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Datasheet for the decision of 2 October 2014

Case Number: T 2135/10 - 3.4.03

99303256.4 Application Number:

Publication Number: 0984672

IPC: H05K3/38

Language of the proceedings: ΕN

Title of invention:

Process for treating metal surfaces

Patent Proprietor:

MacDermid, Incorporated

Opponent:

Atotech Deutschland GmbH

Headword:

Relevant legal provisions:

EPC 1973 Art. 54(1), 56 EPC Art. 123(2)

Keyword:

Novelty (yes) Inventive step (no) main request (patent as granted) and first auxiliary request Inventive step (yes) - second auxiliary request

Decisions cited:

Catchword:



Beschwerdekammern Boards of Appeal Chambres de recours

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Case Number: T 2135/10 - 3.4.03

D E C I S I O N
of Technical Board of Appeal 3.4.03
of 2 October 2014

Appellant: Atotech Deutschland GmbH

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Representative: Wonnemann, Jörg

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Respondent: MacDermid, Incorporated

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Decision under appeal: Decision of the Opposition Division of the

European Patent Office posted on 19 August 2010 rejecting the opposition filed against European patent No. 0984672 pursuant to Article 101(2)

EPC.

Composition of the Board:

Chairman G. Eliasson Members: R. Bekkering

P. Mühlens

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Summary of Facts and Submissions

I. The appeal is against the rejection of the opposition against European patent EP 0 984 672.

The opposition was filed against the patent as a whole. The grounds for opposition invoked were lack of novelty and lack of inventive step, Articles 100(a), 52(1), 54 and 56 EPC.

- II. As requested by both the appellant opponent and the respondent patent proprietor, oral proceedings were held before the board on 2 October 2014.
- III. The appellant opponent requested that the decision under appeal be set aside and that the patent be revoked.
- IV. The respondent patent proprietor requested that

Main request:

the appeal be dismissed (maintenance of the patent as granted),

First auxiliary request:

the patent be maintained in amended form on the basis of claims 1 to 16 of the first auxiliary request filed with letter dated 6 May 2011,

Second auxiliary request:

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the patent be maintained in amended form in the following version:

- Claims: Nos. 1 to 16 of the second auxiliary request filed with letter dated 6 May 2011;
- Description: Pages 2 to 7 filed in the oral proceedings.
- V. Claim 1 as granted reads:
 - "A process for treating a metal surface, said process comprising:
 - (a) contacting the metal surface with an adhesion-promoting composition comprising:
 - 1. an oxidizer;
 - 2. an acid;
 - 3. a corrosion inhibitor;
 - 4. optionally, a source of halide ions;
 - 5. optionally, a water soluble polymer; and thereafter;
 - (b) contacting the metal surface with an alkaline solution."

Claim 19 as granted reads:

- "A process for bonding a metal surface to a polymeric layer, said process comprising:
- (a) contacting the metal surface with an adhesion-promoting composition comprising:
- 1. an oxidizer;
- 2. an acid; and
- 3. a corrosion inhibitor; thereafter

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- (b) contacting the metal surface with an alkaline solution; and thereafter
- (c) bonding the polymeric layer to the metal surface."
- VI. Claim 1 according to the respondent's first auxiliary request corresponds to claim 1 as granted with the following feature added at the end of the claim:

"wherein said alkaline solution is an aqueous solution of at least one alkali metal hydroxide selected from the group consisting of sodium hydroxide and potassium hydroxide."

Claim 15 according to the respondent's first auxiliary request corresponds to claim 19 as granted with the same feature above added to the claim.

VII. Claim 1 according to the respondent's second auxiliary request corresponds to claim 1 as granted with the following feature added at the end of the claim:

"wherein said alkaline solution is an aqueous solution of 25 to 50 grams per liter of an alkali metal hydroxide selected from the group consisting of sodium hydroxide and potassium hydroxide."

Claim 15 according to the respondent's second auxiliary request corresponds to claim 19 as granted with the same feature above added to the claim.

VIII. Reference is made to the following documents:

D1: WO 96/19097 A

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D9: Hartinger L., "Handbuch der Abwasser- und Recyclingtechnik für die metallverarbeitende Industrie", 2nd edition, Hanser, 1991, pages 429 to 432

D10: Jelinek T. W., "Praktische Galvanotechnik", 5th edition, 1997, pages 208 to 211

IX. The appellant opponent submitted in substance the following:

From document D1 a process for treating a metal surface was known, comprising contacting the metal surface with an adhesion-promoting composition comprising an oxidizer, an acid and a corrosion inhibitor. Moreover, document D1 indicated that it might be desirable to follow the adhesion promoting step with a rinse step. As it was generally known to the skilled person that a rinse with an alkaline solution was the most effective way of removing any acid residues, it was implicit to the skilled person that the rinse step in D1 was with an alkaline solution. Accordingly, the subject-matter of claim 1 as granted was not new over document D1.

Moreover, the subject-matter of claim 1 as granted lacked an inventive step. The experimental data provided by the respondent patent proprietor were not conclusive in showing that the step of contacting the surface with an alkaline solution improved the peel strength of a polymeric layer bonded to the surface. In fact, experimental data provided by the appellant showed that no such effect was present. The objective problem to be solved relative to D1, thus, was to provide an alternative to a rinse with water. As the skilled person was aware, based on his common general knowledge of basic chemistry as exemplified by

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documents D9 and D10, that an alkaline solution was particularly effective for removing acid residues, it was obvious to use an alkaline rinse in the process of D1.

The amendments to the claim 15 of the respondent's first auxiliary request introduced subject-matter which extended beyond the content of the application as filed, contrary to Article 123(2) EPC. Moreover, since sodium hydroxide and potassium hydroxide were commonly used in alkaline solutions, also the subject-matter of claim 1 according to the respondent's first auxiliary request lacked an inventive step.

Also the amendments to the claim 15 of the respondent's second auxiliary request introduced subject-matter which extended beyond the content of the application as filed, contrary to Article 123(2) EPC. Moreover, since the concentration range of sodium hydroxide and potassium hydroxide claimed did not produce any particular effect, also the subject-matter of claim 1 according to the respondent's second auxiliary request lacked an inventive step.

X. The respondent patent proprietor argued in substance as follows:

Document D1 only disclosed a rinse with water or no rinse. Moreover, at any rate other rinse solutions such as organic solutions were generally available. Accordingly, a rinse with an alkaline solution was not implicit from D1. The subject-matter of claim 1 was, thus, new over D1.

Moreover, the subject-matter of claim 1 as granted also involved an inventive step. The step of contacting the

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surface with an alkaline solution provided an improvement in the peel strength when bonding a polymeric layer to the surface. This was shown by the experimental data provided. There was nothing in D1 or elsewhere suggesting that this increase in peel strength and its resulting reduction in flaws in multilayer boards could be achieved by the use of an alkaline solution.

Claim 1 according to the first auxiliary request further limited the alkaline solution. The subject-matter of this claim was, thus, not obvious.

Claim 1 according to the second auxiliary request further limited the solution to a specific strong alkaline solution. The experimental data clearly showed that this produced an improvement in peel strength. There was nothing in D1 or elsewhere suggesting the use of the claimed alkaline solution to this end.

Reasons for the Decision

- 1. The appeal is admissible.
- 2. Respondent's main request (patent as granted)
- 2.1 Novelty

The appellant has opposed the patent *inter alia* on the ground that the subject-matter of claims 1 and 19 of the patent as granted lacks novelty over document D1, pursuant to Article 100(a) EPC 1973.

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Document D1 relates, like the patent in suit, generally to the fabrication of printed circuit multi-layer boards consisting of metal foils bonded to polymeric layers.

In particular, document D1 discloses, using the terminology of claim 1, a process for treating a metal surface, said process comprising:

- (a) contacting the metal surface with an adhesion-promoting composition comprising:
- an oxidizer (hydrogen peroxide)
- 2. an acid
- 3. a corrosion inhibitor

(cf page 5, lines 16 to page 13, line 13; claim 1).

This is, as a matter of fact, undisputed between the parties.

Contested is whether the remaining feature of claim 1 of,

"and thereafter,

(b) contacting the metal surface with an alkaline solution"

is known from D1.

According to D1, "As mentioned above, the present invention avoids the use of multi-step microetch processes which require additional steps [...] between a microetch step and the subsequent PCB manufacturing step in which a polymeric layer is applied to the copper, including alkali dips, oxide and reducer steps.

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Although it may be desirable to follow the adhesion promotion step with a rinse step, it is often adequate to rinse just with water. The treated surface is optionally subsequently dried. According to a preferred embodiment of the process a polymeric material is subsequently adhered to the micro-roughened surface with no intermediate steps between the adhesion promotion step and the adhesion of the polymeric material, or with a single rinse and/or drying step." (page 12, lines 3 to 17).

In the board's view, and as argued by the appellant, it is clear from this passage that following the treatment with the adhesion-promoting composition there is either:

- no rinse
- a rinse with just water
- a rinse with not just water.

The appellant essentially argued that in the latter case, since the treatment involved an acid, it was implicit that the rinse involved an alkaline solution in order to neutralise the acid. Based on his common general knowledge as exemplified by document D9 (cf page 430, table 6-1) and D10 (cf page 210, chapter 7.5.3, second paragraph), the skilled person would know that a rinse with an alkaline solution was the most effective way of removing any acid residues from the surface.

However, as essentially argued in the decision under appeal and by the respondent, as other rinse solutions, such as organic solvents, are available and could be employed, the use of an alkaline solution in the

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board's judgement is not implicit, ie directly and unambiguously derivable, from document D1.

Accordingly, the subject-matter of claim 1 is new over document D1.

The subject-matter of claim 19 is also new over D1 for essentially the same reasons.

The subject-matter of claims 1 and 19 is also new over the remaining available, more remote prior art.

2.2 Inventive step

2.2.1 The patent was, moreover, opposed on the ground that the subject-matter of claims 1 and 19 of the patent as granted lacked an inventive step, in particular over document D1, pursuant to Article 100(a) EPC 1973.

The respondent argued that the technical effect of contacting the metal surface with an alkaline solution was an improvement in the peel strength of the polymer laminate to the metal surface. This was disclosed in the patent description and shown by examples 1 and 2 in the patent.

Moreover, the respondent provided experimental data relating to the peel strength between treated copper panels and pre-preg foils with no post-treatment and with an alkaline post-treatment, using an alkaline solution ranging from mildly to strongly alkaline (cf declaration of Mr J. Cordani, submitted with letter dated 7 June 2010).

The appellant also provided comparative experimental data relating to the peel strength of laminated copper

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foils with a water rinse post-treatment and with a mildly alkaline post-treatment (cf letter dated 4 May 2010). From these data, the appellant concluded that mildly alkaline post-treatment solutions did not show any technical effect as compared to a water rinse.

2.2.2 However, from the examples provided in the patent it is not clear that an improvement in peel strength is consistently achieved as a result of a treatment with an alkaline solution. In fact, in example 1 in which samples were immersed in an aqueous solution of 38 q/l sodium hydroxide the peel strength, measured after 20 seconds solder immersion, was lower than in the comparative example in which samples were only rinsed and dried. It is, moreover, unclear how the measurements results are distributed over the range of values provided. It is, furthermore, also unclear which measurement errors apply to the indicated values. indicated by the appellant, measurements of peel strength are complicated and prone to be subject to relatively large measurements errors. This casts doubts on whether the marginal improvement in peel strength in some of the other samples is meaningful.

Also from the experimental data provided by the respondent it is not clear that an improvement in peel strength is consistently achieved as a result of a treatment with an alkaline solution. In fact, in about 30% of all tests no improvement or even a deterioration is attained.

The appellant argued that it was clear from the data that overall an improvement in peel strength was achieved. In the board's view, however, the data fail to prove that the improvement in peel strength is caused by the treatment with an alkaline solution. The

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high level of "noise" in the data rather indicate that other parameters play a predominant role in the peel strength, which are neither controlled nor documented in the test series provided, leading to the scattered measurement values.

In the board's view, the data provided by the appellant, with the same reservations as to their validity in view of the absence of any indication of measurement distributions and errors, show that the mildly alkaline solution as indicated produces no improvement in peel strength.

Accordingly, the respondent's contention that the technical effect of contacting the metal surface with an alkaline solution was an improvement in the peel strength is unsupported. This effect can, therefore, not serve as a basis for defining the objective problem to be solved relative to document D1.

- 2.2.3 As discussed above, in D1, following the treatment with the adhesion-promoting composition, there is either:
 - no rinse
 - a rinse with just water
 - a rinse with not just water.

The objective problem to be solved relative to D1, thus, is finding a suitable rinse in case it is not just with water.

In the board's judgement, and as essentially argued by the appellant, to a person skilled in the art, familiar with the chemical processes at issue in the present case, since the preceding treatment is with a solution containing an acid, it would be obvious to use a rinse - 12 - T 2135/10

with an alkaline solution, as the skilled person would know based on his common general knowledge, as exemplified by documents D9 and D10, and in fact that of basic chemistry, that a chemical rinsing by means of a neutralising reaction between acid and base would be particularly effective.

In particular, document D9 is concerned with rinsing technology in the context of waste water and recycling technology for the metal processing industry. As an example, a chemical rinse with an alkaline solution (NaOH) is used after an acidic copper etch (cf page 430, table 6-1, second item).

Document D10 is concerned with electroplating. In the context of pre- and intermediate treatments of the products, rinsing, pickling and neutralising processes are discussed. In particular, it is indicated that for neutralising acidic electrolyte residues diluted alkaline solutions are used (cf page 210, chapter 7.5.3., second paragraph).

2.2.4 The respondent argued that a number of rinse solutions were available and that there was nothing to suggest using an alkaline solution in the process of document D1. Moreover, document D1 disclosed rinsing and not neutralising, so that there was nothing to suggest using a solutions with reagents. In fact, document D1 used a different terminology where reagents were used. On page 2, lines 15 to 19, reference was made to a caustic pre-rinse as distinguished from a rinsing step. On page 12, line 3 to 9, reference was made to alkali dips. Furthermore, D1 avoided the use of multi-step microetch processes which required additional steps between a microetch step and the subsequent PCB manufacturing step in which a polymeric layer was

applied to the copper, including alkali dips, oxide and reducer steps. Moreover, in the absence of any information that an alkaline solution would not destroy the carefully prepared adhesion promoting surface, the skilled person would not use such a solution. Moreover, the relevance of documents D9 and D10 was questioned as they related to different technical fields.

2.2.5 These arguments are, however, not found convincing. As discussed above, in the board's view, D1 discloses as one possibility, a rinse with not just water and, thus, with a solution containing reagents (cf page 12, lines 13 to 17). It is thus clear that the term "rinse" is used here in a broader sense than in the discussion of the prior art. Moreover it is clear that the alkali dips referred to in D1 as step of the prior art process are distinct from the rinse step explicitly included as part of the embodiment discussed in D1. In the board's view, moreover, there is no reason why the skilled person should be wary of applying a rinse with an alkaline solution to the treated metal surface. There is nothing suggesting that the prepared surface would be affected by (very) mildly alkaline solutions encompassed by claim 1. If anything, the skilled person would rather be wary of leaving any acid residues on the surface, etching the surface over time.

Finally, as regards the relevance of documents D9 and D10, the board agrees with the respondent that the chemical processes discussed in these documents are different from that of claim 1. Nevertheless, these documents both confirm that acidic residues are effectively removed with a rinse with an alkaline solution, a fact the skilled person would at any rate already know from his knowledge of basic chemistry.

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Accordingly, having regard to the state of the art, the subject-matter of claim 1 as granted is obvious to a person skilled in the art and, therefore, lacks an inventive step (Article 56 EPC 1973).

The same applies, *mutatis mutandis*, to the subject-matter of claim 19, the additional feature (c) of claim 19 being known from document D1 (cf claim 14)

Accordingly, the ground of opposition under Article 100(a) EPC 1973 invoked by the appellant prejudices the maintenance of the patent as granted, Article 101(2) EPC, first sentence.

3. Respondent's first auxiliary request

3.1 Amendments

Claim 1 according to the respondent's first auxiliary request corresponds to claim 1 as granted with the following feature added at the end of the claim:

"wherein said alkaline solution is an aqueous solution of at least one alkali metal hydroxide selected from the group consisting of sodium hydroxide and potassium hydroxide."

Claim 15 according to the respondent's first auxiliary request corresponds to claim 19 as granted with the same feature above added to the claim.

The appellant argued that the amendment of claim 15 introduced subject-matter which extended beyond the content of the application as filed, contrary to Article 123(2) EPC.

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In particular, the feature added to claim 15 was disclosed in the application as originally filed in claims 15 and 16, which, however, were dependent on claim 1. The combination of these features with those of claim 19 as granted was not disclosed in the application as filed.

In the board's judgement, however, the amendment of claim 15 does not introduce subject-matter which extends beyond the content of the application as filed, if not just the claims as originally filed with their respective dependencies are considered, but due account is taken also of the description as originally filed, thereby considering what the application as a whole discloses to a skilled person.

The subject-matter of claim 15 as amended is considered to be derivable from the description of the embodiment of the invention (cf pages 11 to 17 as originally filed), with the features of the process as claimed being taken in particular from page 11, line 1 to page 13, line 2 and page 16, line 9 to page 17, line 8.

The remaining claims are also considered to be duly based on the application as filed.

Accordingly, the amendments to the claims according to the respondent's first auxiliary request meet the requirement of Article 123(2) EPC.

3.2 Inventive step

Aqueous solutions of sodium hydroxide and potassium hydroxide are among the most commonly used alkaline solutions, as would be known to the person skilled in the art based on his common general knowledge.

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Reference is also made to document D9 (page 430, table 6-1) referring to a rinse with a solution of sodium hydroxide.

Accordingly, it would be obvious for a person skilled in the art charged with the problem to be solved of finding an appropriate solution for the rinse in the process of document D1 to resort to these commonly used solutions, the use of an alkaline solution being obvious for the reasons given above with respect to the respondent's main request.

Accordingly, having regard to the state of the art, also the subject-matter of claim 1 according to the respondent's first auxiliary request is obvious to a person skilled in the art and, therefore, lacks an inventive step (Article 56 EPC 1973).

The same applies, mutatis mutandis, to the subjectmatter of claim 15.

The respondent's first auxiliary request is, thus, not allowable.

4. Respondent's second auxiliary request

4.1 Amendments

Claim 1 according to the respondent's second auxiliary request corresponds to claim 1 as granted with the following feature added at the end of the claim:

"wherein said alkaline solution is an aqueous solution of 25 to 50 grams per liter of an alkali metal hydroxide selected from the group consisting of sodium hydroxide and potassium hydroxide."

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Claim 15 according to the respondent's second auxiliary request corresponds to claim 19 as granted with the same feature above added to the claim.

Essentially as for the respondent's first auxiliary request above, the appellant argued that the amendment of claim 15 introduced subject-matter which extended beyond the content of the application as filed, contrary to Article 123(2) EPC.

In particular, the feature added to claim 15 was disclosed in the application as originally filed in claims 15 and 16, which, however, were dependent on claim 1. The combination of these features with those of claim 19 as granted was not disclosed in the application as filed.

However, as argued above for the respondent's first auxiliary request above, the amendment of claim 15 does not introduce subject-matter which extends beyond the content of the application as filed, if due account is taken also of the description as originally filed, thereby considering what the application as a whole discloses to a skilled person.

The subject-matter of claim 15 as amended is considered to be derivable from the description of the embodiment of the invention (cf pages 11 to 17 as originally filed), with the features of the process as claimed being taken in particular from page 11, line 1 to page 13, line 2 and page 16, line 9 to page 17, line 8.

The remaining claims are also considered to be duly based on the application as filed.

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Accordingly, also the amendments to the claims according to the respondent's second auxiliary request meet the requirement of Article 123(2) EPC.

4.2 Inventive step

4.2.1 Having regard to claim 1 of the respondent's first auxiliary request above, by specifying that the solution contains 25 to 50 grams per liter of sodium hydroxide or potassium hydroxide, claim 1 of the respondent's second auxiliary request requires the use of a strong alkaline solution.

Whereas in the board's judgement it is obvious to a person skilled in the art to use a mildly alkaline solution in the rinse step following the adhesion promotion step in D1, as discussed above, this is not so for a strong alkaline solution.

Since according to D1 a rinse may be desirable, but clearly is not indispensable and may in fact be omitted, and thus apparently, if anything, only residues of the acid solution would need removal from the surface, the skilled person would only consider the use of mildly alkaline solutions. There would be no reason to use a strong alkaline solution as now claimed and indeed, as argued by the respondent, the skilled person would be wary to use any such strong alkaline solution on the carefully prepared surface as a substantial alteration or damage to it may be reasonably expected. Clearly, where D1 refers to a rinse, no step substantially altering the adhesion promoting micro-roughened surface is intended.

Accordingly, from this angle the use of a strong alkaline solution as claimed is not obvious.

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4.2.2 The respondent argued that both the examples in the patent and the experimental data of Mr. Cordani showed that a post dip with such a strong alkaline solution produced an improvement in peel strength. In the latter experimental data, reference was made to the measurements with a solution at 5% and a pH value of 12.7, generally showing a substantial improvement in peel strength. The experimental data provided by the appellant were not relevant as they related to the treatment with a mildly alkaline solution of sodium carbonate with a pH of only about 9 to 9.5.

The appellant argued that from the experimental data provided by the respondent there was no effect noticeable for the specific selection of the concentration range as claimed. The selection was thus arbitrary and, therefore, could not support the presence of an inventive step.

4.2.3 In the board's view, as discussed above the respondent's experimental data do not conclusively demonstrate that the post dip causes an improvement in peel strength. The noise in the data rather suggests that some other undocumented factor plays a role in these experiments.

The experimental data provided by the appellant, on the other hand, do not disprove the alleged correlation either, as they do not relate to a strong alkaline solution as claimed.

Notwithstanding the above, the examples provided in the patent, using a strong alkaline solution with 38 g/l sodium hydroxide show that wedging, pink ring formation and to some extent resin voids are avoided. These are

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defects which typically arise when through holes are drilled in the printed circuit multi-layer boards and the boards are further processed (cf patent, paragraph [0015]).

Leaving aside whether an increase in peel strength is responsible for reducing these defects, the problem to be solved relative to D1 can also be formulated to be to reduce the occurrence of these defects.

There is, however, nothing in D1 or elsewhere suggesting contacting the metal surface with a strong alkaline solution as claimed for reducing the occurrence of these defects.

Accordingly, also from this angle contacting the metal surface with a strong alkaline solution as claimed is not obvious.

The subject-matter of claim 1 is also not obvious with respect to the remaining available, more remote prior art.

Accordingly, having regard to the state of the art, the subject-matter of claim 1 according to the respondent's second auxiliary request is not obvious to a person skilled in the art and, therefore, involves an inventive step (Article 56 EPC 1973).

The same applies, *mutatis mutandis*, to the subject-matter of claim 15.

The remaining claims 2 to 14 and 16 are dependent on claims 1 and 15, respectively, and provide further limitations. The subject-matter of these claims, therefore, also involves an inventive step.

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- 4.3 The description has been duly adapted to these claims.
- 4.4 Accordingly, taking into consideration the amendments made by the respondent patent proprietor in accordance with its second auxiliary request, the patent and the invention to which it relates meet the requirement of the EPC, pursuant to Article 101(3)(a) EPC.

Order

For these reasons it is decided that:

- 1. The decision under appeal is set aside.
- 2. The case is remitted to the department of first instance with the order to maintain the patent in amended form in the following version:

Description:

Pages 2 to 7 filed in the oral proceedings on 2 October 2014;

Claims:

Nos. 1 to 16 according to the respondent's second auxiliary request filed with letter dated 6 May 2011.

The Registrar:

The Chairman:



S. Sánchez Chiquero

G. Eliasson

Decision electronically authenticated