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D E C I S I O N
of 12 October 1995

Case Number: T 0825/91 - 3.2.2

Application Number: 83304885.3

Publication Number: 0106459

IPC: C23C 22/73

Language of the proceedings: EN

Title of invention:
Phosphate coating metal surfaces

Patentee:
Nippon Paint Co., Ltd.

Opponent:
Brent International plc

Headword:
Phosphate coating/NIPPON PAINT CO.

Relevant legal provisions:
EPC Art. 54, 56

Keyword:
"Novelty - yes"
"Inventive step - yes"

Decisions cited:
-

Catchword:
-



Case Number: T 0825/91 - 3.2.2

D E C I S I O N
of the Technical Board of Appeal 3.2.2
of 12 October 1995

Appellant: Brent International plc
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Decision under appeal: Decision of the Opposition Division of the
European Patent Office dated 14 August 1991
rejecting the opposition filed against European
patent No. 0 106 459 pursuant to Article 102(2)
EPC.

Composition of the Board:

Chairman: H. J. Seidenschwarz
Members: R. A. Lunzer
J. C. M. de Preter

Summary of Facts and Submissions

I. European patent No. 106 459 was granted on 22 February 1989 on the basis of application No. 83 304 885.3 filed on 24 August 1983 claiming a priority date of 24 August 1982 based on JP Application No. 147266/82. The patent as granted included product Claims 1 to 15 and process Claims 16 to 25. Claims 2 to 25 were dependent on Claim 1, Claims 1 and 16 taking the following form:

"1. An acidic aqueous phosphate solution for phosphating a metal surface which solution contains:

- (a) from 0.5 to 1.5 g/l of zinc ion,
- (b) from 5 to 50 g/l of phosphate ion,
- (c) from 0.2 to 4 g/l of manganese ion,
- (d) at least 0.05 g/l of a fluoride ion, and
- (e) from 0.01 to 0.2 g/l of nitrate ion as phosphating accelerator,

the weight ratio of ion to phosphate ion being 1:(10 to 30).

16. A process for phosphating a metal surface by treating the metal surface with an acidic aqueous phosphate solution as claimed in any one of the preceding claims."

II. An opposition was filed by the Appellant on the ground of Article 100(a) EPC alleging lack of novelty (Article 54 EPC) and lack of any inventive step (Article 56 EPC). The Opponent relied in particular on the following documents:

- (2) FR-A-2 389 683
- (4) EP-A-0 056 881
- (5) GB-A-2 046 312 and
- (7) D. B. Freeman "Pretreatment for electropaint---the facts", Polymers, Paint and Colour Journal, August 8/22 1979, pages 748-757.

III. By its decision given in writing on 14 August 1991 the Opposition Division maintained the patent as granted. It regarded the subject-matter of Claim 1 as novel over the disclosure of document (5) because that document did not explicitly mention a solution containing the elements in the required quantities defined by Claim 1 in suit. Consequently it held that Claims 1 and 16 were novel. As to the existence of an inventive step, document (5), the closest prior art, disclosed ranges which largely overlapped those of the patent in suit, and its Example had all the features of Claim 1, save that it contained Ni within the range claimed by the patent in suit in place of Mn. The tests and Comparative tests disclosed in Table 2 of the patent in suit showed that the Mn present in accordance with Example 1 gave better adhesion and corrosion resistance to coatings produced by cationic electrocoating than a comparable phosphating solution containing Ni and no Mn. Thus the alleged invention provided a solution to the technical problem dealt with by the patent which was not suggested by the disclosure of document (5). The remaining documents were regarded as more remote than document (5) and thus did not lead to any other conclusion.

IV. An appeal against that decision was filed on 17 October 1991, the appeal fee was paid on the next day, and the statement of grounds of appeal was filed on 19 December 1991. In response to a communication from the Board dated 8 August 1995 questioning whether there could be

novelty in the face of the disclosure of document (5), or inventiveness having regard to the disclosure of document (7) both parties amplified their previously-filed written submissions.

In its written statements and during oral proceedings held on 12 October 1995 the Appellant argued that there was a lack of novelty over document (5) which disclosed explicitly the option of using Mn in the solution as an alternative to Ni in the amount of 0.1 to 0.5 g/l. That disclosure overlapped the lower part of the range of Mn expressed in Claim 1 in suit. There could not be novelty in including Mn in the composition of Claim 1 in suit in the face of its actual disclosure as one out of a choice of Ni Mn and/or Ca in document (5).

Even if there were to be novelty, there could be no inventive step because the disclosure of document (5) mentioned the inclusion of Mn in the context of improving the properties with respect to corrosion resistance and adhesion, which were exactly the properties now relied on by the Respondent for asserting that an inventive contribution to the art had been made. Documents (2), (4) and (7) each mentioned the inclusion of Mn in phosphating solutions used for the treatment of surfaces prior to electrocoating. If novelty were to be lacking for the range of up to 0.5 g/l there was no evidence of any qualitative difference when using more than 0.5 g/l of Mn.

V. In its written submissions, and during oral proceedings, the Respondent argued that the alleged invention satisfied the requirements of the EPC, and that when seen in its proper context it reflected a major advance in the art.

Early electrocoating systems for painting automobile bodies involved anodic deposition. That process was soon replaced by cathodic deposition. The body had first to be dipped or sprayed with a phosphate coating before electrocoating. When the change was made from anodic to cathodic treatment, it was found that it was preferable to use lower proportions of Zn:PO₄ than had been used in the past. These solutions were satisfactory for the treatment of ferrous surfaces, but with the trend towards the use of car bodies made up of both galvanised and ungalvanised components, it was found that the adhesion of the electrocoated layer to galvanised areas was inadequate.

That problem was solved by the alleged invention. Document (5) failed to give any clear and unmistakable directions towards the use of Mn, but instead proposed its use as one amongst a host of multivalent ions. The phosphating solutions proposed by the patent in suit were not foreshadowed in any of the cited prior art. These solutions had achieved great commercial success, and were by now the standard product in the motor industry.

- VI. The Appellant requested that the decision under appeal be set aside, and that the European patent be revoked. The Respondent requested that the appeal be dismissed, and by way of an auxiliary request that it be maintained on the basis of the set of claims filed with its letter of 14 September 1995.

Reasons for the Decision

1. The appeal is admissible.
2. As the late-filed documents produced by both parties were in direct response to matters raised by the Board's communication of 8 August 1995, or in answer to further points raised by the opposing party, all were admitted.
3. *Novelty*
 - 3.1 Novelty in the light of the disclosure of document (5) was a substantial issue in the present appeal. That patent specification is so drafted that Example 1 is a comparative example, and the novel contribution to the art is demonstrated by Example 2, which is a variant of Example 1.

It introduces the novel feature of including in a low zinc containing phosphating solution an addition of 0.8 g/l of BF_4 . This Example lacks Mn ion which is essential to Claim 1 of the patent in suit, but apart from that it satisfies all the composition requirements of Claim 1. The issue of novelty turns on whether that deficiency is made good by the passage at page 1 lines 84 to 95. As that passage was subject to detailed scrutiny during the oral proceedings, it is quoted here verbatim:

"The properties of the coating can often be improved by including also in the composition that is applied polyvalent cations such as nickel, manganese and/or calcium ions in an amount of 0.1 to 0.5 grams per litre of the working solution. Such additions improve the corrosion and adhesion of the film. It is particularly desirable to include vanadium compounds, for example in an amount providing 0.1 to 10 mg/l vanadium, especially

when the process is being operated with high throughputs." (The reference to vanadium in terms of mg/l and not g/l is confirmed in Claim 4, and is evidently not a printing error.)

- 3.2 With regard to this passage, the Appellant stressed the fact that there was an actual disclosure of Mn as an alternative to Ni in the combined Examples 1 and 2, while the Respondent emphasised the reference to polyvalent cations in general, the vagueness of the words, "can often be improved", and argued that as four polyvalent cations had been identified by name, there was scope for inventive selection.
- 3.3 In accordance with the case law of the Boards of Appeal, to deprive a later invention of novelty a prior disclosure must contain a clear and unmistakable direction to use the subject-matter of the later invention. The above passage goes no further than equating the use of Mn and Ca with the Ni of the Examples. That is done in the context of a disclosure which points to the advantages of including fluoroborate, which is the essential contribution to the art by document (5). In the Board's view, the reference to the inclusion of Mn would be understood by the skilled reader as doing no more than confirming his general knowledge, that many other polyvalent cations can usefully be included, and frequently are included in phosphating solutions, rather than pointing positively in the direction of using Mn in preference to, or in addition to, Ni or Ca. It is also noteworthy that whereas the inclusion of vanadium is a feature of Claim 4 of document (5), none of its claims refers to the inclusion of other polyvalent cations.

3.4 The Board therefore concludes that the disclosure of document (5) is not sufficiently specific with regard to the use of Mn to deprive Claim 1 in suit of novelty, and that subject-matter of Claim 1 is considered to be novel within the meaning of Article 54 EPC.

4. *Inventive step*

4.1 It was common ground that document (5) was the closest prior art, since it alone disclosed in combination in a single example all of the features of Claim 1 of the patent in suit, apart from the presence of Mn. Its disclosure relates to a low zinc containing phosphating solution which included a source of fluoride ions in the form of fluoroborate, and it demonstrated that the presence of that constituent improved the corrosion resistance of a subsequently applied lacquer, especially the protection against undercreep (page 2 lines 90 to 96).

4.2 In contrast to that disclosure, the patent in suit is directed, not to the improvement of corrosion resistance in general, but instead to the specific problem which had been encountered of securing uniformly good corrosion resistance and adherence of electrocoated layers on both iron-based and zinc-based surfaces (patent in suit page 2 lines 58 to 61) or to put it more clearly, galvanised and ungalvanised steel sheet. In Table 1 the compositions of the solutions used in Examples 1 to 7 and Comparative Examples 1 to 8 are given while Table 2 shows the results obtained with these solutions. Similarly, Table 3 gives the further compositions of solutions used in Examples 8 to 12, and also of that used in Comparative Example 9. The results of these further tests are shown in Table 4.

- 4.3 Each of the above-mentioned solutions was tested in turn on four different surfaces, identified respectively as Galvanized steel plate, Electro-galvanized steel plate, Electro-zinc alloy plated steel plate, and Cold rolled steel plate. They show that the best results are obtained when a combination of nitrite, fluoride and Mn ions are present in solution, and in particular, that when these ions are present, the quality of coating achieved on galvanised materials equals that achieved on cold rolled steel.
- 4.4 Only one Example, Comparative Example 3, includes Ni, used at a level of 0.3 g/l in the absence of Mn. When Mn is present in the remaining Examples and Comparative Examples, it is present at a level of 0.8 or 2.0 g/l. Although Comparative Example 3 shows inferior results, having regard to the much lower proportion of Ni ion present the Board cannot treat this as a convincing demonstration of the superiority of Mn to Ni.
- 4.5 The Respondent made reference in its letter of 14 September 1995 to an experimental report intended to show the comparative effects of the inclusion of Mn and Ni in a low zinc containing phosphating solution. The tests were carried out using two starting solutions, one containing 1.0, and the other containing 1.2 g/l of Ni, with amounts of Mn which were increased in steps, initially of 0.25 g/l, from zero to 1.5 g/l.
- 4.6 The probative value of these experiments was attacked by the Appellant in its letter of 5 October 1995 (page 6) where it observed that they did no more than show that **both** Ni and Mn were useful additions, the graphs showing an appreciable improvement in the desired properties

when the Ni concentration was increased from 1.0 to 1.2% g/l Ni, i.e. demonstrating that a change of 0.2 g/l in the concentration of Ni ions had a considerable positive effect.

4.7 These tests show, however, that when both Mn and Ni ions are present in solution, the addition of Mn has a decisive effect on improving adhesion and corrosion resistance, particularly in the low range of Mn content.

4.8 Against that factual background, the Board has to consider whether a skilled person, confronted with the problem that inferior adhesion and corrosion resistance was experienced with electrocoatings deposited on galvanised surfaces treated with phosphating solutions, which solutions may have included Ni, would have found in document (5) a pointer towards replacing the Ni there disclosed by Mn, or the inclusion of Mn ions in addition to Ni ions. In the Board's view, although the cited passage indicates that Mn ions could be included, it does not contain any sufficient pointer towards the selection of Mn ions, as contrasted with the general class of polyvalent ions, and the individually selected choices of Ni, Ca, and V, such as would lead him to expect that better results would be attained if Mn were to be included.

4.9 The passage from document (5) cited in paragraph 3.1 above invites the skilled reader to investigate possibilities, rather than giving him a specific pointer towards the use of Mn. Furthermore, it is to be observed that although document (5) in its opening words at page 1 line 7 refers to metal surfaces, the only metal exemplified in its examples is steel at page 2 line 37. Given that the problem faced in the present case was that adhesion was adequate on steel surfaces, but inadequate on galvanised surfaces, it does not follow

that the skilled person would automatically extend any proposal made with regard to treating steel surfaces to a problem encountered with a galvanised surface. Accordingly the Board rejects the attack of obviousness based on document (5).

4.10 Other documents in the case, notably documents (2), (4) and (7), make reference to the presence of varying amounts of Mn in phosphating solutions. Document (2), which is acknowledged in the patent in suit at page 1 lines 51 to 55, relates to high zinc containing phosphating solutions, and teaches that Mn ions can advantageously be included in the solution to make it suitable for use at a temperature of up to 43°C. In contrast, the range of temperature proposed in the patent in suit is 30 to 70°C (page 4 lines 53 to 59 and Claim 23), with the tests being carried out at 52°C (page 6 line 7). In the present context, the skilled worker may be expected to look for a phosphating solution which will deposit a coating of the desired thickness within an acceptable time. The advantage of Mn in solutions intended to work at lower temperatures in the presence of much larger proportions of zinc would not suggest to the skilled worker that this feature should be taken from document (2) and combined with the teaching of document (5) in the expectation of improving corrosion resistance and adhesion when coating upon galvanised surfaces. As the disclosure of document (2) relates solely to high zinc containing phosphating solutions, it is not regarded on its own as being a sufficient pointer towards the invention.

4.11 Of the above-mentioned documents, the most pertinent is document (4) (acknowledged in the patent in suit at page 1 lines 44 to 50) which in a Table at page 7 shows various acidic aqueous phosphating solutions. The options of including Mn in the absence of F, or F in the

absence of Mn, are shown by solutions 2 and 4 respectively. Although it might be suggested that the absence of any Mn from solution 4 can be made good by the general teaching at page 5 lines 5 to 9, to the effect that its presence is useful, in particular when phosphating at the lower temperature ranges, that is not a pointer towards Mn solving the problem of improving adhesion and corrosion resistance on galvanised surfaces. Furthermore, as document (4) is directed towards the avoidance of the use of nitrite on environmental grounds (page 1 line 26 to page 2 line 3), it cannot be regarded as a pointer towards a composition in which nitrite is an essential component.

4.12 Document (7) is a review article directed mainly to high zinc containing compositions, although there is a reference at page 751 last paragraph to, Chlorate/nitrite accelerated [solutions] with low zinc content, operating at 55 to 60°C. It goes on to refer to the disadvantage of these solutions in requiring rather long process times by British and American standards. Throughout document (7), the different kinds of baths are discussed in general terms, and the weights of coatings attainable are given in terms of g/m², but there is an absence of information on bath compositions. This document, whether taken alone or in combination with any other, cannot be regarded as leading the skilled worker in the direction of compositions falling within the ranges specified in accordance with the invention.

4.13 For the above reasons, the Board is satisfied that none of the documents taken into account in the present appeal, whether considered alone or in combination with others, makes the subject-matter of Claim 1 obvious. It therefore involves an inventive step, as is required by Article 56 EPC.

5. *Commercial success*

In resisting the present appeal, the Respondent asserted, without substantiation, that the product the subject of the alleged invention had achieved commercial success, and suggested at the oral proceedings, again without evidence, that it had by now become the industry standard. It was also contended that the EPO is unreceptive to evidence of commercial success. In view of that contention the Board draws attention to the fact that the EPO has consistently adhered to the principle of the free evaluation of evidence. In adherence to that principle, unsubstantiated assertions of commercial success have to be disregarded, as they were in the present case. However, cogent evidence of commercial success, if it had been available, could have assisted the Board.

6. *Conclusion*

Claim 1 being allowable, the same applies to dependent product Claims 2 to 15, and process Claims 16 to 25, which are directed respectively to preferred products according to Claim 1, or processes using the products of Claim 1, and whose inventiveness is supported by that of the main claim.

Order

For these reasons it is decided that:

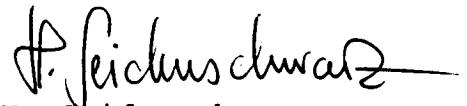
The appeal is dismissed.

The Registrar:



S. Fabiani

The Chairman:



H. Seidenschwarz