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**Datasheet for the decision
of 20 March 2018**

Case Number: T 0187/14 - 3.2.03

Application Number: 01998669.4

Publication Number: 1347075

IPC: C23C8/52

Language of the proceedings: EN

Title of invention:

METHOD OF SALT BATH NITRIDING FOR PRODUCING IRON MEMBER HAVING
IMPROVED CORROSION RESISTANCE AND IRON PARTS

Patent Proprietor:

Parker Netsushori Kogyo K.K.

Opponent:

H.E.F.

Headword:

Relevant legal provisions:

EPC Art. 54, 56

Keyword:

Novelty - (yes) - implicit disclosure (no)
Inventive step - non-obvious alternative

Decisions cited:

Catchword:



Beschwerdekammern

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Case Number: T 0187/14 - 3.2.03

D E C I S I O N
of Technical Board of Appeal 3.2.03
of 20 March 2018

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Decision under appeal: Interlocutory decision of the Opposition
Division of the European Patent Office posted on
27 November 2013 concerning maintenance of the
European Patent No. 1347075 in amended form.

Composition of the Board:

Chairman G. Ashley
Members: B. Miller
D. Prietzel-Funk

Summary of Facts and Submissions

- I. European patent No. 1 347 075 relates to a method of salt bath nitriding for iron or steel parts.
- II. An opposition was filed against the patent, based on the grounds of Article 100(b) and (c) EPC and of Article 100(a) EPC together with both Articles 54 and 56 EPC.

In the interlocutory decision the opposition division found that the contested patent, on the basis of the first auxiliary request filed during oral proceedings in the opposition proceedings, met the requirements of the EPC.

The appellant (opponent) filed an appeal against this decision.

- III. The appellant requested that the decision under appeal be set aside and the patent be revoked. In the statement setting out the grounds of appeal, the appellant challenged novelty and inventive step.
- IV. The respondent requested that the appeal be dismissed. Therefore the first auxiliary request found allowable in the impugned decision forms the main request in the appeal proceedings.
- V. The independent claims according to the main request read as follows:

"1. A nitriding process of iron and steel parts having an improved corrosion resistance by immersing the iron and steel parts in a melting salt bath containing

cationic components of Li^+ , Na^+ and K^+ and anionic components of CNO^- and CO_3^{2-} , wherein an iron-lithium complex oxide layer is formed at the outermost surface of the iron and steel parts simultaneously with forming a nitrided layer on a surface of the iron and steel parts by adding at least one hydroxide being selected from lithium hydroxide, sodium hydroxide and potassium hydroxide to the melting salt bath.

"2. A nitriding process of iron and steel parts having an improved corrosion resistance by immersing the iron and steel parts in a melting salt bath containing cationic components of Li^+ , Na^+ and K^+ and anionic components of CNO^- and CO_3^{2-} , wherein an iron-lithium complex oxide layer is formed at the outermost surface of the iron and steel parts simultaneously with forming a nitrided layer on a surface of the iron and steel parts by adding a salt bath material comprising a hydrate or free water for supply of the components to the melting salt bath wherein the process does not employ an electrolysis."

"3. A nitriding process of iron and steel parts having an improved corrosion resistance by immersing the iron and steel parts in a melting salt bath containing cationic components of Li^+ , Na^+ and K^+ and anionic components of CNO^- and CO_3^{2-} , wherein an iron-lithium complex oxide layer is formed at the outermost surface of the iron and steel parts simultaneously with forming a nitrided layer on a surface of the iron and steel parts by using air having an absolute moisture content of more than $(1 \times 10^{-2} \text{ kg H}_2\text{O}) / (1 \text{ kg dry air})$ for air bubbling for mixing the melting salt bath."

Claims 4 to 6 of the main request relate to preferred embodiments of the process according to claims 1 to 3.

VI. State of the art

The following documents were cited by the appellant:

- D1: JP45 8771;
D1': English translation of D1;
D2: US 3 847 685;
D3: US 3 912 547;
D4: US 3 915 759;
D5: FR 2 280 715;
D6: FR 2 708 623;
D7: EP 0 667 401;
- A: "Quest-ce que le climat ?", Centre de Recherche et de Restauration des Musées de France, Département Conservation préventive Environnement - Climat fiche 1, pages 1 to 5;
B: "Confort thermique, Température et Humidité", page 1, retrieved from the Internet:
[URL:www.medecine-preventive.ch/fra/humidite.php](http://www.medecine-preventive.ch/fra/humidite.php);
B': "Humidité de l'air", retrieved from the Internet:
[URL:www.wikidelta.com/deltaplane, meteorologie/humidite-de-lair.html](http://www.wikidelta.com/deltaplane,meteorologie/humidite-de-lair.html);
B": "Observation de l'humidité relative", retrieved from the Internet:
[URL:www.meteociel.fr/observations-meteo/humi.php?archieve=1&jour=4&mois=3&annee=2014&heures=17&mode=&sub=OK](http://www.meteociel.fr/observations-meteo/humi.php?archieve=1&jour=4&mois=3&annee=2014&heures=17&mode=&sub=OK);
C: US 2 960 421;
D: "Préparation métallographiques des composants nitrurés et carbonitrurés", Struers, pages 1 to 6;
B1': WO 02/44438, claims 1 to 4.

- VII. With the summons to oral proceedings, the Board sent a communication pursuant to Articles 15(1) and 17(2) of the Rules of Procedure of the Boards of Appeal (RPBA) indicating to the parties its preliminary, non-binding opinion of the case.
- VIII. Oral proceedings were held on 20 March 2018. During the oral proceedings the appellant confirmed that it pursues the revocation of the patent solely based on the ground of opposition pursuant to Article 100(a) EPC in combination with both, Article 54 and 56 EPC. At the end of the oral proceedings the Board pronounced its decision.
- IX. The arguments of the appellant can be summarised as follows.

D3 disclosed a method of nitriding which inherently achieved the formation of an iron-lithium complex oxide layer. The molten salt bath used according to D3 comprised free water generated by reaction of the regenerating agent urea. Moreover, the salts used according to D3 were not dried and consequently contained free water and hydrates. Therefore claim 2 lacked novelty over D3. The relative moisture content of air in France was in general above 70 % and therefore the compressed air used according to D3 had a moisture content as required by claim 3.

D6 disclosed a method of nitriding which lead to the formation of an iron-lithium complex oxide layer. D6 made it clear, that electrolysis was not absolutely required to form the complex oxide layer. Water was generated by reactions taking place in the salt bath. This free water introduced into the salt bath of D6

inherently formed hydroxides. Moreover, the air used for bubbling the salt bath inherently had a moisture content as required by claim 3.

Therefore claims 1 to 3 lacked novelty in view of D6.

Starting from D3 as the closest prior art it was obvious

- to add hydroxides for their well-known effects described in D1, D2, D4 and D5,
 - to arbitrarily adjust the moisture content of the air used to bubble the salt bath and
 - to use salts containing hydrates
- in order to promote the formation of an iron-lithium complex oxide layer.

D6 disclosed a method for obtaining an iron-lithium complex oxide layer and therefore solved the problem underlying the contested patent. D6 suggested that electrolysis was not absolutely necessary to form the complex oxide layer. Arbitrarily adjusting the moisture content of the air used to bubble the salt bath and adding hydroxides for their well-known effects was obvious for the skilled person.

- X. The respective arguments of the respondent can be summarised as follows.

D3 neither disclosed that the nitriding process formed an iron-lithium complex oxide layer nor described the process steps leading to its formation as defined in claims 1 to 3.

D6 disclosed that it was essential to use electrolysis for forming an iron lithium complex oxide. No other method for obtaining the iron-lithium complex oxide formation was disclosed or at least suggested by D6.

Therefore the subject-matter of claims 1 to 3 was novel over D3 and D6.

The skilled person had no reason to assume, that the process described in D3 could achieve not only a nitrated iron layer but also an iron-lithium complex oxide layer. Moreover, none of the further documents provided the teaching that by increasing the oxidation strength of the salt bath, a further iron-lithium complex oxide layer could be formed at the same time as an iron nitride layer.

D6 did not provide any hint that an iron lithium complex oxide could be formed without electrolysis. This teaching was also not derivable from any other document.

Increasing the oxidation strength of a salt bath while performing electrolysis technically did not make sense. Therefore the skilled person would neither add hydroxides to the salt bath of D6 nor use air having a relatively high moisture content for bubbling the salt bath.

Reasons for the Decision

1. Article 54 EPC
- 1.1 Concerning D3
- 1.1.1 D3 relates to a nitrating process of ferrous metals by immersing the parts in a molten salt bath comprising carbonate and cyanate anions and Li^+ , Na^+ and K^+

cations. The presence of Li^+ cations in conjunction with Na^+ and/or K^+ cations increases the fluidity of the bath, see column 1, lines 46 to 51. According to the examples of D3, the alkali compounds are present in the form of carbonates, see column 4, lines 37 to 45. The immersion of ferrous parts in the salt bath results in a "white microlayer", see claim 1 and examples.

- 1.1.2 The appellant argues that the term "white microlayer" is used in general for a coating of the nitriding process and does not imply that the layer has to have a white appearance. Furthermore, it argues that any composition comprising carbonate ions, cyanate ions, Li^+ , Na^+ and K^+ cations inevitably leads to the formation of an iron-lithium complex oxide layer.

However, D3 does not disclose that the nitrided steel has a black colour, which is a characteristic for the formation of an iron-lithium complex oxide layer (paragraphs [0038] and [0048] of the contested patent).

The layer defined as "white microlayer" in D3 might not have a completely white appearance, as argued by the appellant, but this does not inevitably lead to the conclusion that in D3 the "white microlayer" refers to an iron-lithium complex oxide layer that is characterized by a distinctive black or grey appearance.

Moreover, example III of D3 describes that the "white microlayer" has "a base mainly of carbides, nitrides and sulphides of iron". A further ingredient may be phosphorus, see column 2, lines 17 to 20 and example VI. Lithium is not mentioned at all as being a component of the "white microlayer" for any of the examples of D3.

It is therefore concluded that there is no clear disclosure that the "white microlayer" obtained by the examples of D3 is composed of an nitrated layer and an iron-lithium complex oxide layer on the outer surface.

1.1.3 In addition, no disclosure can be found in D3 for the specific process steps defined in claims 1 to 3 of the disputed patent, which lead to the formation of the iron-lithium oxides on the iron nitride layer.

(a) Claim 1 requires that at least one hydroxide selected from LiOH, NaOH and KOH is added to the salt bath.

This process step is not disclosed in D3.

(b) Claim 2 defines that a salt component comprising a hydrate or free water is added.

During the process of D3 water can be generated in the salt bath by the reaction of the regenerating agent urea (column 2, lines line 76 to column 3, line 2).

However, the generation of water as a result of a chemical reaction in the salt bath does not correspond to a process wherein water is purposively added in the form of a hydrate or free water of a salt.

The appellant argues that D3 does not describe that the salts are dried and concluded that they have to comprise free water or hydrates, which would thus be added to the bath.

The Board observes in this context that paragraph [0051] of the contested patent explains that the addition of the moisture enhances the oxidizing activity of the salt bath by shifting the basicity. Thus, it is evident that a minimum amount of moisture is necessary to achieve the required enhancement of the oxidizing activity leading to the formation of the iron-lithium complex oxide layer.

However, D3 does not provide any disclosure that the amount of water generated by the reaction of urea or possibly present as hydrate or free water of the salt is sufficient to lead to the formation of an iron-lithium complex oxide layer.

Therefore the subject-matter of claim 2 differs from the disclosure of D3 in that the process leads to the formation of an iron-lithium complex oxide by adding water in the form of a hydrate or free water of a salt.

- (c) Claim 3 requires that air with an absolute moisture content of more than 1×10^{-2} kg H₂O/kg dry air is used for bubbling the mixture.

This moisture content corresponds to a relative humidity in the atmosphere of at least 70% as confirmed by the appellant.

Compressed air is used according to D3 for bubbling the salt bath (column 4, lines 8 to 22). However, the moisture content of the compressed air is not disclosed in D3.

It can be accepted that the moisture content of air can be above 70 % in certain parts of France (see

documents B' and B") and that a healthy working environment has air with a moisture content between 40 and 70 % as described in document B.

However, in the absence of any disclosure concerning the moisture content of the air used according to D3, it cannot inevitably be concluded that the compressed air used according to D3 has a moisture content as defined in claim 3. The air used for the process of D3 could be dried before compression or the process could be performed in a region where the air has a lower moisture content.

1.1.4 Therefore the subject-matter of claims 1 to 3 is novel in view of D3.

1.2 Concerning D6

1.2.1 D6 describes a process of electrochemically nitriding and oxidizing of ferrous metal parts immersed in a salt bath comprising alkali metal cyanates and carbonates (claim 1, page 2, lines 23 to 36 and page 12, lines 12 to 13). The electrochemical process results in the oxidation of the parts and reaction of nitrogen with the iron of the substrates, see page 3, lines 18 to 21. The salt bath is bubbled with air (page 5, lines 22 to 24).

A "dense grey layer" is formed at the surface of the parts treated by the process of D6. According to table 2, the dense grey layer comprises $\text{Fe}_{2-4}\text{N} + \text{Fe}_3\text{O}_4 + \text{Li}_2\text{Fe}_3\text{O}_4$, or $\text{Fe}_{2-4}\text{N} + \text{Fe}_3\text{O}_4 + \text{LiFe}_5\text{O}_8$ and therefore is an iron-lithium complex oxide layer.

The appellant argues that D6 suggests on page 3, lines 15 to 26 that electrolysis is not necessary to achieve

the formation of the iron-lithium complex oxide layer on the iron nitride layer.

However, on page 3, lines 1 to 26 different possibilities for passing the current through the nitriding bath are evaluated. At lines 15 to 29 on page 3 it is indicated that if the parts are at the same potential as the counter-electrode the results are the same as for conventional nitriding, whereas if the parts are at a positive potential relative to the counter-electrode two layers are formed, an iron nitride layer and a complex oxide layer.

Hence, the teaching on page 3 of D6 is consistent with the teaching of claim 1 and of the example on page 7, lines 8 to 28 of D6 that electrolysis is essential for achieving the iron-lithium complex oxide layer, contrary to the interpretation of the appellant.

Therefore the Board concludes that D6 does not contain any disclosure that generates a doubt that electrolysis is an essential feature for forming an iron-lithium complex oxide layer.

- 1.2.2 As indicated above, the iron-lithium complex oxide layer is obtained by an electrochemical reaction according to D6 (claim 1). Claim 2 of the main request excludes electrolysis. The subject-matter of claim 1 is therefore novel.
- 1.2.3 D6 does not disclose that the salt bath comprises a hydroxide as required by claim 1.

The appellant argues

- i) that the term "adding to" has the same meaning as "using in",

- ii) that the salts used for the salt bath disclosed in D6 are not absolutely dry and therefore contain some water in the form of their hydrates or free water and
- iii) that the presence of hydrates implies that hydroxides are present.

However, a step of adding a specific hydroxide is not the same as a step of adding a component containing some moisture.

The subject-matter of claim 1 therefore differs from the process described in D6 in that hydroxide is added to the salt bath.

- 1.2.4 D6 discloses that the salt bath can be homogenised by bubbling air through the bath (page 4, lines 28 to 32). However, similar to D3 (see point 1.2.3 c) above), D6 does not disclose the moisture content of the air and therefore does not disclose that inevitably air having a moisture content of more than 1×10^{-2} kg H₂O/kg dry air is used for bubbling the salt bath as required by claim 3.

Therefore the subject-matter of claim 3 is novel over D6.

- 1.3 In summary, the Board concludes that the subject-matter of claims 1 to 3 as granted is novel over D3 and D6 and therefore fulfils the requirements of Article 54 EPC.

2. Article 56 EPC

2.1 D3 as starting point

2.1.1 The appellant argues that D3 is a suitable starting point, since it relates to a nitriding process and therefore to the same general problem as the contested patent.

2.1.2 As indicated above in point 1.1 the subject-matter of claims 1 to 3 differs from the disclosure of D3 in that the process leads to the formation of an iron-lithium complex oxide layer by

- adding lithium, sodium or potassium hydroxide (claim 1),
- adding salts comprising hydrates or free water and (claim 2) or
- bubbling the salt bath with air having a moisture content of more than 1×10^{-2} kg H₂O/kg dry air (claim 3).

2.1.3 The formation of the iron-lithium complex oxide layer provides a better abrasion resistance (example 5 of the contested patent) and corrosion resistance (paragraphs [0021] to [0026] of the contested patent).

2.1.4 The objective technical problem to be solved can be seen as to provide a nitriding process which further improves the corrosion and wear resistance of the nitride layer.

2.1.5 This problem is solved according to the main request by one of the three processes defined in claims 1 to 3.

(a) concerning claim 1

The appellant argues that the subject-matter of claim 1 is obvious when considering in addition documents D1, D2, D4 and D5.

The Board does not agree with that argument.

D2 does not relate to a nitriding process but describes a process providing an oxide coating on metal elements, such as made from stainless steel, by immersing the parts in a bath of fused alkali hydroxide which can include fused lithium, sodium or potassium hydroxide. A similar teaching can be found in D4 which is also not related to a nitriding process but to a process for forming black oxide coatings on stainless steel parts in a melt bath comprising preferably an eutectic mixture of 50 % NaOH and 50 % KOH.

Starting from D3 and aiming at an improvement of the nitriding process documents D2 and D4 therefore would not be consulted by the skilled person, since they relate to different type of processes and do not teach that an iron-lithium complex oxide layer improves the corrosion and wear resistance and how to produce it at the same time as an iron nitride layer.

D1' discloses that sodium hydroxide provides a cleaning action and the formation of a glossy surface of the nitrated article (D1', page 2, lines 28 to 29) but does not disclose that a further iron-lithium complex oxide layer can be formed by adding the hydroxide in a nitriding salt bath.

D5 proposes a way of stabilising a salt bath by adding hydroxide ions, preferably in form of alkali or

alkaline earth hydroxides (page 2, lines 4 to 12). On page 1, lines 21 to 32 it is disclosed that the stability of melting baths for nitriding ferrous substrates can be improved by the presence of lithium cations in a mixture of cyanates and carbonates. However, in the examples of D5 lithium cations are not present, neither as bath component nor as cation of the added hydroxides. Therefore D5 does not provide any incentive for the skilled person to add alkali hydroxides into the metal bath compositions proposed by D3 in order to provide an iron-lithium complex oxide layer leading to an improved wear and corrosion resistance.

In summary, none of the documents cited by the appellant provides any hint that the addition of hydroxides during the nitriding process of D3 leads to the formation of an iron-lithium complex oxide layer in addition to the iron nitride layer.

(b) concerning claim 3

It can be accepted that in certain parts of France the moisture content of air is frequently above 70 % (see documents A, B' and B'') and that in a healthy working environment air has a moisture content between 40 and 70 % as described in document B.

However, this does not provide any hint to the skilled person that the compressed air used according to D3 should have a high humidity, i.e. a moisture content of above 70 %, in order to provide in addition an iron-lithium complex oxide layer leading to an improved wear resistance.

Therefore the subject-matter of claim 3 is not considered to be obvious when starting from D3.

2.2 D6 as starting point

2.2.1 As indicated above in point 1.2.1, D6 discloses a process of electrochemically nitriding and oxidizing of ferrous metal parts immersed in a salt bath comprising alkali metal cyanates and carbonates (claim 1).

D6 therefore explicitly teaches that the iron-lithium complex oxide layer is formed by electrolysis which is a process requirement the contested patent wants to avoid (paragraph [0013]).

2.2.2 Starting from D6 the objective technical problem can be considered as the provision of an alternative process for providing an iron-lithium complex oxide layer.

2.2.3 That problem is solved according to the contested patent by adding hydroxide (claim 1), by adding moisture in the form of a salt hydrate or free water (claim 2) or by adding moisture with the air used for bubbling the salt bath (claim 3) in order to increase the oxidation strength of the salt bath (see paragraphs [0051] and [0057] of the contested patent).

(a) Concerning claim 1

The appellant argues that starting from D6 the subject-matter of claim 1 is obvious when considering either the description of the prior art on page 2, lines 4 to 9 of D6 itself or the teaching in claim 1 and the examples of D2.

D6 as well as D2 (claim 1) confirm that it is known to the skilled person that an oxidising salt bath can comprise hydroxides.

However, neither D6 itself nor D2 provides any hint that in the nitriding process of D6 using electrolysis the addition of hydroxides would be at least of an benefit, let alone that hydroxides could be added in order to form an iron-lithium complex oxide layer on the surface of an iron nitride layer in the absence of electrolysis.

Therefore the subject-matter of claim 1 is not rendered obvious by D6 when considered on its own or in combination with D2.

(b) Concerning claim 2

The appellant argues that according to the teaching of D6 electrolysis is not required to form an iron-lithium complex oxide layer.

However, as indicated above in point 1.2.1 D6 does not provide any hint that the electrolysis is not essential. Therefore the skilled person has no motivation to go against the explicit teaching in D6 (claim 1, example) and to try to achieve the iron-lithium complex oxide layer in the absence of the electrochemical reaction.

No teaching has been identified in any of the cited documents that the presence of free water or hydrates can increase the oxidation strength of the salt bath to an extent that an iron-lithium complex oxide layer is

formed in the absence of the electrochemical reaction described by D6.

Therefore the subject-matter of claim 2 is not obvious.

(c) Concerning claim 3

According to the appellant it comes within the experimental routine of the skilled person to select arbitrarily a specific moisture content of the air used for bubbling the salt bath in the process of D6.

However, the skilled person has no motivation starting from D6 to use air having a minimum moisture level for bubbling the air. On the contrary in the absence of any indication that electrolysis is unnecessary when increasing the oxidising strength of the salt bath, it is not obvious for the skilled person to use moist air for bubbling the salt bath, in particular since the addition of water in a molten salt bath is usually avoided.

Therefore the subject-matter of claim 3 is not obvious.

2.3 In summary, the Board concludes that the subject-matter of claims 1 to 3 as granted is not obvious when starting from either D3 or D6 and fulfils the requirements of Article 56 EPC.

Order

For these reasons it is decided that:

The appeal is dismissed.

The Registrar:

The Chairman:



C. Spira

G. Ashley

Decision electronically authenticated