NASA TECHNICAL NOTE

NASA TN D-3597

c. 1

NASA TN D-3597

INVESTIGATION OF COLUMBIUM-MODIFIED NASA TAZ-8 SUPERALLOY

by William J. Waters and John C. Freche

Lewis Research Center
Cleveland, Ohio

NATIONAL AERONAUTICS AND SPACE ADMINISTRATION • WASHINGTON, D. C. • SEPTEMBER 1966

INVESTIGATION OF COLUMBIUM-MODIFIED NASA TAZ-8 SUPERALLOY

By William J. Waters and John C. Freche

Lewis Research Center
Cleveland, Ohio

NATIONAL AERONAUTICS AND SPACE ADMINISTRATION

For sale by the Clearinghouse for Federal Scientific and Technical Information
Springfield, Virginia 22151 – Price $2.00

INVESTIGATION OF COLUMBIUM-MODIFIED NASA TAZ-8 SUPERALLOY

by William J. Waters and John C. Freche

Lewis Research Center

SUMMARY

The oxidation resistance of the NASA TAZ-8 alloy was substantially improved, and its workability potential and high-temperature strength characteristics were generally retained by replacing vanadium with columbium and by adding small quantities of boron. This modified version was designated TAZ-8A. The nominal composition of the alloy in weight percent is 2.5 columbium, 8 tantalum, 6 chromium, 6 aluminum, 4 molybdenum, 4 tungsten, 1 zirconium, 0.125 carbon, 0.004 boron, and the balance nickel.

In the vacuum-melted condition after 310 hours in air at 1900° F the alloy showed a weight gain of 1.8 milligrams per square centimeter. An argon-melted 0.0215-inch rolled-sheet oxidation specimen had a weight gain of 0.5 milligram per square centimeter after approximately 8 hours exposure at 1900° F. Substantially improved high-temperature oxidation resistance over other representative nickel-base alloys was observed in tests that measured weight gain and weight loss.

The TAZ-8A alloy was hot rolled from cast strips 0.110-inch-thick to 0.020-inch strips on a conventional rolling mill. Such a reduction indicates at least limited workability potential.

Ultimate tensile strengths of the as-cast alloy ranged from maximum values of 130 500 psi at room temperature to 8000 psi at 2200° F. In the as-rolled condition the ultimate tensile strengths were 240 000 and 6500 psi at these respective temperatures. At a stress of 15 000 psi the 500-, 100-, and 10-hour use temperatures were 1815°, 1895°, and 2010° F, respectively.

INTRODUCTION

Nickel-base alloys have potential for meeting the material requirements of many aerospace applications in an intermediate temperature range between approximately 1500° and 2100° F. Because of their high strengths and generally good oxidation resistance at these temperatures, these alloys are used in turbojet engine components, such as turbine buckets, and in various structural members of space and reentry vehicles.

Research is being conducted at NASA and elsewhere to extend the high-temperature capability of nickel-base alloys. Some of the more recent commercial cast alloys such as IN 100 and Mar M-200 (ref. 1) have very high tensile and stress-rupture properties at elevated temperatures. Research at NASA also led to the development of a series of high-strength cast nickel-base alloys (refs. 2 to 7) and culminated in the development of an 8 percent tantalum-modified alloy (ref. 6), which is referred to in reference 7 as the TAZ-8 alloy. The latter alloy compares in high-temperature strength with the stronger commercial alloys.

Although TAZ-8 is basically a cast alloy, it also has some workability potential. Thickness reductions of 50 percent were obtained with 1/2-inch-diameter as-cast bars by unidirectional forging techniques at room temperature without appreciable edge cracking (ref. 6). Cast sheets of this alloy approximately 0.100 inch thick were rolled to thicknesses of 0.015 inch (ref. 8) by specialized techniques.

Since the oxidation resistance of the TAZ-8 alloy was less than that of some of the commercial alloys, further improvement in oxidation resistance was considered desirable. For example, after 50 hours at 1900° F, the oxidation resistance of TAZ-8 was less than that of René 41 and Nicrotung (ref. 6).

The present investigation was directed toward improving the oxidation resistance of the TAZ-8 alloy without the loss of its workability potential and good elevated-temperature strength. In order to accomplish this end, systematic changes in composition were investigated. Oxidation data were obtained from 1900° to 2200° F for one of the more promising alloys. Rollability was also investigated, and elevated-temperature tensile data (up to 2200° F) and stress-rupture data (up to 2125° F) were obtained with this composition. Experimental melts were made by high-frequency induction heating. Melts were generally made under an argon cover although some melts were also made in vacuum. Investment casting techniques were employed to make test speciments.

MATERIALS, APPARATUS, AND PROCEDURE

Alloys Investigated

The nominal compositions of the alloys studied are given in weight percent in table I. The composition of TAZ-8 is 8 percent tantalum, 6 percent aluminum, 6 percent chromium, 4 percent tungsten, 4 percent molybdenum, 2.5 percent vanadium, 1 percent zirconium, and 0.125 percent carbon. Vanadium was removed and replaced by columbium in quantities ranging from 0.5 to 20.0 percent. Columbium additions were made in substitution for equal amounts of nickel. A constant small amount of boron (0.004 percent) was also added to each alloy.

TABLE I. - NOMINAL COMPOSITIONS OF ALLOYS INVESTIGATED

Alloys	Composition, weight percent								
	Ta	Cr	Al	Mo	W	Cb	Zr	C	B	Ni
1	8	6	6	4	4	0	1	0.125	0.004	Balance
2						0.5				
3						1.0				
[a]4						2.5				
5						3.5				
6						5.0				
7						7.5				
8						10.0				
9						20.0				

[a]Alloy referred to as TAZ-8A.

TABLE II. - PURITY OF ALLOYING ELEMENTS

[The purities are those reported by the supplier.]

Element	Minimum purity, percent
Ni	99.9
Ta	99.7
W	99.9
Mo	99.5
Cr	99.8
Al	99.88
Cb	99.6
C	99.5
B	99.5

The purities of the various alloying elements used are given in table II. All commercial alloys used in the oxidation and stress-rupture tests were supplied by the manufacturers.

One of the more promising alloys was selected for more extensive evaluation after reviewing the results of tensile, stress-rupture, oxidation, impact-resistance, and hardness tests. This alloy contained 2.5 percent columbium, and was designated TAZ-8A. Chemical analyses of randomly selected heats of this alloy were made by an independent laboratory and are shown in table III. In general, the analyses indicated that the compositions of the various heats were close to the nominal composition.

TABLE III. - COMPOSITION OF TYPICAL HEATS OF TAZ-8A AS DETERMINED BY CHEMICAL ANALYSIS

Condition	Composition, weight percent								
	Ta	Cr	Al	Mo	W	Cb	Zr	C	B	Ni
Vacuum melted	8.02	5.93	5.70	3.70	3.84	2.21	0.64	0.15	0.004	Balance
Argon melted	7.89	5.74	5.66	3.99	(a)	2.44	.94	.136	.004	
Argon melted	7.96	5.91	5.59	4.03	3.83	2.38	.85	.13	.006	
Argon melted	7.92	5.92	5.54	3.94	3.81	2.45	1.06	.14	.005	

[a]Tungsten analysis not definitive.

Casting Techniques

The casting procedures were generally similar whether melting was done under an inert gas (argon) cover or in vacuum. Melting was carried out in 50-kilowatt, 10 000-cycle-per-second, water-cooled induction units. Stress-rupture and tensile bars were cast to final dimensions. Unnotched Charpy impact bars were cast oversize. Rolling blanks were cast to dimensions of 3 by 1.5 by 0.115 inches.

Melting under inert gas cover. - Melts were made in 6-inch stabilized zirconium oxide crucibles with an outside diameter of 2 inches and an inside diameter of $1\frac{3}{4}$ inches. Tungsten, carbon, and boron were added in the form of powder that had been packed in aluminum foil containers. The other alloying elements were in the form of roundels, granules, platelets, or chips. First tungsten, columbium, carbon, and boron were melted together with some of the nickel. Then the remainder of the nickel was added and melted. Next tantalum, chromium, and molybdenum were added, in that order. The melt was then held at 3150° F for 3 minutes to assure complete solutioning of all the alloying elements. Aluminum was added last just before pouring the melt. As in the earlier investigations (refs. 2 to 6), zirconium was not added as an alloying element but was picked up from the stabilized zirconia crucibles. The pouring temperature of all melts, as determined by an optical pyrometer, was $3050^{\circ}\pm50^{\circ}$ F. All melts were top poured into investment molds that had been preheated to 1600° F. The molds were not protected by an inert gas cover during pouring. A silica slurry with a binder was used as the investment material. Castings were allowed to air cool to room temperature before the investment material was removed.

Melting under vacuum. - Melts were made in 6-inch stabilized zirconium oxide crucibles with a $2\frac{1}{2}$-inch outside diameter and a $2\frac{1}{8}$-inch inside diameter. The alloying elements were supplied in the same forms as described in the argon melting procedure. All the alloying elements except molybdenum, aluminum, and some of the nickel were first charged into the crucible. When this charge was melted, the remainder of the nickel was added, followed by the molybdenum and finally the aluminum. Contrary to the argon melting practice, a nominal 1/2 percent of zirconium was added to the original charge. Despite a longer exposure time in the crucibles, approximately 40 minutes as compared with 20 minutes, the melt did not pick up the nominal 1 percent zirconium when melting was done under vacuum. The pouring temperature, as determined by an optical pyrometer, was $3100^{\circ}\pm50^{\circ}$ F. Melts were top poured into zircon shell molds, preheated, and maintained at 1600° F by a mold heater. A pressure of approximately 10 microns was maintained over both the crucibles and the molds during melting and pouring. Castings were removed from the furnace and cooled in air to room temperature before removing the investment material.

Rolling Procedure

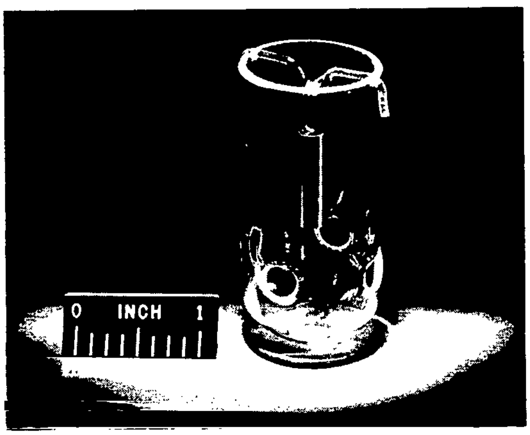
Workability of TAZ-8A was investigated by hot rolling the cast rolling blanks. The cast blanks, 3 by 1.5 by 0.115 inches, were cut into three sections 3 by 0.5 by 0.115 inches, and each of these was rolled into sheet strips approximately 0.020 inch thick. Rolling was done in a conventional four-high rolling mill at a surface speed of 80 feet per minute. These specimens were reduced 0.001 inch per pass, approximately 1 percent of the original thickness. Prior to each pass the specimens were heated in a protective (argon) atmosphere to 1900° F and transferred to the rolls as quickly as possible to minimize heat loss. Rolling was continued until strips approximately 0.020 inch thick were obtained.

Inspection of Test Specimens

All cast test specimens as well as rolled sheet strips were vapor blasted prior to inspection. All specimens were inspected by X-ray and by fluorescent-dye penetrant techniques prior to testing.

Heat Treatments

In order to improve ductility, the as-rolled sheet was given a heat treatment of 1/2 hour at 2000° F followed by aging for 24 hours at 1300° F. The heat treatment was done under an inert gas (argon) cover.



C-72043

Figure 1. - Oxidation test specimen in container.

Alloy Property Determinations

Oxidation tests. - A 20-cubic-foot resistance-wound, hearth-type furnace was used for the oxidation studies. Figure 1 shows an oxidation specimen supported in a Vycor container, closed at one end to catch any spalled oxide scale. These containers were supported in Inconel fixtures during testing. The oxidation specimens were either cylinders ground to 0.225-inch diameter by 0.875-inch length or vapor-blasted segments

TABLE IV. - SUMMARY OF TENSILE DATA

Alloy	Columbium content, percent	Condition	Temperature, °F	Ultimate tensile strength, psi	Elongation, percent
As-cast					
1	0	Argon melted	Room	127 000	5
1	0		Room	142 000	6
1	0		1900	58 000	8
2	.5		1900	62 000	--
3	1.0		Room	118 000	3
3	1.0		Room	125 000	8
3	1.0		1900	63 500	9
5	3.5		Room	125 000	3
5	3.5		Room	130 000	3
6	5.0		Room	120 000	1
6	5.0		Room	133 000	1
6	5.0		1900	49 000	--
7	7.5		Room	75 500	1
7	7.5		Room	100 000	1
8	10.0		Room	74 500	1
TAZ-8A	2.5	Argon melted	Room	126 000	2
			Room	130 500	5
			Room	126 000	3
			Room	129 000	8
			Room	131 000	4
			1400	124 000	2
			1400	134 500	3
			1600	117 500	3
			1600	89 000	2
			1600	120 500	6
			1800	83 000	4
			1800	77 000	7
			1900	64 500	6
			1900	62 000	3
			1900	65 000	7
			1900	66 000	2
			1900	65 000	2
			2000	49 500	3
			2000	47 000	5
			2000	52 000	6
			2000	47 500	3
			2000	50 000	6

TABLE IV. - Concluded. SUMMARY OF TENSILE DATA

Alloy	Columbium content, percent	Condition	Temperature, °F	Ultimate tensile strength, psi	Elongation, percent
TAZ-8A	2.5	Vacuum melted	2100	32 500	3
		Argon melted	2100	30 500	2
			2100	33 500	2
			2100	35 000	1
			2200	3 500	1
			2200	8 000	4
			2200	7 500	3
Sheet					
TAZ-8A	2.5	(a)	1900	37 500	16
		(a)	1900	37 000	20
		(a), (b)	1900	46 500	12
		(a), (c)	1900	54 000	10
		(a), (d)	1900	47 000	13
		(a), (e)	1900	32 000	16
		(a), (f)	1900	26 000	--
		(a), (c)	Room	190 000	3
		As-rolled	Room	231 000	2
		As-rolled	Room	240 000	2
		(a), (c)	1400	181 000	4
		(a), (c)	1400	190 000	4
		As-rolled	1400	181 000	16
		As-rolled	1400	191 000	8
		(a), (c)	1600	125 500	8
		As-rolled	1600	130 500	8
		As-rolled	1800	68 000	--
		As-rolled	1800	60 500	8
		(a), (c)	1800	66 500	8
		As-rolled	1900	51 500	7
		As-rolled	1900	56 500	8
		(a), (c)	1900	54 000	10
		(a), (c)	2000	20 500	18
		As-rolled	2000	29 000	10
		As-rolled	2000	30 500	6
		(a), (c)	2100	12 500	37
		As-rolled	2100	12 000	19
		As-rolled	2100	18 000	22
		As-rolled	2200	6 500	14
		As-rolled	2200	6 500	26
		(a), (c)	2200	3 000	55

[a]Heat treated 1/2 hr at 2000° F, air cooled.
[b]Heat treated 24 hr at 1200° F, air cooled.
[c]Heat treated 24 hr at 1300° F, air cooled.
[d]Heat treated 24 hr at 1400° F, air cooled.
[e]Heat treated 24 hr at 1600° F, air cooled.
[f]Heat treated 24 hr at 1800° F, air cooled.

of rolled sheet 0.75 by 0.6 by 0.016 to 0.022 inch. The specimens were suspended from quartz rods by platinum wires that were spot welded to the specimen ends. The quartz rods in turn were supported by the Vycor tubes. Specimens were weighed prior to insertion into and after removal from the furnace. After testing, the specimens were removed from the furnace and allowed to cool in air to room temperature. The weight gain of the specimen (including spalled oxide) was determined first. Oxidized specimens were then cleaned of surface oxide by microsandblasting with a dental cleaner. Weight loss was determined by subtracting the specimen weight after cleaning from its original weight.

Tensile and stress-rupture tests. - Tensile tests were conducted in air with both the as-cast and as-rolled specimens over a range of temperatures up to 2200° F. Table IV (pp. 6 and 7) summarizes the tensile test conditions. Two or more tests at each temperature were made with both the as-cast and as-rolled alloys. Generally, one test was run at each temperature with the heat-treated rolled sheet specimens. Drawings of the test specimens used for tensile and stress-rupture tests with the cast and rolled alloys are shown in figure 2. Elongations for both as-cast and sheet specimens were determined for gage lengths equivalent to the test sections shown in figure 2. Tensile tests were made with a standard hydraulically regulated tensile testing machine. The average strain rates ranged from approximately 0.2 to 0.002 inch per inch per minute and were calculated from the measured elongation after fracture and total test time. In order to determine if a significant difference existed between tests run as described and a controlled strain-rate test (0.005 in./in./min in the elastic portion and 0.05 in./in./min after yield), a controlled strain-rate test was also run at 2000° F in an Instron machine. The tensile strengths and elongations obtained from both types of tests were essentially the same.

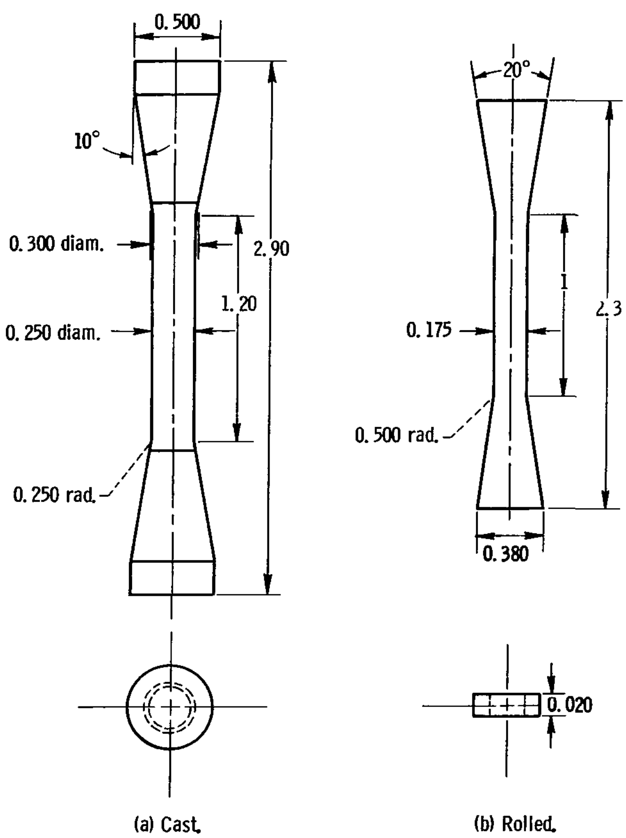


Figure 2. - Tensile-test specimens. (All dimensions are in inches.)

Stress-rupture tests were made with alloys in the as-cast condition over a range of temperatures at stresses of 15 000 and 8000 psi. The test conditions are summarized in table V.

Impact tests. - The unnotched Charpy impact resistance was measured at room temperature for all alloys in the as-cast condition. A standard Charpy impact tester was used. The total capacity of the tester was 220 foot-pounds. Specimens were ground from oversize investment blanks to 0.395 by 0.395 by 2.25 inches.

Hardness determinations. - Hardness data (Rockwell A) were obtained for all alloys in the as-cast condition.

Metallographic studies. - Metallographic studies were made of TAZ-8A in the as-cast, as-rolled, and heat-treated conditions, as well as after oxidation tests. Photomicrographs were taken at magnifications of 250 and 750.

RESULTS AND DISCUSSION

In order to improve the oxidation resistance of the TAZ-8 alloy, the replacement of vanadium by another alloying element was considered desirable. Although vanadium is an excellent strengthener (ref. 3), it was believed to adversely affect oxidation resistance.

TABLE V. - SUMMARY OF STRESS-RUPTURE DATA

Alloy	Columbium content, percent	Condition	Temperature, ^{o}F	Stress, psi	Life, hr
1	0	Argon melted	2000	15 000	4.4
1	0				8.3
1	0				1.3
1	0				1.8
3	1.0				17.2
3	1.0				6.9
5	3.5				1.1
5	3.5				3.0
5	3.5				2.7
6	5.0				1.8
6	5.0				3.3
6	5.0				2.7
7	7.5				.3
7	7.5				.4
8	10.0				.4
8	10.0				.4
8	10.0				.1
TAZ-8A	2.5	Vacuum melted	2000	8 000	97.4
			2000		346.1
			2000		330.0
			2125		18.2
			2125		5.9
			2125		14.4

TABLE V. - Concluded. SUMMARY OF STRESS-RUPTURE DATA

Alloy	Columbium content, percent	Condition	Temperature, ^{o}F	Stress, psi	Life, hr
TAZ-8A	2.5	Argon melted	1800	15 000	450.3
					400.0
					310.0
					784.3
		Vacuum melted			657.8
		Vacuum melted			659.1
		Vacuum melted			757.8
		Argon melted	1850		174.5
					96.7
					257.8
					238.2
		Vacuum melted			399.7
		Vacuum melted			208.6
		Argon melted	1900		121.5
		Argon melted			71.2
		Argon melted			107.4
		Vacuum melted			104.7
		Vacuum melted			65.9
		Argon melted	1950		50.2
					33.1
					29.1
					11.3
					14.2
		Vacuum melted			55.0
		Vacuum melted			41.4
		Vacuum melted			42.4
		Argon melted	2000		16.3
		Argon melted			9.6
		Argon melted			19.5
		Vacuum melted			12.0
		Argon melted	2050		6.5
		Argon melted			3.3
		Vacuum melted			4.0
		Vacuum melted			3.5
		Argon melted	2100		2.2
		Argon melted	2100		0.5
		Vacuum melted	2100		0.6

Vanadium forms several oxides. One of these, vanadium pentoxide (V_2O_5), melts at 675° C and is relatively volatile. An iron-nickel-chromium alloy when heated in contact with this oxide experienced catastrophic oxidation at 900° C in still air (ref. 9, pp. 218, 278). For high-temperature strength, columbium was substituted for vanadium and was varied from 0.5 to 20 percent. Columbium forms carbides and also combines with nickel to form a relatively high melting-point intermetallic compound Ni_3Cb (ref. 10). It is used as an alloying constituent in a number of nickel-base alloys. A small quantity of boron (0.004 percent) was added to all the alloys investigated. Other investigators have shown that boron has beneficial effects on the stress-rupture life and hot workability of some nickel-base alloys when added in small amounts (ref. 11).

The effect of varying amounts of columbium on oxidation resistance, stress-rupture, and tensile properties, impact resistance, and hardness was determined. Because of casting difficulties and the brittleness of the 20-percent-columbium alloy, no property data other than hardness readings were obtained. A composition, based on these screening studies, was selected (alloy 4, table I, p. 3), which afforded a good balance of oxidation resistance and high-temperature strength. This alloy is referred to as TAZ-8A, and more extensive mechanical and physical property data were obtained for this composition.

Screening Studies

Oxidation resistance. - Before considering the oxidation data, some aspects of the oxidation tests, which have a bearing upon these results, should be pointed out. Although both weight gain and weight loss measurements were made, neither measurement provides a wholly satisfactory picture of oxidation resistance. For this reason microstrucural studies of oxidized specimens and an electron microprobe analysis were used to supplement the weight-gain and the weight-loss data. A disadvantage of the weight-gain data is that evaporative losses of alloying constituents are not measured. On the other hand, a test in which weight loss is determined by subtracting the oxidized specimen weight after cleaning from its original weight is subject to error of another type, even though any evaporative losses are included in total weight loss. This error stems from the possible loss of some unoxidized metal during the cleaning process. The maximum degree of metal removal by cleaning was established by sandblasting an unoxidized specimen in a manner similar to that used for the oxidized samples, so that a surface texture comparable to that of cleaned oxidized specimens was obtained. The weight loss per unit area in this case was 0.3 milligram per square centimeter. As will be seen from the weight loss oxidation data, this small amount of metal loss cannot account for the high weight-loss values associated with many of the alloys investigated.

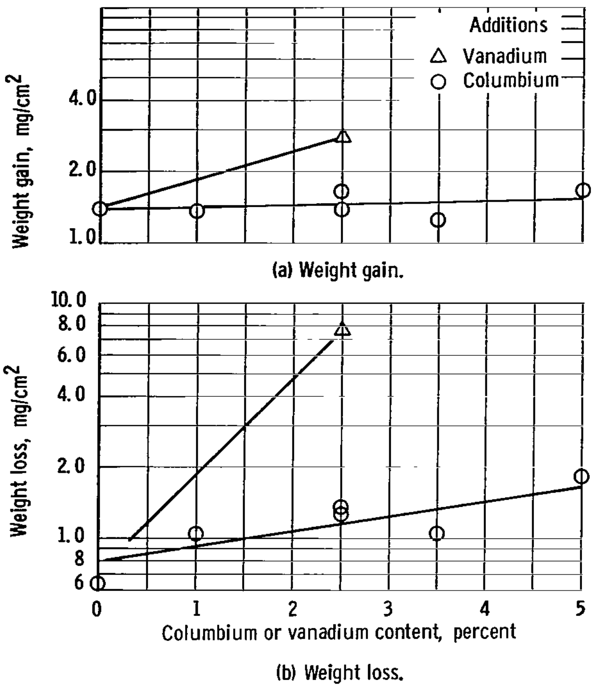


Figure 3. - Effect of columbium and vanadium additions on oxidation resistance of modified TAZ-8 alloy after 50 hours at 1900° F.

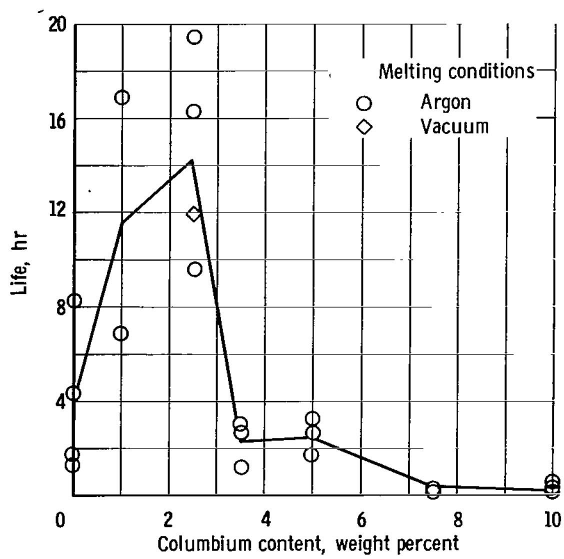


Figure 4. - Effect of columbium additions on stress-rupture life at 2000° F and 15 000 psi of modified TAZ-8 alloy.

The results of the oxidation screening studies for 50 hours at 1900° F are shown in figure 3. The relative effects of both columbium and vanadium on oxidation resistance are compared by using a modified TAZ-8 alloy (the TAZ-8 composition with zero vanadium) as a base. The figure shows that a marked improvement in oxidation resistance occurred when vanadium was removed from the TAZ-8 alloy. After 50 hours, the weight gain per unit area was reduced from 2.8 to 1.4 milligrams per square centimeter (fig. 3(a)). On the basis of weight loss (fig. 3(b)), approximately an order of magnitude improvement resulted when vanadium was removed. Columbium was not nearly as detrimental to oxidation resistance as vanadium. In fact, on a weight-gain basis the oxidation resistance was not markedly changed by columbium additions up to 5 percent over that of the baseline (zero vanadium) alloy. When measured on the basis of weight loss, a small adverse affect due to increasing columbium additions occurred. At a 5-percent columbium content, the weight loss was approximately double that obtained at zero-percent columbium content.

Stress-rupture life. - The stress-rupture capability of the columbium-modified alloys was screened by tests at 2000° F and 15 000 psi. Figure 4 shows rupture life plotted as a function of columbium content. Of the alloys considered, the maximum stress-rupture life was obtained with a 2.5-percent columbium content alloy. Average life increased from 4 to 14 hours as columbium content was increased from 0 to 2.5 percent. Stress-

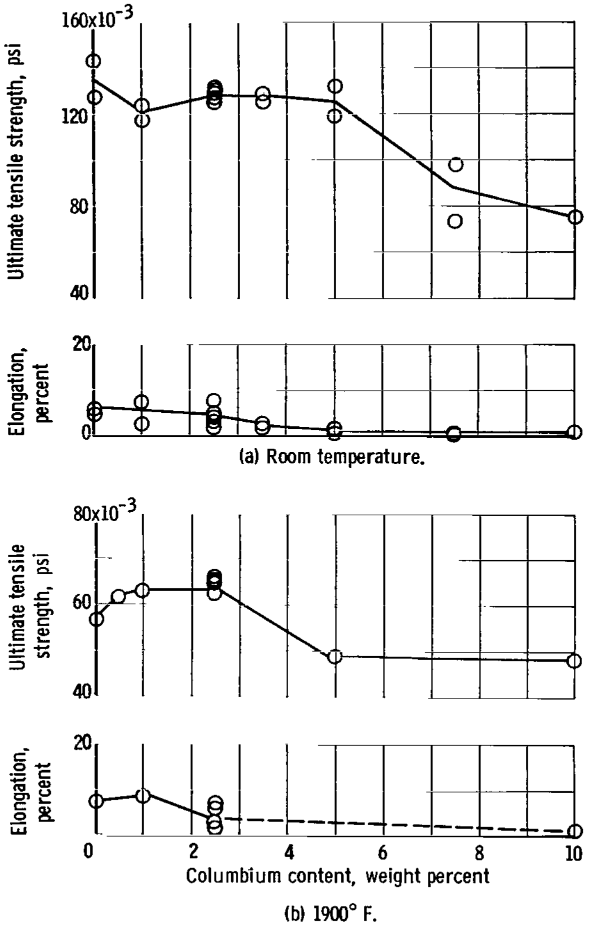


Figure 5. - Effect of columbium additions on as-cast tensile properties of modified TAZ-8 alloy.

TABLE VI. - ROOM TEMPERATURE UNNOTCHED

CHARPY IMPACT RESISTANCE

Alloy	Columbium content, percent	Impact resistance, ft-lb			
		Measured values			Average
1	0	28.0	26.0	----	27
3	1	38.0	47.0	49.0	45
TAZ-8A	2.5	19.0	20.0	34.0	24
5	3.5	14.0	8.0	14.0	12
6	5.0	12.0	13.0	11.0	12
7	7.5	7.0	9.0	7.0	8
8	10.0	8.5	7.0	7.0	8

rupture lives were reduced to less than 3 hours as columbium content was increased to 3.5 percent. At columbium contents of 7.5 percent and greater, the alloys had lives of less than 1 hour.

Tensile test data. - Tensile test data for the columbium-modified alloys are plotted in figure 5. Ultimate tensile strength and elongation data at room temperature (fig. 5(a)) and 1900° F (fig. 5(b)) are shown as a function of columbium content. At room temperature, a 2.5-percent columbium addition appeared to provide close to the maximum tensile strength. At 1900° F, both the 1- and 2.5-percent columbium-modified alloys had similarly high tensile strengths. Average elongation was approximately 5 percent for both alloys at room temperature and about 8 and 4 percent, respectively, at 1900° F. Higher columbium contents resulted in decreased strength and lower elongations.

Impact resistance. - Impact tests were made at room temperature with unnotched specimens of the various alloys in a Charpy impact tester. The results are tabulated in table VI. The highest impact resistance, an average of 45 foot-pounds, was obtained with alloy 3, which contained 1 percent columbium. Impact resistance was lower for all the other alloys. Above a 1 percent columbium content, the impact resistance of the alloys generally decreased.

Hardness data. - Table VII shows the Rockwell A hardness data for alloys with columbium contents ranging from 0 to 20 percent. Average hardness gradually increased from 69.7 for the alloy with

TABLE VII. - SUMMARY OF HARDNESS DATA

Alloy	Columbium content, percent	Rockwell A hardness					
		Measured values					Average
1	0	69.4	69.5	69.6	70.0	70.0	69.7
3	1.0	69.5	69.8	70.0	70.4	70.8	70.1
TAZ-8A	2.5	71.0	71.1	71.2	71.3	71.5	71.2
5	3.5	70.5	71.0	71.2	71.3	71.8	71.1
6	5.0	71.9	72.0	72.5	72.6	73.0	72.4
7	7.5	71.5	72.5	73.2	73.8	75.0	73.2
8	10.0	72.8	73.5	74.2	74.4	75.5	74.1
9	20.0	77.6	79.0	79.2	79.8	81.0	79.3

zero percent columbium (alloy 1) to 79.3 for the alloy with 20 percent columbium.

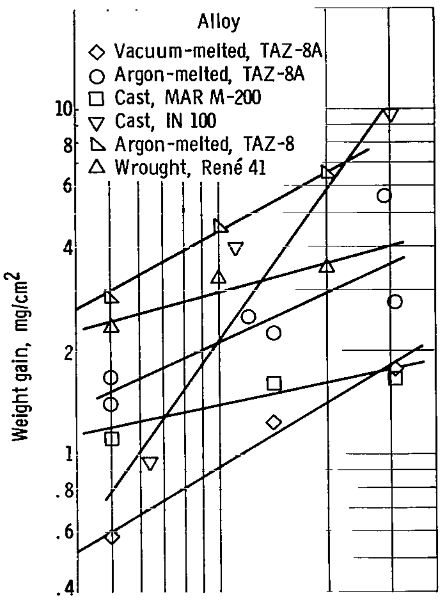
Selection of promising composition for further study. - The 2.5-percent-columbium-modified alloy (TAZ-8A) was selected for further evaluation. This alloy performed most favorably in stress rupture at 2000° F and 15 000 psi and was also one of the best insofar as tensile strength was concerned. Its tensile elongation and impact resistance were considered to be adequate. Its oxidation resistance was not markedly different from the other columbium-modified alloys. Because of the different mechanical properties associated with different values of columbium content, other compositions might, of course, be selected, dependent on the particular properties desired.

Properties of TAZ-8A

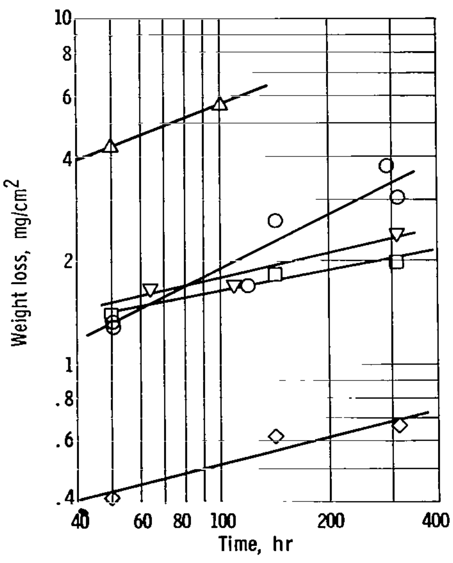
The TAZ-8A alloy (measured density 8.65 g/cm^3) was further evaluated and extensively compared with other alloys.

Oxidation resistance. - Oxidation specimens of representative commercial alloys supplied by the manufacturers were tested concurrently with TAZ-8A. Its 1900° F oxidation resistance in the as-cast condition is compared in figure 6 with that of three of the strongest known cast nickel-base alloys, MAR M-200, IN 100, TAZ-8, and a representative wrought alloy, René 41. Comparison is made on the basis of weight gain per unit area in figure 6(a) and weight loss per unit area in figure 6(b). On a weight gain basis only MAR M-200 shows an overall improved oxidation resistance over that of argon-melted TAZ-8A. In the vacuum-melted condition TAZ-8A compared favorably with all the other alloys up to 310 hours, the maximum test time considered, although the slope of its weight-gain curve is somewhat steeper than that of MAR M-200. On the basis of weight loss, the argon-melted TAZ-8A has generally the same oxidation resistance as the other cast alloys, although the steeper slope of its weight-loss curve suggests poorer oxidation resistance at exposure times over 100 hours. Vacuum-melted TAZ-8A, however, showed substantial improvement over all the other alloys. For example, in the vacuum-melted condition TAZ-8A showed about an order of magnitude improvement over René 41 and about a threefold improvement over the other cast alloys at all test times (fig. 6(b)). The improvement in oxidation resistance of vacuum-melted TAZ-8A over argon-melted TAZ-8A is most probably due to microstructural differences.

Figure 7 shows the oxidation resistance of argon-melted TAZ-8A 0.0215-inch sheet compared with that

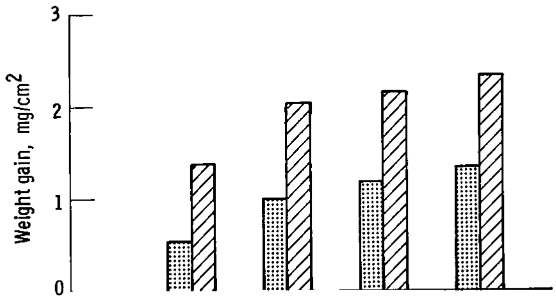


(a) Weight-gain comparison.

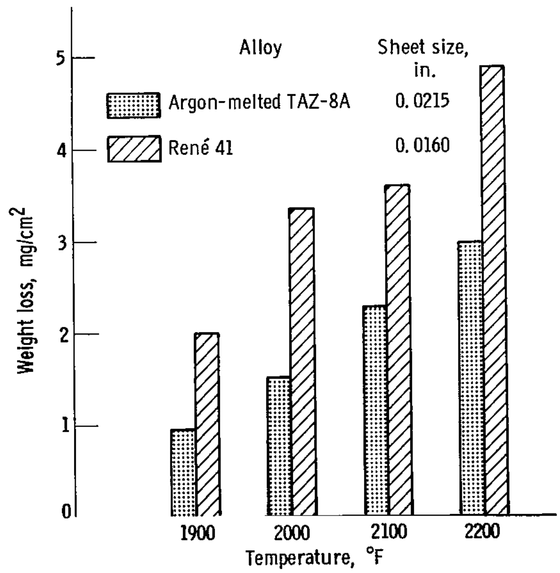


(b) Weight-loss comparison.

Figure 6. - Oxidation behavior of several nickel-base alloys at 1900° F.



(a) Weight-gain comparison.

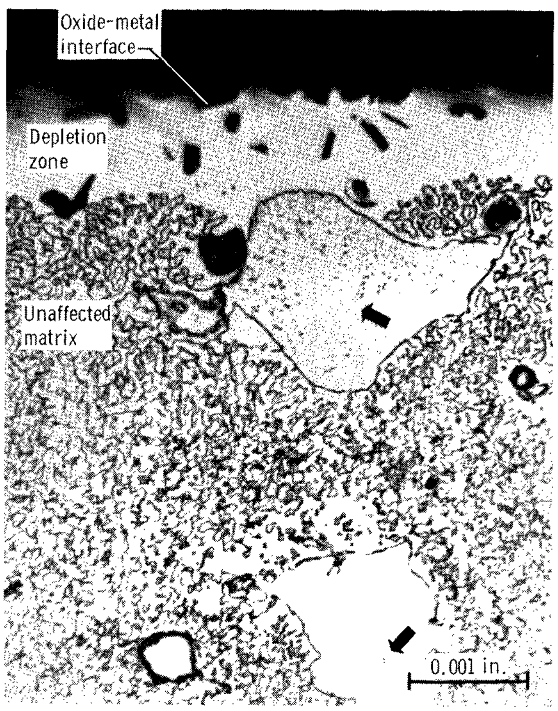


(b) Weight-loss comparison.

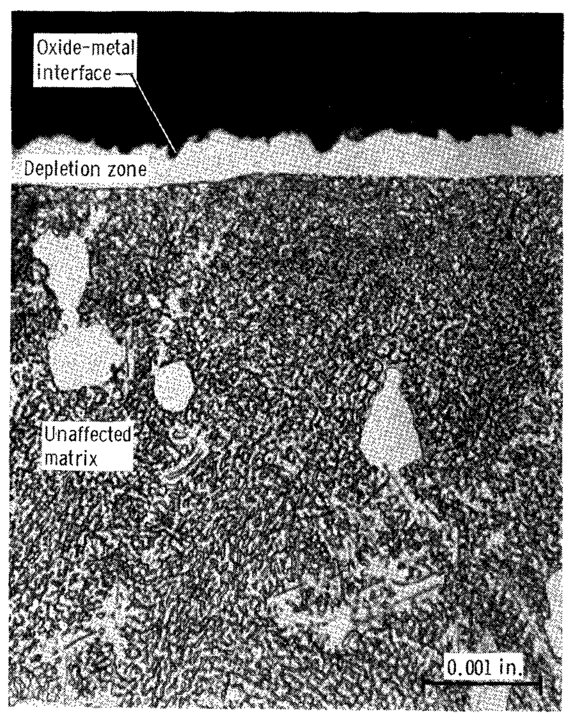
Figure 7. - Comparison of oxidation behavior of TAZ-8A and Rene' 41 sheet after 8 hours exposure at several temperatures.

of 0.016-inch René 41 sheet after exposure for 8 hours at various temperatures. The short exposure time was believed to be significant as an indication of oxidation resistance for shorttime applications (i.e., surface panels on reentry vehicles). On the basis of weight gain per unit area (fig. 7(a)), TAZ-8A sheet has approximately the same degree of oxidation resistance at 2200° F as does René 41 at 1900° F. This difference in oxidation resistance is not as marked when the comparison is made on the basis of weight loss per unit area (fig. 7(b)), although a substantial improvement in oxidation resistance of TAZ-8A over René 41 is still evident.

Figure 8 shows photomicrographs of the metal-oxide interface, depletion zone, and unaffected matrix of several of the alloys that were compared after oxidation at 1900° F. The surface oxide is not evident because it was removed to obtain weight-loss measurements. Several interesting aspects pertaining to the oxidation process in these alloys may be observed from the micrographs. The interface between the affected matrix and the depletion zone is more clearly defined for vacuum-melted TAZ-8A than for the argon-melted material. This suggests that the oxidation process from which the depletion zone arises proceeds more uniformly in the vacuum-melted material. A marked difference exists between the microstructures of the vacuum- and argon-melted TAZ-8A alloy. The aluminum-rich (white) particles, shown by arrows in figure 8, are more massive in the argon-melted than in the vacuum-melted material. Electron-probe microanalyses



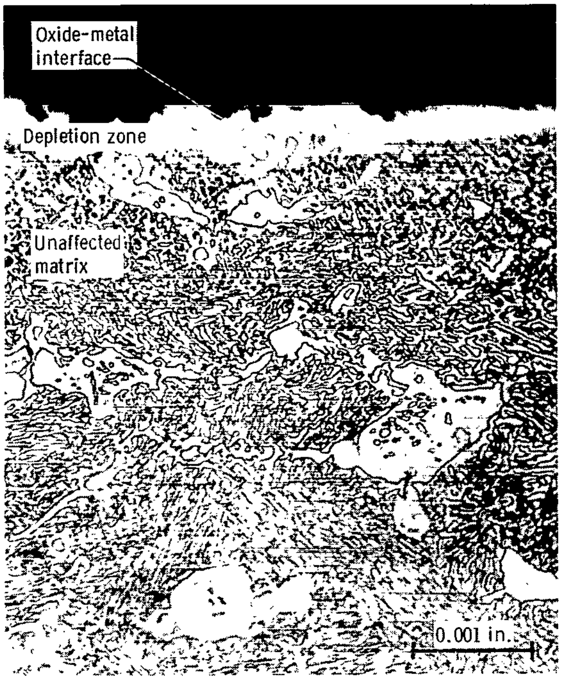
(a) Argon-cast TAZ-8A; exposure, 310 hours.



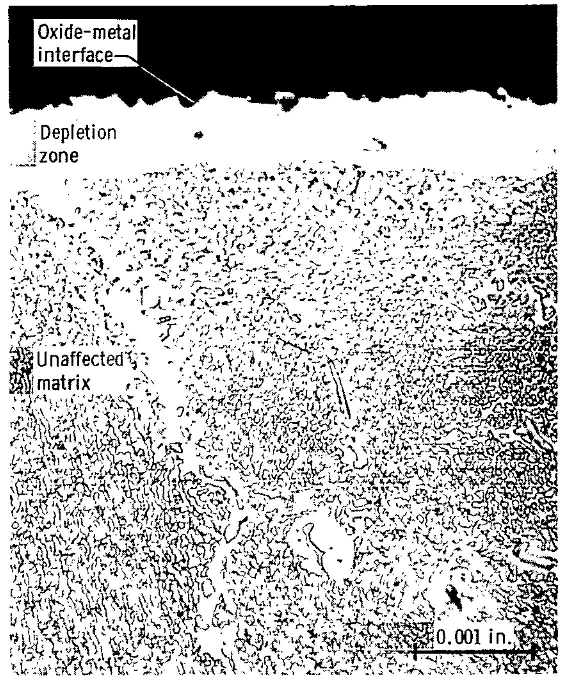
C-66-2405

(b) Vacuum-cast TAZ-8A; exposure, 310 hours.

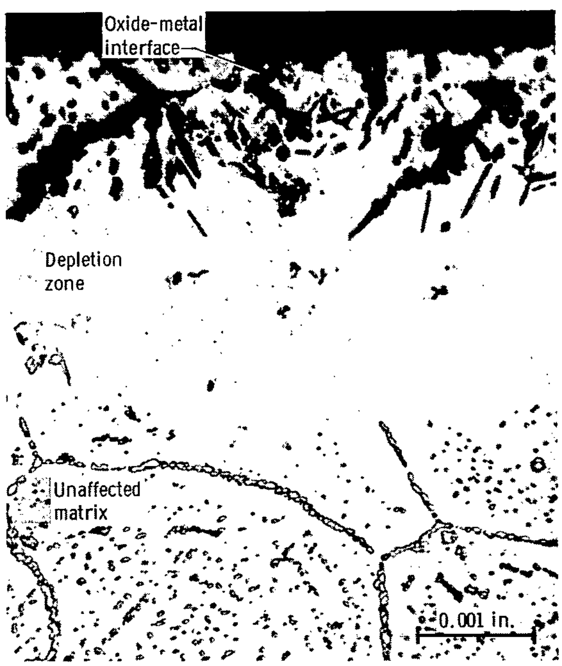
Figure 8. - Photomicrographs of oxidation specimens in vicinity of exposed surface after oxidation at 1900° F. X750.



(c) IN 100; exposure, 304 hours.



(d) MAR M-200; exposure, 310 hours.



(e) Rene' 41; exposure, 100 hours.

C-66-2406

Figure 8. - Concluded.

indicated that these regions contain approximately 50 percent more aluminum than the nominal composition of 6 percent. Both the size and distribution of the aluminum-rich white phases in the vacuum-melted alloy as compared with the argon-melted alloy suggest that aluminum would be made more readily and uniformly available to the surface in the vacuum-melted material. The vacuum-melted material, therefore, might be expected to have improved oxidation resistance, as observed in figure 6 (p. 15).

The micrographs of figure 8 also show that the depletion zone depth is approximately the same for all the alloys except René 41. The latter alloy, after exposure for 100 hours, had a substantially greater depletion zone than the other alloys after 310 hours. This indicates that René 41 may be more subject to a loss of strength because of the depletion of strengthening phases on longtime exposure to elevated temperatures. Since this depletion is a surface associated phenomenon, it could become a severe problem in thin sections exposed to oxidizing environments.

In order to place the oxidation data presented in a more useful perspective, the total affected depths (external scale plus depletion zone) due to oxidation of TAZ-8A and René 41 are compared in table VIII. Since the external scale was removed to obtain weight-loss data and since it is in any case extremely difficult to retain in position to make accurate measurements, its average thickness was calculated from weight-loss and density measurements. Typical depletion zone depths were measured from photomicrographs of oxidized samples. Both the external scale and the depletion zone are substantially smaller for TAZ-8A than for René 41. Of course, these calculations are subject to error because of uncertainty regarding the amount of the external scale and possible dimensional variations in the test specimens. Nevertheless, these calculations give a useful relative indication of the depth to which oxidation can affect the microstructure of the alloys.

The results of an electron-probe microanalysis of an argon-melted TAZ-8A oxidation specimen along a line extending from the exposed surface, through the depletion zone, and into the matrix are shown in figure 9. The analysis was made on a sample that was exposed for 290 hours at 1900° F. The concentration of the major alloying elements is plotted against the distance from the oxide-metal interface. The curve in the figure is stopped at a distance of 6 microns from the outer edge of the specimen because of edge effects. An abrupt change generally occurred in the concentration of the major alloying constituents at the matrix - depletion-zone interface. Unexpected discontinuities at this interface, such as those shown for columbium, tantalum, tungsten, and chromium, may be explained by the presence of an unidentified particle along the probe trace. A reduction in aluminum concentration was evident throughout the depletion zone. This observed depletion can reasonably be assumed to be the result of the formation of an aluminum-rich surface oxide.

Stress-rupture properties. - Table V (pp. 9 and 10) summarizes the stress-rupture

TABLE VIII. - COMPARISON OF AFFECTED ZONE DEPTHS

Alloy	Exposure condition		External scale thickness, mils	Depletion zone thickness, mils	Total affected depth, mils
	Time, hr	Temperature, ^{o}F			
René 41	100	1900	0.3	3.0	3.3
Argon-melted TAZ-8A	310	1900	.1	.8	.9
Vacuum-melted TAZ-8A	310	1900	.1	.3	.4

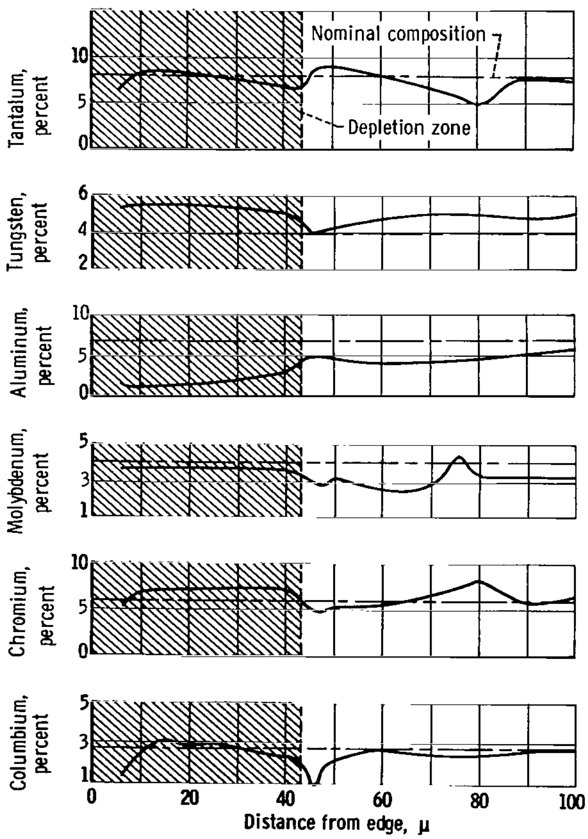


Figure 9. - Distribution of alloying elements near surface of argon-melted TAZ-8A; exposed 290 hours at 1900° F.

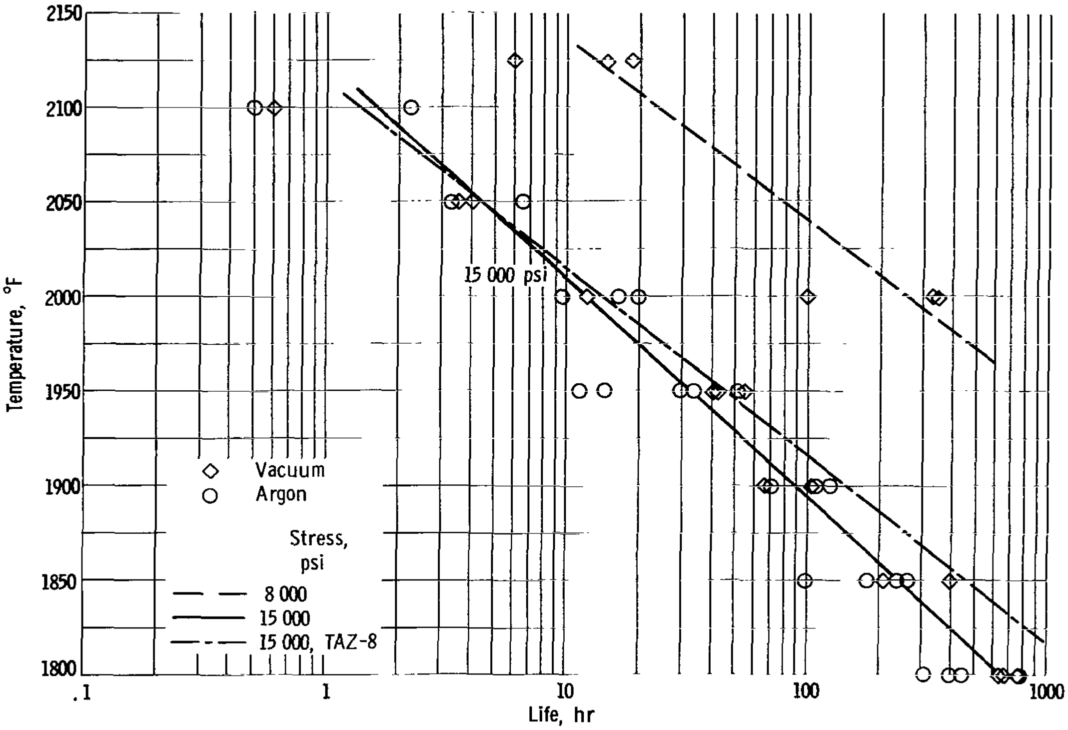


Figure 10. - As-cast stress rupture properties of TAZ-8A with 15 000 psi TAZ-8 isostress line superimposed.

data for TAZ-8A. These data are plotted in figure 10. A least-squares line has been drawn through the 15 000-psi data. Data from both argon- and vacuum-melted specimens are shown. There was no general tendency toward improved life for the vacuum-melted material. Superimposed for purposes of comparison is the TAZ-8 alloy 15 000-psi isostress line (ref. 6). At 15 000 psi the 500-, 100-, and 10-hour use temperatures for TAZ-8A are 1815°, 1895°, and 2010° F, respectively. Use temperature is defined herein as the maximum temperature at which an alloy has a specified average life under specified loading conditions. The TAZ-8A alloy has lower stress-rupture life than the TAZ-8 alloy at temperatures up to 2050° F. The isostress lines for both alloys converge and cross at this temperature. The improved oxidation resistance of TAZ-8A may be reflected in this crossover of the isostress lines for the two alloys at high temperature.

In view of the good high-temperature oxidation resistance of TAZ-8A, the alloy may be of interest for application to high-temperature, low-stress components of turbojet engines such as stator vanes. Consequently, some stress-rupture data were also obtained at a lower stress level, 8000 psi (fig. 10). The use temperature for 100-hour life at 8000 psi is 2040° F. Figure 11 shows a bar-graph comparison of the 8000-psi, 2125° F rupture life of TAZ-8A and four of the strongest known nickel- and cobalt-base alloys. The TAZ-8A alloy has a higher rupture life (13 hr) at these conditions than do all the other alloys. It is particularly interesting to note that its rupture life is greater than

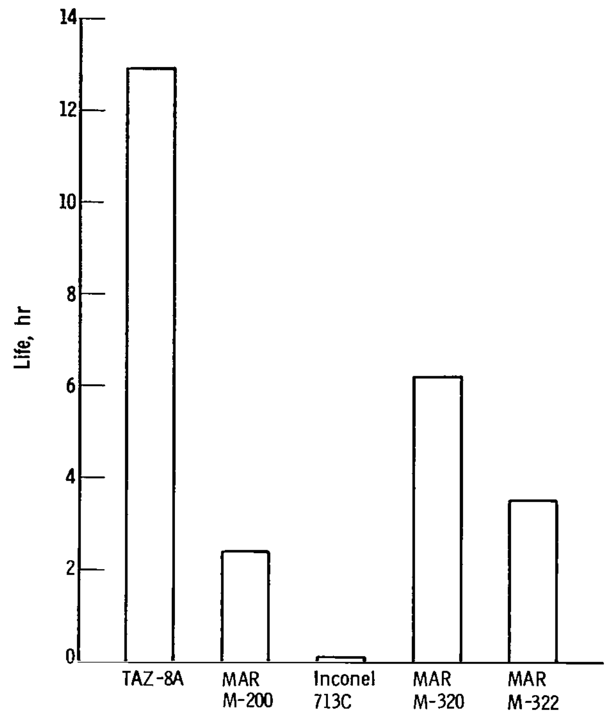


Figure 11. - Stress-rupture comparison of several nickel- and cobalt-base alloys at 8000 psi at 2125° F in air. (MAR M-320 and MAR M-322 data are from Thompson Ramo Wooldridge, Inc.)

that of the cobalt-base alloys (MAR M-320 and MAR M-322). Because of the higher melting point of cobalt and because these alloys do not depend, for high temperature strengthening, upon the gamma prime phase, which generally goes into solution below 2000° F, cobalt-base alloys might be expected to have higher use temperatures than nickel-base alloys. The good high-temperature performance (strength plus oxidation resistance) of TAZ-8A suggests that it may have advantages for low-stress applications in the range of temperatures from 2000° F to somewhat above 2100° F.

Tensile properties. - The tensile properties of TAZ-8A in cast and sheet form are summarized in table IV (pp. 6 and 7). Ultimate tensile strength and percent elongation are plotted as functions of temperature in figure 12 (p. 22). Above 1400° F, the as-cast ultimate tensile strength decreases continually with increasing temperature, ranging from approximately 129 000 psi at 1400° F to 6000 psi at 2200° F (fig. 12(a)). Between 1600° and 2000° F elongation remained reasonably constant at approximately 5 percent although the elongation decreased to about 2 percent at 2100° F. Tensile properties of as-rolled and heat-treated TAZ-8A sheet are plotted as functions of temperature in figure 12(b). It is significant that a heat treatment has been achieved that maintained as-rolled strength at temperatures over 1400° F and substantially improved the ductility of the heat-treated sheet above 1800° F. Percent elongation of the as-rolled sheet ranges

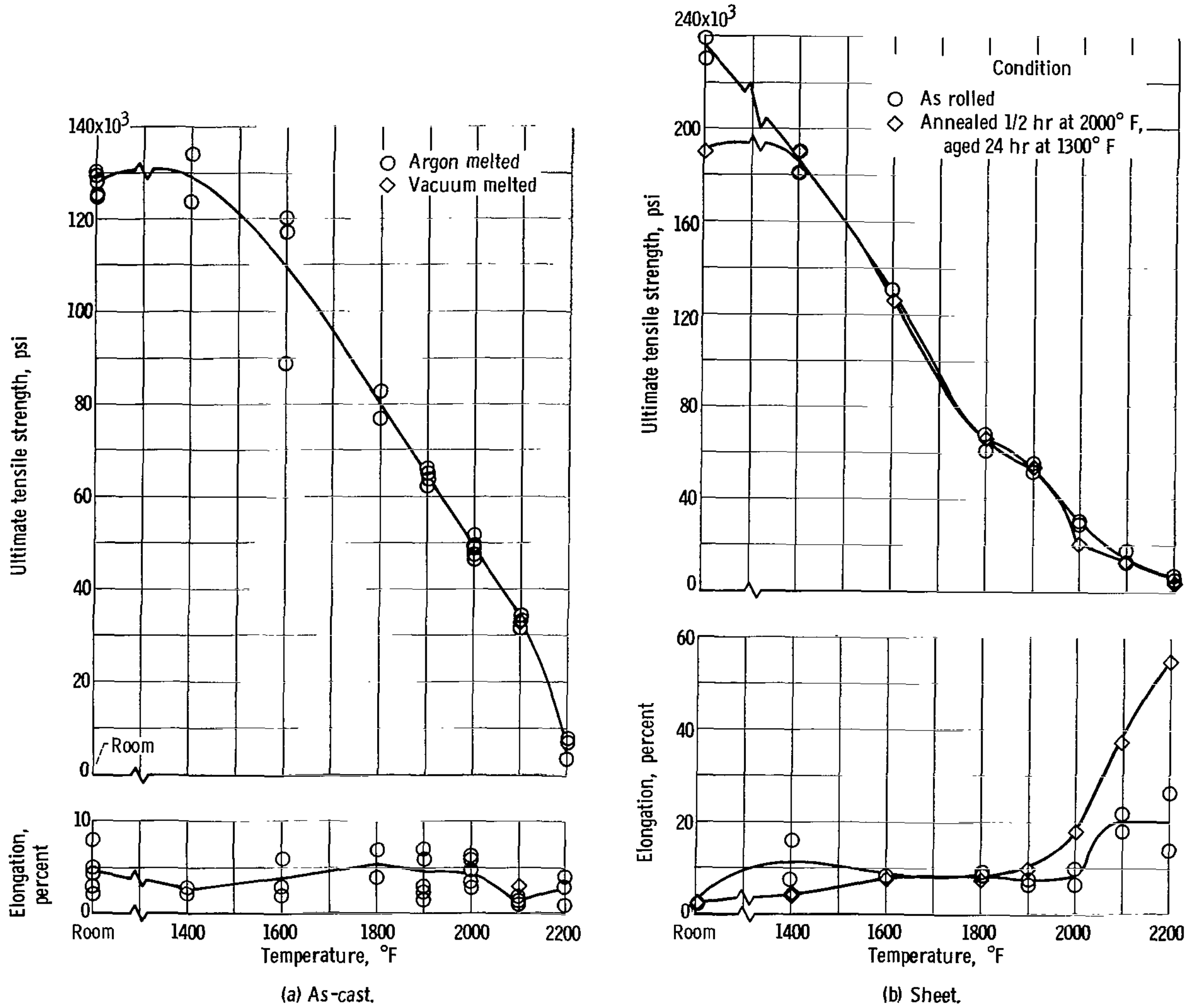


Figure 12. - Tensile properties of TAZ-8A alloy.

from approximately 3 percent at room temperature to 20 percent at 2200° F. The elongation of the heat-treated sheet begins to exceed that of the as-rolled sheet slightly above 1800° F and reaches a maximum of 55 percent at 2200° F. At 1400° F, the heat-treated material has an elongation of about 5 percent as compared with 10 percent for the as-rolled material. The high elongations of the heat-treated sheet at 2100° and 2200° F suggest that it might be fabricable into complex shapes at these temperatures.

The ultimate tensile strengths of TAZ-8A in both cast and sheet form are compared in figure 13 at several temperatures with a wrought nickel-base alloy, René 41, and with three cast nickel-base alloys MAR M-200, IN 100, and TAZ-8. In sheet form TAZ-8A is approximately 50 percent stronger than René 41 sheet at 1600° and 1800° F. At 2000° F its ultimate strength is 30 000 psi, approximately three-fourths that of René 41 at 1800° F. In the cast condition TAZ-8A is comparable to the other cast nickel-base

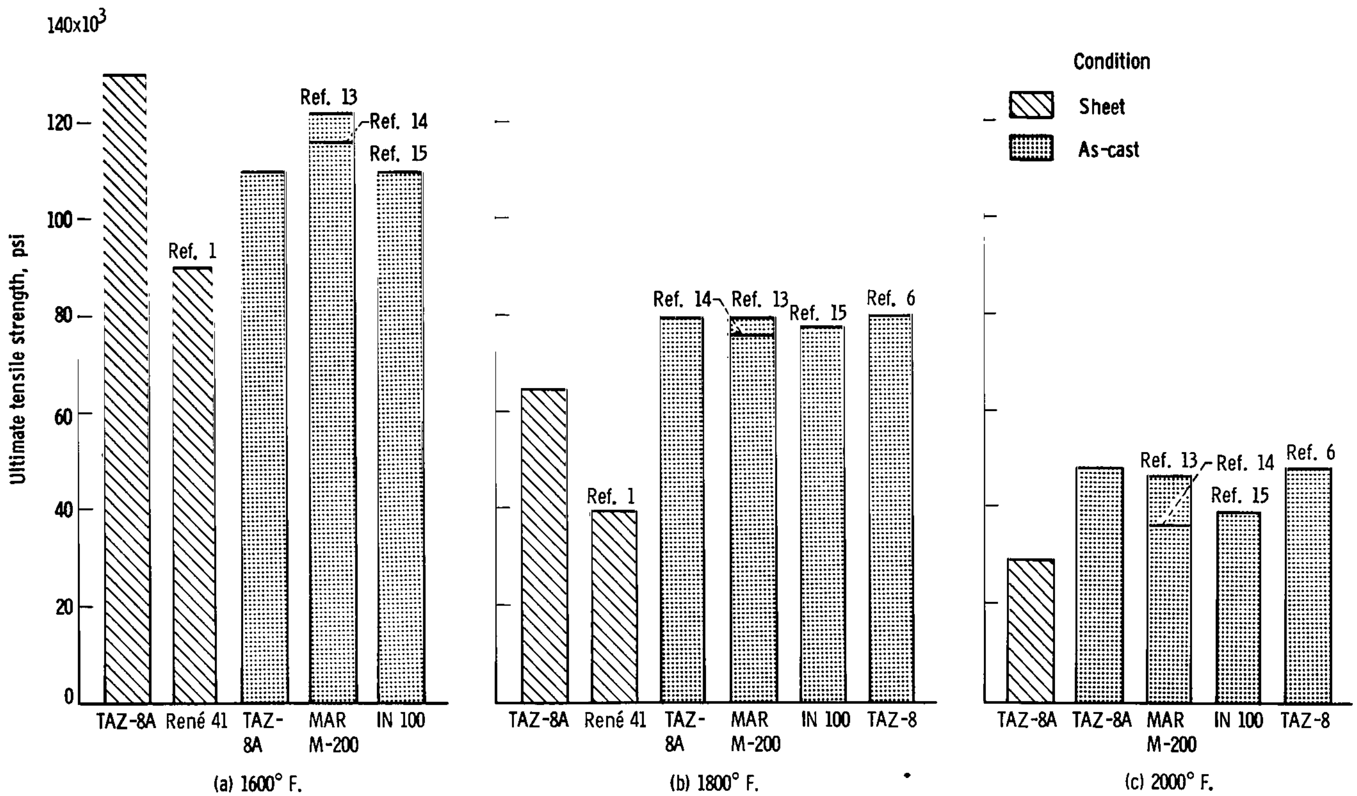


Figure 13. - Ultimate tensile strength of TAZ-8A and several nickel-base alloys in sheet and cast form at various temperatures.

alloys at all three temperatures.

Impact resistance. - The average room-temperature Charpy impact resistance (unnotched) of TAZ-8A in the as-cast condition was 24 foot-pounds (table VI, p. 13). This is somewhat lower than the impact resistance of the TAZ-8 alloy, which had an average Charpy impact resistance of 33 foot-pounds but considerably greater than that of another high-strength cast nickel-base alloy, Nicrotung, which had an average impact resistance of 9.7 foot-pounds (ref. 6). Relating these Charpy impact resistances to Izod values, which are given for two of these alloys in reference 6, and comparing them to the Izod impact resistance of other alloys successfully tested as turbojet engine buckets (ref. 12), indicate that TAZ-8A has better impact resistance than some of these alloys. Whether or not the alloy develops embrittling phases upon longtime exposure at elevated temperatures remains to be investigated.

Hardness. - Hardness data for the as-cast argon-melted TAZ-8A alloy, are presented in table VII (p. 14). Rockwell A hardness readings for this alloy ranged from 71.0 to 71.5 and averaged 71.2. If a standard conversion table for steel is used, the average Rockwell A hardness would be equivalent to Rockwell C hardnesses of 41 to 42. These values are equivalent to the as-cast hardness of the TAZ-8 alloy (ref. 6).

Workability. - Workability potential of TAZ-8A was demonstrated by rolling argon-melted slabs, 0.110 inch thick, into sheet strips approximately 0.020 inch thick. Figure 14 shows an as-cast strip and an untrimmed as-rolled sheet strip. Some edge cracking of the sheet occurred during the 1900° F rolling operation. It should be emphasized that the process of making sheet by rolling a cast thin slab is a somewhat specialized one. A fine grain size can be obtained in a thin slab. This contributes to rollability of the alloy. One benefit of the fine grain size is that impurities that normally congregate at grain boundaries are more widely distributed. Although the alloy was successfully rolled, it has not been demonstrated to be a wrought alloy in the conventional sense of the word. Nevertheless, this study suggests that the alloy may be worked under closely controlled conditions and thereby fulfill requirements that cannot normally be met by other high-strength cast nickel-base alloys.

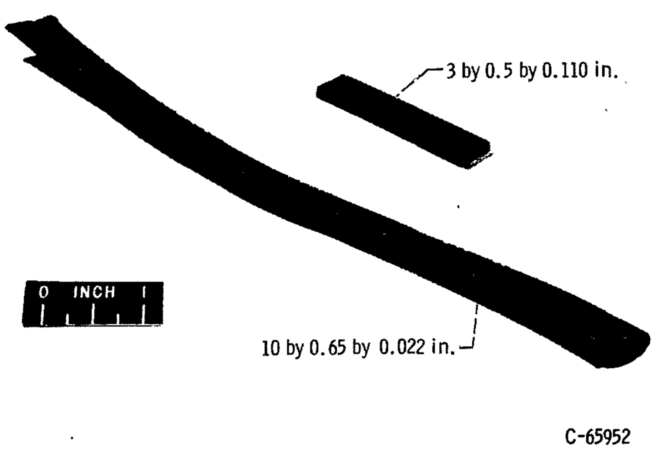
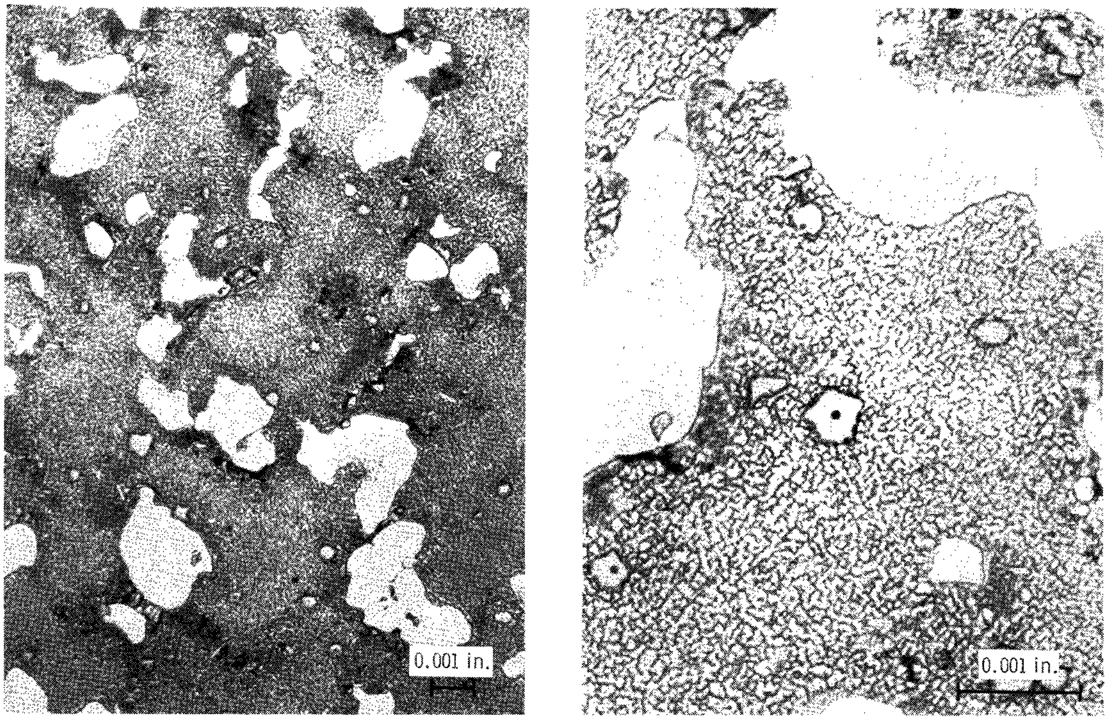


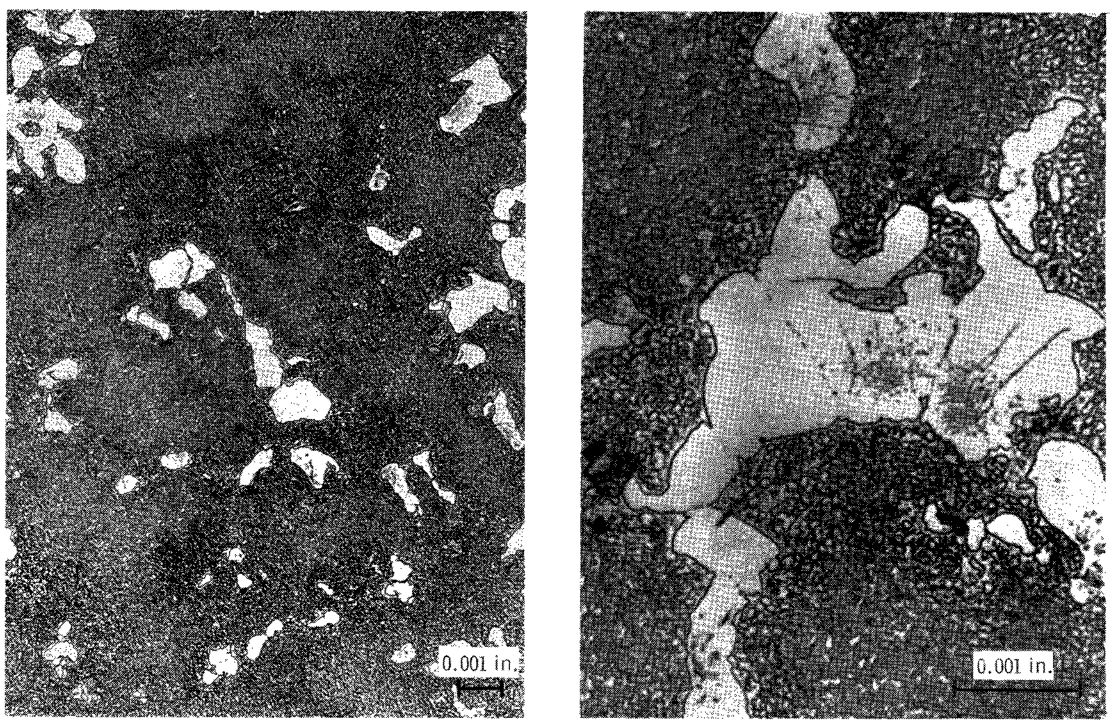
Figure 14. - TAZ-8A alloy before and after rolling at 1900° F.

Metallography. - Figure 15 shows photomicrographs of the argon- and vacuum-melted TAZ-8A alloy taken at magnifications of 250 and 750. Sections taken perpendicular to the longitudinal axis of cast stress-rupture



X250. X750.

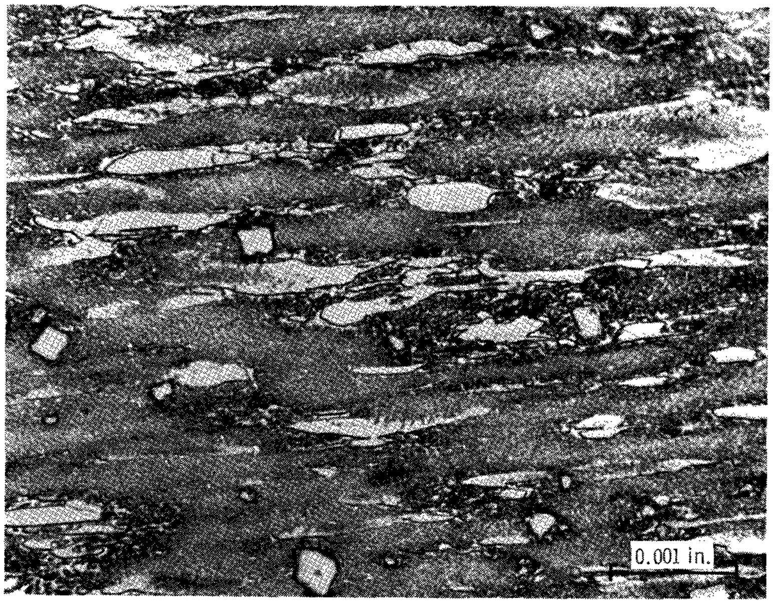
(a) Argon melted.



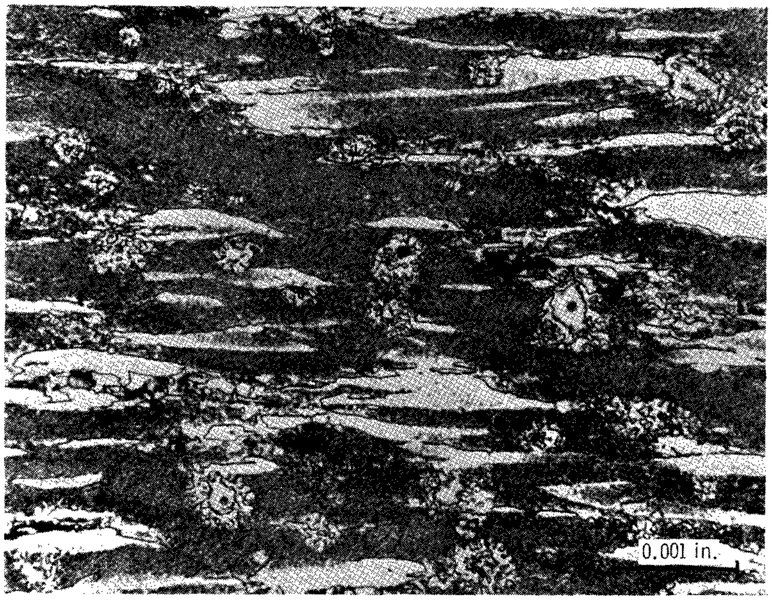
X250. X750. C-66-2407

(b) Vacuum melted.

Figure 15. - As-cast microstructure of TAZ-8A alloy.



(a) As rolled.



(b) Heat treated 1/2 hour, 2000° F; 24 hours, 1300° F. C-66-2408

Figure 16. - Microstructure of 0.020-inch TAZ-8A alloy sheet. X750.

bars are shown. The microstructures of the argon- and vacuum-melted samples are generally similar, although some differences are discernible. The vacuum-melted material has smaller, more evenly distributed gamma-prime type particles. The massive carbides and/or carbonitrides that appear in the argon-melted material are absent in the vacuum-melted condition.

Photomicrographs were taken of the as-rolled and heat-treated TAZ-8A 0.020-inch sheet at a magnification of 750 (fig. 16). The sections were taken transverse to the rolling direction. The gamma-prime type phase has clearly been deformed by the rolling process. The carbides and/or carbonitrides remained unaffected by rolling although subsequent heat treatment greatly affected their appearance. The matrix precipitate has clearly been refined by rolling as compared with the as-cast material.

CONCLUDING REMARKS

The TAZ-8A alloy has a combination of properties that make it of interest for various aerospace applications. Its good high-temperature oxidation resistance, good high-temperature strength, and at least a degree of rollability suggest that it may be useful for surface panels of reentry vehicles. It may also be of interest in turbojet engine ducting and formed stator-vane applications. Of course, additional performance studies are necessary before the alloy can be fully evaluated for these applications. One of the most critical studies would have to consider joining techniques for this alloy. Because of the high alloy content, welding would be very difficult. Weldability can probably be improved by substantially reducing the zirconium content of the alloy. Limited preliminary data indicate that zirconium content may be lowered without significantly affecting either the strength or the oxidation characteristics of the alloy.

SUMMARY OF RESULTS

The following results were obtained from an investigation intended to improve the oxidation resistance of the NASA TAZ-8 alloy without the loss of its workability potential and high-temperature strength:

1. Replacing vanadium in the TAZ-8 alloy by various amounts of columbium and adding a small (0.004) percent of boron altered the oxidation, tensile, stress-rupture, and impact properties of the alloy. Oxidation resistance was substantially improved by removing vanadium and remained approximately constant (on the basis of weight gain data) for columbium additions up to 5 percent. The highest 1900° F temperature tensile strength was obtained for columbium contents of 1 and 2.5 percent. Stress-rupture life

at 2000° F and 15 000 psi was a maximum at 2.5 percent. Impact resistance was a maximum at 1 percent and decreased with increasing columbium additions.

2. A promising alloy, TAZ-8A, was selected based upon considerations of a combination of improved oxidation resistance and good high-temperature strength. This alloy had a nominal composition in weight percent of 2.5 columbium, 8 tantalum, 6 chromium, 6 aluminum, 4 molybdenum, 4 tungsten, 1 zirconium, 0.125 carbon, 0.004 boron, and the balance nickel. Because of the differences in mechanical properties associated with different values of columbium content, other compositions might be selected dependent on the particular properties desired.

3. The TAZ-8A alloy has good high-temperature oxidation characteristics. In the vacuum-melted condition after 310 hours at 1900° F, the weight gain was 1.8 milligrams per square centimeter. An argon-melted 0.0215-inch rolled-sheet oxidation specimen had a weight gain of 0.5 milligrams per square centimeter after 8 hours exposure at 1900° F. The alloy was substantially better in high-temperature oxidation resistance than other representative nickel-base alloys when compared on the bases of weight gain and weight loss.

4. No marked difference occurred in the stress-rupture life of the alloy between the argon- and vacuum-melted conditions. At a stress of 15 000 psi, the 500-, 100-, and 10-hour use temperatures were 1815°, 1895°, and 2010° F, respectively. At the highest temperature for which stress-rupture data were obtained, 2125° F, and a stress of 8000 psi, the alloy had a rupture life of 13 hours.

5. Ultimate tensile strengths of the as-cast alloy ranged from maximum values of 130 500 psi at room temperature to 8000 psi at 2200° F. These values compare with ultimate strengths of 240 000 psi and 6500 psi for the as-rolled material. By heat treating, the as-rolled strength was maintained at temperatures above 1400° F and substantially improved ductility was achieved above 1800° F.

6. The TAZ-8A alloy was hot-rolled from cast slabs 0.110 inch thick to a thickness of 0.020 inch on a conventional rolling mill. The reductions obtained indicate that the alloy has at least limited workability potential.

Lewis Research Center,
National Aeronautics and Space Administration,
Cleveland, Ohio, June 1, 1966.
129-03-01-03-22.

REFERENCES

1. Anon: High Temperature, High Strength, Nickel Base Alloys. The Int. Nickel Co., Inc. Data Sheet (rev. ed.), 1964.

2. Freche, John C.; and Waters, William J.: Exploratory Investigation of Advanced-Temperature Nickel-Base Alloys. NASA MEMO 4-13-59E, 1959.

3. Freche, John C.; Riley, Thomas J.; and Waters, William J.: Continued Study of Advanced-Temperature Nickel-Base Alloys to Investigate Vanadium Additives. NASA TN D-260, 1960.

4. Freche, John C.; Waters, William J.; and Riley, Thomas J.: A New Series of Nickel-Base Alloys for Advanced-Temperature Applications. ASM Trans., vol. 53, 1961, pp. 523-537.

5. Freche, John C.; Waters, William J.; and Riley, Thomas J.: A New Series of Advanced-Temperature Nickel-Base Alloys. High Temperature Materials - II, G. M. Ault, W. F. Barclay and H. P. Munger, eds., Interscience Publ., 1963, pp. 215-228.

6. Freche, John C.; and Waters, William J.: Continued Investigation of an Advanced-Temperature, Tantalum-Modified, Nickel-Base Alloy. NASA TN D-1531, 1963.

7. Freche, John C.: Progress in Superalloys. NASA TN D-2495, 1964.

8. Greenewald, H., Jr.; and Riley, T. J.: Development of a Nickel-Base Alloy Sheet for High Temperature Application. Final Rep. (AFASD-TDR-62-869), Chance Vought Corp., Apr. 1963.

9. Kubaschewski, O.; and Hopkins, B. E.: Oxidation of Metals and Alloys. 2nd ed., Butterworth and Co. Ltd., 1962, pp. 218, 278.

10. Hansen, Max; and Anderko, Kurt: Constitution of Binary Alloys. 2nd Ed., McGraw-Hill Book Co., Inc., 1958.

11. Lund, C. H.: Physical Metallurgy of Nickel-Base Superalloys. DMIC Rep. 153, Battelle Memorial Institute, May 5, 1961.

12. Waters, William J.; Signorelli, Robert A.; and Johnston, James R.: Performance of Two Boron-Modified S-816 Alloys in a Turbojet Engine Operated at 1650° F. NASA MEMO 3-3-59E, 1959.

13. Anon: Martin Metals Data Folder.

14. Simmons, W. F.; and Van Echo, J. A.: Mechanical and Physical Properties of Three Superalloys - MAR M-200, MAR M-302, and MAR M-322. DMIC Memo. 193, Battelle Memorial Institute, May 6, 1964.

15. Anon: A New Cast 1850° to 1900° F Turbine Blade Alloy, "IN 100". The Int. Nickel Co. Data Sheet, Feb. 1961.

NASA-Langley, 1966 E-3331

"The aeronautical and space activities of the United States shall be conducted so as to contribute . . . to the expansion of human knowledge of phenomena in the atmosphere and space. The Administration shall provide for the widest practicable and appropriate dissemination of information concerning its activities and the results thereof."

—NATIONAL AERONAUTICS AND SPACE ACT OF 1958

NASA SCIENTIFIC AND TECHNICAL PUBLICATIONS

TECHNICAL REPORTS: Scientific and technical information considered important, complete, and a lasting contribution to existing knowledge.

TECHNICAL NOTES: Information less broad in scope but nevertheless of importance as a contribution to existing knowledge.

TECHNICAL MEMORANDUMS: Information receiving limited distribution because of preliminary data, security classification, or other reasons.

CONTRACTOR REPORTS: Technical information generated in connection with a NASA contract or grant and released under NASA auspices.

TECHNICAL TRANSLATIONS: Information published in a foreign language considered to merit NASA distribution in English.

TECHNICAL REPRINTS: Information derived from NASA activities and initially published in the form of journal articles.

SPECIAL PUBLICATIONS: Information derived from or of value to NASA activities but not necessarily reporting the results of individual NASA-programmed scientific efforts. Publications include conference proceedings, monographs, data compilations, handbooks, sourcebooks, and special bibliographies.

Details on the availability of these publications may be obtained from:

SCIENTIFIC AND TECHNICAL INFORMATION DIVISION

NATIONAL AERONAUTICS AND SPACE ADMINISTRATION

Washington, D.C. 20546