BESCHWERDEKAMMERN DES EUROPÄISCHEN PATENTAMTS

BOARDS OF APPEAL OF THE EUROPEAN PATENT OFFICE

CHAMBRES DE RECOURS DE L'OFFICE EUROPEEN DES BREVETS

Internal distribution code:
(A) [ ] Publication in OJ
(B) [ ] To Chairmen and Members
(C) [ ] To Chairmen
(D) [X] No distribution

Datasheet for the decision of 28 September 2006

Case Number: T 0820/04 - 3.2.02

Application Number: 93904252.9

Publication Number: 0628088

IPC: C22C 19/03

Language of the proceedings: EN

Title of invention:
Nickel-molybdenum alloys

Patentee:
HAYNES INTERNATIONAL, INC.

Opponent:
ThyssenKrupp VDM GmbH

Headword:
-

Relevant legal provisions:
EPC Art. 54, 56

Keyword:
"Inventive step (yes, after amendment)

Decisions cited:
T 0074/98

Catchword:
-

Europäisches Patentamt | European Patent Office | Office européen des brevets

Beschwerdekammern | Boards of Appeal | Chambres de recours

Case Number: T 0820/04 **-** 3.2.02

D E C I S I O N
of the Technical Board of Appeal 3.2.02
of 28 September 2006

Appellant: (Patent Proprietor)
HAYNES INTERNATIONAL, INC.
1020 West Park Avenue
P.O.Box 9013
Kokomo
Indiana 46904-9013 (US)

Representative:
Trueman, Lucy Petra
Barker Brettell
138 Hagley Road
Edgbaston
Birmingham B16 9PW (GB)

Respondent: (Opponent)
ThyssenKrupp VDM GmbH
c/o Dipl.-Ing. Wolfgang Cichy
Schulstrasse 52
D-58332 Schwelm (DE)

Representative:
-

Decision under appeal:
Decision of the Opposition Division of the European Patent Office posted 22 April 2004 revoking European patent No. 0628088 pursuant to Article 102(1) EPC.

Composition of the Board:

Chairman: T. K. H. Kriner
Members: R. Ries
A. Pignatelli

Summary of Facts and Submissions

I. Opposition was filed against the whole patent No. 0 628 088 and was based on Article 100(a) EPC, (lack of novelty and inventive step).

The opposition division held that the subject matter of claim 1 of the main request and the first to sixth auxiliary requests then on file lacked novelty with respect to the technical disclosure given in either document

D4: US-A-4 861 550 or

D17: Lee, W. T. "Stress corrosion cracking of a Ni-Mo-Fe alloy (Hastelloy B2) in a fluoride salt, Proceedings conf. Corrosion 89, April 17 to 21, 1989, New Orleans, Louisiana, Paper 96, pages 1 to 12; or

D17': conversion from weight% to atom% of the composition of over-check 2665-3, given in Table 1 of D17

and revoked the patent. The decision was dispatched on 22 April 2004.

II. The appellant (patent proprietor) lodged an appeal against this decision of the opposition division. The appeal and the fee for the appeal were received on 22 June 2004. The statement setting out the grounds of appeal was received on 31 August 2004 and included in the annex amended sets of claims, test results of

comparative alloys and alloys according to the patent and the declarations of Mr Klarstrom and Mr Gillette.

III. Enclosed with its reply to the appellant's statement, the respondent (opponent) referred to document

D18: Standard Practice for Using Significant Digits in Test Data to Determine Conformance with Specifications, Designation E29-02, 2004.

IV. Oral proceedings before the board took place on 28 September 2006, at the end of which the following requests forming the basis of the decision were submitted:

The appellant requested that
- the decision under appeal be set aside and
- the patent be maintained on the basis of the main request or
- of the auxiliary request 1 both requests filed on 31 August 2004, or
- of the second auxiliary request filed during the oral proceedings.

The respondent (opponent) requested that the appeal be dismissed.

V. Independent claim 1 of the main request reads a follows:

"1. A crystalline metal alloy having the general formula $Ni_aMo_bX_cY_dZ_e$ where:
"a" is more than 73, but less than 77, atom percent of nickel;

"b" is more than 18, but less than 23 atom percent of molybdenum;
"X" is two or more substitutional alloying elements of chromium, tungsten, manganese, iron or cobalt in amounts "c" being at least two atom percent in total but not exceeding five atom percent for any one such element;
"Y" is one or more optional metallic substitutional elements of aluminium, copper, silicon, titanium, vanadium or zirconium in amounts "d" not exceeding one atom percent for any one such element;
"Z" is present and is one or more interstitial elements of boron, carbon, nitrogen, oxygen, phosphorus or sulphur in amounts "e" not exceeding 0.1 atom percent for any one such element;
wherein the sum of "c" plus "d" is between 2.5 and 7.5 atom percent,
and
wherein the sum of "c" plus "d" plus 0.7 times "b" is between 17 and 21 atom percent."

Independent claim 1 of the first auxiliary request reads:

"1. A metal alloy having the general formula $Ni_aMo_bX_cY_dZ_e$ where:
"a" is between 73.5 and 76.5 atom percent of nickel;
"b" is more than 19, but less than 22 atom percent of molybdenum;
"X" is two or more substitutional alloying elements from Groups VIA, VIIA or VIII of the Periodic Table, in amounts "c" being at least two atom percent in total but not exceeding five atom percent for any one such element;

"Y" is one or more optional substitutional alloying elements of aluminium, copper, silicon, titanium, vanadium or zirconium in amounts "d" not exceeding one atom percent for any one such element;
"Z" and is one or more interstitial elements of boron, carbon, nitrogen, oxygen, phosphorus or sulphur in amounts "e" not exceeding 0.05 (amended during the oral proceedings) atom percent for any one such element; and wherein the sum of "c" plus "d" is between 3 and 7 atom percent."

Independent claim 1 of the second auxiliary request reads as follows:

"1. A metal alloy having the general formula $Ni_aMo_bX_cY_dZ_e$ where:
"a" is between 73.5 and 76.5 atom percent of nickel;
"b" is more than 19, but less than 22 atom percent of molybdenum;
"X" is two or more substitutional alloying elements of chromium, tungsten, manganese, iron or cobalt, in amounts "c" being at least two atom percent in total but not exceeding five atom percent for any one such element;
"Y" is one or more optional substitutional alloying elements of aluminium, copper, silicon, titanium, vanadium or zirconium in amounts "d" not exceeding one atom percent for any one such element;
"Z" is present and is one or more interstitial elements of boron, carbon, nitrogen, oxygen, phosphorus or sulphur in amounts "e" not exceeding 0.05 atom percent for any one such element;
wherein the sum of "c" plus "d" is between 3 and 7 atom percent; and

wherein the sum of "c" plus "d" plus 0.7 times "b" is between 18 and 20 atom percent."

VI. The appellant argued as follows:

In the decision the opposition division objected to the novelty of the claimed alloy in view of document D4, Table 1, alloy 10 and over-check alloy 2665-3 given in document D17.
Novelty of the claimed alloy composition however arose as claim 1 of the main request required interstitial elements like C to be present in amounts not exceeding 0.1 at% and preferably not exceeding 0.05 at%. The carbon content of sample 10 in D4 of 0.14 at% (or 0.135 at% according to a second calculation by the opponent) clearly exceeded that limit. As to claim 1 of the first and second auxiliary requests, the Mo-content of 18.40 at% of sample 10 was below the lower limit of 19 at% specified therein.
Turning to document D17, the nickel content of 77.05 at% of the over-check 2336-5 analysis exceed the limit of 77 at% set out in claim 1 of the main request.
Moreover, the carbon content of 0.087 at% was above the upper limit of 0.05 at% specified in claim 1 of the first and second auxiliary requests and the total of c+d+0.7b of 17.001 at% was outside the range of 18 to 22, given in claim 1 of the second auxiliary request.

Following the considerations given in T 74/98, rounding up or down the amounts of the components, as done by the opposition division in the impugned decision, was not admissible given that the elemental amounts were obtained by converting the original composition from weight percent into atom percent. Such rounding off the

converted figures (in at%) would affect the accuracy of the original composition (in wt%).

Moreover, only the mill certificates 1 and 2 given in Table 1 of document D17 reflected the true and accurate chemical analysis of the Hastelloy B-2 tubes referred to in this document. Both compositions fell, however, clearly outside the claimed ranges. The expert on this field of technology very well knows that the alloy composition designated as "over-check 2665-3" also given in Table 1 of D4 was simply an inaccurate composition check performed by the purchaser simply to show that the analysis of the tubes actually met the required standard. Since an alloy with the composition of analysis 2665-3 never existed, it could not destroy the novelty of claim 1 of all requests. This position was corroborated by the declarations of Mr Klarstrom and Mr Gillette.

As to inventive step, the problem to be solved by the patent was the provision of a high Mo-Ni-base alloy which - compared to Hastelloy B-2 - was not prone to an order induced, grain boundary embrittlement. This disadvantage resulted from the formation of ordered intermetallic phases such as Ni_2Mo, Ni_3Mo and Ni_4Mo. Since this problem was likewise addressed in document D4, this document could be regarded as closest prior art. However, document D4 offered a different solution, i.e. a Ni-base alloy in which aluminium and or iron were used separately or in combination to give the desired overall effect. The skilled person faced with the above mentioned problem thus would take from the whole teaching of document D4 the need to control the amounts of aluminium and of iron to obtain the desired

combination of properties. Nothing in D4 would however suggest to the skilled person that it was critical to select at least two substitutional elements in amounts satisfying the relationships featuring in claim 1 of the main, first or second auxiliary requests. Document D17 was even more remote since it simply dealt with Hastelloy B-2 without giving any motivation to alter this alloy composition. The subject matter set out in claim 1 of the main, first and second auxiliary request therefore was novel and inventive with respect to the technical teaching given in the prior art D4 and D17.

VII. The respondent argued as follows:

The patent at issue was essentially concerned with a modified Hastelloy B-2 which was also mentioned in document D17. Over-check analysis 2665-3 of Hastelloy B-2 was within the claimed elemental ranges except for 77.05 at% Ni which was extremely close to the claimed range for nickel. The chemical analysis of the over-check and of the mill certificates were performed by competent laboratories and could not be doubted as to their accuracy. The alloy set out in claim 1 of the main and the first auxiliary request was therefore anticipated by over-check analysis 2665-3 of alloy B-2.

Document D4 related to a high Mo-Ni-base alloy and even addressed the same problem underlying the patent at issue, i.e. to avoid the precipitation of deleterious intermetallic compounds even after thermal processing. Example 10 given in document D4, Table 1 fell within the alloy claimed in the patent except for 0.135 at% carbon. The considerations given in T 74/98 did not

apply to the present case, since they related to different subject matter. Thus rounding down this value to 0.1 at % C as proposed by the opposition division was admissible and sensible, in particular since in document D4 carbon was rated as being an undesirable impurity that should not exceed 0.02 wt%. The objection of lack of novelty of the claimed alloy composition held by the opposition division vis-à-vis over-check analysis 2665-3 of D17 and sample 10 of D4 was therefore justified.

As to inventive step, D4 qualified as closest prior art. Starting from the composition of example 10 given in Table 1 of D4, it would not involve an inventive step to reduce the carbon content to a level as low as possible for the reasons mentioned above. For the same reason, the skilled person would reduce the carbon content of Hastelloy B-2 referred to in document D17. In so doing, the skilled metallurgist would - by combining the technical teaching of D4 and D17 - arrive at the alloy composition claimed in the patent. Selecting the elemental area of alloy compositions set out in claim 1 of the main, first and second auxiliary request therefore did not involve an inventive step.

Reasons for the Decision

1. The appeal is admissible.

2. *Amendments; Article 123(2),(3) EPC*

Claim 1 of the main request results from a combination of the subject matter featuring in claims 1, 5 and 13

as granted, whereas claim 1 of the first auxiliary request is based on claims 1 and 2 as granted.

Claim 1 of the second auxiliary request is supported by claims 1, 2, 4 and 13 as granted (claims 1, 2, 4, 16 as originally filed). The selection of "chromium, tungsten, manganese or cobalt" from the elements in Groups VIA, VIIA, or VIII of the Periodic Table in claim 1 is derivable from claim 3 as granted (claim 3 as originally filed). Dependent claims 2 to 4 correspond to claims 3, 6 and 7 as granted (claims 3, 6 and 7 as originally filed.

Hence, there are no formal objections to the claims with respect to Article 123 EPC.

3. *Novelty*

3.1 According to the position of the opposition division and the respondent, either example 10 of document D4 or the composition of the over-check 2665-3 disclosed in document D17 (D17') anticipate the Ni-Mo alloy set out in claim 1 of all requests.

The alloy compositions of the prior art D4 and D17 are expressed in weight percent (wt%). A re-calculation into atom percent (at%) (calculations made by the opponent) leads to the following values:

	D4, Table 1, ex. 10 (at%).	D17' 2663-3 (at%)
Ni	76,37	**77.05**
Mo	18,38	19.53
Fe	2.229	1.50

Cr	1,284	0.50
Al	0.889	0.60
Si	0.508	0.10
C	**0.135**	0.087
P	0.104	0.00
S	0.104	0.00
total	100.00	99.997
c+d =	4.91	3.33
c+d+0.7(%Mo)	17.776	17.001

Applying the usual rules for rounding, as for instance given in Designation E29-02 (document D18), to sample 10 and alloy 2663-3, the amounts of 0.135 at% C and 77.05 at% Ni are to be read - in the respondent's view - as 0.1 at% C and 77 at% Ni, respectively, since claim 1 of the main request defines the upper limits for C and Ni without any indication of decimals, thus including also the values of 0.135 at% or 77.05 at% Ni.

Moreover, the respondent is of the opinion that the same principle could be applied also to claim 1 of the first and second auxiliary request which restrict the upper limit for carbon to 0.05% and for nickel of 76.5 at% which are extremely close to 0.135 at C and 77.05 at% Ni.

3.2 The board cannot follow this line of argument, since it is not in conformity with the standard practice of the boards of appeal (see T 74/98, in particular point 3).

3.2.1 Turning to the patent under consideration, the skilled reader is unambiguously taught that carbon in the claimed Ni-Mo alloy represents an undesirable interstitial element which should be kept as low as

possible. More specifically, carbon can be tolerated at levels up to 0.05 at% (cf. the patent, page 6, lines 14 to 17) but in any case, it should not exceed 0.1 at% (see claim 1). This statement is also true for the nickel content of the alloy which should be less 77 at%. The wording of claim 1 itself therefore makes it clear that the ranges for carbon and nickel are to be interpreted as they stand: values exceeding 0.1 at% carbon or 77 at% nickel are stated not to be included in the claim. To interpret an upper limit of 0.1 at% carbon or 77 at% Ni, respectively, so as to include all values which, upon application of rounding rules, would have those numbers as the outcome, would inevitably result in expanding the scope of the claim beyond the indicated limits. In so doing serious doubt would be cast upon the meaning of ranges in general.

3.2.2 Moreover, the contents of 0.135 at% carbon (D4, Table 1, sample 10) and of 77.05 at% nickel (of alloy 2665-3 of D17') have been re-calculated from original compositions expressed in weight percentage. It is immediately evident that rounding down these figures (in at%) would imply a modification of the original alloy composition (in wt%), given that 0.1 at% C no longer corresponds to 0.025 wt% C of example 10 in Table 1 of document D4. The true meaning of a specific disclosure cannot, however, be influenced by the units chosen to express it. Consequently, the objection of lack of novelty relies on an ambiguity introduced by rounding up or down re-calculated values.

The same statements apply also to the alloys set out in claim 1 of the first and second auxiliary requests in which carbon and nickel have been further restricted to

not more than 0.05 at% C and not more than 76.5 at% Ni, respectively, and wherein the sum of "c" + "d" = 3 to 7 at% (1. auxiliary request) and the sum of ("c" + "d" + 0.7 • "b") = 18 to 20 at% (second auxiliary request).

3.3 It is therefore concluded that the subject matter of claim 1 according to the main, the first and second auxiliary requests is not anticipated by the disclosure of documents D4 or D17 and hence is novel.

4. *Inventive step*

4.1 Main and first auxiliary requests:

4.1.1 Document D17 deals with the storage of eutectic CaF_2-LiF fluoride salts in corrosion resistant canisters of Hastelloy B2. Table 1 of document D17 includes (in wt%) mill certificates 1 and 2 of alloy B2 provided by the manufacturer (Haynes Int. Inc) and three over-check analysis performed by the client (Rockwell Int.) in which the tubes have been verified to comply with ASME requirements (cf. Table 1, SB626, in weight %: ≤0.02% C, ≤1.0% Mn, ≤0.03% S, ≤0.04% P, ≤0.10% Si, ≤1.0% Cr, ≤1.0% Co, 26 to 30% Mo, ≤2.0% Fe, balance Ni). In the appellant's view which is corroborated by the declarations of Mr Klarstrom and Gillette, "over-check" is a term in industry to identify a particular type of analysis a customer performs on a product to determine if the product conforms to the applicable specification. However, he is further of the opinion that the over-check analysis is not carried out with a very high accuracy and consequently, over-check analysis 2663-3 is to be rated only as an inaccurate report of mill

certificate 1 which represents the most accurate and true chemical analysis of the Hastelloy B2 tubing delivered.

4.1.2 However, the board cannot see any specific reason as to why the accuracy of over-check analysis 2665-3 should be put in doubt in relation to that of the mill certificate 1, all the more so since both companies (Haynes International Inc. and Rockwell International /Rocketdyne Division) are regarded as being fully competent in that field of metallurgy. Contrary to the position of the appellant, the conclusion must be drawn that a product exhibiting the composition "heat no 2665-3" disclosed in D17 within the Hastelloy B-2 limits actually existed. Except for the nickel content of 77.05 at% which is 0.05 at% (i.e. slightly) above the claimed upper limit of 77 at% Ni, this composition satisfies all elemental limitations for the alloy defined in claim 1 of the main request and it is also very close to the Ni-Mo alloy composition defined in claim 1 of the first auxiliary request in which the upper limits for Ni and C are 76.5 at% and 0.05 at%, respectively.

The question therefore arises whether such a small compositional difference between the claimed alloy and that of the prior art is actually associated with a change of properties, in particular a reduced order induced grain boundary embrittlement without sacrifice in corrosion resistance (cf. page 3, lines 18 to 20). At the oral proceedings, the appellant could not satisfy the board that the mechanical and anti-corrosion properties of the claimed alloy set out in claim 1 of the main request are significantly different

from those of D17, heat no. 2665-3. In this respect, the appellant pointed to D17, Table 5, disclosing a very low elongation of 2.5% at a test temperature of 1300°F (704°C) for the Hastelloy B-2 tubes, whereas the claimed alloy compositions disclosed in Table D of the patent exhibited much higher values for the 700°C-1h-elongation.

However, as it is apparent from Table D of the patent, examples 13 and 14 likewise exhibit a 700°C/1h elongation of 1.7 and 1.3%, respectively. Hence, a technical effect which could justify an inventive step of the claimed Ni-Mo alloy vis-à-vis the example of Hastelloy B2 given in document D17 is not discernable. The subject matter of claim 1 of the main request and of the first auxiliary request therefore lacks an inventive step with respect to the disclosure of document D17.

4.2 Second auxiliary request:

Claim 1 of the second auxiliary further specifies a relationship between the Mo-content and the amount of the alloying elements expressed by "the sum of "c" plus "d" plus 0.7 times "b" = between 18 and 20 atom percent." This means that lower total amounts of alloying elements are needed with increasing amounts of Mo in order to obtain a good ductility or elongation of above 10 % (cf. page 5, lines 38 to 46 and Figure 7).

4.2.1 The opponent has not submitted any evidence to prove the contrary or that the additional limitation is redundant, but pointed to the D17 alloy 2665-3 with c+d+0.7b = 17,001% and to D4, sample 10 with c+d+0.7b =

17.776%. In its view, both compositions are very close to the claimed alloy and should, therefore, exhibit the same technical properties. Hence, it was obvious to design Ni-base alloys having a composition close to those given in D17 and D4.

4.2.2 Document D17 could qualify as closest prior art since this document discloses the technical properties of Hastelloy B-2, which likewise has been chosen as a starting point in the patent at issue (cf. the patent, page 2, lines 50 to 58). As the patent observes on page 3, lines 1 to 11, very rapid order reactions can take place in Hastelloy B-2 forming ordered intermetallic phases Ni_2Mo, Ni_3Mo and Ni_4Mo which result in a severe age embrittlement and catastrophic failures in stressed structures such as cold worked vessels when exposed to high temperatures for even a short time. Starting from Hastelloy B-2, the problem underlying the patent at issue therefore resided in providing a high-Mo-Ni-base alloy which is not prone to rapid, order induced, grain boundary embrittlement but which still exhibits a high corrosion resistance (cf. the patent, page 3, lines 18 to 20).

The solution to this problem is the narrowly restricted area of Ni-base alloys set out in claim 1 of the second auxiliary request which - compared with commercial alloy B-2 - exhibit a greatly enhanced thermal stability, i.e. a significantly reduced age embrittlement as well as a superior corrosion resistance and which are designated as "Alloy B-3". Having regard to the test results of the many examples given in Tables B, C and D of the patent, the board has no reason to doubt that these objects are successfully

obtained by the claimed alloy composition, i.e. a reduced embrittlement and, in consequence thereof, such a significant improvement in elongation.

Document D17 actually deals with cannister tubes made of Hastelloy B-2 which generally comprises 26-30wt% Mo - Ni and can include up to 1 wt% Cr, 1 wt% Co and up to 2.0 wt% Fe. It appears from the examples 2 to 5 according to the patent and also from mill certificates 1 and 2 which are all representatives of alloy B-2 that the nickel content of alloy B-2 is generally greater than 77 at% and the molybdenum content lower than 19 at%. As to the composition of over-check 2663-5, the total of c+d+0.7b is 17.001 which is outside the range of 18 to 20 for the relationship given in claim 1 of the second auxiliary request. Moreover, the contents of Ni and C fall outside claimed ranges. In document D17, the tested samples of alloy B-2 were found suitable for the containment of fluoride salts and the canisters were cycled through a maximum of 7231 cycles without failure (cf. D17, abstract). There is, however, no suggestion whatsoever in this document as to how the composition of alloy B-2 could be modified to prevent order induced, grain boundary embrittlement. The teaching of document D17 thus could not lead to the claimed high Mo-Ni-base alloy.

4.2.3 Turning to document D4, this document aims at providing a Ni-base alloy which is highly resistant to stress corrosion cracking without precipitating any intermetallic compounds even after subjecting the alloy to thermal processing. Given that D4 addresses the same problem underlying the patent at issue, it also qualifies as closest prior art. The problem is solved

by the corrosion resistant Ni-base alloy set out in claim 1 of document D4. It is, however, noted that the Ni-base alloys defined in the claims of D4 are iron-free, as are the (46) examples listed in Tables 2 and 3. Contrary thereto, D4 mentions in column 2, line 60 to column 3, line 10 that 0.5 to 6.0 wt% Fe is desirable but that Al can substitute for Fe. Examples containing Fe and Al are given in Table 1 which are, however, outside the scope of claim 1 of D4. Considering the whole teaching of document D4 the skilled reader is, therefore, taught that aluminium in the range of 0.3 to 2 wt% is the most important element which should be present together with at least one of W, Cr and Cu. When producing an alloy having the desired properties, a person skilled in the art would thus in the first place turn to the iron-free, Al-containing alloys rather than to the Fe-containing examples given in Table 1 since the latter are outside the scope of claim 1 of D4. And even if he did, nothing in this document would motivate the skilled practitioner to select (among 36 examples) alloy 10 of Table 1, all the more so since many other examples of Table 1 exhibit a better corrosion resistance.

More importantly, however, document D4 does not teach or suggest that the relationship between the amounts "c" and "d" of the compulsory and optional substitutional alloying elements and the molybdenum content "b" is important to achieve the desired combination of the properties. Hence, this document fails to comprises any indication towards designing alloy compositions falling within the restricted area of alloys set out in claim 1 of the second auxiliary request.

4.3 The subject matter of claim 1 of the second auxiliary request therefore involves an inventive step.

4.4 Dependent claims 2 to 4 are concerned with preferred embodiments of the Mo-Ni-base alloy set out in claim 1 of the second auxiliary request 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 department with the order to maintain the patent on the basis of claims 1 to 4 according to the second auxiliary request filed during the oral proceedings and the description and drawings to be adapted to these claims.

The Registrar: The Chairman:

V. Commare T. K. H. Kriner