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D E C I S I O N
of 8 November 2001

Case Number: T 1133/97 - 3.2.2

Application Number: 87311012.6

Publication Number: 0306578

IPC: C22C 38/28

Language of the proceedings: EN

Title of invention:

Ferritic stainless steel and process for producing

Patentee:

ALLEGHENY LUDLUM CORPORATION

Opponent:

Société UGINE S.A.

Headword:

-

Relevant legal provisions:

EPC Art. 52(1), 54, 56

Keyword:

"Inventive step (yes)"

"Novelty (yes)"

Decisions cited:

-

Catchword:

-



Case Number: T 1133/97 - 3.2.2

D E C I S I O N
of the Technical Board of Appeal 3.2.2
of 8 November 2001

Appellant:
(Opponent)

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Respondent:
(Proprietor of the patent)

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Decision under appeal:

**Interlocutory decision of the Opposition Division
of the European Patent Office posted 16 October
1997 concerning maintenance of European patent
No. 0 306 578 in amended form.**

Composition of the Board:

Chairman: W. D. Weiß
Members: S. S. Chowdhury
R. T. Menapace

Summary of Facts and Submissions

I. The appellant (opponent, Ugine S. A.) lodged an appeal against the decision of the opposition division to maintain the patent No. 0 306 578 in amended form. The decision was dispatched on 16 October 1997.

The appeal and the fee for the appeal were received on 19 November 1997. The statement setting out the grounds of appeal was received on 13 February 1998.

The opposition was filed against the whole patent and based on Article 100(a) EPC (lack of novelty and inventive step) and Article 100(b) EPC (the patent did not disclose the invention sufficiently clearly and completely for it to be carried out by the person skilled in the art), but the latter objection was dropped during the appeal procedure.

With the notice of opposition the opponent had cited three documents, D1 to D3, and after expiry of the period of opposition, cited the further documents D4 to D13. The opposition division decided that documents D4 to D13 did not prima facie jeopardise the claims and were to be disregarded under Article 114(2) EPC. The opponent's argument, that the late-filed documents were necessary further evidence as a direct result of a shift in the emphasis of the invention, was not accepted since the amended main claims were based on features contained in the dependent claims of the granted patent.

The opposition division decided that, having regard to documents D1 to D3, the amended claims submitted during the opposition procedure met all the requirements of

the EPC, in particular those of Article 52(1) EPC and Article 100(b) EPC.

The appellant has cited the following prior art documents during the appeal proceedings:

- D2: Wada et al., "Nitrogen Solution and Titanium Nitride Precipitation in Liquid Fe-Cr-Ni Alloys", Met. Trans. B, Vol. 8B, September 1977, pages 443 to 450.
- D4: Johnson, J. N., "Influence of Columbium on the 870°C Creep Properties of 18% Chromium Ferritic Stainless Steel", Soc. of Motor Engineers, Inc., Warrendale, Pennsylvania US, from the Int. Congress and Exposition Cobo Hall, Detroit, Michigan, February 23 to 27, 1981.
- D9: Gates et al., "Absorption of gaseous contaminants by welds and weld simulations in ferritic stainless steels", Mat. Sci. and Technology, May 1987, Vol.3, pages 386 to 393.
- D15: "Niobium in ferritic stainless steels", Keown, S. R., Niobium Technical Report, NbTR - 09/86, December 1986, pages 1 to 31.

II. Oral proceedings took place on 8 November 2001, at the end of which the following requests forming the basis of the decision were put forward:

The appellant requested that the decision under appeal be set aside and that European patent No. 0 306 571 be revoked.

The respondent (patent proprietor, Allegheny Ludlum Corp) requested that the appeal be dismissed and that the patent be maintained in the form set out in the decision under appeal.

III. The independent claims 1, 8, 18, and 19 read as follows:

"1. A method of producing a weldable ferritic stainless steel sheet or strip product having improved surface quality, characterised in the method comprising: preparing a steel melt containing, by weight percent, up to 0.03 carbon, 0.012 to 0.05 nitrogen, 10 to 13 chromium, up to 1.0 manganese, up to 0.5 nickel, up to 1.0 silicon, 0.03 to 0.35 titanium, 0.10 to 0.6 niobium, optionally up to 1.2 aluminium, balance iron, the maximum amounts of the titanium and nitrogen varying inversely in amounts not more than necessary to satisfy the following Equation 1:

$$6.194 - 16437/T = \log \%N + \log \%Ti + \log f_N + \log f_{Ti}$$

where $\log f_N$ is described in Equation 2 herein and $\log f_{Ti}$ is described in Equation 3 herein;

casting and solidifying the steel without the precipitation of detrimental intermetallic or nonmetallic titanium compounds during the molten phase; and working the steel by hot rolling and cold rolling to final gauge strip or sheet without grinding the hot rolled band for removal of surface defects attributable to the titanium compounds; said cold rolled steel product having good surface quality substantially free of open surface defects."

"8. A weldable ferritic stainless steel sheet or strip product having improved surface and elevated temperature oxidation resistance and strength, characterised by the steel containing, by weight percent, up to 0.03 carbon, 0.012 to 0.05 nitrogen, 10 to 13 chromium, up to 1.0 manganese, up to 0.5 nickel, up to 1.0 silicon, 0.03 to 0.35 titanium, 0.10 to 0.6 niobium, optionally up to 1.2 aluminium, balance iron, titanium and nitrogen present in amounts which vary inversely and not more than necessary to satisfy the following Equation 1:

$$6.194 - 16437/T = \log \%N + \log \%Ti + \log f_N + \log f_{Ti}$$

where $\log f_N$ is described in Equation 2 herein and $\log f_{Ti}$ is described in Equation 3 herein."

"18. An automotive exhaust article for elevated temperature service having improved oxidation resistance and surface quality, the article being made from a steel alloy consisting of, by weight percent, up to 0.01 carbon, up to 0.03 nitrogen, 10 to 13 chromium, up to 1.0 manganese, up to 0.5 nickel, 0.5 to 1.0 silicon, optionally up to 1.2 aluminum, 0.03 to 0.1 titanium, 0.1 to 1.0 niobium, balance iron, and the titanium and nitrogen present in amounts which vary inversely and not more than necessary to satisfy the following Equation 1:

$$6.194 - 16437/T = \log \%N + \log \%Ti + \log f_N + \log f_{Ti}$$

where $\log f_N$ is described in Equation 2 herein and $\log f_{Ti}$ is described in Equation 3 herein."

"19. An automotive exhaust article for elevated

temperature service having improved surface oxidation resistance and surface quality, the article being made from a steel alloy consisting of, by weight percent, up to 0.01 carbon, up to 0.03 nitrogen, 16 to 19 chromium, up to 1.0 manganese, up to 0.5 nickel, 0.5 to 1.0 silicon, optionally up to 1.2 aluminium, 0.03 to 0.1 titanium, 0.1 to 1.0 niobium, balance iron, and the titanium and nitrogen present in amounts which vary inversely and not more than necessary to satisfy the following Equation 1:

$$6.194 - 16437/T = \log \%N + \log \%Ti + \log f_N + \log f_{Ti}$$

where $\log f_N$ is described in Equation 2 herein and $\log f_{Ti}$ is described in Equation 3 herein."

Equations 2 and 3, which feature in the independent claims, are defined on page 6 of the patent specification.

Claims 2 to 7 and 9 to 17 are dependent on claims 1 and 8, respectively.

IV. The appellant argued as follows:

Relevancy of the late filed documents: The change of the scope of the claims, particularly as regards the chromium content, necessitated a new search, which was the reason for the late filing of documents D4 onwards. Document D4 was novelty destroying for claim 19 and should, therefore, be admitted into the procedure.

Documents D9 disclosed an alloy (alloy C in Table 1) whose composition was in accordance with the opposed claims, and also Equation 1, and document D15 disclosed

an alloy (alloy 12 SR) whose composition also fell within the terms of the claims and it was intended for the same use (car exhausts etc.) as the steel of the patent in suit, and this document also disclosed the problem of surface defects. These documents demonstrated the lack of inventive step of the claimed subject-matter and were also highly relevant.

Novelty: The compositions of the steels L-1 and L-2 in Table 1 of document D4 fell within the range of product claim 19. It was not clear how a temperature restriction as defined in the claim, or the inverse relationship between the titanium and nitrogen contents, which were considerations during manufacture, was manifest in the product. All steels having the required composition and including a low titanium amount, would have an adequate surface quality. Therefore document D4 was novelty destroying for claim 19.

Inventive step: The alloys L-1 and L-2 in Table 1 of document D4 had compositions within the claimed range. Document D2 gave the activity coefficients for chromium and nitrogen, and also the fact that the titanium and nitrogen amounts varied inversely. The quantity of titanium nitride precipitated could be determined simply and this precipitation avoided if necessary.

V. The respondent argued as follows:

Relevancy of the late filed documents: At least the silicon content of the alloys L-1 and L-2 of document D4 was outside the claimed range, so that this document was not novelty destroying for claim 19, and therefore not relevant.

The appellant's arguments regarding the need for a further search was not valid in view of the fact that the chromium contents of the new claims was taken from the dependent claims of the granted patent. Documents D9 and D15 were not relevant since they provided background information only and did not disclose either the present technical problem nor taught a solution therefor, which was to maintain the titanium and nitrogen contents below the stoichiometric level. Therefore, none of the late filed documents should be admitted.

Novelty: The compositions L-1 and L-2 of document D4 did not fall within the composition range of the article of claim 19 owing to the silicon and carbon contents being outside the claimed range. Moreover, Equation 1 of the patent did represent a limitation of the scope of the claim, which was not satisfied by any composition of document D4.

Inventive step: Documents D2 and D4 taught away from the claimed invention since they disclosed titanium and nitrogen contents that varied in the same direction and not inversely. Document D2 also says that precipitation of titanium nitride was desirable, contrary to what was sought after in the patent in suit.

Reasons for the Decision

1. The appeal is admissible.
2. *Amendments*

After grant, the claims were amended as follows:

Claim 1:

- (i) The chromium content has been narrowed from 10 to 25% to 10 to 13%.
- (ii) The words "during the molten phase" have been added to the casting and solidifying step.

Claims 18 and 19: Although the granted claims contained three independent claims, there are now four independent claims. The reason for this is that granted claim 20, for an automotive exhaust article, is now split up into two alternative claims, 18 and 19, which differ only in the chromium content (10 to 13% in claim 18 and 16 to 19% in claim 19), and claim 19 mentions surface oxidation resistance.

The basis for the amendment (i) and to the chromium content in claims 18 and 19 is found in claims 8 and 9 as originally filed. Support for the amendment (ii) to claim 1 is on page 5, lines 12 to 21 of the A1 publication.

The above changes were made in response to a novelty attack and are therefore allowable. The amendment (ii) has been effected in order to clarify that the precipitation of the detrimental compounds is avoided during the molten phase, in order to indicate more clearly the aim of the invention and hence emphasise the distinction over the prior art. Moreover, the scope of the claims has been restricted by the amendments.

Granted claim 20 mentions "improved oxidation resistance", as does present claim 18, but present claim 19 mentions "improved surface oxidation

resistance". This is allowable since surface oxidation resistance is addressed in original claim 7.

The lower limit of nitrogen of 0.012% in the claims of the granted patent was an amendment over the claims as originally filed, where the nitrogen content was defined as "up to 0.05". This amendment is supported by the examples since this is the lowest amount of nitrogen used therein.

The amendments are allowable under Article 123(2,3) EPC, accordingly.

3. Admissibility of the late filed documents
 - 3.1. Although the opposition division stated in the decision under appeal, that the documents D4 to D13 were not relevant and therefore not admitted into the procedure under Article 114(2) EPC, it had in fact already cited, in its communication dated 10 July 1996, document D4 (at that time referred to as document D) as being novelty destroying against the then pending claims 2 and 10. Therefore, this document was already in the proceedings and could not subsequently be disqualified therefrom.
 - 3.2. It remains only to be examined whether documents D9 and D15 are of sufficient relevance that they should be admitted under Article 114(2) EPC. In order to do this it is first necessary to analyse the invention of the patent in suit.
 - 3.3. The patent in suit relates to a weldable ferritic stainless steel sheet or strip with improved surface quality for use in automobile exhaust and emission

systems, which has high temperature strength and resistance to oxidation and corrosion. In order to improve surface appearance and formability while minimising roping, niobium is added to the steel, but this contributes to weld cracking, against which titanium is, therefore, also added. A well known alloy of this type is the USS-Type 409 alloy.

The disadvantages of prior art methods of making titanium and niobium stabilised steel alloys are set out on page 3, lines 35 to 42 of the patent in suit, and may be summarised as follows: The titanium used to stabilise alloys such as USS-Type 409 has an extremely high affinity for nitrogen and oxygen and forms and precipitates nonmetallic oxides and intermetallic titanium nitride during melting, refining and casting. These precipitates coalesce into large chunks or clusters and float to the surface of the cooling molten metal in the mould, and upon freezing they are trapped in or near the surface of the cast slabs. Costly grinding is required to minimise rolling these clusters into detrimental surface defects. Another defect that arises is the open surface defect, which appears as a grey or dark streak parallel to the rolling direction in the hot rolled band.

- 3.4. The problem that the patent in suit seeks to solve is set out in the paragraph linking pages 3 and 4 of the patent specification is, accordingly, to manufacture an alloy comparable to the USS-Type 409 alloy in terms of fabricability and oxidation and corrosion resistance, but which does not exhibit the open surface defects of titanium-bearing stainless steels. Such steels should be capable of being produced in light gauges of the order of less than 0.381mm (0.015 inch) without surface

defects or holes.

- 3.5. The solution proposed is that the titanium content of the ferritic stainless steel should be minimised whereby the titanium nitride is soluble in the melt down to the liquidus temperature within the normal nitrogen content range available with conventional AOD practice, so as to avoid precipitates which affect surface appearance. The reduced titanium content is compensated by the addition of niobium, and the stabilisation effect of titanium and niobium is achieved by their combination with carbon and nitrogen to avoid their adverse affects on corrosion resistance. Thus, the amounts of titanium and nitrogen are below the stoichiometric levels so as not to precipitate in the liquid phase, yet play an important role in the solid phase.

The independent claims accordingly feature an equation that embodies upper limits for the permitted maximum amounts of nitrogen and titanium, and specify that the amounts of nitrogen and titanium should be selected so as to vary inversely with each other.

Examples are given of ferrite steel alloys having constituents falling within the claimed composition ranges. These have good surface characteristics without the need for grinding, while having mechanical properties and corrosion resistance comparable with USS-Type 409 steel.

Thus, the steel may be made using conventional AOD practices and no grinding procedures are necessary to improve surface appearance. The steel may be rolled to thinner gauges than was feasible for the USS-Type 409

steel.

- 3.6. Document D9 presents studies of the factors influencing weldability of USS-Type 444 ferritic stainless steel, including contamination of welds with nitrogen. Stabilisation with both titanium and niobium is recommended to reduce nitrogen pick-up. The alloy C listed in Table 1 is not in accordance with the claims of the patent in suit in that the chromium content is too high and there is a substantial amount of molybdenum present.

Equations 4 and 7 of the appendix are alleged to amount to equation 1 of the patent in suit, but this is not the case, even though Equation 7 does define an inverse relationship. However, there is no information concerning solving the problem of surface defects by keeping the titanium and nitrogen contents below the stoichiometric level so as to avoid precipitation of nitrides during the molten phase.

- 3.7. Document D15 reviews the role of niobium as a stabilising element and its influence on the mechanical and chemical properties of ferritic stainless steel. Section 6.5 discusses dual stabilisation by titanium and niobium and recommends their combined stabilisation for toughness and ductility. Section 6.6 mentions the problem of surface defects due to inclusions and seems to suggest favouring niobium over titanium to reduce this. This document does not suggest the steel compositions as defined in the claims of the patent in suit, nor the inverse relationship between the titanium and nitrogen contents so as to avoid precipitation of nitrides during the molten phase, nor keeping the titanium and nitrogen contents below the stoichiometric

level so as to avoid precipitation of nitrides during the molten phase.

- 3.8. For the foregoing reasons documents D9 and D15 are not relevant to the problem or solution of the patent in suit, and since they are late filed they are not considered in the procedure under Article 114(2) EPC.

4. *Novelty*

Only document D4 was cited against claim 19, it being alleged that steels L-1 and L-2 in table 1 have the composition of the steels of these claims. However, at least the carbon and silicon contents of the steels L-1 and L-2 lie outside the claimed ranges. It is also not clear that the titanium and nitrogen contents satisfy Equation 1 of the patent in suit. Therefore, document D4 is not novelty destroying for claim 19.

5. *Inventive step*

Only documents D2 and D4 remain to question the inventive step of the claimed subject-matter. Document D2 presents studies of the effects of alloying elements and temperature on nitrogen solubility and the solubility product of titanium nitride in liquid Fe-Cr alloys. Although nitrogen is important for determining the properties of the steel, soluble nitrogen is undesirable and to stabilise the dissolved nitrogen, titanium should be added to precipitate titanium nitride in the solid steel.

The first paragraph on page 443, therefore, mentions the precipitation of titanium nitride as being desirable in the structure of the solid steel, but it

does not suggest inhibiting such precipitation in the molten phase, which is the aim of the opposed patent. The solubility product of titanium nitride in Fe-Cr alloys at different temperatures is calculated (Fig.7 and Table IV), though the inverse relation between nitrogen and titanium is disclosed, this information is not associated with any practical measures such as overcoming the problem of surface defects by keeping the nitrogen and titanium contents low so as to avoid precipitation of nitrides during the molten phase.

Document D4 was cited only as an anticipation of the subject-matter of claim 19. If anything, this document teaches away from the patent in suit since the alloys L-1 and L-2 have titanium and nitrogen contents that change in the same direction rather than inversely. Also, the chromium contents of these alloys lie outside the claimed range, and in any case, there is no disclosure of the present problem or its solution.

To summarise, whereas in the prior art enough titanium was added to the melt to form nitrides, wherein in the molten phase excess titanium in the form of precipitated nitrides floated to the top of the melt, the claimed invention, in contradistinction to the prior art, suggests the prevention of detrimental precipitates during the molten phase, by setting limits on the titanium and nitrogen contents, whilst still enabling the carbon and nitrogen to be bound up with titanium and niobium in the solid phase. The present problem and solution are, therefore, new in the art and involve an inventive step.

The subject-matter of the method claim 1 and the device claims 8, 18, and 19 involves an inventive step,

accordingly.

6. Since, in view of the above, the grounds of opposition raised by the appellant do not prejudice the maintenance of the patent in amended form, the patent in suit is maintained on the basis of the claims upheld by the opposition division.

Order

For these reasons it is decided that:

The appeal is dismissed.

The Registrar:

The Chairman:

V. Commare

W. D. Weiß