March 24, 1959 T. W. EICHELBERGER 2,879,194

METHOD OF AGING IRON-BASE AUSTENITIC ALLOYS

Filed July 12, 1957 3 Sheets-Sheet 1

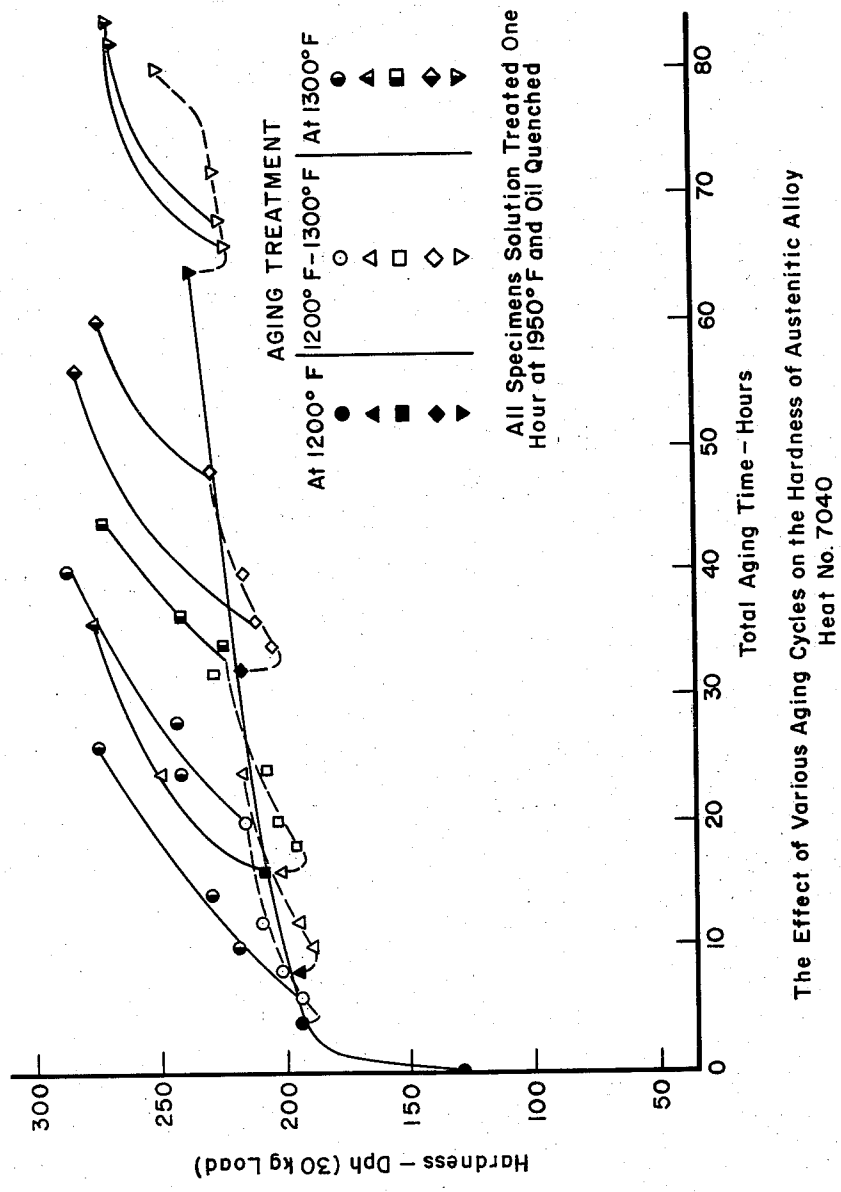

Fig. 1.

WITNESSES

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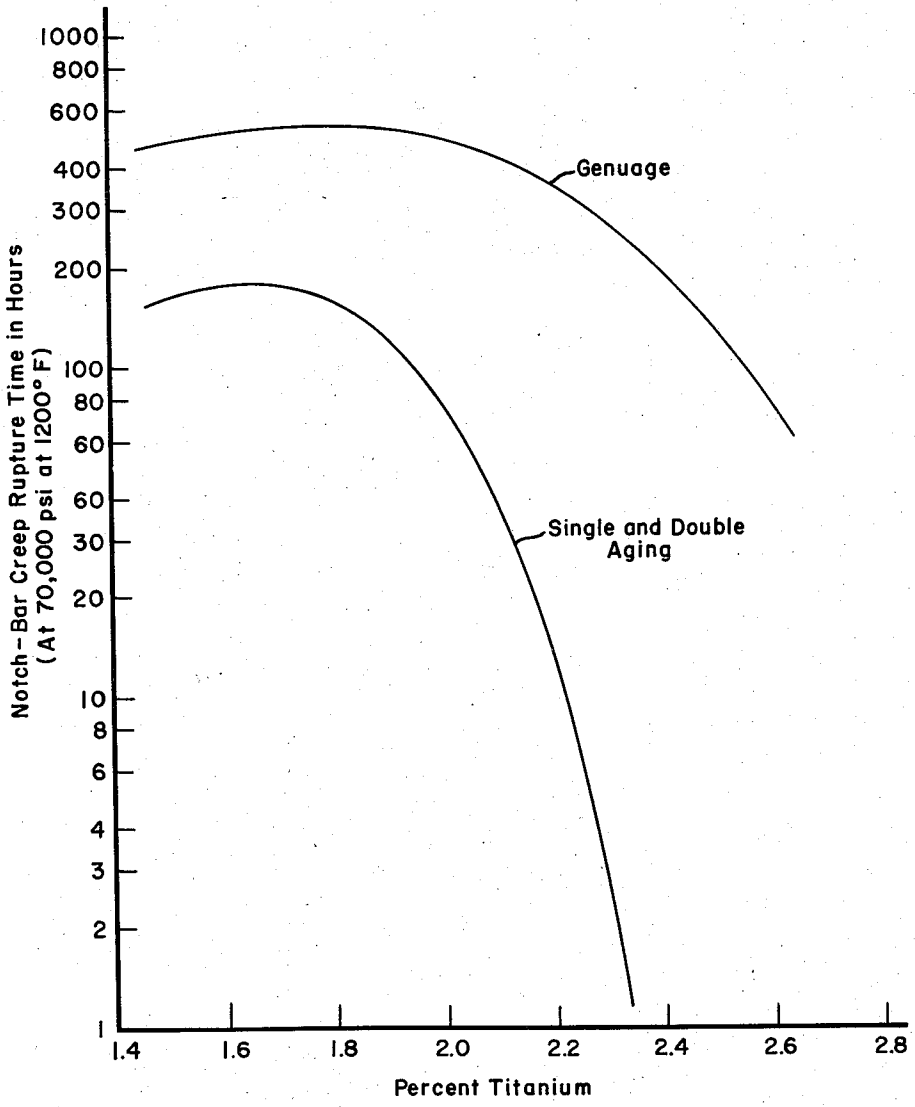
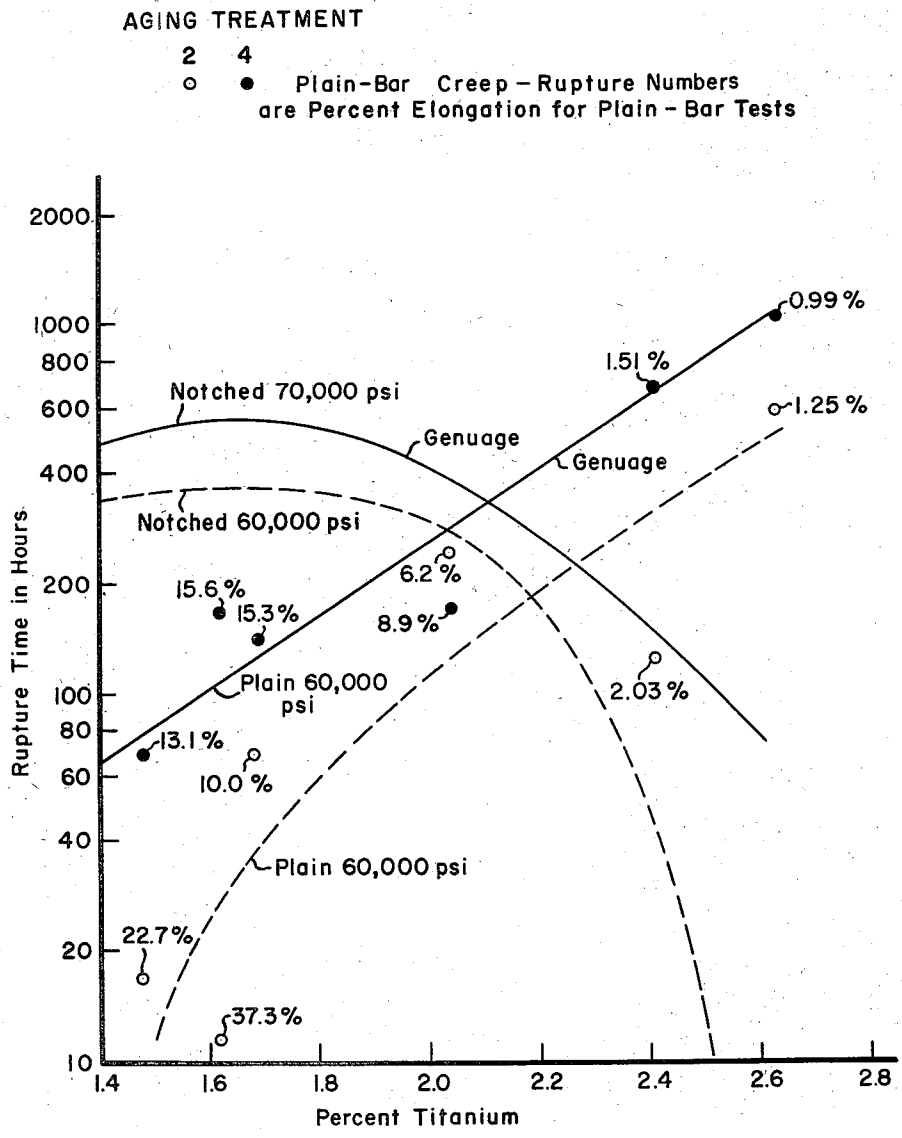
Fig. 2.



Notch Creep – Rupture Tests of Austenitic Alloys

METHOD OF AGING IRON-BASE AUSTENITIC ALLOYS

Filed July 12, 1957 3 Sheets-Sheet 3



1200° F Creep – Rupture Tests on Austenitic Alloys with Varying Titanium Contents

Fig. 3.

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2,879,194

METHOD OF AGING IRON-BASE AUSTENITIC ALLOYS

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Application July 12, 1957, Serial No. 671,598

7 Claims. (Cl. 148—21.54)

This invention relates to a heat treatment process for the hardening of precipitation hardening alloys.

At the present time high temperature age-hardenable alloys are employed extensively for applications in gas turbines and jet engines. In attaining the desired physical properties such as creep-rupture strength and ductility, members of the wrought alloys are subjected to selected heat treatment procedures. Such previously employed heat treatment procedures usually have comprised the heating of the member initially to a solution treatment temperature of from about 1700° F. to 2200° F., and then the members have been subjected to either a double aging treatment or a single aging treatment. The known commercial double aging heat treatments have resulted in an excess of age-hardening precipitate forming both at grain boundaries and at imperfections in the crystal structure. Consequently, the alloys have not been completely satisfactory because the excessive grain boundary precipitate has resulted in low ductility on creep-rupture tests. To avoid difficulties, the amount of precipitation-hardening component in the alloy is critically restricted.

The previously employed single aging heat treatment has not been entirely satisfactory for a number of reasons. A primary drawback has been the fact that such single heat treatments have required excessively long aging times to secure the best creep-rupture properties obtainable therefrom. At least 100 hours and as much as 1000 hours in the aging furnace is required to secure good creep-rupture properties for the given alloy.

The maximum hardness and the optimum creep-rupture properties for a given alloy being given a single aging treatment can be secured only at the lower aging temperatures applied for very long periods of time. For example, when a given titanium hardened austenitic alloy was aged 400 hours at 1200° F., it had a creep-rupture time of 108 hours at 60,00 p.s.i. at 1200° F., and an elongation of 17.2%. Other specimens of the same alloy aged for 20 hours at 1300° F., when tested at 60,000 p.s.i. loads at 1200° F. had a creep-rupture time of only 19 to 35 hours with the elongation being from 15% to 17%. This example emphasizes the long aging times required to produce an above average aged product by a single aging treatment.

It will be appreciated that single aging treatments requiring such lengthy periods of time add greatly to the cost of the wrought members and are not commercially acceptable.

The object of the present invention is to provide a multiple stage aging heat treatment procedure for precipitation-hardenable high temperature alloys which may be carried out in a relatively short total period of time and which will develop optimum creep-rupture properties for the given alloy.

A further object of the invention is to provide an aging treatment for high temperature austenitic alloys containing a precipitation-hardening component wherein the alloys after solution treatment are heat treated initially in a temperature range of from 1100° F. to 1250° F. for a period of time of the order of from 2 to 50 hours and then slowly raised to a higher temperature of from 1300° F. to 1450° F. and maintained in this latter temperature range to attain optimum creep-rupture properties.

A still further object of the invention is to provide a two-stage aging treatment of a solution-treated wrought member of a high temperature precipitation-hardening austenitic alloy wherein the first step comprises aging the alloy in the range of from 1100° F. to 1250° F. to cause nucleation of the precipitation-hardening component whereby to increase the hardness of the member and then slowly increasing the temperature of the member to from 1300° F. to 1450° F. and maintaining the member in this last temperature range to cause the full hardness of the member to be developed with very little grain boundary precipitate.

Other objects of the invention will in part be obvious and will in part appear hereinafter.

For a better understanding of the nature and objects of the invention, reference should be had to the following detailed description and drawing, in which:

Figure 1 is a graph plotting hardness against the total aging time of an alloy processed in accordance with the present invention;

Fig. 2 is a graph plotting notch bar creep-rupture time against the titanium content of a given precipitation-hardened alloy following the practice of the present invention and following the practice of the prior art; and

Fig. 3 is a graph plotting rupture time in hours against the 1200° F. creep-rupture properties of a precipitation-hardened alloy in which the titanium hardener content is varied.

The present invention particularly relates to high temperature austenitic alloys in which titanium alone or in combination with other components functions as the precipitation-hardening component. More specifically, these alloys will contain from 1.3% to 3.5% by weight of titanium. The alloys may, and usually will, also contain molybdenum and/or tungsten, and aluminum which exhibit some precipitation-hardening characteristics, though not to as marked a degree as that of the titanium.

Broadly, the austenitic alloys to which the present invention is particularly adapted are composed of from 10% to 35% by weight of nickel, from 7% to 23% by

weight of chromium, a total of up to 6% by weight of at least one metal from the group consisting of tungsten and molybdenum, up to 2.5% by weight of manganese, between 0.3% and 1.5% by weight of silicon, up to 0.5% by weight of aluminum, up to 0.5% by weight of vanadium, up to 0.5% by weight of boron, from 1.3% to 3.5% by weight of soluble titanium, less than 0.15% of carbon, and the balance being iron except for incidental impurities such as phosphorous, sulfur, nitrogen, oxygen and the like. A desirable range of alloy compositions for processing in accordance with the invention comprises from 22% to 28% nickel, from 12% to 18% chromium, a total of from 1% to 3% of at least one metal from the group consisting of tungsten and molybdenum, from 0.5% to 1.5% manganese, from 0.1% to 0.35% vanadium, less than 0.10% carbon, from 0.8% to 1% silicon, up to 0.5% aluminum, from 1.5% to 2.8% soluble titanium, from 0.03% to 0.15% boron, and the balance being iron except for incidental impurities. Outstanding properties have been obtained in heat treating alloys containing from 22% to 28% by weight of nickel, from 12% to 18% by weight of chromium, from 1% to 3.5% by weight of molybdenum, from 0.03% to 0.15% by weight of boron, from 0.1% to 0.50% by weight of vanadium, less than 0.08% carbon, from 1% to 1.5% by weight of manganese, from 0.8% to 1% of silicon and up to 0.4% by weight of aluminum, the balance being iron except for incidental impurities. If the alloys are vacuum melted lesser amounts of manganese, silicon and aluminum may be employed to accomplish the usual scavenging functions thereof. "Soluble titanium" may be determined, for practical purposes, by subtracting from the total titanium content an amount equal to 4 times the carbon content of the alloy.

The alloy may be prepared by induction melting either in the open air or under a protective atmosphere, or under vacuum, or by arc melting in an evacuated vessel or with a protective inert gas such as helium or argon. Ingots or castings may be prepared from the alloy. The ingots or castings may be subjected to forging, hot rolling, contour die forging or other appropriate hot working procedures. The resulting wrought members which may be in the form of discs, bar stock, bolts, sheets and the like, must be subjected to an aging heat treatment in order to develop the physical properties to the desired extent.

In accordance with the present invention, the wrought members of the precipitation-hardenable austenitic alloy are initially solution treated at a temperature above 1700° F. In practice the solution treatment may be carried out at a temperature from 1700° F. to 2200° F. in a period of time of from ½ to 8 hours. After solution treating, the alloy member is rapidly cooled to a temperature below 1250° F. Water or oil quenching has given excellent results. The temperature of the member after the rapid cooling may be at approximately room temperature, though this is not important.

The cooled solution-treated member is then heated to a first aging temperature of from 1100° F. to 1250° F. The alloy members are maintained at this first aging temperature for a period of time of from 2 to 50 hours, and preferably from 4 to 20 hours. The temperature need not be constant in this range. During this first aging treatment it has been discovered that very fine, evenly distributed particles of the precipitation-hardening components are nucleated. There is no excessive segregation of the hardening components near the grain boundaries or other imperfections. The extent of this initial nucleation precipitation can be measured by the increase in hardness of the alloy member. The hardness will usually increase from 30 to 125 points as determined by the Diamond Pyramid Hardness Test, abbreviated as DPH or VHN (Vickers Hardness Number). Ordinarily, the first aging treatment has given excellent results when the hardness has increased from 50 to 100 DPH.

After the nucleation of the precipitation-hardening components has taken place to the desired extent, the alloy member is then slowly increased in temperature to a second aging temperature of from 1300 to 1450° F. In order to prevent redissolution of some of the fine hardening precipitate, the temperature should be raised relatively uniformly and for a prolonged period of time of from 2 to 32 hours to the desired second aging temperature.

Once the desired second aging temperature has been reached, the member is held or maintained at this second aging temperature until it has attained the desired optimum hardness. The rate of increase of hardness of the alloy is much more rapid than during the first aging temperature. The hardness should increase at least 25 DPH points. The ordinarily desired optimum hardness of the members after the second aging heat treatment will be at least 225 DPH for an alloy containing 1.3% soluble titanium and exhibit progressively higher hardness with increasing soluble titanium content to 330 to 360 DPH for an alloy containing 2.4% soluble titanium.

After the alloy member has been subjected to the second aging temperature, it may then be cooled to room temperature or otherwise put into condition for use.

The aging treatment of this invention is designated as "genuage" heat treatment.

In some cases it may be desirable to secure a further increase of hardness for the given alloy. To accomplish this it has been discovered that by reducing the temperature of the alloy member from the second aging temperature, that is from 1300° F. to 1450° F., to a temperature of about 1200° F. and holding the member for a period of time of at least 2 hours at this third temperature, a marked increase in hardness will occur.

Heats of the several alloys shown in the attached Table I were prepared by melting the components in an induction furnace in air.

TABLE I

Chemical composition of alloys

Heat No.	Ti	Mo	Ni	Cr	Mn	Si	C	Al	V	Fe
6929	1.62	3.32	24.8	14.2	1.38	.76	.036	.11	.068	Balance.
6931	2.04	3.33	24.8	14.1	1.38	.82	.032	.12	.078	Do.
6932	1.72	2.28	24.9	14.1	1.41	.87	.032	.11	.078	Do.
6966	2.41	3.22	25.2	14.4	1.28	.74	.024	.10	------	Do.
6968	2.63	1.23	25.5	14.2	1.28	.74	.02	.19	------	Do.
6969	1.48	3.40	25.2	14.5	1.17	.88	.038	.06	------	Do.
7040	1.69	3.03	26.4	13.7	1.60	.71	.04	.15	.08	Do.

Ingots were prepared from each of the alloys of Table I. The ingots were soaked for four hours at 2100° F., and then subjected to 8 series of hot rolling stages with intermediate reheating, to produce ⅝-inch round bar. Specimens were machined from the round bar and subjected to a variety of heat treatments. Table II indicates the variety of heat treatments of this invention applied to the alloy of heat No. 7040, and the resulting hardness of each specimen.

TABLE II

Aging cycle tests of heat No. 7040. Solution treated 1 hour at 1950° F.

[Hardness in DPH; time in hours.]

Specimen Number	Hardness as Solution Treated	Aging Time at 1,200° F.	Hardness After 1,200° F.	Heating Time from 1,200° to 1,300° F.	Hardness After Reaching 1,300° F.	Aging Time at 1,300° F.					
						2	4	8	12	16	20
A-2	129	4	197	2	195	------	219	230	------	------	274
A-4	128	4	192	4	202	------	------	------	------	------	277
A-8	130	4	192	8	209	------	238	242	------	------	278
A-16	128	4	192	16	216	------	242	243	------	------	286
B-2	130	8	198	2	191	------	------	------	------	------	278
B-4	128	8	198	4	196	------	------	------	------	------	276
B-8	130	8	200	8	206	------	------	250	------	------	276
B-16	130	8	196	16	217	------	------	247	------	------	274
C-2	129	16	211	2	196	------	------	------	------	------	270
C-4	130	16	210	4	203	------	------	------	------	------	276
C-8	128	16	209	8	208	------	240	245	------	------	284
C-16	130	16	209	16	229	224	241	------	272	------	------
D-2	127	32	221	2	206	------	------	------	------	------	272
D-4	128	32	218	4	211	------	------	------	------	------	282
D-8	130	32	223	8	216	------	------	------	------	------	273
D-16	131	32	220	16	229	------	------	------	274	------	------
E-2	129	64	236	2	222	------	------	------	------	------	------
E-4	132	64	234	4	224	------	------	------	------	------	------
E-8	130	64	234	8	226	------	------	------	------	------	------
E-16	127	64	239	16	248	------	------	268	------	------	------

Referring to Fig. 1 of the drawings, the curves there shown graphically illustrate the data of Table II, in terms of the change in hardness of the alloy of heat No. 7040 which contains 1.69% total titanium as the major precipitation-hardening ingredient and 3.0% molybdenum which cooperates therewith. The curves on Fig. 1 show the change in hardness as a function of time in hours for the wrought members (*a*) during the first aging treatment, (*b*) during the period of time when the temperature is being changed from 1200 to 1300° F., and (*c*) finally while aging at 1300° F. Thus, it will be noted that the initial hardness of the alloy is approximately 130 D.P.H. before any aging treatment. After approximately 4 hours at 1200° F., the hardness has increased to about 195 D.P.H.—an increase of approximately 65 points D.P.H. A slow increase in hardness occurs with further aging after 4 hours at 1200° F. so that even after 64 hours aging time it has only reached 235 D.P.H. It will be noted that as the period of time consumed in raising the temperature of the alloy from 1200 to 1300° F. varies from 2 to 16 hours, there is an initial slight drop in hardness, which is believed to be due to some of the hardening precipitate redissolving, soon, however, an increase in hardness is observed. A rather high rate of increase in hardness occurs while the alloy is being held at 1300° F.

It will be noted further that when the first aging heat treatment at 1200° F. exceeds 8 hours, there is a definite drop in hardness if the temperature is increased from 1200 to 1300° F. in from 1 to 8 hours. However, there is no decrease in hardness if a period of time of the order of 8 to 16 hours is used to increase the temperature from 1200 to 1300° F.

Prlonged aging times employing the three stages of heat treatment are not required to attain optimum hardness. Thus, for a total aging time of approximately 26 hours, the hardness of the alloy is approximately 275 D.P.H., while for a total aging time of 40 hours, the maximum hardness is approximately 285 D.P.H. and for 84 hours it attains only 265 D.P.H.

Since the ultimate hardness of the alloy, regardless of the length of the total heat treatment time, is between about 265 and 285 D.P.H., here is no need to employ any heat treatment schedule that exceeds 40 hours. Figure 1 of the drawing therefore emphasizes the fact that the process of the present invention produces outstanding hardness for a relatively short total aging time of between 20 and 40 hours.

Creep-rupture tests of the heat treated alloy No. 7040 are set forth in the following Table III.

TABLE III

Creep tests of heat No. 7040 at 1200° F. All specimens solution treated 1 hour at 1950° F.

PLAIN-BAR SPECIMENS—AT 60,000 P.S.I.

Specimen Number	Aging Treatment	Hardness as Aged DPH	Rupture		Minimum Creep Rate, Percent Hour
			Time, Hours	Elongation, Percent	
28	D	289	69	10.0	0.40
35	A	280	169	9.8	0.014
					0.04
39	B	283	181	10.3	0.017
43	C	272	141	15.3	0.02

NOTCH-BAR SPECIMENS—AT 70,000 P.S.I.

Specimen Number	Aging Treatment	Hardness as Aged DPH	Time, Hours
27	D	282	127
36	A	282	668
40	B	278	602
44	C	268	736

The aging treatment applied to the specimens is indicated in the attached Table IV. It will be observed that aging treatments A, B and C are those of the present invention, while treatment D is a standard prior art heat treatment in wide use. Aging treatments A, B and C resulted in two to three times the rupture time for plain-bars as obtained with treatment D. In notch-bars, aging treatments A, B and C result in 5 times or more rupture time as from treatment D, while the elongation is as good or better in A, B and C as compared to aging treatment D.

TABLE IV

Aging Treatment	
A	4 hours at 1,200° F., temperature increased to 1,265° F. in 1⅓ hours, held at 1,265° F. for 2 hours, raised to 1,300° F. in 10 minutes, held at 1,300° F. for 20 hours and air cooled.
B	8 hours at 1,200° F., temperature increased to 1,300° F. in 8 hours, held at 1,300° F. for 20 hours and air cooled.
C	16 hours at 1,200° F., heated to 1,300° F. in 16 hours, held at 1,300° F. for 12 hours and air cooled.
D	20 hours at 1,300° F. cooled to 1,200° F. in 4 hours, held at 1,200° F. for 20 hours and air cooled.

The effect of the aging treatment of the present invention as compared to other widely used aging treatments for various specimens of some of the alloys of Table I is set forth in the attached Table V.

of the present invention. Further, it will be noted that alloys may be processed in accordance with the present invention with much higher titanium contents then would be considered usable when aged by prior procedures.

TABLE V

The effect of various aging treatments on the creep properties of some austenitic alloys at 1200° F. and 60,000 p.s.i.

Specimen Number	Aging Treatment	Hardness		Transition		Time to Given Strain in Hours				Rupture		Red. in Area, Percent	Minimum Creep Rate, Percent/Hour
		As Aged DPH	After Testing DPH	Time, Hours	Elongation Percent	0.3%	0.5%	1.0%	2.0%	Time, Hours	Elongation, Percent		
6929-5	1	259	282	4.7	1.33		0.7	3.0		44.2	20.8	24.3	
6929-11	5	193	265	9	9.36			0.2	0.7	15	20.8	22.2	0.80
6929-27	4	276	298	51	1.1	6.0	20	48	123	168	15.6	21.6	0.016
6931-7	1	334	335	58.5	2.45	1.0	7.0	19.0	46.0	97.2	6.1	9.0	0.04
6931-25	5	272	329	12.5	0.94	4	7	13	21.5	62	13.6	18.4	0.065
6931-27	4	299	321	26	0.4	19	31	55	86	173	8.9	11.2	0.0085
6966-3	1*	343	329			25	40			65	0.83	2.7	0.012
6966-15	5	307	343	144	1.15	18	49	126	176	176	2.03	3.0	0.0065
6966-17	4	320	344	610	0.92	150	305	643		692	1.51	2.7	0.0013
6968-15	5	315	353	90	0.4	6	132			184	0.78	4.4	
6968-17	4	325	366	800	0.42	560	880	1,074		1,074	0.99	3.2	0.00033
6969-3	1*	254	263	16.5	5.41	0.5	0.8	1.0	5.0	24	8.9	13.6	0.29
6969-17	4*	270	301	20	2.8	0.0	0.0	0.1	12	69	13.1	20.2	0.09

Heat treatment **1** is a standard heat treatment widely used at the present time. It comprises aging for 20 hours at 1350° F., cooling to 1200° F. in 4 hours and holding at 1200° F. for 20 hours followed by air cooling. Heat treatment **1*** is the same as heat treatment **1** except the time to cool from 1350° F. was carried out in 3 hours. Heat treatment **5**, another widely used commercial heat treatment for these alloys, comprises aging the alloy for 16 hours at 1325° F. Heat treatment **4** constitutes a heat treatment in accordance with the present invention wherein the alloy is heated for 16 hours at 1200° F., heated to 1300° F. over a period of 16 hours, then held at 1300° F. for 12 hours and air cooled. Heat treatment **4*** comprises heating 16 hours at 1200° F., then increasing the temperature over a period of 8 hours to 1250° F. and holding at 1250° F. for 16 hours and air cooling.

With respect to alloys having low soluble titanium content of below 1.4%, it is preferred to modify slightly the second aging temperature to a range of from 1250° F. to 1350° F. This corresponds to heat treatment **4***. The first aging temperature range for such low titanium alloy should be preferably from 1100° F. to 1200° F. Best aged alloys having soluble titanium contents of 1.3% to 1.4% have been obtained with such modified aging treatment.

It will be observed from Table II that the first four alloys exhibited much superior creep-rupture times when heat treated in accordance with the present invention than when the same alloy was heat treated by other known processes. The ductility as measured by the elongation was favorably comparable to that of the members with inferior rupture times.

Notch-bar creep-rupture tests established that the alloys aged by the process of the present invention were less notch sensitive than the same alloys processed by other commercially used aging treatments. The loads in many cases had to be increased to 70,000 p.s.i. to secure rupture in reasonable periods of time.

Referring to Fig. 2 of the drawings, there are plotted curves based on data obtained from a considerable number of notch-bar creep-rupture tests of similar alloys with varying total titanium contact. It will be noted that the notch-bar creep-rupture times using the single and double aging heat treatments of the prior art are considerably inferior to the creep-rupture times, for similar alloys with the same titanium content, obtained by the aging process Thus, for alloys having a 2.3% titanium content, the notch-bar creep-rupture time is of the order of 2 hours when aged by either the single or double aging treatments of the prior art. Using the aging treatment of the present invention, a 2.3% titanium alloy exhibits a creep-rupture time of approximately 280 hours. The curves on Fig. 2 therefore emphasize the outstanding improvement in the alloy properties obtainable by the practice of the present invention.

TABLE VI

The effect of various aging treatments on the notch-bar creep-rupture properties of some austenitic type alloys at 1200° F.

Specimen Number	Aging Treatment	Hardness		Load, p.s.i.	Rupture Time, Hours
		As Aged DPH	After Test DPH		
6929-6	1	297	299	70,000	180
6929-9	5	186	282	70,000	36
6929-28	5	179	287	60,000	300
6929-30	4	277	295	70,000	403
6931-3	1	328	326	70,000	54.5
6931-9	5	311	333	70,000	38
6931-28	5	298	329	60,000	261
6931-30	4	295	331	70,000	508
6966-4	1*	344	331	70,000	1.5
6966-6	1*	333	349	60,000	23
6966-18	5	304	334	60,000	35.5
6966-20	4	336	355	70,000	74
6968-18	5	320	347	60,000	153
6968-20	4	336	355	70,000	123
6969-4	1*	253	269	70,000	33
6969-6	1*	246	268	65,000	199
6969-18	5	144	278	60,000	914
6968-20	4*	248	294	70,000	521

A summary of the various physical properties obtainable by the practice of the present invention on various precipitation-hardenable alloys as compared to those obtainable by the best prior art practices, for varying titanium content is presented the curves of Fig. 3. Aging treatment **2** of Fig. 3 comprises 20 hours at 1300° F., cool to 1200° F. in 4 hours, hold at 1200° F. for 20 hours and air cool; it has been widely used. Aging treatment **4** is that of the present invention and is that set forth above in Table V. The aging treatments of Fig. 3 were applied to alloys corresponding to those in Table I.

It will be observed that aging treatment 4 results in members whose creep-rupture times at stresses of 70,000 p.s.i. exceeds by a substantial amount that of alloy members aged by treatment 2 but loaded to only 60,000 p.s.i. It will be apparent that even at these higher strengths, the ductility of the alloy members is superior in general when aging treatment 4 of this invention is employed as contrasted to the prior art heat treatment.

The following alloy was melted and cast into an ingot: nickel 26.1%; chromium 13.1%; molybdenum 3.13%; carbon 0.018%; silicon 1.01%; manganese 1.33%; titanium 2.4%; boron 0.12%; and 52.4% iron, balance incidental impurities. The ingot was forged at 2000° F. to bars which were hot rolled to ⅝ diameter stock. The bars were solution treated one hour at 1950° F., and quenched in oil. The grain size of the solution treated alloy was 4 to 5 A.S.T.M. Part of the bars were aged by heating 20 hours at 1350° F., cool to 1200° F. in 5 hours, hold 20 hours at 1200° F., and air cool—this is indicated as treatment X. Other bars were aged by heating at 1200° F. for 16 hours, slowly increasing the temperature to 1300° F. over 16 hours, holding 12 hours at 1300° F. and finally air cooling. This latter is aging treatment Y. Creep-rupture tests at 1200° F. at 70,000 p.s.i. load resulted in the following results:

Aging Treatment	Hours to Rupture	Elongation, Percent	Notched Bar, Hours to Rupture
X	25	18.1	56
Y	95	8.5	156

It will be understood that the above description and drawing are illustrative and not in limitation of the invention.

I claim as my invention:

1. In the process of producing a precipitation hardened member from an iron base austenitic alloy containing from 1.3% to 3.5% by weight of soluble titanium as a precipitation hardening component, the steps comprising (1) solution treating the alloy member at a temperature above 1700° F., (2) rapidly cooling the alloy member to a temperature below 1250° F., (3) reheating the cooled member to a first aging temperature of from 1100° F. to 1250° F. and preferably between 1100° F. and 1200° F. for alloys having less than 1.4% of soluble titanium, and maintaining the member in this temperature range for a period of time of from 2 to 50 hours sufficient to cause nucleation of the precipitation hardening components whereby the hardness of the alloy increases from 30 to 125 points by the diamond pyramid hardness test, (4) then heating the alloy member over a period of from 2 to 32 hours to increase its temperature slowly to a temperature of from about 1300° F. to 1450° F., and preferably from 1250° F. to 1350° F. for alloys having less than 1.4% of soluble titanium, and maintaining the alloy member in this range until it increases in hardness at least 25 points more than the hardness after the first aging temperature.

2. The process of claim 1 wherein after the heating at from 1300° F. to 1450° F., the temperature of the member is reduced to about 1200° F. and maintained at this temperature for a period to produce a further increase in hardness.

3. In the process of producing a precipitation hardened member from an austenitic alloy composed of from 10% to 35% nickel, from 7% to 23% of chromium, a total of up to 6% of at least one metal from the group consisting of tungsten and molybdenum, up to 2.5% manganese, between 0.3% and 1.5% silicon, up to 0.5% aluminum, up to 0.5% vanadium, less than 0.15% carbon, up to 0.5% boron, from 1.3% to 3.5% soluble titanium, the balance being iron except for incidental impurities, the steps comprising (1) solution treating the alloy member at a temperature above 1700° F., (2) rapidly cooling the alloy member to a temperature below 1250° F., (3) bringing the member to a first aging temperature of from 1100° F. to 1250° F. and maintaining the alloy member at this temperature for a period of from 2 to 50 hours to cause nucleation of the precipitation hardening components whereby the hardness of the alloy increases from 30 to 125 points by the DPH test, and (4) thereafter heating the alloy member over a period from 2 to 32 hours to increase its temperature slowly to a second aging temperature of from 1300° F. to 1450° F. and maintaining the member at this second temperature to cause the hardness to increase at a more rapid rate than when at the first aging temperature to a value proportional to at least from 225 DPH for a 1.3% soluble titanium alloy to 330 DPH for a 2.4% soluble titanium alloy.

4. In the process of producing a precipitation hardened member from an austenitic alloy composed of from 10% to 35% nickel, from 7% to 23% of chromium, a total of up to 6% of at least one metal from the group consisting of tungsten and molybdenum, up to 2.5% manganese, between 0.3% to 1.5% silicon, up to 0.5% aluminum, up to 0.5% vanadium, less than 0.15% carbon, up to 0.5% boron, from 1.3% to 3.5% soluble titanium, the balance being iron except for incidental impurities, the steps comprising (1) solution treating the alloy member at a temperature above 1700° F., (2) rapidly cooling the alloy member to a temperature below 1250° F., (3) bringing the member to a first aging temperature of from 1100° F. to 1250° F. and maintaining the alloy member at this temperature for a period of from 2 to 50 hours to cause nucleation of the precipitation hardening components whereby the hardness of the alloy increases from 30 to 125 points by the DPH test, (4) thereafter heating the alloy member over a period of from 2 to 32 hours to increase its temperature slowly to a second aging temperature of from 1300° F. to 1450° F. and maintaining the member at this second temperature to cause the hardness to increase at a more rapid rate than when at the first aging temperature to a value proportional to at least from 225 DPH for a 1.3% soluble titanium alloy to 330 DPH for a 2.4% soluble titanium alloy, and (5) thereafter reducing the temperature of the alloy member to about 1200° F. for a period of time of at least 2 hours to produce a further increase in hardness of the alloy.

5. In the process of producing a precipitation hardened member from an austenitic alloy composed of from 10% to 35% nickel, from 7% to 23% of chromium, a total of up to 6% of at least one metal from the group consisting of tungsten and molybdenum, up to 2.5% manganese, between 0.3% and 1.5% silicon, up to 0.5% aluminum, up to 0.5% vanadium, less than 0.15% carbon, up to 0.5% boron, from 1.3% to 3.5% soluble titanium, the balance being iron except for incidental impurities, the steps comprising (1) solution treating the alloy member at a temperature above 1700° F., (2) rapidly cooling the alloy member to a temperature below 1250° F., (3) bringing the member to a first aging temperature of from 1100° F. to 1250° F. and maintaining the alloy member at this temperature for a period of from 2 to 50 hours to cause nucleation of the precipitation hardening components whereby the hardness of the alloy increases from 30 to 125 points by the DPH test, and (4) thereafter heating the alloy member over a period of from 2 to 32 hours to a second aging temperature of from 1300° F. to 1450° F. and maintaining the member at this second temperature to cause the hardness to increase at a more rapid rate than when at the first aging temperature to increase its temperature slowly to a value proportional to at least from 225

DPH for a 1.3% soluble titanium alloy to 330 DPH for a 2.4% soluble titanium alloy.

6. In the process of producing a precipitation hardened member from an austenitic alloy composed of from 22% to 28% of nickel, from 12% to 18% chromium, a total of from 1% to 3% of at least one metal selected from the group consisting of tungsten and molybdenum, from 0.5% to 1.5% manganese, from 0.1% to 0.35% vanadium, less than 0.10% carbon, from 0.8% to 1% silicon, up to 0.5% aluminum, from 1.5% to 2.8% soluble titanium, from 0.03% to 0.15% boron, and the balance being iron except for incidental impurities, the steps comprising (1) solution treating the alloy member above 1800° F. for at least half an hour, (2) quenching the alloy member to a temperature below 1250° F., (3) reheating the quenched alloy member to a first aging temperature of from 1100° F. to 1250° F. and maintaining the member at this first aging temperature for from 2 to 32 hours to cause nucleation of the precipitation hardening components whereby the hardness of the alloy increases from 50 to 100 D.P.H. points, (4) slowly increasing the temperature of the alloy member over a period of from 2 to 32 hours until the member is at a second aging temperature in the range of from 1300° F. to 1450° F., and maintaining the alloy member at this second aging temperature to cause the hardness to increase at a more rapid rate than during the first aging temperature to a value proportional to a value of at least 225 D.P.H. for an alloy having 1.3% soluble titanium and 330 D.P.H. for an alloy having 2.4% soluble titanium.

7. In the process of heat treating a wrought member to an optimum precipitation hardened condition in a total time of from 20 to 50 hours, the wrought member comprising an austenitic alloy composed of from 10% to 35% nickel, from 7% to 23% of chromium, a total of up to 6% of at least one metal from the group consisting of tungsten and molybdenum, up to 2.5% manganese, between 0.3% and 1.5% silicon, up to 0.5% aluminum, up to 0.5% vanadium, less than 0.15% carbon, up to 0.5% boron, from 1.3% to 3.5% soluble titanium, the balance being iron except for incidental impurities, the steps comprising (1) solution treating the wrought member from ½ to 8 hours at a temperature of from 1700° F. to 2200° F., (2) quenching the wrought member to a temperature below 1250° F., (3) reheating the wrought member to a first aging temperature of from 1100° F. to 1250° F. and maintaining the member in this temperature range for a period of time of from 2 to 20 hours to cause nucleation of the precipitation hardening components, (4) slowly increasing the temperature of the wrought member to a second temperature from 1300° F. to 1450° F. over a period of from 2 to 16 hours, and (5) maintaining the wrought alloy members in this second temperature for a period of from 2 to 20 hours, the total aging period of steps (3) to (5) being from 10 to 50 hours, to provide a highly hardened member.

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