lines 61 to 65). Since iron precipitates from the phosphating baths D5 teaches to replace it by nickel and manganese ions (see column 2, lines 15 to 21). However, the person skilled in the art knows from D11 that ferrous ions, which are enriched due to the pickling action of the phosphate bath on iron and steel substrates, do not precipitate (see page 3, lines 33 to 36). Likewise the bath according to D3 comprises ferrous ions (see column 4, lines 28 to 36 and line 75 to column 5, line 3) which do not precipitate. Consequently, no prejudice against using ferrous ions in phosphating baths exists. Therefore, the person skilled in the art when considering environmental needs would substitute the nickel of the bath according to D5 with ferrous ions and arrive at the concentrate of claim 14 in an obvious manner when combining with the teaching of D3. Claim 14 comprises no limiting feature concerning the free acidity of the concentrate and does not specify any nickel. The fluoride content of D3 starts at 150 ppm and thus greatly overlaps with that of the claims.

The conclusion with respect to claim 14 also applies to claim 1 which uses the diluted concentrate of claim 14. Taking account of a comparison of the examples A and C, and G and H no synergistic effect can be seen (see paragraph [0019] and tables I and III).

XII. The respondent argued essentially as follows:

The first question to be answered is whether or not it is possible to reproduce the claimed subject-matter. The first instance acknowledged that the requirements

of Article 83 EPC are met so that the onus of proof is on the side of the appellant and the party as of right.

The appellant has not shown that the person skilled in the art cannot make the composition of claim 14 by weighing the specified components. The question what is meant by the feature "fluoride ion measured as F⁻" represents a clarity issue. According to the patent the fluoride may be added or measured (see paragraph [0024]). As proven by the appellant the interpretation of "F⁻" as meaning only F⁻-ions would be nonsense so that it is clear that it has to be understood as the total fluorine content. The measuring method is to use a buffered solution to determine the fluoride with an ion selective electrode which indicates the total fluorine content. The technical experts in the USA would use it that way. That the total fluorine content is meant can also be derived from table III since the fluoride ions are calculated from the added amount of NH₄FHF. This is sufficient for the reproducibility of the examples. During the opposition proceedings the appellant and opponent I had the understanding of this "F⁻" as meaning the total fluorine content. There exists only the method with a fluorine sensitive electrode known for measuring either the actual fluoride content or the content in a buffer solution. Other methods are not known. In any case it is measured against a reference solution.

With respect to claim 1 the appellant failed to prove that the person skilled in the art cannot run the phosphating process. Although there exist complicated equilibria in the bath as shown by the appellant they have nothing to do with the issue of reproducing the

claimed invention. Examples G and H clearly disclose the composition of the concentrates to be used for making the phosphating baths. There is no necessity to make calculations based on the density. The skilled person only has to mix the specified components in the specified amounts, which do not represent weight% but parts by weight (see paragraph [0047]) and thus need not to add up to 100%, and thereby he is able to reproduce these compositions. The patent in suit discloses all the necessary parameters for carrying out the examples including the temperature (see paragraph [0041]). Despite these clear indications in the patent in suit of for example a temperature of 55°C for cleaning the aluminium panel by spraying for one minute, the panels according to comparative tests 2 were made at a higher temperature of 60°C by dipping for the longer time of 7-8 minutes (see comparative tests 2, page 1) while it was possible to apply the spraying treatment according to comparative tests 3 (see comparative tests 3, page 1). The much longer cleaning treatment with a mild alkaline cleaner according to comparative tests 2 may cause considerable etching of the aluminium surface - aluminium is liable to alkaline attack - thereby resulting in the formation of aluminium hydroxide at the surface which then can be coated with the phosphate coating; however, the obtained phosphate coated aluminium hydroxide is wipeable from said aluminium panel as explained by the technical expert. The use of the mild alkaline cleaner instead of Chemkleen® is in line what the skilled person would do. Furthermore, the panels according to the comparative tests 2 were conditioned with a Ti-phosphate activator for 30 seconds instead of one minute as stated in the patent. Reducing the activation

time, however, can influence the coating. The use of the Ti-phosphate activator is within the common general knowledge of the person skilled in the art. The free acid of the specified phosphating baths has to be reduced by neutralization as was correctly done by the appellant taking account of the general description in the patent. The best results of comparative tests 2, however, were obtained with samples G8 and J4N which comprised the required amount of ferrous and ammonium ions. It appears that the appellant did not have problems with the addition of the ferrous ions when repeating the prior art D1 (see comparative tests 3, table 1) but that these problems were actually caused by the analysis of the ferrous ions after some hours or days which explains the Merckoquant results of the same baths (see comparative tests 3, table 1a). It is well known that ferrous ions can easily be oxidized by air to ferric ions. Thus the data presented are not plausible. Furthermore, comparative tests 3 were made with a different free acidity and total acidity than example V of D1. From the aforesaid it is, however, evident that the comparative tests 2 and 3 have neither been made in agreement with the examples of the patent in suit nor of example V of D1.

Claim 14 relates to the use of concentrates for making phosphating baths and the etching in steel equipment does not necessarily result in a ferrous ion content as required by claim 14. No evidence has been submitted in this context.

The appellant allegedly repeated example V of D1 but the disclosure concerning ammonium is not part of the disclosure of D1. Particularly, the example V of D1 has

not been reproduced taking account of the data given in comparative tests 3 which for example used different free acid/total acid points and did not get the result of example V of D1. Furthermore, for compensation a replenisher R 2820 E18 of unspecified composition was added to the used bath which might have contained ferrous and ammonium ions (see comparative tests 3, page 1, last sentence).

The presence of ferrous and ammonium ions is essential to the baths of the invention. The objective problem to be solved is the one stated by the opposition Division (see point 7.3.2 of the decision) but there is no hint in the prior art that a combination of the said ions leads to the preferred type of morphology of the phosphate coating. D1 found this morphology on steel substrates but not on aluminium substrates. The addition of the hydroxylamine increases the range of the zinc ions to produce said morphology (see page 4, lines 47 to 50). However, D1 is the only reference dealing with this problem. Consequently, there exists no motivation for the person skilled in the art to add for example ammonium ions to the bath. Furthermore, although it appears that the appellant does not know what happens in such baths concerning the $\text{Fe}^{2+}/\text{Fe}^{3+}$ equilibrium it nevertheless argues that it is obvious what will happen when ferrous ions are added (see letter dated 26 April 2010, page 20, eighth paragraph). Likewise it argues with respect to the generation of ammonium ions (see letter dated 26 April 2010, page 9, seventh paragraph) that during the treatment of the metallic substrates in the phosphatising bath a particular amount of ammonium ions is inevitably produced from the nitrate and nitrite content therein

which is dependent upon many parameters. However, there is no such disclosure in the prior art, let alone one that by adjusting the ferrous and ammonium ion amounts allows modification of the crystal size and morphology of the phosphate coating. On the other hand the appellant with its "comparative example G" produced a coating weight well above the minimum coating weight required by claim 1.

D5 is accepted as the closest prior art for the concentrates of claim 14 which can be used for start up or as replenisher. Thus the concentrate is linked to the process of claim 1 and there is no hint in the prior art to add ferrous ions in order to obtain the desired morphology. D5 clearly teaches that the incorporation of iron is not wanted but it was the object to substitute nickel or manganese for the iron (see D5, examples and claims). Thus the person skilled in the art has no reason to replace the nickel by iron as argued by the party as of right. Furthermore, the argumentation of the party as of right is not acceptable as it is not the object of the patent in suit to substitute nickel since nickel can be added as is apparent from the examples. The subject-matter of claims 1 and 14 does also not exclude nickel. Furthermore, the low-zinc technology of D5 is combined with the high-zinc technology of D3 which is not allowable as such since it requires an adaption of all the components (compare in this context D13, lines 21 to 29), and even if one were to combine the two documents then they should be combined completely and not by taking the concentration range of only one component of the bath. It needs also to be considered that for phosphating of aluminium a higher acidity and

fluorine range than the general range is preferred according to D3 (see column 5, lines 8 to 13). Thus the total fluorine content would be close to 8 g/l fluoride. D11 teaches away from the patent in suit since the ferrous ions are not added.

The throughput test is not relevant at all since it is not related to a specific prior art which was to be reproduced.

Therefore even if one takes the least ambitious objective problem of providing an alternative to D1 the subject-matter claimed is not rendered obvious by the available prior art.

Reasons for the Decision

1. *Sufficiency of disclosure (Articles 100(b) and 83 EPC)*

Taking account of the arguments presented by all the parties as well as taking account of the comparative test results on file the Board considers that it has not been shown that the Opposition Division's conclusion was wrong with respect that the description of the invention according to the patent in suit being sufficient for the person skilled in the art to carry out the invention. The reasons are as follows:

- 1.1 The question of what is meant by the feature "fluoride ion **measured as F⁻**" of independent claims 1 and 14 is considered to represent only a clarity issue which does **not** result in that the person skilled in the art is unable to carrying out the teaching of the patent in

suit. Although this definition "**measured as F⁻**" could, at least in theory, be interpreted as meaning that only the fluorides in the form of "**F⁻**" should be measured in the phosphating solution or concentrate the person skilled in the art would refrain from doing so for the reasons given below.

- 1.1.1 First of all, there exists only **one** method using a fluorine sensitive electrode for measuring the fluoride content of a solution which works, however, only within a certain pH range of about 3-8. Thus, it is not relevant that the patent in suit is silent in this respect since the person skilled in the art knows this standard method. Furthermore, it belongs to the general knowledge of the person skilled in the art that, in order to determine the (unknown) fluoride content of a solution by using this ion sensitive electrode, it is in any case measured against a reference solution, i.e. at least one other measurement with said electrode with a solution containing a known total fluoride content of a selected compound, such as e.g. NaF, is made. It further belongs to the general knowledge that this method - which is also regularly used in industry in e.g. phosphatising or pickling plants to determine the fluoride content of the used bath - indicates the total fluoride content in the solution or bath, although it actually measures the free fluoride content.

Finally, taking account of the fact that at least one (or several) reference solution(s) having a similar pH has (have) to be used in order to determine the unknown fluoride content of the solution to be measured, it is without relevance whether the solution in question has to be diluted with water to a certain extent in order

to produce a solution having a pH within the aforementioned range of about 3-8, or is mixed with a buffer solution, as long as the same procedure is applied to both the reference solution and to the solution having the unknown content. On the one hand it is thus self-evident that the reproducibility of this standard method is given, while on the other hand it is likewise clear that the same result should be obtained within the error margins of the method irrespective of whether the solution has been diluted or mixed with a buffer solution. Therefore the arguments submitted by the appellant and the party as of right cannot hold, particularly as no evidence to the contrary has been submitted as admitted by the party as of right during the oral proceedings before the Board.

- 1.1.2 With respect to the interpretation of the feature "fluoride ion measured as F⁻" - which in the entire proceedings has been presented for the first time by the appellant at the oral proceedings before the Board but has not been objected to by the respondent - the Board considers the points which follow.
- 1.1.3 Firstly, during the entire opposition proceedings and in its grounds of appeal the appellant had the understanding that this feature means the total fluoride content (see grounds of appeal dated 25 July 2007, page 3, first paragraph). In its response to the Board's summons to oral proceedings the appellant had still the same understanding (see letter dated 26 April 2010, pages 4 to 6, paragraph "3.) Fluorid-Gehalte und Zusatz von Bifluorid") although the NMR-measurements of an aqueous solution comprising HF and the pH dependency of the equilibrium of the ions F⁻ and HF₂⁻ were already

known from the opposition proceedings. The same holds true with respect to the party as of right who likewise submitted similar arguments for the first time at the oral proceedings before the Board.

The Board similarly to the Opposition Division interpreted this feature as meaning the total fluorine content (see e.g. point 3.2 of the Board's communication annexed to the summons).

- 1.1.4 Secondly, the appellant itself argued that taking account of said equilibrium the interpretation of "... measured as F^- " - namely that only the simple fluoride ions " F^- " should be measured - would imply unrealistic high total fluorine content in the phosphating bath or concentrate. Therefore this interpretation is not credible for the person skilled in the art and it is thus clear that this feature should be understood as meaning the total fluorine content.
- 1.1.5 This conclusion and the interpretation of the feature "fluoride ion measured as F^- " is additionally supported by the examples of the patent in suit. The three tables I, III and IV specify in the first column the ingredients of the bath while in the last two columns the concentrations for the listed ions in grams per liter in the concentrate from the addition of the parts by weight of the listed ingredient are given (see patent, paragraphs [0040], [0047] and [0051]). According to example G of table III, for example 2.27 parts by weight of the acid ammonium bifluoride NH_4FHF was added to 61.94 parts by weight water and the other listed ingredients. This addition resulted in a

concentrate G containing 18 g/l "ions" of "F" (see table III).

Consequently, it is clear to the person skilled in the art that the total fluorine content is meant since the said concentration of fluoride ions can be calculated from the specified amount of NH_4FHF and the total volume of the concentrate.

1.1.6 Neither the appellant nor the party as of right disputed that it is possible to make concentrates as specified in the examples or comparative examples of the patent in suit or as defined in claims 1 and 14 by simply mixing the weighed specified ingredients, i.e. without any measurement. According to the patent in suit the amounts of the various ions added to the coating bath (including the fluoride ions) may be determined theoretically and then added or may be measured analytically by techniques known to the person skilled in the art (see paragraph [0024]).

Furthermore, in order to make said concentrates by simply mixing the specified parts by weight there is no need to make any calculations such as for the density.

Consequently, the person skilled in the art is able to make the concentrates as specified in independent claim 14 either by mixing the required ingredients in the necessary amounts or by measuring their added contents, e.g. the fluoride ion concentration by the aforementioned ion sensitive electrode method.

Furthermore, by diluting these concentrates he is likewise able to produce the phosphating bath as specified in claim 1.

- 1.2 The onus on proving that the person skilled in the art is not able to carry out the claimed invention lies with the appellant who, however, failed to do so.
- 1.2.1 As already mentioned in the Board's communication (see points 3.1 to 3.9 thereof) the comparative tests 2 and 3 submitted by the appellant at the appeal stage were **not** made in full agreement with the examples G, H, I and J of the patent in suit, and example V of D1, respectively, and thus cannot prove that the process of claim 1 cannot be carried out.
- 1.2.2 Although the respondent at the oral proceedings before the Board admitted that the adjustment of the free acidity to a value of 0.9 to 0.85 points according to said examples G to J as carried out by the appellant is in agreement with the general teaching of the patent in suit (see paragraph [0012]) there remained several severe differences between the conditions explicitly specified in the patent in suit and those applied according to comparative tests 2.

For example, despite the clear indications in the patent in suit of a temperature of 55°C for cleaning the aluminium panel by spraying for one minute (see paragraph [0041]) the panels according to comparative tests 2 were cleaned with a mild alkaline cleaner - which is in agreement within the common general knowledge of the person skilled in the art - at a higher temperature of **60°C** by **dipping** for **7-8** minutes (see comparative tests 2, page 1). The much longer cleaning treatment according to comparative tests 2 may, however, cause considerable etching of the aluminium

surface - aluminium is liable to alkaline attack - thereby resulting in the formation of aluminium hydroxide at the surface which then can be coated with the phosphate coating. The thereby obtained phosphate coated aluminium hydroxide is, however, wipeable from said aluminium panel as explained by the respondent's technical expert. Furthermore, the panels according to the comparative tests 2 were conditioned with a Ti-phosphate activator - which is in agreement within the common general knowledge of the person skilled in the art - but only for **30 seconds** instead of one minute as stated in the patent. Reducing the activation time, however, can influence the coating.

1.2.3 Since the comparative tests 2 were not in agreement with the examples of the patent in suit there is no need to go into further details such as the problems with the addition of the ferrous ions which were not plausible. It appears that they have been caused by the analysis of the ferrous ions after some hours or days which explains the Merckoquant results of the same baths (see comparative test 3, table 1a). It is well known that ferrous ions can easily be oxidized by air to ferric ions.

1.2.4 Furthermore, comparative test 3 was made with a free acidity of 0.4 points and a total acidity of 18.9 points which differs substantially from the **0.3** points free acid and **25.8** points total acidity according to example V of D1 (compare comparative test 3, page 1, "bath composition"; and D1, example V) so that it is not surprising that the appellant did not obtain the result according to example V of D1. Furthermore, according to comparative test 3 a

replenisher of unspecified composition has been added to the phosphating solution (see comparative test 3, page 1, penultimate paragraph).

Consequently, it is evident that comparative test 3 has **not** been made in agreement with example V of D1.

1.3 The results of the so-called "throughput test" as filed with letter dated 27 July 2005 are considered to be lacking any relevance for the present case as the phosphating solution of this test was neither made in agreement with any of the examples of the patent in suit nor in agreement with a bath of any of the submitted prior art documents.

1.4 Consequently, the Board sees no reason to deviate from the Opposition Division's conclusion that the patent in suit meets the requirements of Article 83 EPC.

1.5 From the above considerations it is also clear that the appellant - based on said comparative tests 2 - failed to prove the non-existence of an effect of the combination of ferrous and ammonium ions as described in the patent in suit.

2. *Inventive step (Article 56 EPC)*

Process claim 1

2.1 D1 represents undisputedly the closest prior art for process claim 1 for disclosing a low-zinc phosphatising process for ferrous substrates. Its phosphating bath comprises a hydroxylamine agent and produces a phosphate coating having columnar and/or nodular

crystal morphology on steel substrates and can also be applied to zinc and aluminium surfaces (see page 3, line 56 to page 4, line 2; page 5, lines 25 to 27; examples I to V, and claims 1, 8 and 9). According to example V of D1 phosphate coatings with platelet morphologies were produced on aluminium substrates.

- 2.2 The subject-matter of claim 1 is distinguished from the process according to D1 in that
- i) 0.01-2 g/l ammonium ions, and
 - ii) 4-400 mg/l ferrous ions have to be present in the phosphate bath.

These two differences i) and ii) allow that on aluminium substrates zinc phosphate coatings can be obtained which have a smaller crystal size with a columnar or nodular morphology (see patent in suit, paragraph [0006]). Example J shows that the presence of 0.6 g/l ferrous ions and of 9.4 g/l ammonium ions in the concentrate which after dilution with water (1:22.8) resulted in a phosphating bath (containing calculated amounts of about 30 mg/l Fe^{2+} and 0.41 g/l NH_4^+ ions) which produced a phosphate coating having smaller crystals with a nodular morphology (see table IV and paragraph [0052]).

- 2.3 Therefore the objective technical problem starting from the phosphating process of D1, which requires the presence of a hydroxylamine agent in the phosphating bath, is the provision of a phosphating process which produces a zinc phosphate coating having smaller crystals and a columnar or nodular morphology on aluminium substrates.

- 2.4 This problem is solved by the process as defined in claim 1 of the patent as granted.
- 2.5 The subject-matter of claim 1 is not rendered obvious for the following reasons:
- 2.5.1 The presence of both ferrous and ammonium ions is essential to the baths according to the patent in suit. There is, however, no hint in the prior art that a combination of the said ions leads to the preferred types of columnar or nodular morphology of the phosphate coating.
- 2.5.2 According to D1 the addition of the hydroxylamine increases the range of the zinc ions to produce said morphology (see page 4, lines 47 to 50) which was only obtained on steel substrates but not on aluminium substrates (see examples).

According to example V of D1 a phosphating bath, comprising in [g/l] 0.5 Ni²⁺, 0.47 Zn²⁺, 13.3 PO₄³⁻, 1.4 F⁻, and 2.3 hydroxylamine sulfate and a total acid of 25.8 points and free acid 0.3 points, was used for phosphatising cold rolled steel at 137°F (=58.3°C) for 60 seconds with a coating weight of 174 mg/ft² (= 1873 mg/m², containing mostly nodular and a few columnar crystals). When used on aluminium and hot dip galvanized steel, coatings with platelet morphologies with coating weights of 180 mg/ft² (=1938 mg/m²) and 195 mg/ft² (= 2099 mg/m²), respectively, were produced.

The bath of example V thus does 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 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 phosphatised 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, if any, thereof, let alone the content of any ferrous ions.

D1 is, however, the only reference dealing with this problem of providing a columnar or nodular morphology. The disclosure of comparative test 3 concerning the generation of ammonium ions is not part of the disclosure of D1 (and is also not considered to be a reproduction of example V of D1; see point 1.2.4 above) and there is no such disclosure in the prior art, let alone one that adjusting the ferrous and ammonium ion amounts allows modification of the crystal size and morphology of the phosphate coating. Likewise the throughput test is not relevant at all in this context since it is neither related to a specific prior art which was to be reproduced nor does it belong to the prior art. Consequently, there exists no motivation for the person skilled in the art to add for example ammonium ions to the bath of D1.

- 2.5.3 Furthermore, it needs to be considered that the person skilled in the art would not combine the teachings of the low-zinc technology of D1 (or of D5 if first a concentrate has to be made which is then diluted to form the phosphate bath; see column 3, lines 37 and 38) with that of the high-zinc technology according to D3 since it requires an adaptation of all the components

in order to be present in a well-balanced manner in the phosphate bath. The respondent refers in this context to D13 which explicitly states that the normal-zinc technology could not be transferred to the low-zinc technology for phosphating aluminium substrates (see D13, lines 21 to 29).

For similar reasons the person skilled in the art would not cherry-pick concentration ranges of a certain ingredient from another piece of prior art as suggested by the appellant or by the party as of right since he has no reason to do so. Furthermore, the essential ingredients of the bath influence each other and thus have to be balanced. Consequently, all the arguments in that direction cannot hold, particularly in view of the effect caused by the combination of the ferrous and ammonium ions according to the patent in suit (see point 1.5 above).

2.5.4 The arguments of the party as of right regarding the non-existence of an effect of these two ions cannot hold in view of the phosphate morphology obtained by the examples I, J and K of the patent in suit. Each of examples I, J and K comprised identical amounts of ferrous ions in the bath but only example J comprised ammonium ions whereas I and K comprised similar amounts of potassium ions, all of them added as the bifluoride salt. However, only example J comprising the combination of ferrous and ammonium ions resulted in a nodular morphology with a smaller crystal size of $< 10\mu\text{m}$ whereas the examples I and K produced a platelet morphology of 20-50 μm size (see table IV).

Concentrate claim 14

3. Documents D5 (e.g. concentrate A1; see example 1) or D12 (concentrate 169, see column 8, lines 44 to 57) were considered by the parties to represent the closest prior art for the zinc phosphate concentrate of composition claim 14 of the patent as granted.
- 3.1 The aqueous zinc phosphate conversion concentrate of claim 14 of the patent as granted is distinguished over the concentrates according to D5 or D12 in that it contains from 0.1-10 g/l ferrous ions.
- 3.2 The concentrate of claim 14 is linked to the process of claim 1 since it can be diluted to thereby provide the phosphate bath (or a replenisher therefor) for forming the zinc phosphate coating according to claim 1. The addition of this amount of ferrous ions - in combination with the ammonium ions - permits obtaining the desired morphology of the zinc phosphate coating (compare point 2.2 above).
- 3.3 Therefore the objective technical problem starting from the concentrates of D5 or D12 is the provision of a concentrate, which when diluted, allows production of a zinc phosphate coating having smaller crystals and a columnar or nodular morphology on aluminium substrates.
- 3.4 This problem is solved by the concentrate as defined in claim 14 of the patent as granted.

3.5 The subject-matter of claim 14 is not rendered obvious for the following reasons:

3.5.1 According to D5 the incorporation of iron in the phosphating bath is not desired in order to avoid its precipitation (see column 2, lines 14 to 21). D5 teaches to substitute nickel or manganese for the iron to produce phosphophyllite-like phosphate conversion coatings on substrates not including iron ions (see D5, column 1, lines 56 to 61; column 2, lines 6 to 9 and lines 61 to 68; examples and claims).

Since D5 is silent with respect to adjusting the ferrous and ammonium ion amounts so that a smaller crystal size and the desired columnar or nodular morphology can be obtained the person skilled in the art has no reason to add ferrous ions to its concentrates.

3.5.2 Likewise the person skilled in the art has no reason to combine the teachings of D5 and D3 and to replace the nickel by iron as argued by the party as of right.

Firstly, this argumentation is not acceptable since it is not the object of the patent in suit to substitute nickel since nickel can be added to the phosphating bath of the patent in suit as is apparent from the examples. The subject-matter of claims 1 and 14 does also not exclude any nickel.

Secondly, the person skilled in the art would not combine the concentration ranges of the low-zinc technology of D5 with the totally different high-zinc technology of D3. Even if it were assumed that the

person skilled in the art theoretically would do so then such a theoretical combination would require an additional adaption of all the components (compare point 2.5.3 above). In this context it needs also to be considered that for phosphating of aluminium according to D3 a higher acidity and fluorine range than the general range is preferred so that the total fluorine content in the phosphating bath would be close to 8 g/l fluoride (see column 5, lines 8 to 13 in combination with column 4, lines 49 to 57) which is outside the claimed range.

Furthermore, D11 - quoted by the party as of right as common general knowledge that no prejudice existed against using ferrous ions in phosphating baths (see also D3, column 4, lines 28 to 36) - is considered to teach away from the patent in suit since the ferrous ions are not deliberately added and additionally may be removed by precipitation (see D11, page 3, lines 33 to 36).

3.5.3 Likewise the appellant's arguments that by using stainless steel equipment the required ferrous ion content in the concentrate of D5 would inevitably be reached cannot be accepted since no evidence has been submitted in this context which would prove a particular ferrous ion content in such concentrates. This deficiency was, however, already mentioned in the communication accompanying the summons for oral proceedings wherein the Board also explained why in the Board's opinion the appellant's arguments concerning D12/D14 and the use of a stainless steel equipment for the making and storage of the phosphate concentrate are not credible (see point 5.3 of the communication).

The arguments based on the so-called throughput test cannot be accepted since this test is not related to a specific prior art disclosure that was to be reproduced.

- 3.5.4 Similarly the person skilled in the art would not combine the teachings of D5 and D14, or D12 and D14 for the reasons given in point 2.5.3 above, since D14 relates to the different normal-zinc technology (see D14, claim 2) while D5 (see column 3, lines 37 and 38) and D12 (see column 2, lines 52 to 60) relate to the low-zinc technology.

Furthermore, neither D12 nor D14 gives a hint with respect to a modification of the phosphate morphology. Consequently, also these arguments do not hold.

- 3.6 The Board thus concludes that the onus lay with the appellant and the party as of right to show that the Opposition Division's conclusion with respect to inventive step was wrong but they failed to show this.

Order

For these reasons it is decided that:

The appeal is dismissed.

The Registrar:

The Chairman:

V. Commare

P. O'Reilly