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3,065,068

AUSTENITIC ALLOY

Filed March 1, 1962

3 Sheets-Sheet 1

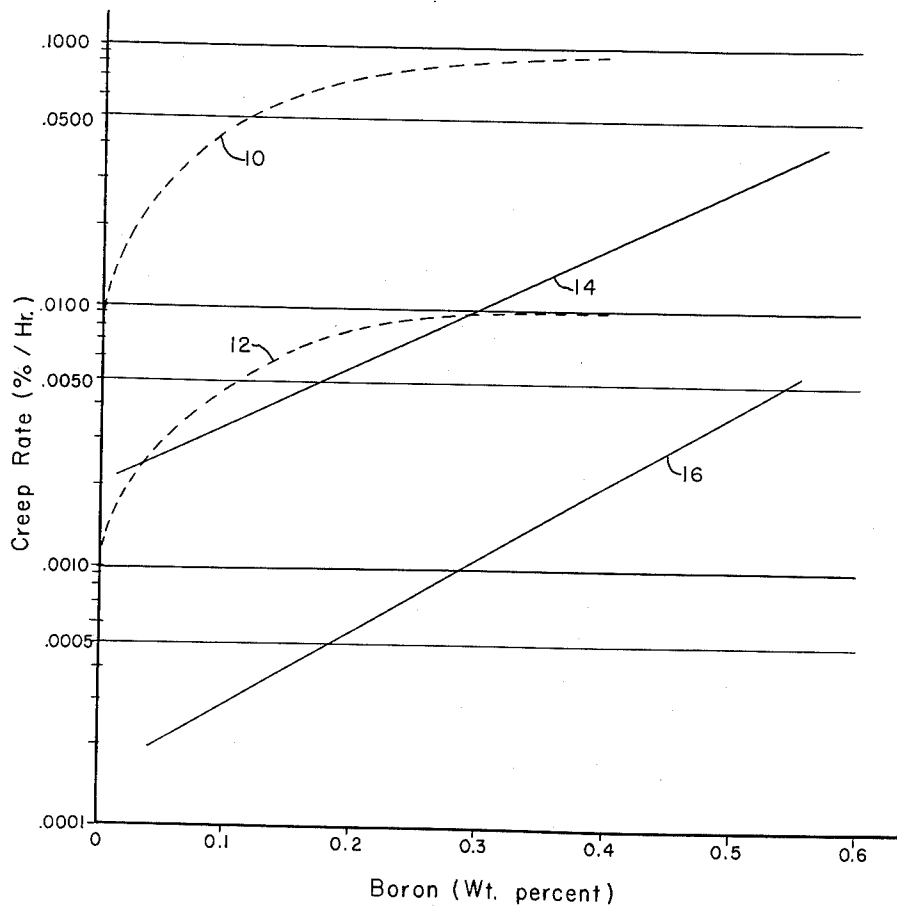


FIG. 1

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3 Sheets-Sheet 2

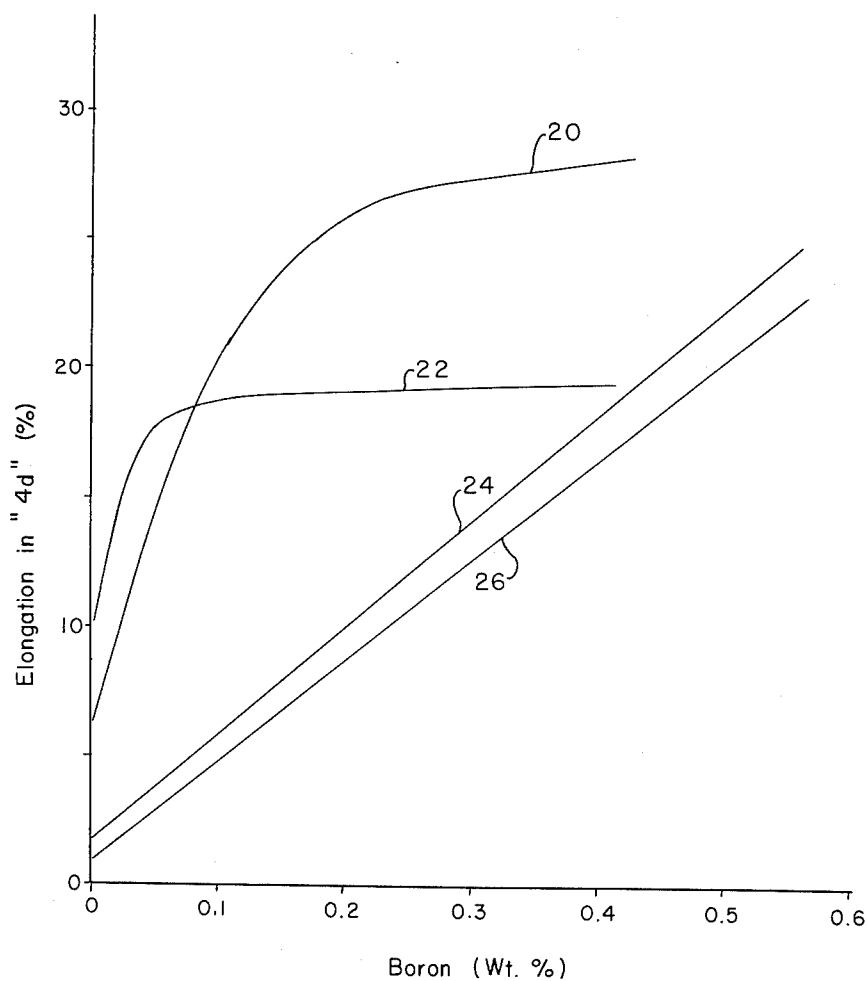


FIG. 2

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3 Sheets-Sheet 3

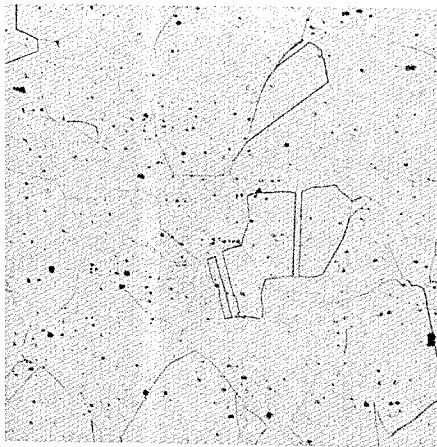


FIG. 3

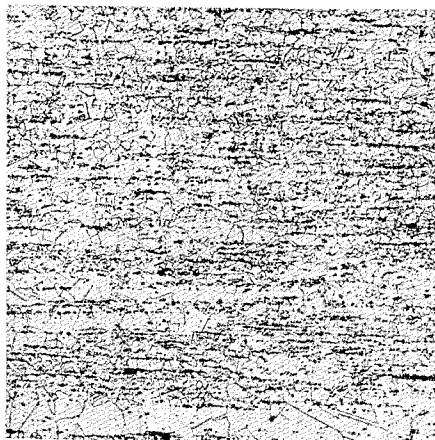


FIG. 4

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AUSTENITIC ALLOY

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8 Claims. (Cl. 75-124)

This invention relates to an improvement in austenitic iron base alloys and in particular to improvements in austenitic iron base alloys for use at elevated temperatures of up to 1400° F.

With the advent of the jet age, metal manufacturers have been constantly developing and testing new alloys for use at elevated temperatures. In particular, alloys for use in such applications demand certain criteria, the most predominant of which concerns rupture strength and rupture ductility, freedom from notch sensitivity, oxidation and corrosion resistance, high strength, hardness and toughness when these alloys are used at elevated temperatures. Considerations of conservation of strategic alloying elements, ease of fabrication and costs have been motivating factors in the development of iron base alloys which are suitable for use at such elevated temperatures yet it is a primary requisite for the alloy to possess an acceptable combination of the hereinbefore specified required mechanical properties. An example of an outstanding alloy which has found considerable use under the above described conditions is the austenitic iron base alloy known in the trade as A-286 alloy disclosed and claimed in Patent No. 2,641,540. A great degree of success and wide acceptance of this alloy has followed its use in such applications as gas and steam turbine components and in particular for such items as wheels, blades, housings, bolts and structural components.

As used in these applications, A-286 alloy has found a wide acceptance, yet manufacturers of turbines and components thereof forecast the need for alloys with higher strengths at room and elevated temperatures in order to make it possible to reduce the weight of the turbine parts and thereby increase the pay load when such turbines are used in jet aircraft applications. In addition, both competitive and tactical considerations indicate that higher temperatures will be necessary for increased efficiency of turbines. While A-286 alloy, as it is presently manufactured, is more than adequate to meet some of these requirements, none the less certain technological considerations indicate that it is rapidly approaching the limit of its practicable usable characteristics in some of the components in which it is now used. In line with these considerations considerable experimental work has been performed in an attempt to improve the mechanical characteristics of the known alloy in order to provide an alloy which will be satisfactory when used in the intended high temperature applications foreseeable within the near future.

An object of this invention is to provide an austenitic iron base alloy which is suitable for use at temperatures up to 1400° F.

Another object of this invention is to provide an austenitic iron base alloy which is suitable for use in the manufacture of parts and components of gas and steam turbines which operate at a temperature of up to 1400° F.

Another object of this invention is to provide an austenitic iron base alloy suitable for use at temperatures of up to 1400° F. which is heat treatable to provide high strength and ductility without adversely affecting its other mechanical properties.

A more specific object of this invention is to provide an austenitic iron base alloy suitable for use at temperatures of up to 1400° F., said alloy containing carbon, titanium and boron and having a critical relation therebetween.

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Other objects will become apparent when taken in conjunction with the drawings in which:

FIGURE 1 is a graph, the curves of which illustrate the effect of boron on the creep strength for certain alloys.

FIG. 2 is a graph, the curves of which illustrate the effect of boron on the rupture ductility for the same alloys as in FIG. 1;

FIG. 3 is a photomicrograph taken at a magnification of 100 times of A-286 alloy; and,

FIG. 4 is a photomicrograph taken at a magnification of 100 times of an alloy of this invention containing about 0.2% boron.

In its broader aspects, the alloy of this invention contemplates a precipitation hardenable iron base austenitic alloy containing up to 0.20% carbon, from 1.0% to 3.0% manganese, up to 1.5% silicon, from about 10% to about 22% chromium, from about 15% to about 50% nickel, from about 0.25% to about 2% molybdenum, from about 0.5% to about 4.5% titanium, up to about 1.0% aluminum, from about 0.1% to about 1.5% vanadium, from about 0.1% to about 0.8% boron and the balance iron with incidental impurities. In order to more clearly illustrate the compositional limitations of the alloy of this invention reference may be had to Table I which illustrates the general range of alloying elements, an Optimum Range A which is a preferred embodiment containing nominally about 2.2% titanium, and an Optimum Range B which is a preferred embodiment containing nominally about 3% titanium.

TABLE I.—COMPOSITION

[Percent by weight]

	General Range	Optimum Range A	Optimum Range B
C.....	0.01- 0.20	0.01- 0.10	0.10- 0.20
Mn.....	1.0- 3.0	1.0- 1.75	1.0- 1.75
Si.....	0.05- 1.5	0.05- 1.5	0.05- 1.5
Cr.....	10.0-22.0	12.0-18.0	12.0-18.0
Ni.....	15.0-50.0	20.0-30.0	20.0-30.0
Mo.....	0.25- 2.0	1.0- 1.5	1.0- 1.5
Ti.....	0.50- 4.5	1.5- 3.5	2.5- 3.5
Al.....	1.0	0.35	0.35
V.....	0.10- 1.5	0.10- 0.5	0.10- 0.50
B.....	0.10- 0.80	0.10- 0.40	0.10- 0.40
Fe.....	Bal.	Bal.	Bal.

¹ Maximum.

Each of the alloying elements present within the general range as set forth in Table I performs a specific function. Carbon in combination and cooperation with boron and titanium contributes materially to increasing the rupture ductility and the notch rupture life of the alloy when each of the elements, carbon, boron and titanium is properly proportioned within the alloy. The manganese within the range stated is necessary for conferring hot workability upon the alloy and may enter the solid solution to increase the rupture ductility of the alloy. Manganese is essential where the alloy of this invention is commercially produced by air melting methods, for example, carbon electrode electric arc furnace melting, in that it contributes to the hot workability of the alloy. Silicon is present within the alloy of this invention and contributes to the strength thereof and is effective for contributing to the oxidation resistance of the alloy.

Chromium is the predominant element for providing corrosion resistance and oxidation resistance to the alloy when it is used at elevated temperature. Chromium also enters the solid solution and materially contributes to the strength of the matrix when it is present within the general range. Chromium contents in excess of 22% tend to form intermetallic phases which when present reduce the room temperature tensile ductility and adversely affect the rupture strength. Although chromium is a strong ferrite forming element, when it is in solution at elevated temperatures it reacts to retard structural changes and thus tends to stabilize the alloy.

Nickel is the predominant austenite forming element and acts in cooperation with the chromium to provide sufficient oxidation and corrosion resistance. Nickel is also essential for providing the precipitation hardening reaction which is the major strengthening process occurring in this alloy when sufficient titanium is present. In this respect, it is to be noted that a portion of the nickel can be replaced by up to 25% cobalt, the substitution being in direct proportion to each other. Where there is no cobalt present within the alloy a minimum of about 15% nickel is necessary for the precipitation hardening process to occur in the alloy.

Molybdenum within the range given materially contributes to the strengthening of the solid solution and in this respect is particularly effective for offsetting the embrittling effect which is normally expected with the addition of certain of the alloying elements. Titanium is highly critical to this alloy and is present within the solid solution to substantially strengthen the alloy and contribute to the precipitation of a transition phase of an intermetallic compound of the formula Ni_3Ti . Although aluminum is generally considered to contribute towards the embrittlement of this type of alloy, small amounts of aluminum cooperate with nickel and titanium in the precipitation hardening reaction. Aluminum also contributes to the oxidation resistance thereof. Vanadium enters into the solid solution of this alloy and also contributes to reduce the embrittling effect encountered with the use of titanium and aluminum.

Boron within the range disclosed is highly critical in that it permits not only a hardening of the matrix of the alloy thereby contributing to the strength, but is also particularly effective for inhibiting grain growth when the alloy is solution heat treated at high temperatures and exerts an extremely pronounced effect in increasing the rupture ductilities of these alloys when they are used at elevated temperatures and under high stresses. The balance of the alloy consists predominantly of iron with not more than 2% of incidental impurities such as nitrogen, phosphorus, sulfur, copper and other impurities normally found in the commercial production of such alloys.

It is significant to point out that in the alloys of both Optimum Range A and Optimum Range B the manganese, silicon chromium, nickel, molybdenum, aluminum and vanadium ranges are the same. These elements within the preferred ranges given are deemed necessary in order to provide the alloy with its optimum balance of properties. Specifically, the chromium content may be varied from a minimum of 10% where there is a high amount of nickel and/or cobalt present up to a maximum amount of 22.0%. However, at least 12% chromium is necessary in order to obtain optimum corrosion and oxidation resistance consistent with good elevated temperature, strength, ductility and hardness. While chromium contents in excess of 18% can be effectively used, it has been found that the preferred balance of the mechanical properties of the alloy is obtained when the chromium content is limited to about 18%. Nickel which in the general range may vary from a minimum amount of about 15% which has been found to be necessary in order to insure a completely austenitic structure and insure precipitation hardening within the alloy, up to about 50% beyond which no further substantial improvement is noted in any of the properties of the alloy, also contributes to the corrosion and oxidation resistance of the alloy. Within this range a nickel content between 20.0% and 30.0% appears to provide the optimum balance of the required properties. Substantially the same considerations are involved with respect to the manganese, silicon, molybdenum, aluminum and vanadium contents. On the other hand, the elements carbon, titanium and boron present in the alloy require a very critical balance in order to obtain the optimum combination of properties.

Specifically, Optimum Range A, the preferred embodi-

ment where the greatest creep strength, excellent rupture life consistent with great rupture ductility is available, contains between about 0.01% and 0.1% carbon, 1.5% and 3.5% titanium and 0.1% to 0.4% boron. It has been found that the greatest creep strength consistent with excellent rupture life, hardness, corrosion and oxidation resistance is obtained when the carbon content is maintained within the range between 0.01% and 0.10%. In this range with the titanium being present in an amount between 1.5% and 3.5% any increase in the carbon content above 0.10% effects the formation of increasing amounts of titanium carbide which adversely affects the strength and decreases the rupture life of the alloy. While the carbon content is preferably maintained low for alloys in the Optimum Range A, practical considerations require that the alloy contain at least 0.01% carbon. The titanium content must be maintained within the range between 1.5% and 3.5% because titanium is directly related to the strength of the alloy when used at elevated temperatures. A minimum of 1.5% is necessary in order to significantly strengthen the matrix phase to a degree sufficient to make the alloy useful at elevated temperatures. While titanium is not the only element which contributes to the strength of this alloy, its effect, however, is outstanding in this respect. Titanium contents in excess of about 3.5% in the alloys of Optimum Range A appear to contribute to a reduction in rupture ductility.

The boron content within the range between 0.1% and 0.4% with the corresponding carbon content within the range between 0.01% and 0.10% and the titanium content within the range between 1.5% and 3.5% is extremely effective for inhibiting the grain growth of the alloys of Optimum Range A thereby materially increasing the short-time tensile strength and the fatigue strength thereof. A minimum of 0.10% boron is necessary in order to provide for the smaller grain size of the alloy in its heat treated form and also for contributing to a very great extent to the rupture ductility of the alloy. Further, boron contents in the range of below 0.1% down to 0.1% while having been found to be effective for forming an excess of an intermetallic compound which may be sufficient for inhibiting grain growth during heat treatment, such small quantities were insufficient to obtain the requisite good ductility. In addition, the boron residues of below 0.01% which are normally present in A-286 alloy as a result of the use of ferrotitanium containing boron while giving some improvement in hot workability of air melted alloys are insufficient to impart good ductility after treatment at the high solution temperature to which the alloys of this invention are subjected as will be described hereinafter. While boron contents in excess of 0.4% can be used it is preferred to maintain the upper limit of the boron content at 0.40% within the Optimum Range A in order to prevent an excess precipitation of a complex titanium-boron phase which may materially contribute to the loss of strength of the alloy by combining with the titanium thereby decreasing its effectiveness.

Substantially somewhat different considerations are involved with respect to Optimum Range B. The alloys of Optimum Range B have the characteristics of extremely high rupture ductility with a correspondingly adequate creep strength and rupture life and without any adverse effect on notch rupture sensitivity as well as the other mechanical properties of the alloy. In the alloys of Optimum Range B, the carbon content is maintained between about 0.10% and 0.2% and cooperates with the titanium and boron contents in order to provide extremely good rupture ductility in the alloy. In no event should the carbon content exceed an amount of about 0.2%. Carbon contents in excess thereof have a tendency to form excess amounts of titanium carbide depleting the matrix of titanium which is necessary for elevated temperature strength.

The titanium content of the alloys of Optimum Range B is preferably maintained within the range between 2.5% and 3.5% in that it has been found that where the alloy is heat treated as will be more fully set forth hereinafter, at the high temperature which is considerably in excess of that heretofore believed desirable in this particular type of alloy, a substantially greater solubility for the titanium content exists within the matrix phase of this alloy. At least 2.5% titanium is necessary in this preferred embodiment in order to insure the adequate rupture strength for the alloy. Increasing the titanium content to greater than 3.5% has the effect of precipitating increasing amounts of a secondary phase which has no useful effect on the mechanical properties of this alloy, and results in poorer hot workability.

The corresponding boron content found to be necessary in the alloys of Optimum Range B is within the range 0.10% and 0.40%. Boron contents below 0.1% and particularly below .01% such as were found as residues from ferrotitanium utilized in making A-286 alloy may be effective for inhibiting grain growth but do not impart to the alloy the extremely high degree of rupture ductility desired at this strength level. The optimum combination between the highest rupture ductility, adequate creep strength, rupture life and grain growth inhibition is obtained when the alloy of this invention has a boron content of at least 0.10%. While boron contents in excess of 0.40% can be used within the alloy of this invention the optimum combination of mechanical properties appears to be obtained when the boron content does not exceed about 0.40%. While boron contents in excess thereof within the limits of the general range will increase the rupture ductility, the rupture strength and creep strength are adversely affected as well as the other mechanical properties of the alloy. The foregoing considerations governing the preferred embodiments of Optimum Range A and Optimum Range B are more clearly evident from the data contained in Table III.

As was stated hereinbefore, the alloy of this invention is a precipitation hardening alloy and as such it requires a preferred heat treatment in order to develop its optimum properties of rupture life, rupture ductility and creep strength. In general, the heat treatment consists of a solution heat treatment followed by quenching and thereafter the alloy is aged for a given period of time.

Heretofore, the commercial A-286 alloy containing residues of boron was heat treated by a solution heat treatment at a temperature of up to about 1850° F. maximum followed by quenching, usually in oil or water, and thereafter aging at a temperature of about 1325° F. The low solution heat treatment temperature was found necessary because of the high degree of grain growth

during heat treatment at higher temperatures. This grain growth at temperatures in excess of 1850° F. and up to 2050° F. was sufficient to produce a grain size in the A-286 alloy of about ASTM #1 or larger which adversely affected the mechanical properties, especially the ductility and fatigue strength. The solution heat treatment was therefore limited to a maximum temperature of about 1850° F. which limited the amount of titanium which was soluble within the solid solution at this heat treatment temperature to about 2.4% maximum. With such low maximum solubility of titanium, the rupture strength was affected by having less titanium in solid solution and the full capability of age hardening through the precipitation of a coherent transition phase of a titanium intermetallic compound was not fully realized.

On the other hand, the alloy of this invention requires a higher solution heat treatment temperature in order to develop optimum properties and this has been accomplished without an abnormally large grain size and, in fact, the grain size of the alloy of this invention in its heat treated form is substantially smaller than that of the prior art A-286 alloy. In particular, it is preferred to solution heat treat the alloy of this invention by heating to a temperature in the range between 1850° F. and 2150° F. and preferably between 1950° F. and 2100° F. for a time period ranging between ¼ and 8 hours depending upon the thickness of the metal being heat treated and the alloy is thereafter cooled to room temperature by quenching in air, oil or water or any other medium sufficient to prevent any precipitation of an intermetallic compound of an element which has been taken into solution at the solution heat treatment temperature. The quenched alloy is then aged at a temperature within the range between 1200° F. and 1500° F. and preferably between 1250° F. and 1350° F. for a time period ranging between 4 and 50 hours after which it is air cooled to room temperature. The heat treatment described hereinbefore is effective for producing an alloy having a grain size of no larger than ASTM #5 and at the same time producing an outstanding combination of mechanical properties as will be more clearly set forth hereinafter.

In order to illustrate the effect of carbon, titanium and boron on the rupture life, rupture ductility and creep rate of the alloy of this invention, a number of heats of this alloy were made and tested to show the outstanding effects of these elements on the mechanical properties of the alloys. Reference is directed to Table II which illustrates the chemical composition of the heats of the alloys used to illustrate the effects of these elements on the mechanical properties.

TABLE II

Heat No.	C	Mn	Si	Cr	Ni	Mo	Ti	Al	V	B	Fe
D-375-----	.070	1.23	.75	14.88	25.74	1.30	2.00	.13	.35	0	Bal.
D-379-----	.092	1.34	.84	15.73	25.81	1.26	2.12	.08	.30	.095	Bal.
D-380-----	.076	1.25	.82	15.52	25.96	1.20	2.12	.08	.32	.234	Bal.
D-381-----	.091	1.20	.78	14.70	25.55	1.22	2.30	.16	.35	.356	Bal.
D-516-----	.024	1.28	.62	14.92	26.30	1.32	2.38	.16	.36	0	Bal.
D-517-----	.062	1.31	.71	14.84	25.96	1.35	2.06	.14	.32	.014	Bal.
D-518-----	.034	1.20	.69	14.32	25.88	1.33	2.24	.15	.32	.027	Bal.
D-519-----	.050	1.38	.56	14.76	25.96	1.30	2.00	.11	.33	.054	Bal.
D-520-----	.042	1.37	.75	15.22	26.14	1.31	2.28	.16	.33	.126	Bal.
D-562-----	.045	1.66	.89	15.08	25.71	1.34	2.76	.18	.37	.126	Bal.
D-621-----	.033	1.21	.68	14.81	25.72	1.25	3.08	.49	.31	.014	Bal.
D-623-----	.028	1.03	.24	15.14	25.77	1.30	3.24	.14	.36	.050	Bal.
D-625-----	.038	1.34	.80	15.04	26.02	1.36	2.94	.18	.33	.153	Bal.
D-626-----	.037	1.45	.69	15.12	26.02	1.34	2.92	.18	.30	.281	Bal.
D-627-----	.037	1.38	.61	14.73	26.04	1.34	2.84	.18	.38	.362	Bal.
D-628-----	.060	1.33	.86	14.82	26.00	1.33	2.84	.17	.30	.516	Bal.
D-629-----	.036	1.24	.90	15.06	26.14	1.33	2.44	.11	.33	.799	Bal.
D-756-----	.105	1.54	.64	15.00	25.96	1.30	3.23	.18	1.30	0	Bal.
D-757-----	.126	1.58	.82	15.16	26.44	1.37	3.14	.12	.33	.095	Bal.
D-758-----	.121	1.45	.84	14.88	25.88	1.30	2.77	.09	.33	.276	Bal.
D-759-----	.188	1.56	.76	14.66	25.92	1.33	2.92	.13	.29	0	Bal.
D-760-----	.186	1.50	.75	15.16	25.93	1.25	2.65	.09	.29	.168	Bal.
D-761-----	.162	1.54	.75	15.16	25.96	1.30	3.23	.17	.25	.326	Bal.
4X-170-----	.056	1.20	.52	14.83	26.38	1.23	2.33	.16	.30	.172	Bal.
9X-141-----	.042	1.45	.53	14.72	25.94	1.34	2.44	.20	.31	.201	Bal.
9X-178-----	.063	1.53	.89	14.87	25.33	1.28	2.23	.16	.32	.140	Bal.

¹ Aim analysis—Actual value not reported.

It is to be noted that the alloys set forth hereinbefore in Table II have a composition which is both within and outside of the general range of the alloying elements as set forth hereinbefore in Table I.

In evaluating the alloys set forth in Table II, creep-rupture tests are utilized. In making the tests the alloy is formed into a creep-rupture bar having a diameter of about 0.195 inch. The bar is subjected to a given stress at a given temperature and the time required to produce rupture in the alloy is measured in order to determine the rupture life of the alloy. This is the standard creep-rupture test. In the same test, the creep rates were also measured. Reference is directed to Table III which sets forth the test results of the creep-rupture tests used to evaluate the alloys set forth in Table II.

therein indicates that for the corresponding alloys in subsection 1 and subsection 2 a great increase is noted in the rupture ductility as measured by the percentage elongation and the reduction of area with increasing amounts of boron present within the alloy. The rupture life appears to be unchanged and the creep strength only slightly decreased with additions of up to 0.2% boron. In particular, it is seen that by increasing the boron content from 0% up to about 0.2% there has been produced an outstanding increase in the proper balance of the rupture ductility and creep rate in the alloys. Specific note must be taken of Alloys D-518, D-520 and 4X-170 in subsection 2 of section A wherein it appears that about 0.02% boron is effective for producing an extremely long rupture life and an extremely low creep rate. However,

TABLE III.—CREEP-RUPTURE PROPERTIES

Effect of Boron

A. 0.04% C; 2.2% Ti

Heat No.	Heat treat	Test temp. (° F.)	Test stress (p.s.i.)	C (percent)	Ti (percent)	B (percent)	Rupture life (hrs.)	Elong. in "4d" (percent)	Red. of area (percent)	Min. creep rate (percent/hr.)
1. D-516-----	(1)	1,200	62,500	0.02	2.4	0.0	163	3.5	9.0	0.003
D-517-----	(1)	1,200	62,500	0.06	2.1	0.014	378	4.7	7.7	0.002
D-518-----	(1)	1,200	62,500	0.03	2.2	0.027	254	5.3	7.1	0.002
D-519-----	(1)	1,200	62,500	0.05	2.0	0.054	175	6.2	12.4	0.008
D-520-----	(1)	1,200	62,500	0.04	2.3	0.126	279	10.5	18.5	0.011
9X-141-----	(1)	1,200	62,500	0.04	2.4	0.201	245	17.2	26.6	0.016
D-629-----	(1)	1,200	62,500	0.04	2.4	0.799	63	31.2	52.3	0.127
2. D-516-----	(2)	1,200	62,500	0.02	2.4	0.0	1,209	2.0	4.1	0.0002
D-518-----	(2)	1,200	62,500	0.03	2.2	0.027	1,550	4.2	7.2	0.0001
D-520-----	(2)	1,200	62,500	0.04	2.3	0.126	1,173	12.4	30.0	0.0006
4X-170-----	(2)	1,200	62,500	0.06	2.3	0.172	1,431	11.5	20.2	0.0008
D-629-----	(2)	1,200	62,500	0.04	2.4	0.799	80	29.5	51.0	0.0840

B. 0.08% C; 2.2% Ti

1. D-375-----	(1)	1,200	62,500	0.07	2.0	0.0	124	6.1	13.6	0.008
D-379-----	(1)	1,200	62,500	0.09	2.1	0.095	76	20.1	27.0	0.046
D-380-----	(1)	1,200	62,500	0.08	2.1	0.234	81	27.6	49.5	0.078
D-381-----	(1)	1,200	62,500	0.09	2.3	0.356	81	27.2	39.0	0.088
2. D-375-----	(2)	1,200	62,500	0.07	2.0	0.0	831	9.0	19.7	0.001
D-379-----	(2)	1,200	62,500	0.09	2.1	0.095	468	18.1	45.6	0.003
9X-178-----	(2)	1,200	62,500	0.06	2.2	0.140	425	32.8	35.2	0.008
D-380-----	(2)	1,200	62,500	0.08	2.1	0.234	326	16.1	48.4	0.007
D-381-----	(2)	1,200	62,500	0.09	2.3	0.356	310	19.6	50.0	0.009

C. 0.04% C; 3.0% Ti

1. D-621-----	(1)	1,200	62,500	0.03	3.1	0.014	388	3.1	7.8	0.002
D-623-----	(1)	1,200	62,500	0.03	3.2	0.050	605	3.0	7.1	0.002
D-624-----	(1)	1,200	62,500	0.05	2.9	0.126	280	6.3	12.2	0.005
D-625-----	(1)	1,200	62,500	0.04	2.9	0.153	297	8.4	12.7	0.007
D-626-----	(1)	1,200	62,500	0.04	2.9	0.281	317	12.1	14.2	0.009
D-627-----	(1)	1,200	62,500	0.04	2.8	0.362	255	12.5	32.3	0.007
D-628-----	(1)	1,200	62,500	0.06	2.8	0.516	129	25.5	54.5	0.029
2. D-623-----	(2)	1,200	63,500	0.03	3.2	0.050	969	3.4	4.1	0.0002
D-625-----	(2)	1,200	62,500	0.04	2.9	0.153	1,353	1.2	1.6	0.0006
D-626-----	(2)	1,200	62,500	0.04	2.9	0.281	1,664	8.7	25.7	0.0004
D-627-----	(2)	1,200	62,500	0.04	2.8	0.362	904	13.3	36.0	0.0016
D-628-----	(2)	1,200	62,500	0.06	2.8	0.516	502	20.9	50.2	0.0040

D. 0.12% C; 3.0% Ti

D-756-----	(3)	1,200	75,000	0.11	3.2	0.0	420	1.5	3.1	0.0006
D-757-----	(3)	1,200	75,000	0.13	3.1	0.095	206	4.0	11.4	0.0052
D-758-----	(3)	1,200	75,000	0.12	2.7	0.276	37	26.7	40.0	0.0125

E. 0.18% C; 3.0% Ti

D-759-----	(2)	1,200	75,000	0.19	2.9	0.0	219	4.1	6.9	0.0021
D-760-----	(2)	1,200	75,000	0.19	2.7	0.168	57	18.3	33.4	0.0640
D-761-----	(2)	1,200	75,000	0.16	3.2	0.326	54	21.3	31.0	0.1350

¹ 1,800° F., 1 hr., oil quench + 1,300° F., 16 hrs., air cool.

² 2,050° F., 1 hr., oil quench + 1,300° F., 16 hrs., air cool.

³ 2,050° F., 1 hr., oil quench + 1,325° F., 16 hrs., air cool.

Referring now to section A of Table III and in particular to the data for the alloys of Heat Nos. D-516, D-517, D-518, D-519, D-520, 9X-141, D-629 and 4X-170, each of which is contained in subsections 1 and 2 of section A the effect of increasing the boron content on the rupture life, ductility and creep rate in an alloy containing nominally about 0.04% carbon and about 2.2% titanium is clearly set forth. The data contained

it will be noted that for Heat D-518 the elongation and reduction of area values which are used as the criteria for the measurement of ductility are extremely low. While arbitrary figures of about 8% elongation and about 15% reduction of area have been used as desirable minimum limits in evaluating these alloys, it is clearly apparent from Heats D-520 and 4X-170 that at least 0.1% boron and preferably higher amounts not to exceed 0.40%

boron are effective for imparting substantially high rupture life with excellent rupture ductility and a low creep rate. Indeed, the creep rate is outstanding for Heats D-520 and 4X-170 which have a creep rate of 0.0006% and 0.0008% per hour, respectively. It is thus apparent that the high solution heat treatment temperatures which are preferred as set forth hereinbefore are extremely effective for producing the optimum combination of mechanical properties for the alloys of this invention. Of the alloys set forth in subsections 1 and 2 of section A it is seen that only Alloys D-520, 9X-141 and 4X-170 are within the Optimum Range A which exhibit the extremely good creep rates. While Alloy D-629 is within the general range it is seen that the optimum combination of properties is not present within that alloy since it has inferior rupture life and creep rate but has an extremely good ductility. From the foregoing, it is apparent that the boron content is effective for imparting excellent rupture ductility to the alloys of this invention. The boron content is also effective in cooperation with the carbon and titanium contents for imparting excellent creep rates and an adequate rupture life to these alloys.

Referring to section B of Table III and subsections 1 and 2 thereof, the test results for the alloys of Heat Nos. D-375, D-379, D-380, D-381 and 9X-178 are set forth therein and clearly illustrate the effect of increasing the boron content on the mechanical properties in alloys containing nominally about 0.08% carbon and about 2.2% titanium. By comparing the alloys in subsection 1 and subsection 2, it is seen that the high heat treatment temperatures are effective for producing an alloy having an optimum combination of mechanical properties. This again is corroborative of what has been said concerning section A with respect to heat treatment. The increased carbon content appears to impart greater ductility to the alloy with titanium and boron contents in substantially the same range. It is to be noted, however, that while the carbon content has produced an increase in the ductility, the creep rate and rupture life while having been decreased in these alloys are still outstanding when compared to A-286 alloy containing residues of boron of up to .01% boron. In particular, it is to be noted that only Alloy D-375 is outside the preferred embodiment of Optimum Range A. Comparing the test results it is clear that the addition of boron is effective for producing excellent rupture ductility together with excellent creep rates and rupture life. While somewhat higher rupture life and a low creep rate are obtainable when the carbon content is near the lower end of the preferred range as set forth in Optimum Range A, where considerations of high ductility are required with a correspondingly excellent creep rate, the carbon content is preferred to be near the upper end of the range.

As was stated hereinbefore, the higher solution heat treatment temperatures make it possible for a greater amount of titanium to be taken into solid solution. This has the effect of strengthening the matrix phase of the alloy and at the same time produces higher mechanical properties when the alloy is properly aged. Referring in particular to section C of Table III and to subsections 1 and 2 thereof, the effect of the boron content on the mechanical properties of alloys containing nominally 0.04% carbon and 3.0% titanium is clearly illustrated. As was shown by the data contained in sections A and B, the alloys of section C are confirmatory in that the high solution heat treatment temperatures are effective for imparting extremely good mechanical properties to these alloys. This is clearly illustrated by comparing Alloys D-621, D-623, D-562, D-625, D-627 and D-628 of subsection 1 with the similar alloys of subsection 2 in section C. As clearly set forth therein the alloys having less than 0.1% boron have far inferior rupture ductilities and in some cases inferior rupture life. However, where the boron content is increased to more than 0.1% and in par-

ticular up to about 0.4% it is seen that a great increase is noted in the rupture ductility and rupture life in these alloys which are within the preferred embodiment of Optimum Range A. Alloy D-626 appears to have an outstanding combination of mechanical properties. Thus the lower carbon content is effective for producing extremely low creep rates, high rupture life and high ductility. This substantially corresponds to what was observed with respect to the data contained for the alloys in sections A and B.

As was stated hereinbefore, the considerations involved as respects the characteristics of the alloys of the preferred embodiment of Optimum Range B as set forth in Table I are extremely high rupture ductility with adequate rupture life and creep rate. These characteristics are essential where the alloy is used, for example, in the form of a bolt in a steam turbine. The primary concern of manufacturers in considering such applications is the rupture ductility of the alloy although the creep rate and rupture life cannot be too low. Section D contains the data for Alloys D-756, D-757 and D-758 and clearly illustrates the effect of boron on the mechanical properties of alloys containing nominally about 0.12% carbon and about 3.0% titanium, it being noted that these alloys correspond to the preferred embodiment of Optimum Range B. Increasing the boron content to greater than 0.10% is effective for substantially increasing the rupture ductility of these alloys. Correspondingly, however, the creep rate is increased and the rupture life is decreased. The alloys of section E, that is, D-759, D-760 and D-761 which contain nominally about 0.18% carbon and 3.0% titanium with increasing boron contents effectively illustrate the influence of higher carbon content with increasing boron contents in these alloys. It is clear that Alloys Nos. D-760 and D-761 have a very high rupture ductility. Contrasted thereto, Alloy No. D-759 with a boron content outside of the general range taught in Table I has good rupture life but very poor rupture ductility. It is apparent therefore that for the highest rupture ductility it is preferred to have the carbon content near the upper end of Optimum Range B with a corresponding boron content in the range between 0.10% and 0.40%.

As was stated previously, the higher carbon content with high boron content in the range taught herein is effective for imparting extremely high rupture ductility to these alloys although such high rupture ductility is attained at the expense of the rupture life and creep rate. It is to be noted that with respect to sections D and E of Table III the rupture life and creep rate properties appear to be lower and higher, respectively, than those set forth in sections A, B and C. While the same test temperatures were used in all cases, the test data set forth in sections D and E were obtained where the alloys were under substantially higher stresses than the stresses used for the data recorded in sections A, B, and C. It is clear from sections D and E and the test results recorded therein that Optimum Range B is particularly suitable for use in applications which require extremely high rupture ductility. While the test conditions under which the data set forth in sections D and E of Table III are different from those of sections A, B and C, thus prohibiting a direct comparison of the test data, it is apparent that the alloys of sections D and E possess an adequate rupture life so as to be usable where rupture ductility is of prime importance and in particular in steam turbine applications.

In order to more clearly illustrate the effect of boron, titanium and carbon on the mechanical properties of these alloys, reference is directed to the graphical illustration of the curves contained in FIGS. 1 and 2. In FIG. 1, curve 10 illustrates the effect of increasing the boron content on the alloys contained in subsection 1 of section B of Table III on the creep rate. It is apparent that increasing the boron content up to about 0.3% produces a corresponding increase in the creep rate.

Beyond 0.4% boron, no significant further increase in the creep rate was noted. Curve 12 illustrates the effect of increasing the boron content on the creep rates for the alloys contained in subsection 2 of section B of Table III. It is at once apparent that the higher solution heat treatment temperatures are effective for substantially decreasing the creep rates of these alloys. Curve 14 shows the effect of the boron content on the creep rate for the alloys contained in subsection 1 of section C, and curve 16 illustrates the same effect for the alloys of subsection 2 of section C. Here again, the effect of the high solution temperature heat treatment is readily apparent. The lower carbon content is effective for substantially decreasing the creep rate while the increase in boron content substantially increases the creep rate. In any event, FIG. 1 clearly illustrates the outstanding advantage of both the solution heat treatment temperatures and the corresponding boron and carbon contents on the creep rate.

FIG. 2 is a graphical illustration of the effect of boron on the rupture ductility as measured by the percentage elongation. Curve 20 graphically illustrates the effect of the boron content on the rupture ductility for the alloys contained in subsection 1 of section B whereas curve 22 illustrates the effect of the boron content for the alloys contained in subsection 2 of section B. While it appears that the lower solution heat treatment temperature is effective for increasing the ductility with a corresponding increase in the boron content, the corresponding strengths of these alloys are far inferior. However, it will be noted from curves 20 and 22 that the maximum rate of increase in ductility is obtained with boron contents of up to about 0.1% boron, there being so appreciable increase in ductility when the boron content is increased beyond about 0.1%. Curve 24 and curve 26 of FIG. 2 illustrate the effect of the boron content on the rupture ductility for the alloys contained in subsection 1 and subsection 2, respectively, of section C of Table III. With the lower carbon content an increase in the boron content of the alloy produces a correspondingly higher ductility in the alloy. While it appears that the lower heat treatment temperatures produce slightly higher ductilities, it is apparent from Table III as well as FIG. 1 that the other mechanical properties are far inferior unless the higher solution heat treatment is used. It is also apparent by comparing curve 22 with curve 26 having a nominal carbon content of 0.08% and 0.04%, respectively, that the higher carbon contents impart greater ductility with increasing amounts of boron of up to about 0.4%. This same trend is noted irrespective of the heat treatment given to these alloys.

Reference is directed to the photomicrographs contained in FIGS. 3 and 4 which illustrate the effect of boron on the grain size of the alloy of this invention as quenched after solution heat treatment for 1 hour at a temperature of 2050° F. FIG. 3 is a photomicrograph taken at a magnification of 100 times of A-286 alloy without intentional additions of boron whereas FIG. 4 is a photomicrograph of Heat 9X-141 which contains about 0.2% boron, said magnification also being 100 times. A comparison of the grain sizes of the alloys of the photomicrographs of FIGS. 3 and 4 clearly illustrates the effect of boron on the inhibition of grain growth. Thus the photomicrograph of FIG. 3 clearly illustrates that the solution heat treated A-286 alloy which is devoid of intentional additions of boron has a grain size of ASTM #4 to larger than ASTM #1. FIG. 4 shows that the addition of 0.2% boron in the alloy of this invention is effective for controlling the grain size of the solution heat treated alloy so that it is smaller than ASTM #5 and, in particular, in the range of ASTM #6 to finer than ASTM #8. It is apparent from FIGS. 3 and 4 that the boron content is highly effective for restricting the grain growth in this alloy during solution heat treatment. This in turn has the corresponding effect of producing better mechanical properties and, in particular, hardness,

short-time tensile strength, rupture ductility and fatigue strength.

It is apparent from the foregoing that the alloys of this invention is quite versatile in that an optimum combination of properties is available through controlled variations of the carbon, titanium and boron contents within the ranges given in Table I. No special difficulties are encountered in melting or in working the alloy and special emphasis has been placed on minimizing the amount of critical and strategic alloying elements used therein consistent with the obtaining of excellent mechanical properties. The alloy is particularly suitable for use at temperatures of up to about 1400° F. under extremely high loads. Since the alloy has substantial freedom from notch rupture sensitivity this characteristic makes it extremely attractive from the standpoint of use in turbine components.

This application is filed as a continuation-in-part of application Serial No. 673,056, now abandoned.

We claim:

1. An austenitic iron base alloy containing from 0.01% to 0.20% carbon, from 1.0% to 3.0% manganese, from 0.05% to 1.5% silicon, from 10.0% to 22.0% chromium, from 15.0% to 50.0% nickel, from 0.25% to 2.0% molybdenum, from 0.50% to 4.5% titanium, from 0.05% to 1.0% aluminum, from 0.10% to 1.5% vanadium, from 0.10% to 0.80% boron, and the balance iron with incidental impurities.

2. An austenitic iron base alloy consisting of from 0.01% to 0.10% carbon, from 1.0% to 1.75% manganese, from 0.05% to 1.5% silicon, from 12.0% to 18.0% chromium, from 20.0% to 30.0% nickel, from 1.0% to 1.5% molybdenum, from 1.5% to 3.5% titanium, up to 0.35% aluminum, from 0.1% to 0.5% vanadium, from 0.10% to 0.40% boron, and the balance iron with incidental impurities.

3. An austenitic iron base alloy consisting of from 0.10% to 0.20% carbon, from 1.0% to 1.75% manganese, from 0.05% to 1.5% silicon, from 12.0% to 18.0% chromium, from 20.0% to 30.0% nickel, from 1.0% to 1.5% molybdenum, from 2.5% to 3.5% titanium, up to 0.35% aluminum, from 0.10% to 0.5% vanadium, from 0.10% to 0.40% boron, and the balance iron with incidental impurities.

4. An austenitic iron base alloy consisting of from 0.01% to 0.20% carbon, from 1.0% to 3.0% manganese, from 0.05% to 1.5% silicon, from 10.0% to 22.0% chromium, from 15.0% to 50.0% nickel, from 0.25% to 2.0% molybdenum, from 0.50% to 4.5% titanium, from 0.05% to 1.0% aluminum, from 0.10% to 1.5% vanadium, from 0.10% to 0.80% boron, and the balance iron with incidental impurities, the alloy being characterized by having excellent rupture ductility, strength, creep rates and a grain size smaller than ASTM #5 resulting from quenching the alloy from a solution heat treating temperature in the range between 1850° F. and 2150° F. followed by aging at a temperature in the range between 1200° F. and 1500° F.

5. An austenitic iron base alloy consisting of from 0.01% to 0.10% carbon, from 1.0% to 1.75% manganese, from 0.05% to 1.5% silicon, from 12.0% to 18.0% chromium, from 20.0% to 30.0% nickel, from 1.0% to 1.5% molybdenum, from 1.5% to 3.5% titanium, up to 0.35% aluminum, from 0.1% to 0.5% vanadium, from 0.10% to 0.40% boron, and the balance iron with incidental impurities, the alloy being characterized by having excellent rupture ductility, strength, creep rate and a grain size smaller than ASTM #5 resulting from quenching the alloy after solution heat treatment at a temperature in the range between 1950° F. and 2100° F. for a time period of between ¼ and 8 hours followed by an aging treatment at a temperature in the range between 1250° F. and 1350° F. for a time period of between 4 and 50 hours.

6. An austenitic iron base alloy consisting of from

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0.10% to 0.20% carbon, from 1.0% to 1.75% manganese, from 0.05% to 1.5% silicon, from 12.0% to 18.0% chromium, from 20.0% to 30.0% nickel, from 1.0% to 1.5% molybdenum, from 2.5% to 3.5% titanium, up to 0.35% aluminum, from 0.10% to 0.5% vanadium, from 0.10% to 0.40% boron, and the balance iron with incidental impurities, the alloy being characterized by having excellent rupture ductility, strength, creep rate and a grain size small than ASTM #5 resulting from quenching the alloy after solution heat treatment at a temperature in the range between 1950° F. and 2100° F. for a time period of between ¼ and 8 hours followed by an aging treatment at a temperature in the range between 1250° F. and 1350° F. for a time period of between 4 and 50 hours.

7. A ferrous base alloy comprising essentially 20 to 35% nickel, 10 to 22% chromium, 1.0 to 2.0% molybdenum, 1.6 to 3.5% titanium, 0.10 to 0.15% boron, up to 0.40% aluminum, 0.01 to 0.10% carbon, 1.0 to 2.5% manganese, 0.3 to 1.5% silicon, 0.10 to 0.50% vanadium, and the balance being iron with incidental impurities.

8. In the process of producing an alloy member having improved stress-rupture properties at elevated tempera-

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tures, the steps comprising forging a member of an alloy comprising essentially 20 to 35% nickel, 10 to 22% chromium, 1.0 to 2.0% molybdenum, 1.6 to 3.5% titanium, 0.10 to 0.15% boron, up to 0.40% aluminum, 0.01 to 0.10% carbon, 1.0 to 2.5% manganese, 0.3 to 1.5% silicon, 0.10 to 0.50% vanadium, and the balance being iron with incidental impurities, solution treating the forged member at a temperature of 1800° F., quenching the solution treated member, and aging the quenched member at a temperature of 1325° F. for 16 hours.

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