Aug. 22, 1950 H. SCOTT ET AL 2,519,406

WROUGHT ALLOYS

Filed July 30, 1948 2 Sheets-Sheet 1

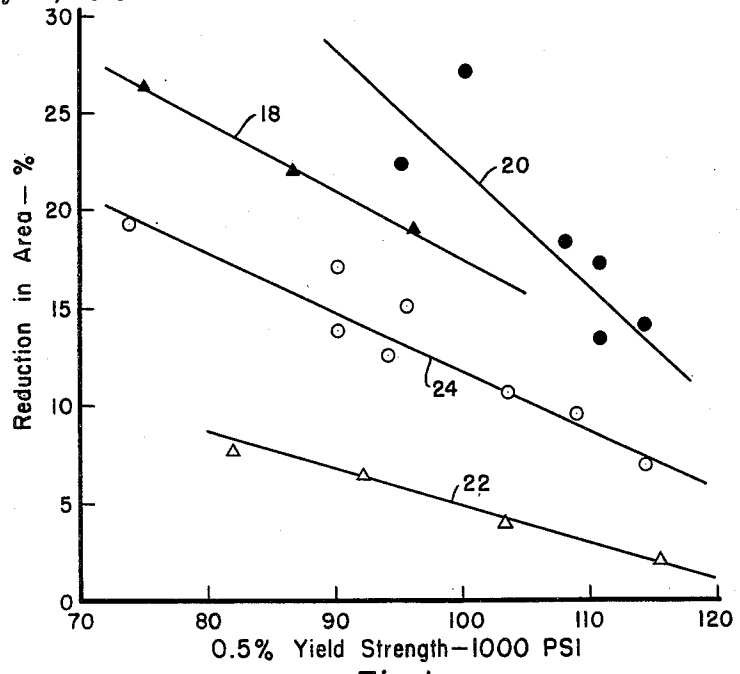


Fig. 1.

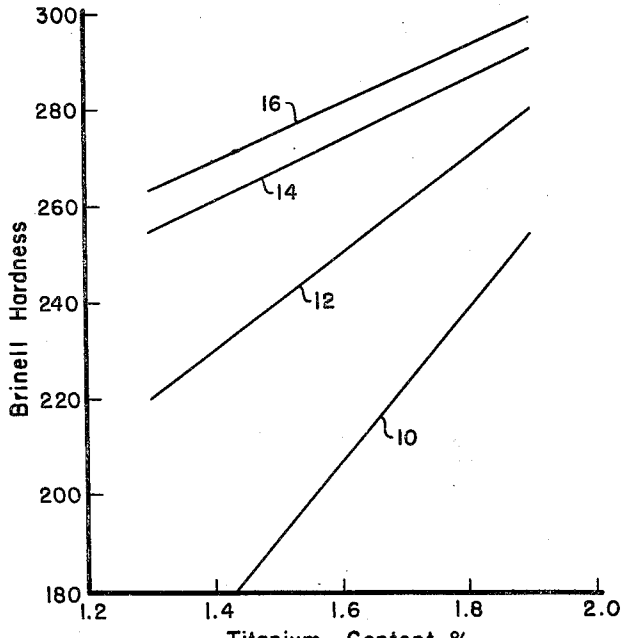


Fig. 2.

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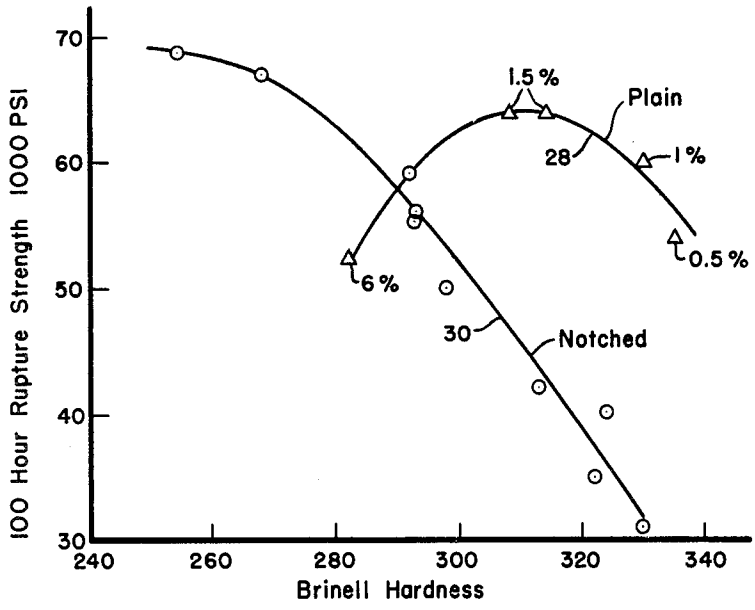


Fig.3.

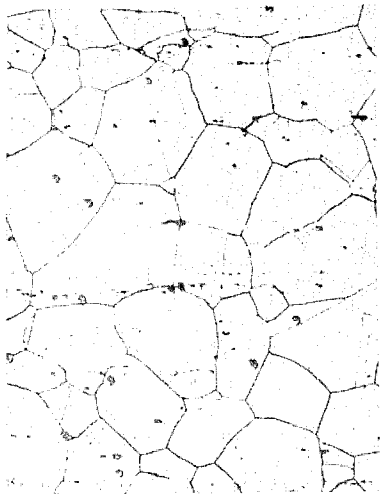


Fig.4a.

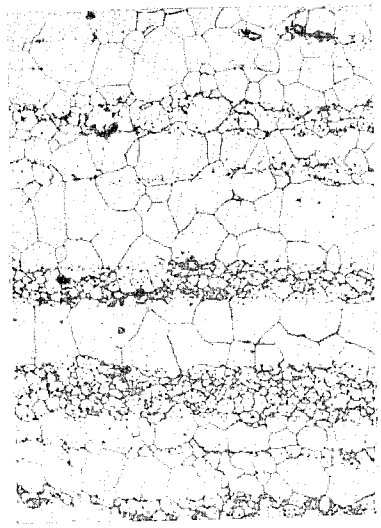


Fig.4b.

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Patented Aug. 22, 1950 2,519,406

UNITED STATES PATENT OFFICE

2,519,406

WROUGHT ALLOY

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Application July 30, 1948, Serial No. 41,467

5 Claims. (Cl. 148—31)

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This invention relates to alloys and in particular to austenitic alloys having high strength and ductility at elevated temperatures.

Many alloys have recently been developed for use in gas turbines and jet engines. Such devices include bolts, replaceable rotating blades or buckets keyed into a rotor periphery and the rotor discs. Because of interlocking projections between the blades and the rotor for resisting the high centrifugal forces encountered, it is found that large bending stresses are imposed on the rotor material under conditions of stress concentration. Also because of bolt holes or other discontinuities, stress concentrations may exist near the axis of the rotor where normal stress is at a maximum. In the known alloys, brittle fractures of the rotor have occurred even though the alloys apparently had adequate ductility under tensile tests. Investigations of such failures indicate that considerably higher ductility as measured by tensile test is required in rotors than in other components subjected only to simple tensile loading.

An object of this invention is the provision of an austenitic alloy having high strength as well as high ductility and resistance to scaling and oxidation at elevated temperatures around 1200° F. combined with superior forgeability and machinability characteristics.

Another object of this invention is the provision of a wrought austenitic precipitation hardened alloy that has a high resistance to yielding and creep at elevated temperatures of 1200° F. while maintaining good ductility.

A further object of this invention is the provision of a wrought austenitic precipitation hardened alloy that has a high resistance to yielding at elevated temperatures of 1200° F. while maintaining good ductility and which will retain such characteristics during long exposures at such elevated temperatures.

Another object of this invention is the provision of articles for use at elevated temperatures of up to 1300° F. formed of a wrought austenitic precipitation hardened alloy that has been completely recrystallized to a uniform fine grain size.

A particular object of this invention is to provide a high grade alloy for the rotors of jet engines which requires a minimum of scarce or strategic alloying elements.

A more specific object of this invention is the provision of a wrought austenitic precipitation hardened alloy of nickel, chromium, molybdenum with or without tungsten, titanium and the balance iron with incidental impurities

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and deoxidizers, the alloy being completely recrystallized and having a grain size finer than No. 4 ASTM.

Other objects of this invention will become apparent from the following description when taken in conjunction with the accompanying drawing in which,

Figure 1 is a graph, the curves of which illustrate the effect of molybdenum content and solution treatment on the yield strength and ductility as measured in reduction in area of the alloy at service temperatures of 1200° F.

Figure 2 is a graph the curves of which illustrate the effect of ageing temperatures and stabilization treatment on the hardness of the alloy.

Figure 3 is a graph the curves of which illustrate 100 hour rupture strength vs. hardness for plain specimens and for notched specimens of the alloy of this invention.

Figures 4*a* and 4*b* are photomicrographs at a magnification of 200 times of alloys showing an effect of aluminum in the alloys.

The alloy of this invention is a wrought austenitic heat resistant alloy precipitation hardened by titanium present in a preferred amount. Fundamentally, the alloy comprises nickel, chromium, molybdenum and/or tungsten, titanium, less than .1% carbon and the balance iron with incidental cobalt not exceeding 2% and controlled amounts of incidental impurities, deoxidizers and scavengers. These elements are present in the following proportions; 15% to 35% nickel, 7% to 20% chromium, metal selected from the group consisting of molybednum and/or tungsten in an amount to equal 2% to 5% by weight, 1.3% to 1.9% titanium, and the balance iron with not more than 0.4% aluminum, .3% to 3% of deoxidizers and scavengers such as manganese and silicon, and not over .5% of incidental impurities such as phosphorus, copper, sulphur, nitrogen and the like. The compositions given are by weight.

Preferably the alloy comprises about 25% nickel, about 13% chromium, about 3% molybdenum, from 1.3% to 1.9% titanium, less than 0.3% aluminum, less than 0.08% carbon, about 0.7% manganese, less than 1% silicon, less than 0.05% copper and the balance iron with traces of incidental impurities. Nickel in amount equal about 25% is found to avoid any possibility of phase changes during the fabrication or heat treatment of the alloy and in use. The chromium is employed within the range given to confer resistance to oxidation, 13% chromium being quite effective for such purpose at the elevated temperatures as referred to hereinafter.

The molybdenum and/or tungsten content is employed as a substitute for iron in the alloy of this invention it being found that the strength-ductility relation of the alloy at elevated temperatures of 1200° F. is greatly improved by such substitution as shown by the curves of Figure 1 and referred to hereinafter in greater detail. Molybdenum in amounts lower than 2% does not offer any significant advantage whereas contents higher than 5% detract from forgeability and machinability of the alloy. The tungsten can be substituted in equal amounts for any or all of the molybdenum content, it being found that tungsten has the same effect on the resulting alloy as molybdenum. The presence of manganese and silicon within the range given hereinbefore does not appear to be critical with respect to the mechanical properties of the alloy of this invention in the absence of a phase change.

Titanium is found to be quite critical in the alloy, relatively small amounts in the range of 1.3% to 1.9% effecting useful precipitation hardening of the alloy as referred to hereinafter. This hardening agent does not introduce a low melting phase but instead permits the heating of the alloy at temperatures up to at least 2200° F. without internal damage to the micro-structure of the alloy. The ageing characteristics imparted to the alloy by the presence of titanium in such a range are substantially independent of variations in the other alloying constitutents within the prescribed limits.

In adding titanium to the alloy, it is preferred to add it in the form of ferroalloys as such materials are relatively inexpensive and are non-strategic. Ferro-titanium containing between 27% and 32% titanium is preferred as such ferro-alloys are readily dissolved. In making the alloy it is preferred to employ a ferro-titanium alloy having a specification of 30% titanium and less than 2.4% aluminum, with the balance substantially iron and the usual impurities in order to maintain the aluminum content of such resulting alloy below 0.4%. The reason for such limitation on the aluminum content will be explained more fully hereinafter.

In making the alloy, special precautions are necessary in melting and casting to obtain the desired results. In practice, carefully selected raw materials are melted in a high frequency induction furnace of suitable capacity having a magnesia or mixed magnesia and alumina crucible or lining. Silica is avoided in both lining and slag since it is readily reduced by titanium in the melt. The iron, nickel and molybdenum contents are first melted and deoxidized with a calcium-silicon alloy after which an inert slag of suitable melting point is added. Ferroalloys such as ferro-chromium and ferro-titanium as well as manganese and other deoxidizers are then added in predetermined amounts through a protective layer of slag to reduce reaction between the surface of the molten metal and oxygen as well as nitrogen in the air, stirring being employed to promote rapid solution of the titanium.

In order to eliminate inclusions of non-metallic matter, the heat is preferably poured into a ladle and held for a suitable time before casting to permit levitation of non-metallic inclusions. During such holding the surface of the metal in the ladle may be insulated to reduce heat loss therefrom. Thereafter the metal is cast from the ladle by bottom pouring into ingots in a chill mold. Other practices have been used however with comparable and satisfactory results.

In processing the alloy thus formed into useful articles of manufacture such as rotors for gas turbines, bolts or other articles for use at elevated temperatures in the range of 1100° F. to 1300° F. under load conditions of stress concentrations, it is preferred to so process the alloy as to recrystallize it completely to a uniform grain size as fine as No. 2 ASTM. Ingots or billets of the alloy may be processed by an alternate working and reheating cycle at elevated starting temperatures at which successive recrystallizations effect a substantial refinement of the grain size of the original coarse-grained ingot or billet. This is followed by a controlled working to final shape at a lower elevated temperature which decreases resistance to plastic flow as compared to working at room temperature but avoids recrystallization during working. The worked alloy is thereafter subjected to a solution treatment to remove the work hardening and to recrystallize the alloy completely to a uniform fine grain size followed by an ageing treatment. This process is fully described and claimed in the copending application Serial No. 41,466 of Scott et al. filed concurrently herewith.

Where it is desired to produce rotor discs of the alloy, it is found that the normal working as by upsetting a cylinder into a disc under an open die forging hammer or press is unsatisfactory since there is inadequate working at and near the centers of the faces and midway between faces on the circumference. Instead it has been necessary to develop a new method of processing discs of the alloy described herein in order to effect complete recrystallization of the alloy at a solution temperature low enough to develop the maximum properties thereof.

In processing rotor discs, a forged cylinder of the alloy is heated to a temperature which will not produce recrystallization and preferably between 1550° F. and 1700° F. The heated cylinder is placed on the anvil of a forging hammer or press and the top surface is worked by a spreader bar placed between the disc and the hammer or ram whereby the load applied is concentrated, friction is minimized and a deep penetration of work hardening is obtained. The other face of the disc is worked in a like manner so as to equalize the working of both faces. In working the alloy in this manner the spreader bar is either moved progressively across the face of the cylinder or disc or it is held in a fixed position over the axis of the disc and the disc is rotated slightly under the bar after each blow of the hammer. As many reheatings at 1550° F. to 1700° F. are applied to the disc as is necessary to complete the working of the disc to size and to introduce work hardening in an amount equivalent to a reduction in height of at least 20% and preferably about 40%.

After the discs are thus produced they are subjected to a solution treatment to remove the work hardening and to recrystallize completely the alloy to a uniform fine grain size and to cause the precipitation hardening constituent to go into solution in the matrix. Preferably the solution treatment is at a temperature between 1800° F. and 2000° F. for a period of time between ½ and 2 hours. The articles thus produced and treated are then subjected to an ageing treatment at a temperature between 1300° F. and 1450° F. for a period of time producing maximum hardness, usually 20 hours.

Sound discs of the alloy of this invention hav-

ing a uniform recrystallized grain size have been produced in sizes as large as 25 inches in diameter and 5½ inches thick. Ingots as small as 2½ inches square and as large as 12 inches square have been successfully upset and finished upset using the spreader bar technique into discs which when precipitation hardened have outstanding physical characteristics at elevated temperature of 1200° F. hardly distinguishable from those of heat treated specimens prepared from rolled bar stock of the same alloy.

While excellent results have been achieved with all sizes of rotors forged, it has been found more difficult to control work hardening in the larger sizes or more complex shapes than in a small disc. In small discs we have obtained a grain size of No. 5 to No. 8 whereas in the large discs and more complex shapes a grain size of No. 4 to No. 6 ASTM is obtained. The effect of the coarser grain size is to reduce ductility. Therefore in order to offset this effect and to maintain ductility at its optimum value in the larger sizes we prefer to use a lower titanium content in combination with the medium grain size. With a grain size of ASTM No. 4 to No. 6, for example, we prefer a titanium content between 1.3 and 1.7 percent and with a grain size of No. 5 to No. 8 between 1.5 and 1.9 percent.

Referring to Fig. 2 of the drawing the effect of titanium on the hardness of the alloys of this invention is illustrated for different ageing treatments. Curve 10 represents the hardness obtained with an ageing treatment of 20 hours at 1350° F. which produces approximately maximum hardness that can be obtained in 20 hours at a single fixed temperature. The hardness so obtained however is not the most stable at elevated temperatures. Therefore a further treatment at 1200° F. for 20 hours is utilized to further stabilize the alloy and the results thus obtained are shown in curve 12 of Fig. 2. With such a treatment the hardness materially increases especially so at the lower titanium contents. Further improvement in hardness can also be obtained by slow cooling the alloy from the 1350° F. ageing temperature to the 1200° F. ageing temperature as illustrated by curves 14 and 16, curve 14 being obtained with a cooling period of 1½ hours between the ageing temperatures whereas curve 16 is obtained with an 8 hour cooling period between the ageing temperatures.

The use of the dual ageing treatment plus slow cooling from the first ageing temperature to the second in addition to improving stability reduces materially the titanium content required for maximum strength, thereby reducing production difficulties and improving forgeability and machinability. Also, the slope of the stress versus time to rupture curve is reduced improving long time rupture strength significantly and titanium content is less critical. Our preferred ageing treatment to secure this result is the following continuous ageing sequence after solution treatment:

(1) Moderately rapid heating to the ageing temperature;
(2) Ageing at 1350 to 1450° F. for 4 to 24 hours;
(3) Cooling from the initial ageing temperature to a lower ageing or stabilizing temperature of 1175 to 1250° F. in 1 to 10 hours;
(4) Holding at the stabilizing temperature for 8 to 24 hours; and
(5) Cooling in the furnace or in air to room temperature.

The effect of the molybdenum content on alloys of this invention is shown in Fig. 1 in which curves 18 and 20 are representative of comparable alloys worked as described and given a solution treatment at 1800° F. and aged at 1350° F. for 20 hours, the alloys of curve 18 being devoid of molybdenum and the alloys of curve 20 containing 3% molybdenum. The alloys of curves 18 and 20 also have a uniform grain size of between No. 6 and No. 8 ASTM. Curves 22 and 24 are also based on similar alloys, treated in identical manner except that the solution treatment was applied at 1950° F., the alloys of curve 22 being devoid of molybdenum whereas the alloys of curve 24 contained 3% molybdenum. The alloys of curves 22 and 24 have a uniform grain size of between No. 4 and No. 5 ASTM. Fig. 1 clearly demonstrates that at a particular yield strength, ductility is materially increased by either the addition of molybdenum or the refinement of grain size or by both. We prefer to use both effects to obtain as high an inherent ductility as possible at 1200° F., at which temperature ductility is at or near its minimum.

The gain in tensile ductility is particularly advantageous where high strength in the presence of stress concentration is desired. In Fig. 3, 100 hour rupture strength values at 1200° F. for both plain (standard) creep test specimens and notched specimens are plotted. The notch of the specimen was .072 inch deep in a 0.50 inch diameter cylinder with the sides of the notch forming an angle of 60° and the radius of the base of the notch was .010 inch. Contrary to prior conceptions, rupture strength in plain specimens does not increase continuously with hardness which is varied chiefly by the titanium content but goes through a maximum. In the absence of stress concentration this maximum occurs at 310 Brinell when ductility measured by elongation is only 1.5 percent as shown in curve 26 of Fig. 3. With the notched specimen, however, maximum strength is obtained at a much lower hardness, 250 Brinell or less as shown by curve 30. Ductility as measured by elongation of the unnotched specimens, values of which are entered for each plotted point on curve 28, increases continuously as hardness diminishes. Undoubtedly the improvement in ductility is responsible for the increase in notched bar strength as hardness diminishes from 310 to 250 Brinell.

The foregoing observations provide a substantial basis for the selection of composition and heat treatments. Molybdenum content and treatment for minimum grain size and maximum stability have already been selected on this basis. Titanium content is chosen to provide notched bar strength at least equal to plain bar rupture strength, hardness 290 Brinell or less, but not too low for adequate resistance to creep at 1200° F. In the case of bolting stock, however, with its extremely sharp service notch, a considerable sacrifice in creep strength for gain in notched bar strength is indicated. Thus use of titanium contents at the lower end of our range with an extremely fine grain size, No. 7 to No. 9 ASTM, is preferred for bolting stock to be used at temperatures approaching 1200° F.

As stated hereinbefore, the aluminum content of the alloy is maintained below 0.4% and preferably below 0.3%. This is because it has been found that where more than 0.4% aluminum is present, the aluminum forms alumina inclusions in the alloy which render the alloy difficult to machine and dull or chip the expensive cutting

tools required in machining the alloy. It has also been found that aluminum in quantities greater than 0.4% in the alloy inhibits normal grain growth and introduces directional effects which adversely affect the mechanical properties of the alloy.

The effects of alumina inclusions resulting from the presence of substantially more than 0.4% aluminum in the alloy are apparent from the photomicrographs shown in Fig. 4*a* and Fig. 4*b*. Fig. 4*a* is a photomicrograph at a magnification of 200 times taken along a longitudinal section of a hot rolled bar of the alloy having an aluminum content of 0.26% whereas Fig. 4*b* is a similar photomicrograph of the same basic alloy but containing 0.70% aluminum, both alloys being treated identically including rolling and solution treatment 1 hour at 1950° F. and ageing 20 hours at 1350° F. The directional strata effects of the inclusions are clearly shown in the latter photomicrograph.

As representative of alloys of this invention, reference may be had to the following table of composition, it being understood that in addition to the elements listed the balance is iron with incidental impurities.

Alloy No.	Composition by Per Cent Weight								
	Ni	Cr	Mn	Si	Mo	W	Ti	Al	C
D-109	25.5	13.1	.64	.92	2.8	-----	1.83	.17	.03
D-110	25.5	13	.64	.68	3.0	-----	1.84	.15	.04
D-128	25	13	.75	.90	2.9	-----	1.64	.18	.017
D-136	25.4	13.9	.79	.79	0.66	2.4	1.54	.19	.029
D-138	25.5	13.6	.75	.76	2.88	-----	1.85	.15	.052
D-139	26.3	13.7	.74	.69	2.9	-----	1.74	.18	.021
D-141	27.6	14.1	.90	.97	3.18	-----	1.51	.15	.048
D-145	25.6	13.5	.80	.74	2.88	-----	1.83	.19	.028
D-147	26.1	13.7	.75	.81	2.69	-----	1.84	.23	.026
D-148	25.7	13.8	.74	.80	2.70	-----	1.85	.15	.030

The alloys of the foregoing table have a high hardness when precipitation hardened in the completely recrystallized condition, the hardness values being shown in the following table together with the treatment applied to the alloys.

Alloy No.	Form Treated	Solution Treatment		Ageing 1350° F.—20 Hrs.			Hardness BHN
		Temp., °F.	Time, Hr.	Cooling 1350° F. to 1200° F.	Stabilization		
					Temp., °F.	Time, Hr.	
D-109*a*	Disc	1,950	1	Air	None	----------	242
D-109*b*	do	1,800	1	do	None	----------	243
D-109*c*	do	1,900	1	In Furn. 8 hrs	1,200	16	295
D-110	do	1,950	1	Air	None	----------	236
D-110	do	1,800	1	do	None	----------	228
D-110	do	1,900	1	In Furn. 8 hrs	1,200	16	295
D-128	Rolled Bar	1,800	1	Air	1,200	20	262
D-136	Disc	1,800	2	In Furn. 1¼ hrs	1,200	20	269
D-138	do	1,800	2	do	1,200	20	289
D-139	do	1,800	2	do	1,200	20	281
D-141	do	1,800	2	do	1,200	20	269

These alloys have the following room temperature tensile properties:

Alloy No.	Strength—1000 p. s. i.		Elongation, Per Cent	Red. in Area, Per Cent
	0.2% Yield	Ultimate		
D-109c	107.5	145.0	18.4	23
D-128	104.0	140.5	23.1	28
D-138	104.5	140.0	21.3	30
D-141	98.2	136.4	24.6	41

As representative of the strength of the alloys of this invention at 1200° F. reference may be had to the following table in which the time to rupture when subjected to a temperature of 1200° F. under loads of 60,000 pounds per square inch is given together with the elongations found after such exposure. Each of these alloys having compositions identified hereinbefore are creep type specimens machined from rotor discs that were formed by forging an ingot 33 inches long by 12 inches square at one end and 9 inches square at the other end at 2100° F. to a 7 inch square billet, and cutting from the billets 3 inch pieces which were then upset forged as described hereinbefore to 1 inch thick, the upsetting being accomplished in two steps, one at 2100° F. followed by an upset at 1600° F. to finished size, after which it was solution treated and aged as described hereinbefore.

Alloy No.	Hours to Rupture Under 60,000 p. s. i. at 1200° F.	Percent Elongation
D-136	60.2	13
D-138	47.8	6.6
D-139	62.4	6.5
D-141	24.5	12.2
D-145	36.0	11.9
D-147	54.0	6.4
D-148	42.0	6.8

In general the alloys utilized in accordance with this invention have the best combination of properties known in any alloy developed to date. They are resistant to scaling and oxidation at temperatures of 1200° F. together with maximum forgeability commensurate with strength at such elevated temperatures. Further they can be formed to shape with uniform properties in large sections regardless of direction or position. The machinability characteristics are superior to those of any alloy known to date having comparable strength and ductility at 1200° F. Such results are obtained with a comparatively simple alloy composition containing a minimum content of strategic elements in controlled amounts, which alloy has the characteristic of retaining its good physical characteristics after long exposures at elevated temperatures of 1100° F. to 1300° F.

The advantage of employing the alloys of this invention in rotors is apparent as it is necessary to employ a highly ductile alloy to avoid explosive failure of such rotors. In such applications it is preferred that the rotors fail by slow creep which gives warning of impending failure instead of failing by instantaneous rupture. A solid disc rotor 12 inches in diameter and 1 inch thick made in accordance with our invention was tested by spinning at a uniform temperature of 1200° F. It withstood three successive 5 hour

periods at speeds increasing from 20,000 to 24,000 revolutions per minute, maximum calculated stress 65,000 p. s. i. After the end of the third run the diameter had grown 0.009 inch and a crack several inches long was discovered near the axis. The crack had grown progressively, thus permitting its detection before disastrous failure.

Our alloy has also been tested under engine operating conditions where competitive alloys failed at 15,000 to 15,500 revolutions per minute in 5 hours. A rotor of the alloy made in accordance with this invention withstood 18.000 revolutions per minute for 5 hours without distress, the test being discontinued because of failure of the best available blading alloys.

We claim as our invention:

1. A wrought austenitic precipitation hardened alloy consisting of 15% to 35% nickel, 7% to 20% chromium, metal selected from the group consisting of molybdenum and tungsten in an amount to equal 2% to 5% by weight, 1.3% to 1.9% titanium, not more than 0.4% aluminum, less than 0.1% carbon, 0.3% to 3% of deoxidizers and scavengers, and the balance iron with not over 1% of incidental impurities. the alloy being completely recrystallized and having a substantially uniform grain size finer than No. 4 ASTM.

2. A wrought austenitic precipitation hardened alloy consisting of 15% to 35% nickel, 7% to 20% chromium, metal selected from the group consisting of molybdenum and tungsten in an amount to equal 2% to 5% by weight, 1.5% to 1.9% titanium, less than 0.4% aluminum, less than 0.1% carbon, manganese and silicon in amounts ranging between 0.3% and 3%, and the balance iron with not over 0.5% incidental impurities, the alloy being completely recrystallized and having a substantially uniform grain size between No. 5 and No. 8 ASTM.

3. A wrought austenitic precipitation hardened alloy consisting of 15% to 35% nickel, 7% to 20% chromium. metal selected from the group consisting of molybdenum and tungsten in an amount to equal 2% to 5% by weight, 1.3% to 1.7% titanium, less than 0.4% aluminum, less than 0.1% carbon, manganese and silicon in amounts ranging between 0.3% and 3%, and the balance iron with not over 0.5% incidental impurities, the alloy being completely recrystallized and having a substantially uniform grain size between No. 4 to No. 6 ASTM.

4. A wrought austenitic precipitation hardened alloy consisting of about 25% nickel, about 13% chromium, about 3% molybdenum, 1.3% to 1.9% titanium, about .7% manganese, about .7% silicon, less than .4% aluminum, and the balance iron with not more than .5% incidental impurities, the alloy being completely recrystallized and having a substantially uniform grain size finer than No. 4 ASTM.

5. A mechanical element which is to be subjected to a temperature of 1100° F. and higher and considerable stress in normal use consisting of a wrought austenitic precipitation hardened alloy consisting of 15% to 35% nickel, 7% to 20% chromium, metal selected from the group consisting of molybdenum and tungsten in an amount to equal 2% to 5% by weight, 1.3% to 1.9% titanium, not more than 0.4% aluminum, less than 0.1% carbon, 0.3% to 3% of deoxidizers and scavengers, and the balance iron with not over 1% of incidental impurities.

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