BESCHWERDEKAMMERN DES EUROPÄISCHEN PATENTAMTS

BOARDS OF APPEAL OF THE EUROPEAN PATENT OFFICE

CHAMBRES DE RECOURS DE L'OFFICE EUROPEEN DES BREVETS

A		B		C	X

File Number: T 370/90 - 3.2.2

Application No.: 82 303 913.6

Publication No.: 0 071 436

Title of invention: Electroless nickel plating

Classification: C23C 18/32

D E C I S I O N
of 9 February 1993

Proprietor of the patent: RICHARDSON CHEMICAL COMPANY

Opponent: Schering Aktiengesellschaft

Headword:

EPC Art. 54 und 56

Keyword: "Novelty and inventive step (yes) - after amendment"

Europäisches Patentamt | European Patent Office | Office européen des brevets

Beschwerdekammern | Boards of Appeal | Chambres de recours

Case Number : T 370/90 - 3.2.2

D E C I S I O N
of the Technical Board of Appeal 3.2.2
of 9 February 1993

Appellant : (Opponent)
Schering Aktiengesellschaft
Postfach 65 03 11
W - 1000 Berlin 65 (DE)

Representative :
Lederer, Franz, Dr.
Lederer, Keller & Riederer
Patentanwälte
Lucile-Grahn-Straße 22
W - 8000 München 80 (DE)

Respondent : (Proprietor of the patent)
RICHARDSON CHEMICAL COMPANY
2400 East Devon Avenue
Des Plaines
Illinois 60018 (US)

Representative :
Harrison, David Christopher
MEWBURN ELLIS
2 Cursitor Street
GB - London EC4A 1BQ (GB)

Decision under appeal :
Decision of the Opposition Division 2.1.06.018 of the European Patent Office dated 23 April 1990 rejecting the opposition filed against European patent No. 0 071 436 pursuant to Article 102(2) EPC.

Composition of the Board :

Chairman : G.S.A. Szabo
Members : W.D. Weiß
J. Van Moer

Summary of Facts and Submissions

I. European patent No. 0 071 436 was granted with effect from 14 October 1987 on the basis of European patent application 82 303 913.6, filed on 23 July 1982.

II. An opposition was filed against the patent on the grounds of lack of novelty and inventive step. In addition to the twelve documents indicated on the title page of the patent in suit (denominated (1) to (12) in the decision under appeal) the following five documents were cited in the course of the opposition proceedings:

(14) DE-A-2 233 276 (US-A-37 782 978 (13) and FR-A-2 144 782 (1) = family members);
(15) CS-A-130 450;
(16) Dr. Speckhardt "Beitrag zur stromlosen Vernickelung mit Natriumhypophosphit", Metall, June 1971, pages 633 to 640; and
(17) Feldstein et al., "A new technique for investigating the electrochemical behaviour of electroless plating baths and the mechanism of electroless nickel plating", J. Electrochem. Soc., Electrochemical Science, 1971, 869 to 874.

III. By its decision of 23 April 1990, the Opposition Division rejected the opposition pursuant to Article 102(2) EPC.

IV. On 7 May 1990, the Appellant (Opponent) filed an appeal against this decision and paid the appeal fee on the same date. The statement of grounds, in which the Appellant cited the documents

(14') DE-B-2 233 276, and
(14a) GB-A-786 175

for the first time, was filed on 3 September 1990.

V. In a communication of 12 August 1992, the Board drew the parties' attention to the fact that it considered document (14a) to be particularly relevant and, therefore, intended to discuss also the disclosure of this document during an oral proceedings summoned for 9 February 1993.

The documents

(8) V.I. Velemitzina "Electroless nickel plating", pages 357 to 367; and
(12) K. Parker and H. Shah, "Residual Stresses in Electroless Nickel Plating" (seven pages), Plating March 1971,

which the Respondent had cited during the examination phase, were also mentioned by the Board in this communication as relevant state of the art.

VI. During the oral proceedings of 9 February 1993, the Respondent finally filed an amended set of claims together with amended pages 2 and 3. Claim 1 in this amended version reads as follows:

"1. A method of electrolessly depositing a nickel phosphorus deposit onto a substrate with reduced stress in the deposit from a bath having a pH of at least about 4.5 and comprising

(a) a bath soluble unsaturated carboxylic acid of the formula $R(COOH)_n$ or bath-soluble derivative thereof wherein R is an unsaturated alkyl group with at least 2 and not more than 6 carbon atoms and is at least 1; and

(b) a bath-soluble reducing agent; and

(c) a bath-soluble source of nickel,

(d) a bath-soluble saturated alkyl or aryl polycarbocylic acid of the formula $R'(COOH)_p$ or a bath-soluble salt ester or anhydride thereof, wherein R' is absent or is a saturated alkyl or aromatic group having from 1 to 20 carbon atoms and p is at least 2;

(e) any sulfur if present being exclusively in the highest oxidation state of sulfur; and

(f) a bath-soluble phosphorus source which may be said reducing agent; the deposit formed having a phosphorus content of at least about 10 per cent by weight."

The dependent Claims 2 to 13 refer to particular embodiments of the method according to Claim 1.

VII. The Appellant's arguments presented against the amended version of the claims can be summarised as follows:

Document (14a) had to be seen as the closest prior art. The bath composition indicated in Claim 1 differed from the one disclosed in document (14a) only in that the unsaturated alkyl group of the unsaturated carboxylic acid had a short chain of two to six carbon atom. According to the tables on page 3 the known bath had a pH value of 4.58. A sulphur content was nowhere mentioned. Document (14), however, disclosed that such short chained aliphatic unsaturated carboxylic acids were to be preferred as additives to suppress the spontaneous decomposition of a bath. The connection of residual stresses and the phosphorous content in nickel platings was known from document (12). The plating rate achieved by the method according to the patent in suit could only be rated as commonly known and usual in view of document

(9) G. Gawrilov, "Chemische (stromlose) Vernicklung", Eugen Leuze Verlag 1974, pages 39 to 43.

VIII. The Respondent emphasised that Claim 1 did not merely refer to a bath composition but to a method which aimed at achieving nickel platings with a high phosporus content and, hence, low inherent tensile stress at high deposition rates.

Although the interdependence between tensile stress and high phosphorus content had been known, it had not been possible to achieve a nickel plating with a phosphorous content of at least about 10 per cent by weight at a deposition rate which is comparable to the rate which was routine at lower phosphorus contents (see also document (9)).

The feature "from a bath having a pH of at least about 4.5" had to be interpreted according to the general knowledge of the skilled expert (document (12), second page, chapter "Results") that this value had to be kept constant during plating. Therefore, document (14a) presented a deviating disclosure in this respect.

Although document (14) mentioned inter alia examples in which maleic acid was used in plating baths, these baths did not meet the other requirements of the present Claim 1. Moreover sulphur containing agents there were recommended as particularly effective stabilisators.

IX. The Appellant requests that the decision under appeal be set aside and the patent be revoked in its entirety. The Respondent requests that the patent be maintained on the basis of the Claims 1 to 13 and amended pages 2 and 3 of the description submitted during the oral proceedings of 9 February 1993 and rest of the description as granted.

Reasons for the Decision

1. The appeal is admissible.

2. Amendments

The amendments of Claim 1 and 2 are based on page 5, first paragraph of the original description. They constitute a restriction with respect to the scope of the claims as granted.

There is, therefore, no objection to the current claims under Article 123 EPC.

3. Interpretation of Claim 1

The description of the patent in suit has indicated from the very beginning that it had been known that a low pH value of the electroless plating bath involved a low plating rate. Consequently, it had already been the original object of the patent in suit to enable the plating of products to have a reduced tensile or even contractile stress, and hence a recommended phosphorus content of at least 10 per cent, without sacrificing the plating rate of the electronic bath (pages 2 and 3 of the original description).

In view of the above, it would make no sense to interpret the feature of Claim 1 concerning the pH value as only referring to the initial condition of the plating bath. The feature can only mean that the pH value must be maintained at a value of "at least 4.5" during the duration of the treatment.

Such an interpretation is in line with the normal practice in this field of technology as reflected by document (12), second page, last complete paragraph.

4. Novelty

None of the documents cited discloses a bath composition comprising a particular unsaturated carboxylic acid in combination with a particular saturated polycarboxylic acid as defined in Claim 1 as amended.

Consequently, the subject-matter of Claim 1 is novel.

5. Closest State of the Art and Differences

5.1 The Board concurs with the Respondent that document (14a) is the closest state of the art.

This document discloses a method of electrolessly depositing a nickel phosphorus deposit onto a substrate from a bath comprising

(a) sodium oleate as a bath soluble derivative of an unsaturated carboxylic acid of the formula $R(COOH)_n$ wherein R is an unsaturated alkyl group with 18 carbon atoms and n is 1;
(b) sodium hypophosphite as a bath-soluble reducing agent;
(c) nickel hypophosphite as a bath-soluble source of nickel;
(d) sodium succinate as a bath-soluble salt ester of a saturated alkyl polycarboxylic acid of the formula $R'(COOH)_p$, wherein R' is a saturated alkyl having 2 carbon atoms and p is 2;
(e) no sulphur; and

(f) hypophosphite as a bath soluble phosphorus source which is identical with the reducing agent.

The bath has an initial pH value of 4.58 which drops during the plating procedure to a final value of about 2.7. The document (14a) is silent about the phosphorus content of the resulting nickel layer.

5.2 Consequently, the method of Claim 1 differs from the disclosure of document (14a) in that

- the nickel phosphorus layer is deposited from a bath having (or being maintained at [see point 3 above]) a pH of at least 4.5;
- the alkyl group of the said unsaturated carboxylic acid has at least 2 and not more than 6 carbon atoms; and
- the method is guided in a manner that the resulting nickel phosphorus layer has a phosphorus content of least 10 per cent by weight.

6. Problem

The disclosure of document (12) represents general technical knowledge of a person skilled in the art of electroless nickel plating.

The skilled person knows from this document (penultimate and last paragraphs) that an increase of the phosphorus content of the deposit decreases the tensile stress and increases the compressive stress. The phosphorus content and thereby the residual stress can be controlled by the most critical variables of the plating bath: the pH and the temperature. According to Figure 3 and the paragraph "Results", it is also known, that, before the priority date, a phosphorus content of more than 10 per cent was

usually only obtainable at pH values below 4.5 (see also document (8), page 359, third paragraph).

However, the practitioner who wanted to produce a nickel plating exhibiting a low or even negative tensile stress, and therefore having a phosphorus content of more than 10% on a substrate using a bath with a basic composition as disclosed in the closest prior art (14a), was faced with the known drawback that at these reduced pH values the rate of deposition was slowed down to an intolerably low level (e.g. EP-B-0 071 436, page 2, lines 18 to 29).

Starting from the closest prior art, the technical problem therefore consisted of eliminating this drawback by developing a method which permitted to produce an electroless nickel plating (with a phosphorus content of at least 10%) at a tolerable plating rate.

Therefore, the patent in suit particularly aims at producing plating with high phosphorus content at a plating rate comparable to the one common for low phosphorus contents. Referring to document (9), in particular Figure 11, the Appellant argues that the plating rates achieved with the claimed method are not higher or even lower as those obtained by conventional methods. This argument neglects, however, that document (9) obviously describes the production of conventional nickel platings (with less than 10% phosphorus) which was effected in a slightly acid medium of pH 6.5 to 7.0 (document (9), page 41, second paragraph) and hence with a tolerable plating rate anyway.
This problem is solved by the features enumerated in point 5.2. above.

7. Inventive Step

Document (12) (see point 6. above) discusses the tensile stress reducing effect of phosphorus contents of at least 10 percent in nickel platings. The formulation of the plating bath used to perform the tests reported there has, however, less in common with the composition used ir connection with the claimed method than has the bath composition disclosed in document (14a) (closest state of the art). According to this document (second page, right column) pH values of less than 4.5 have to be chosen to produce nickel platings with a phosphorus content of 10 per cent and more at normal bath temperatures. This document does not even mention the plating rates at these low pH values and, therefore, does not offer any solution to the stated problem.

Document (17) (page 871, right column, Fig. 4, Table III; Appendix, Bath C) has also recognised the problem of the reduced plating rate, when nickel platings with a phosphorus content above 10% are produced at pH 4. This document, however, suggests the addition of formate or thiourea to baths as a remedy and thereby differs in its composition from the one used in connection with the claimed method. Consequently, this known solution to the basic problem of the patent in suit is completely different from the subject-matter of Claim 1.

Document (8), on its page 359, third paragraph, discloses the deposition of a nickel plating with 10 to 12 per cent phosphorus on a steel substrate at a pH value of 4.8. The saturated carboxylic acid used when carrying out this known plating method does not meet the requirements of Claim 1 (page 358, the first two paragraphs under Figure 1). Document (8) discloses, however, that the coatings deposited by this known method "are characterised

by residual tensile stresses originating during their deposition" (page 362, last paragraph) and does not even exclude platings with phosphorus contents of at least 10 per cent from this drawback. On the contrary, cold working is suggested to compensate the residual tensile stresses. The interdependence between pH value, plating rate and phosphorus content is not at all revealed in this document.

Document (14), its "Auslegeschrift" version (14') and its family members (1) and (13), are mainly concerned with the stability of the plating bath in particular when used in connection with an aluminum substrate and do not even mention which phosphorus content in the nickel layer is aimed at. The saturated carboxylic acid used in this known bath does not meet the specification of the claimed method. Sulphur in low oxidation states is disclosed as a highly recommended, although optional, stabiliser. The Board can, therefore, not recognise any reason, why a skilled person should take just one component (maleic acid) from the specific bath composition disclosed in document (14) and replace a particular component (sodium oleate) in a different bath composition disclosed in document (14a) and use this bath in a method to produce nickel platings with a phosphorus content of at least 10%.

The documents (15) and (16) and the other documents considered during the examination proceedings lie even farther away from the subject-matter of Claim 1.

The Board, therefore, comes to the conclusion that the subject-matter of Claim 1 cannot be derived in an obvious manner from the documents cited by the Respondents and must accordingly be seen as involving an inventive step in the meaning of Article 52(1) in combination with Article 56 EPC.

8. The independent Claim 1, together with the dependent Claims 2 to 13 and the revised description adapted thereto, can, therefore, form the basis for maintaining the patent as amended.

Order

For these reasons, it is decided that:

1. The decision under appeal is set aside.

2. The case is remitted to the first instance with the order to maintain the patent as amended according to the request of the proprietor:

   Claims 1 to 13 and description, pages 2 and 3, filed during the oral proceedings of 9 February 1993, and rest of the description as granted.

The Registrar

S. Fabiani

The Chairman

G. Szabo

1-4-93

01191