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(54) Title: FABRICABLE, HIGH STRENGTH, OXIDATION RESISTANT NI-CR-CO-MO-AL ALLOYS

(57) Abrégé/Abstract:

Ni-Cr-Co-Mo-Al based alloys are disclosed which contain 15 to 20 wt.% chromium, 9.5 to 20 wt.% cobalt, 7.25 to 10 wt.% molybdenum, 2.72 to 3.9 wt.% aluminum, along with typical impurities, a tolerance for up to 10.5 wt.% iron, minor element additions and a balance of nickel. These alloys are readily fabricable, have high creep strength, and excellent oxidation resistance up to as high as 2100°F (1149°C). This combination of properties is useful for a variety of gas turbine engine components, including, for example, combustors.

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TITLE

FABRICABLE, HIGH STRENGTH, OXIDATION RESISTANT Ni-Cr-Co-Mo-Al ALLOYS

FIELD OF THE INVENTION

This invention relates to fabricable, high strength alloys for use at elevated temperatures. In particular, it is related to alloys which possess excellent oxidation resistance, high creep-rupture strength, and sufficient fabricability to allow for service in gas turbine engine combustors and other demanding high temperature environments.

BACKGROUND OF THE INVENTION

For sheet fabrications in gas turbine engines a variety of commercial alloys are available. These alloys can be divided into different families based on their key properties. Note that the following discussion relates to alloys which are cold fabricable/weldable, meaning that they can be produced as cold rolled sheet, cold formed into a fabricated part, and welded.

Gamma-prime formers. These include R-41 alloy, Waspaloy alloy, 282® alloy, 263 alloy, and others. These alloys are characterized by their high creep-rupture strength. However, the maximum use temperatures of these alloys are limited by the gamma-prime solvus temperature and are generally not used above 1600-1700°F (871 to 927°C). Furthermore, while the oxidation resistance of these alloys is quite good in the use temperature range, at higher temperatures it is less so.

Alumina-formers. These include 214® alloy and HR-224® alloy, but not the ODS alloys (which do not have the requisite fabricability). The alloys in this family have excellent oxidation resistance at temperatures as high as 2100°F (1149°C). However, their use in structural components is limited due to poor creep strength at temperatures above around 1600-1700°F (871 to 927°C). Note that these alloys will also form the strengthening gamma-prime, but this phase is not stable in the higher temperature range.

Solid-solution strengthened alloys. These include 230® alloy, HASTELLOY® X alloy, 617 alloy, and others. As their name implies, these alloys derive their high creep-rupture strength primarily from the solid-solution strengthening effect, as well carbide formation. This strengthening remains effective even at very high temperatures – well above the maximum temperature of the gamma-prime formers, for example. Most of the solid-solution strengthened alloys have very good oxidation resistance due to the formation of a protective chromia scale. However, their oxidation resistance is not comparable to the alumina-formers, particularly at the very high temperatures, such as 2100°F (1149°C).

Nitride dispersion strengthened alloys. These include NS-163® alloy which has very high creep-rupture strength at temperatures as high as 2100°F (1149°C). While the creep-rupture strength of NS-163 alloy is better than the solid-solution alloys, its oxidation resistance is only similar. It does not have the excellent oxidation resistance of the alumina-formers.

What is clear from the above discussion is that there is no cold fabricable/weldable alloy commercially available which combines both high creep-rupture strength and excellent oxidation resistance. However, in the effort to continually push gas turbine engine operating temperatures higher and higher, it is clear that alloys which combine these qualities would be very desirable.

SUMMARY OF THE INVENTION

The principal object of this invention is to provide readily fabricable alloys which possess both high creep-rupture strength and excellent oxidation-resistance. This is a highly valuable combination of properties not found in (or expected from) the prior art. The composition of alloys which have been discovered to possess these properties is: 15 to 20 wt.% chromium (Cr), 9.5 to 20 wt.% cobalt (Co), 7.25 to 10 wt.% molybdenum (Mo), 2.72 to 3.9 wt.% aluminum (Al), and carbon (C), present up to 0.15 wt.%. The elements titanium (Ti) and niobium (Nb) may be present, for instance to provide strengthening, but should be limited in quantity due to their adverse effect on certain aspects of fabricability. In particular, an abundance of these elements may increase the propensity of an alloy for strain-age cracking. If present, titanium should be limited to no more than 0.75 wt.%, and niobium to no more than 1 wt.%.

The presence of the elements hafnium (Hf) and/or tantalum (Ta) has unexpectedly been found to be associated with even greater creep-rupture lives in these alloys. Therefore, one or both elements may be added to these alloys to further improve creep-rupture strength. Hafnium may be added at levels up to around 1 wt.%, while tantalum may be added at levels up to around 1.5 wt.%. To be most effective, the sum of the tantalum and hafnium contents should be between 0.2 wt.% and 1.5 wt.%.

To maintain fabricability, certain elements which may or may not be present (specifically, aluminum, titanium, niobium, and tantalum) should be limited in quantity in a manner to satisfy the following additional relationship (where elemental quantities are in wt.%):

$$Al + 0.56Ti + 0.29Nb + 0.15Ta \leq 3.9$$

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Additionally, boron (B) may be present in a small, but effective trace content up to 0.015 wt.% to obtain certain benefits known in the art. Tungsten (W) may be present in this alloy up to around 2 wt.%. Iron (Fe) may also be present as an impurity, or may be an intentional addition to lower the overall cost of raw materials. However, iron should not be present more than around 10.5 wt.%. If niobium and/or tungsten are present as minor element additions, the iron content should be further limited to 5 wt.% or less. To enable the removal of oxygen (O) and sulfur (S) during the melting process, these alloys typically contain small quantities of manganese (Mn) up to about 1 wt.%, and silicon (Si) up to around 0.6 wt.%, and possibly traces of magnesium (Mg), calcium (Ca), and rare earth elements (including yttrium (Y), cerium (Ce), lanthanum (La), etc.) up to about 0.05 wt.% each. Zirconium (Zr) may be present in the alloy, but should be kept to less than 0.06 wt.% in these alloys to maintain fabricability.

DESCRIPTION OF THE PREFERRED EMBODIMENTS

We provide Ni-Cr-Co-Mo-Al based alloys which contain 15 to 20 wt.% chromium, 9.5 to 20 wt.% cobalt, 7.25 to 10 wt.% molybdenum, 2.72 to 3.9 wt.% aluminum, along with typical impurities, a tolerance for up to 10.5 wt.% iron, minor element additions and a balance of nickel, which are readily fabricable, have high creep strength, and excellent oxidation resistance up to as high as 2100°F (1149°C). This combination of properties is useful for a variety of gas turbine engine components, including, for example, combustors.

Based on the understanding of the requirements of future gas turbine engine combustors, an alloy with the following attributes would be highly desirable: 1) excellent oxidation resistance at temperatures as high as 2100°F (1149°C), 2) good fabricability, such that it can be produced in wrought sheet form, cold formed, welded, etc., 3) high temperature creep-strength as good or better than common commercial alloys, such as HASTELLOY X alloy, and 4) good thermal

stability at elevated temperatures. Historically, attempts to develop an alloy combining all four properties have not been successful, and correspondingly, no commercial alloy is available in the marketplace with all four of these qualities.

We tested 30 experimental alloys whose compositions are set forth in Table 1. The experimental alloys have been labeled A through Z and AA through DD. The experimental alloys had a Cr content which ranged from 15.3 to 19.9 wt.%, as well as a cobalt content ranging from 9.7 to 20.0 wt.%. The molybdenum content ranged from 5.2 to 12.3 wt.%. The aluminum content ranged from 1.93 to 4.30 wt.%. Iron ranged from less than 0.1 up to 10.4 wt.%. Minor element additions including titanium, niobium, tantalum, hafnium, tungsten, yttrium, silicon, carbon, and boron were present in certain experimental alloys.

All testing of the alloys was performed on sheet material of 0.065" to 0.125" (1.6 to 3.2 mm) thickness. The experimental alloys were vacuum induction melted, and then electro-slag remelted, at a heat size of 30 to 50 lb (13.6 to 27.2 kg). The ingots so produced were hot forged and rolled to intermediate gauge. The sheets were annealed, water quenched, and cold rolled to produce sheets of the desired gauge. Intermediate annealing of cold rolled sheet was necessary during production of the 0.065" sheet (1.6 mm). The cold rolled sheets were annealed as necessary to produce a fully recrystallized, equiaxed grain structure with an ASTM grain size between 3½ and 4½.

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Table 1
Compositions of Experimental Alloys (in wt.%)

Alloy	Ni	Cr	Co	Mo	Al	Fe	C	Si	Mn	Ti	Y	Zr	B	Other
A	Bal.	19.9	14.8	7.8	3.64	1.2	0.096	0.15	--	0.25	0.02	0.04	0.004	
B	Bal.	19.8	10.1	7.7	3.56	1.3	0.088	0.14	--	0.25	0.02	0.04	0.004	
C	Bal.	16.1	19.9	7.6	3.65	1.3	0.099	0.14	--	0.24	0.02	0.04	0.004	
D	Bal.	16.1	19.9	7.7	3.54	5.2	0.079	0.14	--	0.25	0.02	0.02	0.004	
E	Bal.	16.0	19.8	7.7	3.62	9.7	0.085	0.14	--	0.25	0.02	0.01	0.004	
F	Bal.	16.0	10.1	7.7	3.46	1.2	0.097	0.14	--	0.22	0.01	0.02	0.004	
G	Bal.	16.1	9.9	7.8	3.51	9.9	0.089	0.13	--	0.23	0.01	0.02	0.005	
H	Bal.	16.0	19.7	9.5	3.56	1.2	0.107	0.17	--	0.24	<0.005	0.02	0.005	
I	Bal.	15.8	19.3	7.5	3.60	1.0	0.110	0.18	--	0.23	0.02	0.02	0.004	1.94 W
J	Bal.	16.0	9.8	9.5	3.58	9.9	0.116	0.17	--	0.22	0.02	0.01	0.005	
K	Bal.	16.3	19.3	7.5	3.50	1.1	0.104	0.14	--	0.22	0.02	0.04	0.004	0.43Hf
L	Bal.	16.2	20.0	7.8	3.48	1.0	0.106	0.22	--	0.23	0.02	0.02	0.005	0.71Ta
M	Bal.	16.6	10.1	7.7	3.75	10.4	0.108	0.15	--	0.23	0.02	0.03	0.004	0.38Hf
N	Bal.	16.7	10.2	7.8	3.64	10.2	0.110	0.19	--	0.23	0.02	0.02	0.005	0.78Ta
O	Bal.	16.0	19.9	7.5	3.60	1.1	0.107	0.17	--	0.23	0.02	0.02	0.004	0.35Nb, 0.69Ta
P	Bal.	16.0	9.9	7.5	3.63	10.0	0.107	0.19	--	0.23	0.02	0.02	0.004	1.93 W
Q	Bal.	16.2	10.1	7.6	3.65	10.2	0.112	0.18	--	0.22	0.02	0.02	0.005	0.35Nb, 0.71Ta
R	Bal.	15.3	20	10.0	3.32	<0.1	0.114	0.19	0.20	0.22	0.01	0.04	0.004	
S	Bal.	15.9	9.9	9.5	3.78	1.0	0.107	0.47	0.19	0.02	0.011	0.04	0.004	
T	Bal.	16.0	9.9	7.6	2.72	4.5	0.120	0.17	0.20	0.22	0.015	0.04	0.004	1.89 W, 0.91 Nb
U	Bal.	19.5	19.9	7.6	3.36	1.1	0.103	0.17	0.20	0.49	0.013	0.04	0.005	
V	Bal.	19.0	9.9	8.0	3.40	1.0	0.090	0.18	0.15	0.21	0.011	0.04	0.005	0.48 Hf
W	Bal.	18.9	19.9	7.5	3.31	1.0	0.086	0.18	0.14	0.21	0.009	0.03	0.004	1.0 Ta
X	Bal.	19.2	19.9	7.7	3.40	1.0	0.088	0.17	0.13	0.21	0.011	0.04	0.004	0.45 Hf
Y	Bal.	16.4	10.2	7.8	2.81	1.1	0.108	0.49	0.50	0.22	0.010	0.04	0.004	
Z	Bal.	19.0	10	7.4	3.19	1.0	0.091	0.18	0.16	0.21	0.008	0.03	0.004	1.0 Ta
AA	Bal.	19.2	20	5.2	3.37	1.0	0.107	0.18	0.20	0.24	0.012	0.04	0.004	
BB	Bal.	19.3	20	12.3	3.67	1.0	0.099	0.51	0.53	0.42	0.011	0.04	0.004	
CC	Bal.	19.4	10	9.6	1.93	1.0	0.107	0.19	0.21	0.24	<0.002	<0.01	0.004	
DD	Bal.	18.9	10	9.5	4.30	1.0	0.117	0.49	0.21	0.43	0.005	0.05	0.004	

To evaluate the key properties (oxidation resistance, fabricability, creep strength, and thermal stability) four different types of tests were performed on experimental alloys to establish

their suitability for the intended applications. The results of these tests are described in the following sections.

Oxidation Resistance Oxidation resistance is a key property for an advanced high temperature alloy. Temperatures in the combustor of a gas turbine engine can be very high and there is always a push in the industry for higher and higher use temperatures. An alloy having excellent oxidation resistance at as high as 2100°F (1149°C) would be a good candidate for a number of applications. The oxidation resistance of nickel-base alloys is strongly affected by the nature of the oxides which form on the surface of the alloy upon thermal exposure. It is generally favorable to form a protective surface layer, such as chromium-rich and aluminum-rich oxides. Alloys which form such oxides are often referred to as chromia or alumina formers, respectively. The vast majority of wrought high temperature nickel alloys are chromia formers. However, a few alumina-formers are commercially available. One such example is HAYNES® 214® alloy. The 214 alloy is well known for its excellent oxidation resistance.

For the purpose of determining the oxidation resistance of the experimental alloys, oxidation testing was conducted on most of the alloys in flowing air at 2100°F (1149°C) for 1008 hours. Also tested alongside these samples were five commercial alloys: HAYNES 214 alloy, 617 alloy, 230 alloy, 263 alloy, and HASTELLOY X alloy. Samples were cycled to room temperature weekly. At the conclusion of the 1008 hours the samples were descaled and submitted for metallographic examination. Recorded in Table 2 are the results of the oxidation tests. The recorded value is the average metal affected, which is the sum of the metal loss plus the average internal penetration of the oxidation attack. Details of this type of testing can be found in International Journal of Hydrogen Energy, Vol. 36, 2011, pp. 4580-4587. For the

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purposes of this invention, an average metal affected value of 2.5 mils/side (64 μm /side) or less was the preferred objective and an appropriate indication of whether a given alloy could be considered as having “excellent” oxidation resistance. Indeed, metallographic examination of the alloys with less than this level of attack confirm their desirable oxidation behavior. Certain minor elements/impurities could possibly result in somewhat reduced (but still acceptable) oxidation resistance, therefore the average metal affected value could probably be as high as 3 mils/side (76 μm /side) while still maintaining excellent oxidation resistance.

Table 2

2100°F (1149°C) Oxidation Test Results

Alloy	Average Metal Affected	
	(mils/side)	(μm /side)
A	0.9	23
B	0.9	23
C	0.7	18
D	1.0	25
E	0.6	15
F	0.9	23
G	0.9	23
H	0.4	10
I	0.6	15
J	0.6	15
K	1.8	46
L	0.7	18
M	1.5	38
N	0.5	13
O	0.6	15
P	0.5	13
Q	0.4	10
R	0.9	23
S	0.6	15

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T	1.1	28
U	1.4	36
V	2.3	58
W	0.5	13
X	1.6	41
Z	0.5	13
CC	4.4	112
263	16.5	419
214	1.3	33
617	5.1	130
230	4.8	122
HASTELLOY X	12.0	305

The results of the oxidation testing of the experimental alloys were very impressive. All of the tested experimental alloys (with the exception of alloy CC) had an average metal affected of 2.3 mils/side (58 μm) or less. Therefore, all of these alloys (with the exception of alloy CC) had acceptable oxidation resistance for the purposes of this invention. Considering the commercial alloys, the experimental alloys were all comparable to the alumina-forming HAYNES 214 alloy, which had an average metal affected value of 1.3 mils/side (33 μm). In contrast, the chromia-forming 617 alloy, 230 alloy, HASTELLOY X alloy, and 263 alloy all had much higher levels of oxidation attack, with average metal affected values of 5.1, 4.8, 12.0, and 16.5 mils/side (130, 122, 305, and 419 μm), respectively. The excellent oxidation resistance of the experimental alloys is believed to derive from a critical amount of aluminum, which was 2.72 wt.% or greater for all of the experimental alloys other than alloy CC. Alloy CC had an Al value of only 1.93 wt. %, illustrating that this is too low an Al level for the desired excellent oxidation resistance. Similarly, the Al levels of the four chromia-forming commercial alloys were quite low (the highest being 617 alloy with 1.2 wt. % Al). In contrast, the alumina forming 214 alloy has an Al content of 4.5 wt.%. In summary, all of the nickel-base alloys tested in this program

with an Al level of 2.72 wt.% or more were found to have excellent oxidation resistance, while those with lower Al levels did not. Therefore, to be considered an alloy of the present invention the Al level of the alloy should be greater than or equal to 2.72 wt. %.

Fabricability One of the requirements of the alloys of this invention is that they are fabricable. As discussed previously, for alloys containing significant amounts of certain elements (such as aluminum, titanium, niobium, and tantalum), having good fabricability is closely tied to the alloy's resistance to strain-age cracking. The resistance of the experimental alloys to strain-age cracking was measured using the modified CHRT test described by Metzler in Welding Journal supplement, October 2008, pp. 249s-256s. This test was developed to determine an alloy's relative resistance to strain-age cracking. It is a variation of the test described in U.S. Patent No. 8,066,938. In the modified CHRT test, the width of the gauge section is variable and the test is performed on a dynamic thermo-mechanical simulator rather than a screw-driven tensile unit. The results of the two different forms of the test are expected to be qualitatively similar, but the absolute quantitative results will be different. The results of the modified CHRT testing performed on our experimental alloys are shown in Table 3. The testing was conducted at 1450°F (788°C), and the reported CHRT ductility values were measured as elongation over 1.5 inches (38 mm). The modified CHRT test ductility of the experimental alloys ranged from 5.9% for alloy DD to 17.9% for alloy X.

Also shown in Table 3 are the modified CHRT test results for three commercial alloys as published by Metzler in Welding Journal supplement, October 2008, pp. 249s-256s. The modified CHRT test ductility values for R-41 alloy and Waspaloy were both less than 7%, while the value for 263 alloy was 18.9%. The R-41 alloy and Waspaloy alloy, while weldable, are both known to be susceptible to strain-age cracking, whereas 263 alloy is considered readily

weldable. For this reason, alloys of the present invention should possess modified CHRT test ductility values greater than 7%. Of the experimental alloys only alloys O and DD had a modified CHRT test ductility value less than 7%; therefore alloys O and DD cannot be considered alloys of the present invention.

Table 3

Results of the Modified CHRT test

Alloy	Modified CHRT Test Ductility (%)
A	13.0
B	11.6
C	7.7
D	13.3
E	13.6
F	8.9
G	10.3
H	8.7
I	9.4
J	10.2
K	8.6
L	8.0
M	9.7
N	10.0
O	6.3
P	9.3
Q	10.2
R	10.8
S	9.4
T	9.9
U	9.5
V	15.1
W	16.3
X	17.9
Y	13.5
Z	11.9
AA	10.5
BB	8.9

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CC	15.3
DD	5.9
R-41	6.9
WASPALLOY	6.8
263	18.9

It was discovered that for these Ni-Cr-Co-Mo-Al based alloys, the resistance to strain age cracking could be associated with the total amount of the gamma-prime forming elements Al, Ti, Nb, and Ta. Therefore, the combined amount of these elements present in the alloy should satisfy the following relationship (where the elemental quantities are given in weight %):

$$\text{Al} + 0.56\text{Ti} + 0.29\text{Nb} + 0.15\text{Ta} \leq 3.9 \quad [1]$$

The values of the left-hand side of equation 1 are shown in Table 4 for all of the experimental alloys. All alloys where $\text{Al} + 0.56\text{Ti} + 0.29\text{Nb} + 0.15\text{Ta}$ was less than or equal to 3.9 can be seen to have greater than 7% modified CHRT test ductility and therefore pass the strain-age cracking resistance requirement of the present invention. Only alloys O, Q, and DD were found to have values greater than 3.9. For alloys O and DD, the values of 3.93 and 4.54 can be correlated with poor modified CHRT test ductility. On the other hand, alloy Q was found to have acceptable modified CHRT test ductility. It is believed that this is a result of the alloy's high Fe content. Fe additions are known to suppress the formation of gamma-prime and could therefore help to improve the modified CHRT test ductility. Nevertheless, a lower amount of gamma-prime forming elements is generally beneficial for fabricability. Therefore, the value of $\text{Al} + 0.56\text{Ti} + 0.29\text{Nb} + 0.15\text{Ta}$ should be kept to less than or equal to 3.9 for all alloys of the present invention. Note that one implication of this is that the maximum aluminum content of the alloys of this invention must be 3.9 wt.% (which corresponds to the case where titanium, niobium, and tantalum are all absent).

Table 4

Experimental Alloys - Eq. [1] value (left-hand side)

Alloy	$Al + 0.56Ti + 0.29Nb + 0.15Ta$
A	3.78
B	3.70
C	3.78
D	3.68
E	3.76
F	3.58
G	3.64
H	3.69
I	3.73
J	3.70
K	3.62
L	3.72
M	3.88
N	3.89
O	3.93
P	3.76
Q	3.98
R	3.44
S	3.79
T	3.11
U	3.63
V	3.52
W	3.58
X	3.52
Y	2.93
Z	3.46
AA	3.50
BB	3.90
CC	2.06
DD	4.54

Creep-Rupture Strength The creep-rupture strength of the experimental alloys was determined using a creep-rupture test at 1800°F (982°C) under a load of 2.5 ksi (17 MPa). Under these conditions, the creep-resistant HASTELLOY X alloy is estimated (based on interpolated data

from Haynes International, Inc. publication #H-3009C) to have a creep-rupture life of 285 hours. For the purposes of this invention, a minimum creep-rupture life of 325 hours was established as the requirement, which would be a marked improvement over HASTELLOX alloy. It is useful to note that the test temperature of 1800°F (982°C) is greater than the predicted gamma-prime solvus temperature of the experimental alloys, thus any effects of gamma-prime phase strengthening should be negligible.

The creep-rupture life of the experimental alloys is shown in Table 5 along with those of several commercial alloys. Alloys A through O, R through Z, and BB, were all found to have creep-rupture lives greater than 325 hours under these conditions, and therefore meet the creep-rupture requirement of the present invention. Alloys P, Q, AA, CC and DD were found to fail the creep-rupture requirement. Considering the commercial alloys, 617 alloy and 230 alloy had acceptable creep-rupture lives of 732.2 and 915.4 hours, respectively. Conversely, the 214 alloy had a creep-rupture life of only 196.0 hours – well below that of the creep-rupture life requirement which defines alloys of the present invention.

Table 5

Creep-Rupture Life at 1800°F (982°C)/2.5 ksi (17 MPa)

Alloy	Rupture Life (hours)
A	1076.7
B	534.7
C	486.1
D	447.0
E	331.9
F	402.8
G	722.0
H	2051.1
I	360.0
J	1785.7

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K	5645.5
L	566.7
M	1317.4
N	1197.3
O	340.3
P	134.3
Q	254.4
R	> 500
S	> 500
T	> 330
U	> 500
V	1624.0
W	693.8
X	> 500
Y	> 500
Z	909.4
AA	276.0
BB	> 500
CC	224.3
DD	138.6
617	732.2
214	196.0
230	915.4
HASTELLOY X	285 (estimated)

Certain experimental alloys containing either hafnium or tantalum, were found to exhibit surprisingly greater creep-rupture lives than many of the other experimental alloys. For example, the hafnium-containing Alloy K has a creep-rupture life of 5645.5 hours, and the tantalum-containing alloy N has a creep-rupture life of 1197.3 hours. A comparison of alloys with and without hafnium and tantalum additions is given in Table 6. For comparative purposes, the alloys are grouped according to their nominal base composition. A clear benefit of hafnium and tantalum additions on the creep-rupture life can be seen for all base compositions. However, any beneficial effect of tantalum on the creep-rupture strength must be weighed against any negative effects on the fabricability as described previously in this document.

Table 6

Effects of Hafnium and Tantalum Additions on Creep-Rupture Life

1800°F (982°C)/2.5 ksi (17 MPa)

Nominal Base Composition	Alloy	Addition	Creep-Rupture Life (h)
Ni-16Cr-20Co-7.5Mo-3.5Al-1Fe	C	-	486.1
	L	0.43 Hf	5645.5
	K	0.71 Ta	566.7
Ni-16Cr-10Co-7.5Mo-3.5Al-10Fe	P	-	134.3
	M	0.38 Hf	1317.4
	N	0.78 Ta	1197.3
Ni-19.5Cr-10Co-7.5Mo-3.5Al-1Fe	B	-	534.7
	V	0.48 Hf	1624.0
	Z	1 Ta	909.4

As mentioned above, the experimental alloys P and Q, both of which contain around 10 wt.% iron, failed the creep-rupture requirement. These alloys contained minor element additions of tungsten and niobium, respectively. It is useful to compare these alloys to alloy G which is similar to these two alloys, but without a tungsten or niobium addition. Alloy G was found to have acceptable creep-rupture life. Therefore, when alloys from this family are at their upper end of the iron range (~10 wt.%) the elements tungsten and niobium appear to have a negative effect on the creep-rupture life. However, when the iron content is lower, for example alloys I and T, tungsten additions do not result in unacceptable creep-rupture lives. Similarly, niobium additions do not result in unacceptable creep-rupture lives when the iron content is lower (alloy T). For these reasons, the alloys of this invention are limited to 5 wt.% iron or less when tungsten or niobium are present as minor element additions. For alloys with greater than 5 wt.%

iron, niobium and tungsten should be controlled to impurity level only (approximately 0.2 wt.% and 0.5 wt.% for niobium and tungsten, respectively).

Also mentioned above, alloys AA, CC, and DD failed the creep-rupture requirement. Alloy AA has a Mo level below that required by the present invention, while all the other elements fall within their acceptable ranges. Therefore, it was found that a critical minimum Mo level was necessary for the requisite creep-rupture strength. Similarly, alloys CC and DD both have Al levels which are outside the range of this invention, while all the other elements fall within their acceptable ranges. The mechanisms responsible for the low creep-rupture strength when the Al level is outside the ranges defined by this invention are unclear.

Thermal Stability The thermal stability of the experimental alloys was tested using a room temperature tensile test following a thermal exposure at 1400°F (760°C) for 100 hours. The amount of room temperature tensile elongation (retained ductility) after the thermal exposure can be taken as a measure of an alloy's thermal stability. The exposure temperature of 1400°F (760°C) was selected since many nickel-base alloys have the least thermal stability around that temperature range. To have acceptable thermal stability for the applications of interest, it was determined that a retained ductility of greater than 10% is a necessity. Preferably the retained ductility should be greater than 15%. Of the 30 experimental alloys described here, 28 of them had a retained ductility of 17% or more – comfortably above the preferred minimum. Alloys BB and DD were the exceptions, both having a retained ductility of less than 10%. Alloy BB has a Mo level greater than the maximum for the alloys of the present invention, while all the other elements fell within their acceptable ranges. Thus, it is believed that this high Mo level was responsible for the poor thermal stability. Similarly, alloy DD had an Al level greater than

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the maximum for the alloys of the present invention, while all the other elements fell within their acceptable ranges. Thus, the high Al level is believed responsible for the poor thermal stability.

Table 7

Thermal Stability Test

Alloy	% Elongation (retained ductility) after 1400°F (760°C) / 100 hours
A	24
B	25
C	23
D	25
E	25
F	23
G	23
H	23
I	21
J	19
K	24
L	22
M	20
N	22
O	23
P	20
Q	20
R	21
S	17
T	23
U	23
V	21
W	23
X	21
Y	23
Z	20
AA	22
BB	2
CC	29
DD	7

Summarizing the results of the testing for the four key properties (oxidation resistance, fabricability, creep-rupture strength, and thermal stability), alloys A through N, alloys R through X, and alloy Z, (22 in all) were found to pass all four key property tests and are thus considered alloys of the present invention. Also considered part of the present invention is alloy Y, which passed the creep-rupture, modified CHRT, and thermal stability tests, but was not tested for oxidation resistance (its aluminum level indicates that alloy Y would have excellent oxidation resistance as well according to the teaching of this specification). Alloys O and DD failed the modified CHRT test and thus were determined to have insufficient fabricability (due to poor resistance to strain age cracking). Alloys P, Q, AA, CC, and DD were found to fail the creep-rupture strength requirement. Alloy CC failed the oxidation requirement. Finally, alloys BB and DD failed the thermal stability requirement. Therefore, alloys O, P, Q, AA, BB, CC, and DD (7 in all) are not considered alloys of the present invention. These results are summarized in Table 8. Additionally, seven different commercial alloys were considered alongside the experimental alloys. All seven commercial alloys were found to fail one or more of the key property tests.

Table 8

Experimental Alloy Summary

Alloy	Failed Key Property Test(s)	Alloy of the Present Invention
A		YES
B		YES
C		YES
D		YES
E		YES
F		YES
G		YES
H		YES

I		YES
J		YES
K		YES
L		YES
M		YES
N		YES
O	Modified CHRT	NO
P	Creep-Rupture	NO
Q	Creep-Rupture	NO
R		YES
S		YES
T		YES
U		YES
V		YES
W		YES
X		YES
Y		YES
Z		YES
AA	Creep-Rupture	NO
BB	Thermal Stability	NO
CC	Oxidation, Creep-Rupture	NO
DD	Modified CHRT, Creep-Rupture, Thermal Stability	NO

The acceptable experimental alloys contained (in weight percent): 15.3 to 19.9 chromium, 9.7 to 20.0 cobalt, 7.5 to 10.0 molybdenum, 2.72 to 3.78 aluminum, less than 0.1 up to 10.4 iron, 0.085 to 0.120 carbon, as well as minor elements and impurities. The acceptable alloys further had values of the term $Al + 0.56Ti + 0.29Nb + 0.15Ta$ which ranged from 2.93 to 3.89.

Perhaps the most critical aspect of this invention is the very narrow window for the element aluminum. A critical aluminum content of at least 2.72 wt.% is required in these alloys to promote the formation of the protective alumina scale – requisite for their excellent oxidation resistance. However, the aluminum content must be controlled to 3.9 wt.% or less to maintain the fabricability of the alloys as defined, in part, by the alloys' resistance to strain-age cracking.

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This careful control of the aluminum content is a necessity for the alloys of this invention. The narrow aluminum window was also found to be important for the creep-strength of these alloys, as well as the thermal stability. In addition to the narrow aluminum window, there are other factors crucial to this invention. These include the cobalt and molybdenum additions, which contribute greatly to the creep-rupture strength – a key property of these alloys. In particular, it was found that a critical minimum level of molybdenum was necessary in this particular class of alloys to ensure sufficient creep-strength. Chromium is also crucial due to its contribution to oxidation resistance. Certain minor element additions can provide significant benefits to the alloys of this invention. This includes carbon, a critical (and required) element for imparting creep strength, grain refinement, etc. Also, boron and zirconium, while not required to be present, are preferred to be present due to their beneficial effects on creep-rupture strength. Likewise, rare earth elements, such as yttrium, lanthanum, cerium, etc. are preferred to be present due to their beneficial effects on oxidation resistance. Finally, while all alloys of this invention have high creep-rupture strength, those with hafnium and/or tantalum additions have been found to have unexpectedly pronounced creep-rupture strength.

The criticality of certain elements to the ability of the alloys of this invention to meet the combination of the four key material properties is illustrated by comparison of the present invention to that described by Gresham in U.S. Patent No. 2,712,498 which overlaps the present invention. In the Gresham patent wide elemental ranges are described which cover vast swaths of compositional space. No attempt is made to describe alloys which possess the combination of the four key material properties required by the present invention. In fact, the Gresham patent describes many alloys which do not meet the requirements of the present invention. For example, the commercial 263 alloy was developed by Rolls-Royce Limited (to whom this patent

was assigned) and has been used in the aerospace industry for decades. However, this alloy does not have the excellent oxidation resistance required by the present invention – as was shown in Table 2 above. Furthermore, there is no teaching by Gresham et al. that a critical minimum aluminum level is necessary for oxidation resistance. Another example is alloy DD described in Table 1. This alloy falls within the ranges of the Gresham patent. However, this alloy fails three of the four requirements of the present invention: creep-rupture, resistance to strain-age cracking (as measured by the modified CHRT test), and thermal stability. The failure of alloy DD to pass the strain-age cracking requirement, for example, has been shown in the present specification to be a result of the aluminum level being too high. There is no teaching by Gresham et al. that there is a critical maximum aluminum level (or a maximum combined level of the elements Al, Ti, Nb, and Ta) to avoid susceptibility to strain-age cracking. A third example is that Gresham does not describe the need to limit the maximum molybdenum level to avoid poor thermal stability. In short, Gresham describes alloys which do not meet the combination of four key material properties described herein and does not teach anything about the critical compositional requirements necessary to combine these four properties, including for example, the very narrow acceptable aluminum range.

The alloys of the present invention must contain (in weight percent): 15 to 20 chromium, 9.5 to 20 cobalt, 7.25 to 10 molybdenum, 2.72 to 3.9 aluminum, an amount of carbon up to 0.15, and the balance nickel plus impurities minor element additions. The ranges for the major elements are summarized in Table 9. In addition to carbon, the minor element additions may also include iron, silicon, manganese, titanium, niobium, tantalum, hafnium, zirconium, boron, tungsten, magnesium, calcium, and one or more rare earth elements (including, but not limited

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to, yttrium, lanthanum, and cerium). The acceptable ranges of the minor elements are described below and summarized in Table 10.

Table 9

Major Element Ranges (in wt.%)

Element	Broad range	Intermediate range #1	Intermediate range #2	Narrow
Ni	balance	balance	balance	balance
Cr	15 to 20	16 to 20	17 to 20	17.5 to 19.5
Co	9.5 to 20	15 to 20	17 to 20	17.5 to 19.5
Mo	7.25 to 10	7.25 to 9.75	7.25 to 9.25	7.25 to 8.25
Al	2.72 to 3.9	2.9 to 3.7	2.9 to 3.6	3.0 to 3.5

The elements titanium and niobium may be present, for instance to provide strengthening, but should be limited in quantity due to their adverse effect on certain aspects of fabricability. In particular, an abundance of these elements may increase the propensity of an alloy for strain-age cracking. If present, titanium should be limited to no more than 0.75 wt.%, and niobium to no more than 1 wt.%. If not present as intentional additions, titanium and niobium could be present as impurities up to around 0.2 wt.% each.

The presence of the elements hafnium and/or tantalum has unexpectedly been found to be associated with even greater creep-rupture lives in these alloys. Therefore, one or both elements may optionally be added to these alloys to further improve creep-rupture strength. Hafnium may be added at levels up to around 1 wt.%, while tantalum may be added at levels up to around 1.5 wt.%. To be most effective, the sum of the tantalum and hafnium contents should be between 0.2 wt.% and 1.5 wt.%. If not present as intentional additions, hafnium and tantalum could be present as impurities up to around 0.2 wt.% each.

To maintain fabricability, certain elements which may or may not be present (specifically, aluminum, titanium, niobium, and tantalum) should be limited in quantity in a manner to satisfy the following additional relationship (where elemental quantities are in wt.%):

$$\text{Al} + 0.56\text{Ti} + 0.29\text{Nb} + 0.15\text{Ta} \leq 3.9 \quad [1]$$

Additionally, boron may be present in a small, but effective trace content up to 0.015 wt.% to obtain certain benefits known in the art. Tungsten may be added up to around 2 wt.%, but if present as an impurity would typically be around 0.5 wt.% or less. Iron may also be present as an impurity at levels up to around 2 wt.%, or may be an intentional addition at higher levels to lower the overall cost of raw materials. However, iron should not be present more than around 10.5 wt.%. If niobium and/or tungsten are present as minor element additions, the iron content should be further limited to 5 wt.% or less. To enable the removal of oxygen and sulfur during the melting process, these alloys typically contain small quantities of manganese up to about 1 wt.%, and silicon up to around 0.6 wt.%, and possibly traces of magnesium, calcium, and rare earth elements (including yttrium, cerium, lanthanum, etc.) up to about 0.05 wt.% each. Zirconium may be present in the alloy as an impurity or intentional addition (for example, to improve creep-rupture life), but should be kept to 0.06 wt.% or less in these alloys to maintain fabricability, preferably 0.04 wt.% or less.

Table 10

Minor Element Additions (in wt.%)

Element	Broad range	Intermediate	Narrow range
C	present up to 0.15	present up to 0.12	0.02 up to 0.12
Fe	up to 10.5	up to 5	up to 2
Si	up to 0.6	up to 0.5	up to 0.4
Mn	up to 1	up to 1	up to 0.5
Ti	up to 0.75	up to 0.75	0.2 to 0.5
Nb ^a	up to 1	up to 1 ^c	up to 1 ^d
Ta	up to 1.5	up to 1.5 ^c	up to 1 ^d
Hf	up to 1	up to 1 ^c	up to 0.5 ^d
Zr	up to 0.06	up to 0.04	present up to 0.04
B	up to 0.015	up to 0.008	present up to 0.005
W ^a	up to 2	up to 2	up to 0.5
Mg	up to 0.05	up to 0.05	up to 0.05
Ca	up to 0.05	up to 0.05	up to 0.05
REE ^b	up to 0.05 each	up to 0.05 each	one or more present up to 0.05 each

^aAlloys with Nb or W present at higher than impurity levels should also contain ≤ 5 wt.% Fe

^bRare earth elements (REE) include one or more of Y, La, Ce, etc.

^cIn the intermediate range, at least one of niobium, tantalum, and hafnium should be present, and the sum should be between 0.2 and 1.5

^dIn the narrow range, at least one of tantalum and hafnium should be present, and the sum should be between 0.2 and 1.5

A summary of the tolerance for certain impurities is provided in Table 11. Some elements listed in Table 11 (tantalum, hafnium, boron, etc.) may be present as intentional additions rather than impurities; if a given element is present as an intentional addition it should be subject to the ranges defined in Table 10 rather than Table 11. Additional unlisted impurities may also be present and tolerated if they do not degrade the key properties below the defined standards.

Table 11

Impurity Tolerances (in wt.%)

Impurity	Maximum Tolerance
Fe	2*
Si	0.4*
Mn	0.5*
Ti	0.2*
Nb*	0.2*
Ta	0.2*
Hf	0.2*
Zr	0.05*
B	0.005*
W*	0.5*
Cu	0.5
S	0.015
P	0.03

*May be higher if an intentional addition (see Table 10)

From the information presented in this specification we can expect that the alloy compositions set forth in Table 12 would also have the desired properties.

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Table 12

Other Alloy Compositions

Alloy	Ni	Cr	Co	Mo	Al	Fe	C	Si	Ti	Y	Zr	B	Other
1	Bal.	16	15	8	3.9	1	0.1	0.1	--	0.02	0.04	0.004	
2	Bal.	16	15	7.25	3.3	1	0.1	0.1	0.25	0.02	0.04	0.004	0.5 Ta
3	Bal.	16	15	8	3.3	1	0.02	0.1	0.25	0.02	0.04	0.004	0.5 Ta
4	Bal.	16	15	8	3.3	1	0.15	0.1	0.25	0.02	0.04	0.004	0.5 Ta
5	Bal.	15	15	8	3.3	1	0.1	0.1	0.25	0.02	0.04	0.004	0.5 Ta
6	Bal.	20	15	8	3.3	1	0.1	0.1	0.25	0.02	0.04	0.004	0.5 Ta
7	Bal.	16	15	8	3.3	1	0.1	--	0.25	0.02	0.04	0.004	0.5 Ta
8	Bal.	16	9.5	8	3.3	1	0.1	0.1	0.25	0.02	0.04	0.004	0.5 Ta
9	Bal.	16	15	8	3.3	1	0.1	0.1	--	0.02	0.04	0.004	0.5 Ta
10	Bal.	16	15	8	3.3	1	0.1	0.1	0.25	0.02	--	0.004	0.5 Ta
11	Bal.	16	15	8	3.3	1	0.1	0.1	0.25	0.02	0.04	--	0.5 Ta
12	Bal.	16	15	8	3.3	1	0.05	0.1	0.25	0.02	0.04	0.004	0.5 Ta
13	Bal.	16	15	8	3.3	1	0.1	0.1	0.25	0.02	0.04	0.015	0.5 Ta
14	Bal.	16	15	8	3.3	1	0.1	0.1	0.75	0.02	0.04	0.004	0.5 Ta
15	Bal.	16	15	8	3.3	1	0.1	0.1	0.25	0.02	0.04	0.004	1 Nb
16	Bal.	16	15	8	3.3	1	0.1	0.1	0.25	0.02	0.04	0.004	1 Hf
17	Bal.	16	15	8	3.3	1	0.1	0.1	0.25	0.02	0.04	0.004	1.5 Ta
18	Bal.	16	15	8	3.3	10.5	0.1	0.1	0.25	0.02	0.04	0.004	0.5 Ta
19	Bal.	16	15	8	3.3	1	0.1	0.1	0.25	0.02	0.04	0.004	1 Mn, 0.5 Ta
20	Bal.	16	15	8	3.3	1	0.1	0.5	0.25	0.02	0.04	0.004	0.5 Ta
21	Bal.	16	15	8	3.3	1	0.1	0.6	0.25	0.02	0.04	0.004	0.5 Ta
22	Bal.	16	15	8	3.3	1	0.1	0.1	0.25	0.02	0.06	0.004	0.5 Ta
23	Bal.	16	15	8	3.3	1	0.1	0.1	0.25	0.02	0.04	0.008	0.5 Ta
24	Bal.	16	15	8	3.3	1	0.1	0.1	0.5	0.02	0.04	0.004	0.5 Ta
25	Bal.	16	15	8	3.3	1	0.1	0.1	0.25	0.02	0.04	0.004	0.5 Hf
26	Bal.	16	15	8	3.3	1	0.1	0.1	0.25	0.02	0.04	0.004	0.5 Ta, 0.2 W
27	Bal.	16	15	8	3.3	1	0.1	0.1	0.25	0.02	0.04	0.004	0.5 Ta, 0.05 Mg
28	Bal.	16	15	8	3.3	1	0.1	0.1	0.25	0.02	0.04	0.004	0.5 Ta, 0.05 Ca
29	Bal.	16	15	8	3.3	1	0.1	0.1	0.25	0.02	0.04	0.004	0.5 Ta, 0.05 La
30	Bal.	16	15	8	3.3	1	0.1	0.1	0.25	0.02	0.04	0.004	0.5 Ta, 0.05 Ce
31	Bal.	16	15	8	3.3	1	0.1	0.1	0.25	0.05	0.04	0.004	0.5 Ta
32	Bal.	16	15	8	3.5	1	0.1	0.1	0.45	0.05	0.04	0.004	1 Ta

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In addition to the four key properties described above, other desirable properties for the alloys of this invention would include: high tensile ductility in the as-annealed condition, good hot cracking resistance during welding, good thermal fatigue resistance, and others.

Even though the samples tested were limited to wrought sheet, the alloys should exhibit comparable properties in other wrought forms (such as plates, bars, tubes, pipes, forgings, and wires) and in cast, spray-formed, or powder metallurgy forms, namely, powder, compacted powder and sintered compacted powder. Consequently, the present invention encompasses all forms of the alloy composition.

The combined properties of excellent oxidation resistance, good fabricability, and good creep-rupture strength exhibited by this alloy make it particularly useful for fabrication into gas turbine engine components and particularly useful for combustors in these engines. Such components and engines containing these components can be operated at higher temperatures without failure and should have a longer service life than those components and engines currently available.

Although we have disclosed certain preferred embodiments of the alloy, it should be distinctly understood that the present invention is not limited thereto, but may be variously embodied within the scope of the following claims.

We Claim:

1. A nickel-chromium-cobalt-molybdenum-aluminum based alloy having a composition comprised in weight percent of:

15 to 20	chromium
9.5 to 20	cobalt
7.25 to 10	molybdenum
2.72 to 3.9	aluminum
up to 10.5	iron
present up to 0.15	carbon
up to 0.015	boron
up to 0.75	titanium
up to 1	niobium
up to 1.5	tantalum
up to 1	hafnium
up to 2	tungsten
up to 1	manganese
up to 0.6	silicon
up to 0.06	zirconium
up to 0.05	magnesium
up to 0.05	calcium
up to 0.05	rare earth element

up to 0.5	copper
up to 0.015	sulfur
up to 0.03	phosphorous

with a balance of nickel and impurities, the alloy further satisfying the following compositional relationship defined with elemental quantities being in terms of weight percent:

$$Al + 0.56Ti + 0.29Nb + 0.15Ta \leq 3.9.$$

2. The nickel-chromium-cobalt-molybdenum-aluminum based alloy of claim 1, containing hafnium, tantalum, or a combination of hafnium and tantalum, where the sum of the two elements is between 0.2 wt.% and 1.5 wt.%.

3. The nickel-chromium-cobalt-molybdenum-aluminum based alloy of claim 1, containing titanium, from 0.2 to 0.75 wt.%.

4. The nickel-chromium-cobalt-molybdenum-aluminum based alloy of claim 1, containing at least one of hafnium and tantalum at a level ranging from 0.2 wt.% up to 1 and 1.5 wt.%, respectively.

5. The nickel-chromium-cobalt-molybdenum-aluminum based alloy of claim 1, wherein the alloy contains in weight percent:

16 to 20	chromium
15 to 20	cobalt

7.25 to 9.75	molybdenum
2.9 to 3.7	aluminum.

6. The nickel-chromium-cobalt-molybdenum-aluminum based alloy of claim 1,
wherein the alloy contains in weight percent:

17 to 20	chromium
17 to 20	cobalt
7.25 to 9.25	molybdenum
2.9 to 3.6	aluminum.

7. The nickel-chromium-cobalt-molybdenum-aluminum based alloy of claim 1,
wherein the alloy contains in weight percent:

17.5 to 19.5	chromium
17.5 to 19.5	cobalt
7.25 to 8.25	molybdenum
3.0 to 3.5	aluminum.

8. The nickel-chromium-cobalt-molybdenum-aluminum based alloy of claim 1,
wherein the alloy contains in weight percent:

up to 5	iron
present up to 0.12	carbon
up to 0.008	boron

up to 0.5	silicon
up to 0.04	zirconium.

9. The nickel-chromium-cobalt-molybdenum-aluminum based alloy of claim 1,
wherein the alloy contains in weight percent:

up to 2	iron
0.02 to 0.12	carbon
present up to 0.005	boron
0.2 to 0.5	titanium
up to 0.5	manganese
up to 0.4	silicon
present up to 0.04	zirconium.

10. The nickel-chromium-cobalt-molybdenum-aluminum based alloy of claim 1,
wherein the alloy has oxidation resistance such that the average metal affected has a value not
greater than 64 μ m/side (2.5 mils/side) when tested in flowing air at 1149°C (2100°F) for 1008
hours.

11. The nickel-chromium-cobalt-molybdenum-aluminum based alloy of claim 1,
wherein the alloy has modified CHRT test ductility values greater than 7%.

12. The nickel-chromium-cobalt-molybdenum-aluminum based alloy of claim 1,

wherein the alloy has a creep-rupture life of at least 325 hours when tested at 982°C (1800°F) under a load of 17 MPa (2.5 ksi).

13. The nickel-chromium-cobalt-molybdenum-aluminum based alloy of claim 1, wherein the alloy contains greater than 5 wt.% iron and at least one of niobium up to 0.2 weight percent and tungsten up to 0.5 weight percent.

14. A nickel-chromium-cobalt-molybdenum-aluminum based alloy having a composition comprised in weight percent of:

15.3 to 19.9	chromium
9.7 to 20.0	cobalt
7.5 to 10.0	molybdenum
2.72 to 3.78	aluminum
0.1 to 10.4	iron
0.085 to 0.120	carbon
up to 0.005	boron
up to 0.49	titanium
up to 1.0	tantalum
up to 0.48	hafnium
up to 0.49	silicon
up to 0.02	yttrium
up to 0.04	zirconium

up to 0.2	niobium
up to 0.5	tungsten
up to 0.5	copper
up to 0.015	sulfur
up to 0.03	phosphorous
up to 0.05	magnesium
up to 0.05	calcium
up to 0.05	rare earth element

with a balance of nickel and impurities, the alloy further satisfying the following compositional relationship defined with elemental quantities being in terms of weight percent:

$$Al + 0.56Ti + 0.29Nb + 0.15Ta \leq 3.89.$$

15. The nickel-chromium-cobalt-molybdenum-aluminum based alloy of claim 14, containing one or more of niobium up to 0.2 wt.%, tungsten up to 0.5 wt.%, copper up to 0.5 wt.%, sulfur up to 0.015 wt.%, phosphorous up to 0.03 wt.%, magnesium up to 0.05 wt.%, calcium up to 0.05 wt.%, and any rare earth elements up to 0.05 wt.%.