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**D E C I S I O N**  
**of 20 June 2002**

**Case Number:** T 0600/99 - 3.2.2

**Application Number:** 91113628.1

**Publication Number:** 0505606

**IPC:** C23G 1/08

**Language of the proceedings:** EN

**Title of invention:**

Process for pickling and passivating stainless steel without using nitric acid

**Patentee:**

Henkel KGaA

**Opponents:**

CONDOROIL IMPIANTI s.r.l.  
USINOR SACILOR S.A.  
Solvay Interlox Ltd.

**Headword:**

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**Relevant legal provisions:**

EPC Art. 83, 84, 123(1)(3), 56

**Keyword:**

"Sufficiency of disclosure (yes)"  
"Inventive step (yes) after amendment"

**Decisions cited:**

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**Catchword:**

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Case Number: T 0600/99 - 3.2.2

**D E C I S I O N**  
**of the Technical Board of Appeal 3.2.2**  
**of 20 June 2002**

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**Decision under appeal:**

**Decision of the Opposition Division of the  
European Patent Office posted 9 April 1999  
revoking European patent No. 0 505 606 pursuant  
to Article 102(1) EPC.**

**Composition of the Board:**

**Chairman:** W. D. Weiß  
**Members:** R. Ries  
U. J. Tronser

## Summary of Facts and Submissions

- I. European patent No. 0 505 606 was granted on 2 November 1995 on the basis of European patent application No. 91 113 628.1.
- II. The granted patent was opposed by the present respondents (opponents I to IV) on the grounds that its subject-matter was not disclosed in a manner sufficiently clear and complete for it to be carried out by a person skilled in the art (Article 100(b) EPC) and that it did not involve an inventive step with respect to the state of the art (Article 100(a) EPC).
- III. With its decision posted on 9 April 1999, the opposition division held that the subject-matter of the claims as granted (single request) lacked an inventive step and revoked the patent.
- IV. An appeal against this decision was filed by the patentee (the appellant) on 31 May 1999. The fee for appeal was paid and the written statement setting out the grounds of appeal was filed within the time limit under Article 108 EPC.

Of the pre-published documents relied upon at the appeal stage, only the following were still discussed:

D1: JP-A-60 243 289 (English translation)

D2: P. Lacombe: "Les Aciers inoxydables" Les Editions de Physique, 1990, pages 868 to 872

D4: GB-A-2 000 196

D22: US-A-4 059 678

D23a: Ullmann's Encyclopedia of Industrial Chemistry,  
Vth ed. vo. A13, pages 461, 464

D23b: "Das Wasserstoffperoxyd und die  
Perverbindungen", Willi Machu, II. ed. 1951,  
Springer, pages 195 to 202

V. With its letter received on 7 March 2002, Opponent II  
(EKA CHEMICALS AB) informed the Board that its  
opposition was withdrawn.

VI. Oral proceedings before the Board were held on 20 June  
2002 at the end of which the requests were as follows:

- The appellant (patentee) requested that the  
decision under appeal be set aside and that the  
patent be maintained on the basis of claims 1 to 3  
submitted at the oral proceedings marked as "Main  
request (A')" and the specification column 1 to 8  
submitted at the oral proceedings.
- The respondents (opponents) requested that the  
appeal be dismissed.

Claim 1 reads as follows:

"1. A process for pickling and passivating stainless  
steel consisting of bringing the material to be treated  
into contact with a bath maintained at a temperature of  
between 30 and 70°C and preferably between 45 and 55°C,  
and having the following initial composition of the  
bath:

a)  $H_2SO_4$  at least 150 g/l

- b)  $\text{Fe}^{3+}$  at least 15 g/l
  - c) HF at least 40 g/l
  - d)  $\text{H}_2\text{O}_2$  (containing known stabilizers) 1 to 20 g/l and preferably 2 to 5 g/l
  - e) additives of the non-ionic surfactant type (emulsifiers, wetting agents, brighteners) and acid attack inhibitor type: about 1 g/l in total;
- into said bath being continuously fed:
- an air flow of at least 3 m<sup>3</sup>/h per m<sup>3</sup> of bath, using a suitable distributor device for diffusing the flow into the liquid mass;
  - a quantity of stabilized  $\text{H}_2\text{O}_2$  of between 0.3 and 1 g/l per hour, controlled on the basis of the REDOX potential of the bath, which must be maintained at \$ 350 mV;
  - and possibly sufficient quantities of ingredient e) to maintain its concentration in the bath at optimum levels;

and being periodically fed sufficient quantities of ingredients a) and c) to maintain their concentrations in the bath at optimum levels on the basis of the bath analysis, i.e. the free acid and fluoride values, and to maintain the bath pH less 1 and preferably between 0 and 0.5."

VI. The appellant argued as follows:

Amended claim 1 is supported by the description of patent specification, column 4, lines 9 to 13 and column 6, lines 22 to 29. Moreover, the proposed amendment does not broaden the scope of protection conferred by claim 1 as granted since claim 1 has been narrowed by deleting the term "possibly" with respect to the addition of ingredients a) and c) thus making this step compulsory. Hence the amendments satisfy the

requirements of Articles 123(2) and (3).

As to the selection of the appropriate hydrogen peroxide "stabilizer", the pH control and determining of REDOX potential, the skilled person is well expected to consult basic handbooks and textbooks to obtain clear advice as to what to do in the present circumstances. Handbooks such as documents D2, D23a, D23b and also D4, page 2, lines 50 to 52 reflect the classical methods and standard equipment used by a person skilled in this field of chemistry. Moreover, the acid supplementation regimen addressed in the patent makes clear that the two acids ( $H_2SO_4$  and HF) are consumed during the pickling process and, therefore, need to be replenished on basis of the bath analysis. Hence the patent specification meets the requirements of Article 83 EPC.

As Table 1 of document D4 demonstrates, the prior art has to start with a pickling solution which is poor in free acid but heavily loaded with ferric and ferrous sulphate. In contrast thereto, the claimed process, by its active management of the free acid and fluoride ions values, permits to start with a composition of the pickling bath exhibiting only a low content of ferric sulphate and no ferrous sulphate at all. This active control of the free acid and fluoride ion values, together with the air flow fed into the bath, produces a long life time of the bath without sludge formation. Since document D4 does not teach such an active control of its free acid and fluoride values, this pickling bath suffers from the drawback of early sludge formation. Hence the process set out in claim 1 involves an inventive step.



VII. At the oral proceedings, the opponents (respondents) argued as follows:

The obligatory step of feeding "periodically" sufficient quantities of ingredients a) and c) is contrary to what was claimed in claim 1 as granted where these ingredients were fed "continuously". Objection to amended claim 1, therefore, arises under Article 123(3) EPC. Given that the term "at optimum level" is not explained in the patent specification, the concentration level to be aimed at by supplying consumed HF and H<sub>2</sub>SO<sub>4</sub> remains speculative so that the subject-matter of claim 1 lacks clarity (Article 84 EPC). Moreover, without giving a reference electrode and detailed information how to monitor the free sulfuric acid and fluoride ion values in the bath, it is not possible to determine the REDOX potential and to control the composition of the solution. In addition, INTEROX-S 333 used as a preferred stabilizing agent for hydrogen peroxide was not publicly available at the priority date of the patent. On the other hand, as can be seen from document D22, not every "stabilizer" is suitable to be used in a strongly acid, fluoride containing pickling solution. The disclosure of the patent, therefore, is not sufficient to enable the skilled reader to carry out the claimed process so that the patent does not meet the requirements of Article 83 EPC.

Although document D4 is silent about the sulfuric acid concentration in the initial pickling bath, the solution nevertheless comprises considerable amounts of H<sup>+</sup> and SO<sub>4</sub><sup>2-</sup> ions. During the pickling process however, increasing amounts of H<sub>2</sub>SO<sub>4</sub> and H<sub>2</sub>O<sub>2</sub> are added to the pickling bath in a molar ratio 1:1 so that the

concentrations of these ingredients after some time of pickling will eventually attain a level similar to that of the bath claimed in the patent in suit. Since according to document D4, see Example 3, certain amounts of HF are also added at times, the only technical difference between the claimed process and that given in document D4 consists in the continuous air supply to the pickling bath. This measure is, however, widely used in the art to improve the pickling efficiency by creating turbulence in the bath. Moreover, the claimed process is obvious from document D4 when read in combination with the teaching given in document D1 disclosing a pickling bath consisting of HF-H<sub>2</sub>SO<sub>4</sub>-H<sub>2</sub>O<sub>2</sub>-H<sub>2</sub>O. The subject-matter of claim 1, therefore, does not involve an inventive step.

## **Reasons for the Decision**

### *1. Admissibility of the appeal*

Given that Opponent IV has no longer maintained its objections to the admissibility of the appeal, there is no need to deal with this item in more detail. In the Board's judgment, the appeal complies with the requirements of Articles 106 to 108 and Rules 1(1) and 64 EPC and is, therefore, admissible.

### *2. Amendments (Articles 123(2), (3) EPC)*

- 2.1 Compared with the claims as granted, independent claim 1 has been amended by adding to the claimed process the step of periodically feeding into the bath sufficient quantities of sulphuric acid and hydrofluoric acid to maintain their concentrations in

the bath at optimum levels on the basis of the bath analysis and to maintain the pH value below 1. This restricting feature has a basis in the description of the patent specification column 4, lines 9 to 13 and column 6, lines 22 to 29. Therefore, the amendment satisfies the requirements of Article 123(2) EPC.

2.2 The opponents objected that, according to claim 1 as granted, the optional (possible) addition of the ingredient a) and c) ( $H_2SO_4$  and HF) was done "continuously" rather than "periodically" as now claimed. In their view, present claim 1 thus had been amended so as to extend the protection conferred by the patent as granted and, therefore, contravened Article 123(3) EPC.

2.3 Pursuant to Article 69 EPC the scope of protection conferred by a European patent is determined by the terms of the claims, the description and drawings being nonetheless usable for the purposes of interpretation of the claims. Moreover, the extent of protection is defined exclusively by the compulsory technical terms of the claimed subject-matter, irrespective of further optional technical features or preferred embodiments also mentioned in an independent claim. The question to be considered is, therefore, whether the scope of protection conferred by present claim 1 is narrower or wider than the one conferred by claim 1 as granted.

Claim 1 as granted defines a process which is marked by (i) the temperature and minimum initial composition of the pickling bath and (ii) the continuous feeding of an air flow and a precise quantity of stabilized  $H_2O_2$  controlled on the basis of the Redox-potential which is maintained at \$ 350 mV. The extent of protection

conferred by this claim neither includes an obligatory step for continuously or periodically replenishing ingredients (such as  $H_2SO_4$  and HF) consumed during the pickling process, nor a step for controlling the pH-value of the bath. Consequently, incorporating into claim 1 as granted the additional requirement of periodically feeding sufficient quantities of ingredients a) and c) to maintain their concentration at the optimum levels and to control the pH-value represents a restriction of the scope of protection rather than an extension. Therefore, claim 1 meets the requirements of Article 123(2) and 123(3) EPC.

3. *Articles 83, 84 EPC*

3.1 The respondents have objected that claim 1 and also the patent as a whole fail to specify

- (i) a precise initial composition of the bath by defining only minimum limits of the ingredients,
- (ii) the type of stabilizer suitable for stabilizing the hydrogen peroxide in the acid bath containing fluoride ions, and the preferred stabilizer INTEROX S mentioned in the examples was not available before the priority date of the disputed patent,
- (iii) the analytical method how to determine the REDOX-potential and to monitor the actual concentrations of  $H_2SO_4$  and HF within certain periods of operation and
- (iv) the meaning of the "optimum level" for the ingredients anywhere in the patent

specification.

3.2 In its most general form the present invention is expressed by method claim 1 which includes all the steps necessary for pickling and passivating stainless steel. To this end, the claimed method provides the minimum levels of ingredients a) to e) necessary for preparing the initial bath composition. These limits represent the lowest concentration level which still makes up a satisfactory pickling solution. A preferred composition for a pickling bath above these minimum limits is given in Example A in column 6 of the patent specification. As known to the expert and conceded by the parties, the initial pickling bath composition to be actually selected is generally dependent on the type and pre-treatment of the stainless steel material. Hence, there is no need in the Board's view to define a specific initial composition or upper concentration limits for the ingredients in the solution.

As to the H<sub>2</sub>O<sub>2</sub> stabilizing agent (item (ii)), a plethora of organic and inorganic compounds is at the disposal of a skilled person who can resort to the substances disclosed in documents D23a and D23b. Having regard to the fact that the selection of one specific "peroxide stabilizer" is not crucial to the claimed process, the opponents' objection whether or not "Interox-S 333" used as a stabilizer in Example A actually was available at the priority date is of minor importance since it can be replaced by any other known stabilizer included in peroxide products which were purchasable on the market at that time (cf. the patent specification column 4, lines 34 to 44). Moreover, despite the possible interference of halides with the H<sub>2</sub>O<sub>2</sub> stabilizer noted in document D22, column 3, lines 53 to

55 in the strong acid, fluoride-ion containing solution, it is not regarded as being an unsurmountable problem for an expert to select, albeit in narrow cooperation with a supplier, the appropriate "stabilizer" which affords a sufficient effect of preventing or at least retarding the decomposition of peroxide under the operating conditions.

This statement is also true for the "non-ionic surfactants". These agents do not influence the pickling process since they are generally inert and do not decompose or exert any influence on the REDOX potential.

Turning to item (iii), the metallurgical chemist is, in the Board's view, aware of the various analytical methods for chemically analysing the concentrations of the acids and determining the REDOX potential in the pickling solution. This chemical background knowledge is confirmed e.g. by document D4 which like the opposed patent relates to a pickling process but "without the need for chemical analysis of the composition" (meaning that normally a chemical analysis of the bath is carried out; cf. D4, page 1, lines 42 to 45). The technical standard equipment typically used by the expert in the measurement of the REDOX potential is also referred to in document D4 on page 2, lines 50 to 54. Given this situation, there is no need to describe in the patent specification in detail generally known methods for determining the above cited parameters.

In case there is any doubt or dispute as to the true meaning of any expression in the claims, it is the well established practice of the EPO that the description should be consulted in order to establish what was

intended to be protected in accordance with Article 69(1) EPC. Reference is also made in this context to the Guidelines for Examination at the EPO CIII, 4.2, stating that a claim should be interpreted so as to put a reasonable construction on it so that it makes sense in the context.

When putting the claimed method into practice, the skilled reader is led in particular to page 4, lines 9 to 13 of the patent specification. This passage reflects the clear teaching that the (initial) concentrations of HF and H<sub>2</sub>SO<sub>4</sub> decrease during the pickling process and, as a counter-measure, the addition of appropriate amounts thereof is indispensably needed in order to maintain the required free acid and fluoride ion values at their "optimum" levels. Based on the disclosure of the patent as a whole, according to which the acid supplementation regimen is one of the key features of the claimed process, this teaching implicitly could only be interpreted as meaning to aim at maintaining the bath concentration at least close to or above the minimum starting composition or on the composition selected in view of the specific material and its pre-treatment to be pickled with the solution.

- 3.3 In view of these considerations, the skilled reader is, in the present case, presented with sufficient technical information and explanations, in particular those given in the description and the Example A, to put into practice the claimed process. Having regard to this technical information, the Board is unaware of any verifiable facts which could cast a serious doubt on the capability of a skilled person to carry out the claimed process on the basis of what is disclosed in

the patent. The added feature also does not render claim 1 unclear because the "optimum level" objected to in this respect is clearly a predetermined value or range on which the concentration of the ingredients has to be maintained on the basis of the bath analysis. The Board therefore concludes that the requirements of Article 83 and also of Article 84 are met.

4. *Novelty*

None of the prior art documents cited in the opposition and appeal proceedings discloses the process which comprises all the technical features of the process set out in claim 1 of the patent at issue. Given that the novelty of the claimed subject-matter has not been disputed by the opponents in the appeal proceedings, there is no need to discuss this point in more detail.

5. *The closest prior art*

It was common ground in the opposition and appeal proceedings, and it is also the view of the Board, that document D4 represents the closest prior art. Like the disputed patent, this document relates to a method for controlling the composition of a HNO<sub>3</sub>-free acid solution for pickling stainless steel, the initial solution comprising specific amounts of ferric (Fe<sup>3+</sup>) sulfate, ferrous (Fe<sup>2+</sup>) sulfate, hydrofluoric acid and being kept at about 50°C (cf Table 1, examples). After starting the process, the REDOX potential of the pickling solution is held at a predetermined and constant range of at least 300 mV by adding hydrogen peroxide and sulfuric acid in a molar ratio 1:1 to the solution. Moreover, a small amount of HF is replenished at times (cf. D4, page 3, lines 13, 14). In doing so, there is no need



for a chemical analysis of the composition (cf. D4, page 1, lines 36 to 63; page 3, Example 3, Table 1, Example 1, claims 1 and 2) which makes the pickling process simple to control and reduces cost.

The claimed method differs from the one disclosed in document D4

- (i) by an air flow of at least 3 m<sup>3</sup>/h per m<sup>3</sup> continuously supplied to the bath;
- (ii) by analysing the chemical composition of the bath i.e. the free acid and fluoride ion values, and as a consequence thereof periodically feeding HF and H<sub>2</sub>SO<sub>4</sub> on the basis of the bath analysis to maintain their concentrations at optimum levels and
- (iii) maintaining the pH-value at less than 1.

6. *Problem to be solved and solution*

6.1 As set out in document D4 on page 2, lines 30 to 38 and 42 to 46, the ferrous sulfate gradually accumulates in the solution. If concentrations above 200 g/l are reached, it is deposited mainly as FeSO<sub>4</sub>·7H<sub>2</sub>O crystals forming a "sludge". In order to prevent the solution from accumulating too high an amount of ferrous sulfate, at least parts thereof need to be drawn out continuously or intermittently or, alternatively, the bath is cooled for removing the ferrous sulfate as crystals precipitated outside the system.

6.2 Starting from the teaching given in document D4, the problem underlying the patent at issue therefore

resides in providing an acid pickling process which reduces to a minimum or even prevents the formation of "sludge", which is highly efficient while being less costly and can be easily controlled automatically (cf. the patent column 6, lines 14 to 40).

- 6.3 The solution to this problem is to prepare an initial composition of the bath comprising specific amounts of  $\text{Fe}^{3+}$ ,  $\text{H}_2\text{SO}_4$ ,  $\text{H}_2\text{O}_2$  and HF thus constituting a specific level of free acid and fluoride ion values and maintaining these values at an "optimum" level all over the time of operation by periodically supplying appropriate amounts of HF and  $\text{H}_2\text{SO}_4$  to the bath. In addition, an air flow is continuously fed to the acid solution to provide a high agitation of the bath so that the surface to be treated is always brought into contact with a fresh pickling solution. Moreover, the REDOX potential in the bath is maintained at more than 350 mV by continuously feeding air in combination with the peroxide added. As is set out in column 5, lines 35 to 47, this process ensures effective pickling and the formation of a passivation film on the stainless steel material without running the risk of excessive corrosion in the form of "pitting" or "burning".

7. *Inventive step*

- 7.1 Although the most decisive feature of the process disclosed in document D4 is in fact its simplicity (i.e. adding  $\text{H}_2\text{O}_2 + \text{H}_2\text{SO}_4$  1:1 to maintain the REDOX-potential in a constant range), it nevertheless entails the drawback of early "sludge" formation. The reason for this appears to be the obligatory high concentration of ferric sulphate in the starting composition, as is apparent from the experiments given

in Table 1: at least 150 g/l  $\text{Fe}_2(\text{SO}_4)_3$  (= 42 g/l  $\text{Fe}^{3+}$ ) are combined with 30 g/l  $\text{FeSO}_4$  ( $\text{Fe}^{2+}$ ) (experiments 1, 4, 5, 7), or with 100 g/l  $\text{FeSO}_4$  (experiment 2) or even with 200 g/l  $\text{FeSO}_4$  (experiments 3, 6, 8). This means that the initial "load" with iron salts is so high that the capacity for keeping the additional iron salts originating from the pickling process in solution without the formation of sludge is limited. The process claimed in the disputed patent, however, allows to start with an initial content of iron ions in the bath as low as 15 g/l  $\text{Fe}^{3+}$  with no  $\text{Fe}^{2+}$  ions being present, while at the same time a high concentration of  $\text{H}_2\text{SO}_4$  is present (corresponding to a high concentration of  $\text{SO}_4^{2-}$  ions, as set out in claim 1 and Example A of the opposed patent). Due to the active management of the free acid and fluoride ion values, the composition of the bath can be chosen such that its capacity for dissolving iron salts during the pickling process is from the very start so high that large quantities thereof can be kept in solution without the formation of sludge. Moreover, the high concentration of  $\text{SO}_4^{2-}$  and  $\text{F}^-$  ions is maintained by periodically replenishing the amounts of  $\text{HF}$  and  $\text{H}_2\text{SO}_4$  consumed during the claimed process. There is experimental evidence and a theoretical calculation enclosed with the patentee's Statement of Grounds of 9 August 1999 showing that the pickling efficiency of the claimed bath is superior to that disclosed in document D4 and that the simple addition of  $\text{H}_2\text{SO}_4:\text{H}_2\text{O}_2$  in a molar ratio 1:1 to the initial bath composition as proposed in document D4 could not create or restore the substantial excess of sulphuric acid in the pickling bath that is required by the lower limit in claim 1 of the patent at issue. This

was not challenged by the opponents at the oral proceedings.

Based on these considerations it is, therefore, concluded that the general teaching given by the description and the examples in document D4 neither points to the presence of an excess of sulphuric acid, (i.e. a surplus of  $\text{SO}_4^{2-}$  ions over the stoichiometric amount required by the conversion of  $\text{Fe}^{2+}$  to  $\text{Fe}^{3+}$  sulphate) nor to the necessity for creating such a surplus during the time of operation. On the contrary, according to the teaching of document D4, only one single parameter, namely the REDOX potential, is monitored and maintained within a predetermined range and the chemical analysis of the pickling solution is not determined at all. Although some hydrofluoric acid may be replenished in the process according to document D4, there is no statement that the predetermined level of HF in the bath has to be maintained during pickling. Moreover, there is no indication anywhere in this document guiding a skilled person to further improve the process by injecting air into the bath to interact with hydrogen peroxide for creating a high oxidation potential and a strong turbulence. Finally document D4 remains silent about the pH-value which according to claim 1 of the disputed patent is to be held below 1.

- 7.2 Although the issue of inventive step was essentially discussed vis-à-vis the technical teaching given in the most pertinent document D4, reference was also made by opponent III to document D1. However, this document fails to teach a specific initial composition of the bath, the REDOX potential and pH-value of the solution to adhere to, the management of HF and  $\text{H}_2\text{SO}_4$  concentrations during operation and the supply of an

air flow. Hence, the disclosure of this document when read in combination with that of document D4 could not incite the skilled reader to arrive at the process claimed in the patent.

- 7.3 In view of these considerations, the Board concludes that the technical process features defined in claim 1 of the patent at issue neither can be derived from the teaching given in document D4 alone or in combination with D1 nor could it be considered simply as a routine application of a skilled person's knowledge. The subject-matter of claim 1, therefore, involves an inventive step.

The dependent claims 2 and 3 relate to preferred embodiments of the process given in claim 1 and are, therefore, also allowable.

## **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 patent 0 505 606 in amended form on the basis of
  - Claims 1 to 3 submitted at the oral proceedings marked as "Main Request (A')" and
  - Description columns 1 to 8 submitted at the oral proceedings.

The Registrar:

The Chairman:

G. Magouliotis

W. D. Weiß