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RENPEI YODA ET AL

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NICKEL BASE HEAT-RESISTING ALLOY

Original Filed June 10, 1964

2 Sheets-Sheet 1

FIG. 1

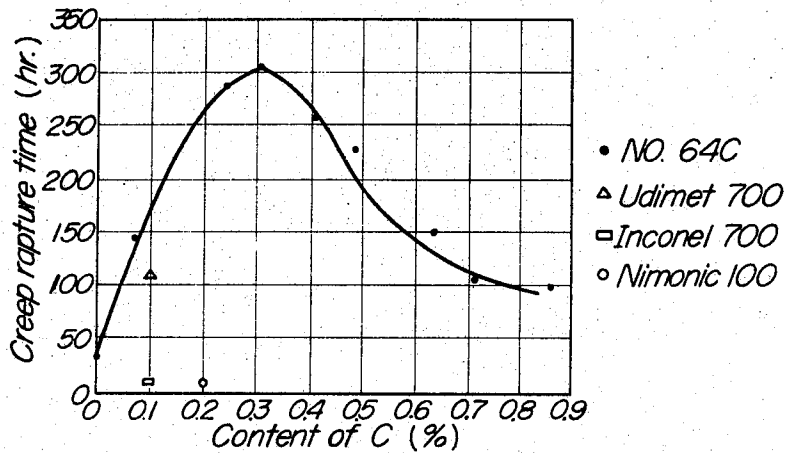
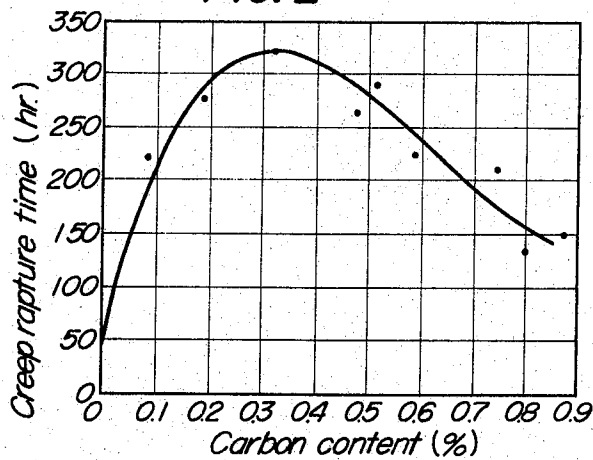


FIG. 2



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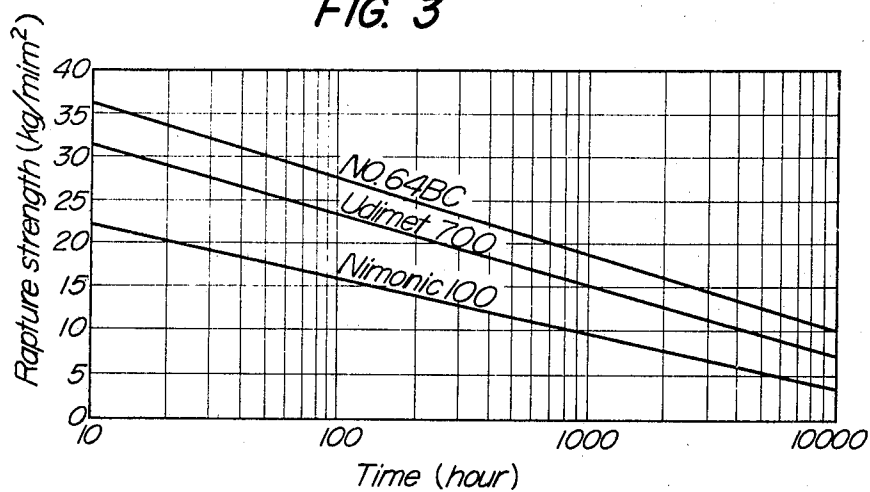
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FIG. 3



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NICKEL BASE HEAT-RESISTING ALLOY

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Continuation of application Ser. No. 374,026, June 10, 1964. This application June 6, 1968, Ser. No. 740,777

Claims priority, application Japan, Jan. 31, 1964,

39/4,495, 39/4,496

Int. Cl. C22c 19/00

U.S. Cl. 75—171

2 Claims

ABSTRACT OF THE DISCLOSURE

Nickel base, heat resisting alloys comprising most especially of carbon and boron in specified ranges, other ingredients also being present within specified ranges. The nickel base, heat resisting alloys are adapted to withstand high temperature ranging from 800° C. to 900° C., and are especially useful as materials for moving blades of gas turbine, jet engines and the like. The following indicates desirable alloy content: from 11 to 13 percent by weight of chromium; from 19 to 21 percent by weight of cobalt; from 4 to 6 percent by weight of molybdenum; from 5 to 6 percent by weight of aluminum; from 3 to 4 percent by weight of titanium; from 0.05 to 0.3 percent by weight of carbon; from 0.1 to 0.45 percent by weight of boron, and the balance being essentially nickel.

This is a continuation of application Ser. No. 374,026 filed June 10, 1964, now abandoned.

This invention relates to nickel base heat resisting alloys containing carbon or carbon and boron. More particularly this invention relates to nickel base heat resisting alloys containing carbon or carbon and boron which can withstand a high temperature ranging from 800° C. to 900° C. to be useful as a material for moving blades of gas turbine, jet engine or the like.

As nickel base heat resisting alloys useful for such purposes, many strengthening materials have been known, e.g. utilizing the strengthening of austenitic matrix structure by addition of cobalt or molybdenum, the precipitation hardening of γ' phase [$\text{Ni}_3(\text{Al}\cdot\text{Ti})$] by addition of titanium and aluminum, the precipitation hardening of carbide phase and the strengthening of crystal grain boundary by addition of boron or carbon. Hastelloy utilizes the strengthening of matrix structure. Inconel and Nimonic utilizes the precipitation hardening by addition of titanium and aluminum. M-252 utilizes the matrix strengthening and precipitation hardening. Such alloys as Udimet 700, DCM, Nicrotung, René 41 or like utilize in addition to these effects the strengthening of grain boundary. However what performs the leading part of the precipitation hardening in the nickel base heat resisting alloys is the above-mentioned γ' phase, and the precipitation hardening of the carbide phase is not notable.

However carbon contents of these nickel base heat resisting alloys are limited generally in the range of lower than 0.1 percent by weight from the point of forgeability. Cast alloys may have a carbon content of up to 0.3% by weight, however, this is at the sacrifice of forgeability. Boron content of the above-mentioned strengthened alloys is also always lower than 0.1 percent by weight, and carbon simultaneously contained never exceeds 0.2 percent by weight. If the contents of boron and carbon are increased in these metals, the metals become brittle and lose their strength.

Thus it has been a continuous desire of those who have been engaged in both the manufacturing and the utiliza-

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tion of nickel base heat resisting alloys to obtain alloys which may be strengthened by inexpensive carbon or boron to withstand a high temperature of 800° C. to 900° C.

It is, therefore, an object of the present invention to provide an alloy which is strengthened by the addition of carbon to exhibit good creep rupture strength at a temperature of from 800° C. to 900° C., and in which the carbon content is generally greater than in conventional strengthened nickel base heat resisting alloys, thereby to achieve better castability. It is another object of the present invention to provide another alloy which is strengthened by the addition of boron and carbon to exhibit good creep rupture strength at a temperature of from 800° C. to 900° C. and in which the contents of boron and carbon are generally greater than conventional strengthened nickel base heat resisting alloys, thereby to obtain better castability.

These and another object can be fulfilled by the present invention there is provided. According to the present invention a nickel base heat resisting alloy having chromium content of from 11~13 percent by weight, cobalt content of from 19~21 percent by weight, molybdenum content of from 4~6 percent by weight, aluminum content of from 5 to 6 percent by weight, titanium content of from 3 to 4 percent by weight and carbon content of from 0.1 to 0.6 percent by weight, and another nickel base heat resisting alloy having the same ranges of chromium, cobalt, molybdenum, aluminum, titanium as the first alloy but having content of carbon from 0.05 to 0.3 percent by weight and boron content of from 0.1 to 0.45 percent by weight.

In the present alloy, the content of chromium lower than 11 percent lowers the oxidation resistant property, and the life for creep rupture. A chromium content higher than 13 percent by weight causes embrittlement. Accordingly the range of from 11 to 13 percent is most suitable. When the content of cobalt is lower than 19 percent by weight or the content of molybdenum is lower than 4 percent, the life for creep rupture is lowered.

When the content of the former is larger than 21 percent by weight, it brings about brittleness and when the content of the latter is larger than 6 percent by weight, it deteriorates oxidation resistant property. In this regard the range of cobalt from 19 to 21 percent by weight and of molybdenum from 4 to 6 percent by weight are required. Further the content of aluminum from 5 to 6 percent by weight and of titanium from 3 to 4 percent by weight are the necessary amounts to promote the precipitation hardening of γ' phase. If the content of aluminum or titanium is apart from these ranges, the creep rupture strength shows sharp drop. The range of carbon content from 0.1 to 0.6 percent by weight or of carbon plus boron content from 0.15 to 0.60 percent by weight performs a leading part in the high temperature strengthening.

METHOD OF CREEP RUPTURE TEST FOR METALS

(1) Scope

This standard specifies method of creep rupture test to determine rupture time by measuring tensile creep strain until metals come to rupture under fixed load and at constant temperature.

(2) Test piece

(2.1) The standard test piece shall be of circular section, as a rule, and shall be of 3 kinds as diameters of 6 mm., 10 mm. and 12 mm., however, shall be allowed to use 8 mm. as the case may be. The gauge length shall be all not less than 5 times the diameter.

(2.2) The test piece shall have the circular section be uniform in every part, shall have the dimensional devia-

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tion (difference between maximum value and minimum value) to inequality of shape in parallel portion be not more than 0.05 mm. and the parallel portion shall be concentric with the grip shank.

(2.3) In case of not capable of sampling the test piece in circular section, a tabular test piece may be used. Provided that the dimension of test piece in this case shall comply with the agreement between the parties concerned.

(2.4) The test piece shall have the parallel portion be smooth and free from cut flaws or such.

(3) Testing sets

(3.1) Tension tester—

(3.1.1) The tester shall be capable of applying the load as 5~100% of the capacity to the test piece at the accuracy within $\pm 1.5\%$.

At the time of applying the load, attention shall be paid to that the impact is not given and eccentric load is not applied.

(3.1.2) The tester shall be firmly set so as not to be affected by vibration and impact from exterior.

(3.1.3.) The tester shall be desirably of direct load type by weight or single lever type, shall not cause the test piece or curve and shall be of a mechanism as capable of applying smoothly the load to the axial direction.

(3.2) *Heating apparatus*—The heating furnace shall be always of capable of heating uniformly through the total range in gauge length of test piece during test within the allowable range in Table 1.

Table 1.—Allowable range of positional variation in temperature between gauge length of test piece unit: ° C.

Test temp.:	Allow. range
400 max -----	± 3
Ex. 400 up to 600 -----	± 4
Ex. 600 up to 800 -----	± 5
Ex. 800 -----	± 8

(3.3) *Temperature measuring apparatus*—The thermometric apparatus shall consist of an indicator and a thermocouple.

(3.3.1.) Indicator: The indicator shall be of having accuracy of $\pm 1.0^\circ$ C. to the total range through measured temperature.

(3.3.2) Thermocouple:

(1) The material of thermocouple shall be of withstanding test temperature well for long time. And the wire size shall be preferably made small, if possible, within a range not to change electromotive force while being in use.

(2) The thermocouple shall be always corrected in compliance with the method under Item (3) before test commencement.

(3) The thermocouple shall be corrected at several fixed points given in Table 2 or shall be corrected in comparison with corrected standard Pt-Pt.Rh thermocouple. In this case the difference of temperature reading shall be within the range of tolerance given in Table 3.

Table 2.—Fixed points for correction of thermocouple, unit: ° C.

Freezing point -----	0
Boiling point of water -----	100
Solidifying melting point of tin -----	231.9
Solidifying melting point of lead -----	327.3
Solidifying melting point of zinc -----	419.5
Boiling point of sulfur -----	444.6000
Solidifying melting point of antimony -----	630.5
Solidifying melting point of silver -----	960.8
Solidifying melting point of gold -----	1063.0

Remarks: This numerical value shall be determined at International Weight and Measures Conference, 1948.

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Table 3.—Tolerance for standard thermocouple and working thermocouple, unit: ° C.

Temp.:	Tolerance
600 max -----	1.5
Exceeding 600 up to 1000 -----	2.5
Ex. 1000 -----	5

(4) The difference of temperature readings of thermocouple before and after working shall be also within the range of tolerance given in Table 3.

(5) The thermocouple shall, at every time when being re-used, be recorrected and may be re-used only in case of the difference from the initial certificated value being within the range in Table 3.

(6) In other case than that of using the Pt-Pt. Rh thermocouple for the test temperature not less than 300° C., a new thermocouple shall be used at every time of the test.

(7) The contact point of the thermocouple shall come thermally well in contact with the surface of test piece and shall, in order to avoid radiation from furnace wall, be properly sheltered. Moreover, the part of the thermocouple inserted in a furnace shall be completely covered with a guard tube and shall be insulated.

(8) Other requirements than specified in the preceding respective items shall comply with JIS C 1602—Thermoelectric Couple.

(3.4) *Temperature regulating apparatus*—The temperature of test piece shall be maintained constant within the allowable range in Table 4 duuring testing time by use of an automatic temperature regulator.

Table 4.—Allowable range in time change for temperature of test piece, unit: ° C.

Test temperature:	Allowable range
400 max. -----	± 3
Exceeding 400 up to 600 -----	± 4
Exceeding 600 up to 800 -----	± 5
Exceeding 800 -----	± 8

(3.5) Elongation measuring apparatus—

(3.5.1.) The elongatometer shall, as a rule, be of capable of measuring the elongation on each end and also shall have an accuracy be within 1% of rupture elongation.

The part of the elongatometer being out of a furnace shall be given attention so as not be affected by the temperature change.

(3.5.2) The deviation of gauge length shall, when fitting the elongatometer, be within $\pm 2\%$ and the numerical value shall be measured up to 0.1%.

(4) Testing method

(4.1) *Heating method*—At the time of heating, heat to 98% of the aiming test temperature in 4~6 hours and then carry out soaking-heating for 20 ± 4 hours, regulate the temperature within the said hours and settle the temperature of test piece accurately to test temperature. Consider the temperature, when being settled, to be the test temperature.

Provided that consider the range of temperature change within soaking hours to be $\pm 4\%$.

(4.2) *Loading method*—After finishing in settling to the test temperature in order to minimize the creep developed in process of loading up to test load, apply the load as quick as possible within the range of avoiding impact.

(4.3) *Measuring method of temperature, rupture time and rupture elongation*—

(4.3.1) Measure the temperature of test piece of gauge length exceeding 50 mm. at 3 points as each end and centre of the gauge length and take the average value.

And, in case of a gauge length being not more than 50 mm., be allowed to omit the thermocouple to be fitted in the centre.

(4.3.2) Let the temperature of test piece be self-recorded continuously throughout the total test duration or read as many as possible and record it.

(4.3.3) Record the elongation continuously during the test or read preferably the many, if possible. Measure the rupture elongation at an accuracy of 0.2 mm. on the basis of each end of the gauge length.

(4.3.4) Let the rupture time have to be measured at an accuracy of $\pm 2\%$.

(4.4) Consider the original sectional area in parallel portion of test piece to be the sectional area in 3 places as each end and centre of the gauge length. Let the diameter or width and thickness to determine the sectional area have to be measured up to the numerical value at least at 0.2% of the specified dimension.

Let the diameter to determine the circular sectional area take the average of measured value in 2 directions to intersect at right angles to each other.

By the content of carbon from 0.1 to 0.6 percent by weight or by the content of carbon coexisting with boron from 0.05 to 0.30 percent by weight and boron content from 0.10 to 0.45 percent by weight, the strength for creep rupture is exceedingly improved in the material. (hereinafter the present alloy containing carbon is referred to No. 64C and the present alloy containing carbon and boron is referred to No. 64BC)

A more comprehensive understanding of the invention can be obtained by referring to the following examples.

EXAMPLE 1

All raw materials of metal used in the preparation of alloy were of highest purities obtained in the market. They were further purified. Namely commercial electrolytic nickel, metallic cobalt and metallic molybdenum were melted in magnesia crucible placed in vacuum high frequency induction furnaces at an absolute pressure of from 10^{-2} to 10^{-3} mm. Hg and at a temperature of

from 1500° to 1600° C. Aluminum was used at the purity of more than 99.9 percent by weight. Titanium was used in plate form of highest purity. Carbon was added in the form of mother alloy consisting of 1.7 percent by weight of carbon and balanced nickel which was obtained by carburizing powder of electrode carbon into electrolytic nickel. The melt was cast in a metal mould in vacuo and 8 kg. of ingot was obtained. Cutting with a electro-spark cutting machine, heating at a temperature of 1150° C. for 15 hours, subjecting to solution heat treatment by use of cold water, the ingot was machined into test pieces stipulated by Japanese Industrial Standard (JIS) for creep rupture.

Result of chemical analysis of each test pieces are shown in Table 1.

TABLE 1.—CHEMICAL COMPOSITIONS OF TEST PIECES, PERCENT

Test pieces of alloys	C	Cr	Co	Mo	Al	Ti	Ni
No. 64.....	0.01	11.90	20.79	4.88	5.76	3.82	The remainder.
No. 64C ₁	0.07	11.70	20.16	4.95	5.68	3.72	Do.
No. 64C ₂	0.24	11.85	19.88	4.67	5.95	3.90	Do.
No. 64C ₃	0.30	12.05	20.13	4.78	5.28	3.76	Do.
No. 64C ₄	0.40	11.67	19.71	4.63	6.01	3.88	Do.
No. 64C ₅	0.48	11.56	20.23	4.53	5.62	3.81	Do.
No. 64C ₆	0.59	11.88	20.67	5.72	5.96	4.00	Do.
No. 64C ₇	0.63	11.54	20.75	4.23	5.95	3.92	Do.
No. 64C ₈	0.71	11.86	20.13	4.65	5.64	3.75	Do.
No. 64C ₉	0.85	11.68	20.54	4.97	5.63	3.84	Do.

Results of creep rupture test at a tensile stress of 23 kg./mm.² and at a temperature of 900° C. are shown in Table 2 and FIG. 1.

TABLE 2.—RESULTS OF CREEP RUPTURE TEST AT A TENSILE STRESS OF 23 kg./mm.² AND AT A TEMPERATURE OF 900° C.

Test pieces of alloys:	Creep rupture life, hours	Creep rupture elongation, percent
No. 64.....	32.4	0.2
No. 64C ₁	143.7	2.7
No. 64C ₂	284.7	4.8
No. 64C ₃	303.1	5.7
No. 64C ₄	258.3	6.6
No. 64C ₅	227.2	6.6
No. 64C ₆	124.4	2.4
No. 64C ₇	150.9	4.6
No. 64C ₈	106.6	4.1
No. 64C ₉	99.1	5.8

The curve in FIG. 1 shows that the addition of carbon to the nickel base alloy containing 12 percent by weight of chromium, 20 percent by weight of cobalt, 5 percent by weight of molybdenum, 6 percent by weight of aluminum and 4 percent by weight of titanium suddenly raises the creep rupture life and exhibits a peak in the neighborhood of 0.3 percent by weight of carbon. This corresponds to 300 hours at a tensile stress of 23 kg./mm.² and at a temperature of 900° C. Since the creep rupture life for the alloy having the same composition except that no carbon was added, was about 30 hours, it was concluded that a life 10 times as long can be achieved by the carbon alloying. The severity of testing condition at a temperature of 900° C. and at a tensile stress of 23 kg./mm.² can be understood by the result attained by several

TABLE 3.—CHEMICAL COMPOSITION OF REPRESENTING NICKEL BASE HEAT RESISTING ALLOYS, PERCENT

Alloys	C	Cr	Co	Mo	Fe	Al	Ti	B	Ni
Incone 700.....	0.10	15.0	29.0	3.0	0.8	2.30	2.20	-----	The remainder.
Nimonic.....	0.20	11.0	20.0	5.0	1.0	5.0	1.50	-----	Do.
Udimet 700.....	0.10	15.0	17.5	5.0	1.0	4.25	3.50	0.08	Do.
No. 64 BC.....	0.20	11.84	20.63	4.71	-----	5.98	3.93	0.27	Do.

strengthened nickel base heat resisting alloys which are now of practical use. Namely Nimonic 100, Inconel 700, and Udimet 700, the chemical compositions of which are shown in Table 3 showed the creep rupture time of 7 hours, 6 hours and 110 hours respectively at the same condition. This means that No. 64C alloy can withstand 40 times as long as Nimonic, 50 times as long as Inconel 700 and about 3 times as long as Udimet 700.

The mechanism of strengthening by the present alloy is due to cumulative effect of precipitation hardening of γ' phase, strengthening of dispersion of titanium carbide (TiC) particles and precipitation hardening of chromium carbide (Cr₂₃C₆).

Since in the present alloy, the carbon content can be raised higher than in the conventional heat resisting alloys, the present alloy has better castability and is suitable for a casting material. It is economical that strengthening effect can be achieved by an inexpensive carbon element instead of expensive special elements.

EXAMPLE 2

Test pieces for creep rupture having chemical compositions shown in Table 4 were made according to the procedure in Example 1 except that boron was added in the form of mother alloy consisting of 16 percent by weight of boron and balanced nickel.

TABLE 4.—CHEMICAL COMPOSITIONS OF TEST PIECES, PERCENT

Test pieces of alloys	C	B	C+B	Cr	Co	Mo	Al	Ti	Ni
No. 64	0.01	0.00	0.01	11.90	20.79	4.88	5.76	3.82	The remainder.
No. 64 BC	0.02	0.06	0.08	12.61	20.22	4.90	5.64	3.94	Do.
No. 64 BC ₂	0.08	0.11	0.19	11.75	20.22	4.96	5.76	3.74	Do.
No. 64 BC ₃	0.08	0.25	0.33	11.62	20.43	4.62	5.83	3.77	Do.
No. 64 BC ₄	0.20	0.27	0.47	11.84	20.63	4.71	5.98	3.93	Do.
No. 64 BC ₅	0.09	0.42	0.51	11.56	20.34	4.83	6.00	3.94	Do.
No. 64 BC ₆	0.18	0.41	0.59	11.77	20.68	4.92	5.92	3.86	Do.
No. 64 BC ₇	0.41	0.34	0.75	11.68	20.73	4.86	5.78	3.75	Do.
No. 64 BC ₈	0.55	0.25	0.80	11.73	20.69	4.45	5.82	3.86	Do.
No. 64 BC ₉	0.53	0.34	0.87	11.55	20.72	4.56	5.74	3.79	Do.

Results of creep rupture test of these test pieces at various condition (900° C. and 1000° C., 10, 18, 23 kg./mm.²) are shown in Table 5, of which the result obtained at a tensile stress of 23 kg./mm.² and at a temperature of 900° C. are illustrated in FIG. 2.

TABLE 5.—RESULTS OF CREEP RUPTURE TEST AT HIGH TEMPERATURES

Test pieces of alloy:	Testing condition	Creep rupture life, hours	Creep rupture elongation, percent
No. 64	900° C., 23 kg./mm. ²	32.4	0.2
No. 64	1,000° C., 10 kg./mm. ²	76.0	1.8
No. 64 BC ₁	900° C., 23 kg./mm. ²	220.4	10.7
No. 64 BC ₁	1,000° C., 10 kg./mm. ²	124.2	2.1
No. 64 BC ₂	900° C., 23 kg./mm. ²	278.7	10.2
No. 64 BC ₂	1,000° C., 10 kg./mm. ²	382.0	8.2
No. 64 BC ₃	900° C., 23 kg./mm. ²	320.0	10.9
No. 64 BC ₄	900° C., 18 kg./mm. ²	1,135.0	16.6
No. 64 BC ₄	900° C., 23 kg./mm. ²	268.5	13.7
No. 64 BC ₄	1,000° C., 10 kg./mm. ²	370.0	15.3
No. 64 BC ₅	900° C., 23 kg./mm. ²	291.9	17.2
No. 64 BC ₆	900° C., 18 kg./mm. ²	1,150.9	11.3
No. 64 BC ₆	900° C., 23 kg./mm. ²	227.7	14.5
No. 64 BC ₆	1,000° C., 10 kg./mm. ²	293.0	14.3
No. 64 BC ₇	900° C., 18 kg./mm. ²	1,164.3	15.3
No. 64 BC ₇	900° C., 23 kg./mm. ²	216.8	16.4
No. 64 BC ₇	1,000° C., 10 kg./mm. ²	212.1	15.5
No. 64 BC ₈	900° C., 23 kg./mm. ²	137.1	18.6
No. 64 BC ₉	do	148.5	18.6
No. 64 BC ₉	1,000° C., 10 kg./mm. ²	101.7	20.5

When carbon and boron were added to nickel base alloy containing 12 percent by weight chromium, 20 percent by weight of cobalt, 5 percent by weight of molybdenum, 6 percent by weight of aluminum and 4 percent by weight of titanium, the creep rupture life showed sharp rise and at the neighborhood of carbon and boron content from 0.15 to 0.6 percent a peak appeared.

The creep rupture time at this peak was more than 8 times as high as at the peak of alloy without addition of carbon and boron. The lower and upper limits of carbon for realizing this peak were 0.05 percent by weight and 0.30 percent by weight respectively. The corresponding limits of boron were 0.10 percent by weight and 0.45 percent by weight respectively. Then test pieces of alloy containing 0.20 percent by weight of carbon and 0.27 percent by weight of boron (C+B=0.47 percent by weight) were tested for their creep rupture time at a temperature of 900° C. under various tensile stresses.

Results of this test are indicated in FIG. 3 as No. 64BC alloy. By comparing the results of this test with those of several strengthened nickel base heat resisting alloys which are now of practical use (FIG. 3), the superiority of the present alloy can readily be understood.

Namely the present alloy can withstand the tensile stress of 14 kg./mm.² for 300 hours (4 months) and the tensile stress of 11.5 kg./mm.² for 6600 hours (9 months) at a temperature of 900° C. At a temperature of 1000° C., it can withstand the tensile stress of 10 kg./mm.² for 370 hours (15 days), but at the same condition Nimonic 100 and Udimet 700 can only stand 17 hours and 70 hours respectively.

By the simultaneous effect of precipitation hardening of γ' phase, the strengthening of crystal grain boundary by boron, the strengthening of dispersion of fine titanium carbide (TiC) particles by carbon and the precipitation hardening of chromium carbide (Cr₂₃C₆) by carbon, the present alloy containing boron and carbon is exceedingly strengthened. Moreover, this alloy has such a low solidifying temperature range (about 1280°~1170° C.) that it has good fluidity and is suitable also as casting materials. As is apparent from FIG. 2, the tolerance of B+C concentration which shows the peak of creep rupture life can be taken in fairly wide range, the chances that the variation of composition may reduce the strength are few. In other words it is another advantage of the present alloy that strengthened materials are readily produced.

What is claimed is:

1. A composition of nickel base heat resisting alloy consisting essentially of from 11 to 13 percent by weight of chromium, from 19 to 21 percent by weight of cobalt, from 4 to 6 percent by weight of molybdenum, from 5 to 6 percent by weight of aluminum, from 3 to 4 percent by weight of titanium, from 0.05 to 0.3 percent by weight of carbon, from above 0.1 to 0.45 percent by weight of boron, and the balance being essentially nickel.

2. A composition of nickel base heat resisting alloy according to claim 1 comprising 12% by weight of chromium, 20% by weight of cobalt, 5% by weight of molybdenum, 6% by weight of aluminum, 4% by weight of titanium, from 0.1 to 0.2% by weight of carbon, from 0.15 to 0.3% by weight of boron, and the balance being essentially nickel.

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