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PRELIMINARY EVALUATION OF TUNGSTEN ALLOY FIBER - NICKEL-BASE ALLOY COMPOSITES FOR TURBOJET ENGINE APPLICATIONS

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16. Abstract

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SUMMARY

The potential of tungsten-2-percent thoria, tungsten-5-percent rhenium-2-percent thoria, 218 CS tungsten, and tungsten-1-percent thoria wire-reinforced nickel-base superalloy composites for turbine bucket applications was evaluated on the basis of stress-rupture strength and oxidation and impact resistance. The results indicated that refractory metal alloy fiber - superalloy composites are potentially useful for turbine bucket applications on the basis of the properties measured. Composites were produced with stress-rupture properties superior to conventional superalloys at 2000° F (1093° C). The 100-hour stress-rupture strength obtained for the strongest composite system was 49 000 psi (338 MN/m^2) as compared with 11 500 psi (79 MN/m^2) for the strongest superalloys. The 1000-hour stress-rupture strength obtained for the composite was 37 000 psi (255 MN/m^2) as compared with 6000 psi (41 MN/m^2) for the superalloys at 2000° F (1093° C). The composites were also much stronger than superalloys when density was taken into consideration. The composite 100-hour specific stress-rupture strength at 2000° F (1093° C) was over two times that of superalloys, and the 1000-hour specific stress-rupture strength was over three times that of the superalloys. The fiber composite has a 200° F (93° C) turbine bucket use temperature advantage over superalloys based on strength-density values.

Advantage can be taken of the good oxidation resistance of the superalloy matrix material by surrounding the fibers with the matrix material or a cladding material so that the fibers are not exposed to an oxidizing environment. The tungsten alloy fiber oxidizes at its normal high rate when exposed to an oxidizing environment. A few thousands of an inch of oxidation resistant material, however, was sufficient to protect the fibers from oxidation at 2000° F (1093° C) for times up to 300 hours in noncyclic furnace tests.

Above 300° F (149° C), the impact resistance of the composite compared favorably with that of superalloys. Considerably lower impact strength values were obtained at temperatures below 300° F (149° C), which is the ductile-brittle transition temperature of the tungsten wire.

INTRODUCTION

A need exists for improved materials to be used as high-temperature components in advanced turbojet engines. Materials currently available are limited in either strength or oxidation resistance. The strength of superalloys, for example, is inadequate and the refractory metals, which have adequate strength at high temperatures, have poor oxidation resistance. One of the most promising types of materials for applications in the temperature range of 2000° to 2400° F (1093° to 1316° C) is refractory fiber-reinforced superalloy composites, if advantage can be taken of the high strength of the refractory metal fiber and the relatively good oxidation resistance of the superalloy matrix.

The principal property requirements for bucket materials are high creep rupture strengths and adequate oxidation resistance. The effects of alternate or contributory failure mechanisms must also be considered: these include impact, thermal, and mechanical fatigue; thermal shock; and hot corrosion damage. The evaluation of the potential of new materials for turbine bucket use is normally performed by laboratory tests to determine these properties.

Previous work conducted at the NASA Lewis Research Center demonstrated that 70-volume percent refractory metal fiber - superalloy composites could be produced to have a 100-hour rupture strength of 35 000 psi (241 MN/m^2) as compared with 12 000 psi (83 MN/m^2) at 2000° F (1093° C) for conventional cast superalloys (ref. 1). The results of this work also demonstrated the necessity to minimize the reaction between the fiber and matrix material by proper fabrication procedures and proper fiber diameter selection in the design of a composite to obtain high-strength properties. The lamp filament wire used in the composite, 218 CS tungsten and tungsten-1-percent thoria (ref. 1), was the strongest wire commercially available at the time. Improved high-strength wire has been made available as part of a continuing contract effort by the Lewis Research Center to obtain higher strength fiber materials.

The objectives of the present investigation were to determine the 2000° and 2200° F (1093° and 1204° C) stress-rupture strength and to conduct exploratory studies on impact and oxidation resistance of tungsten alloy fiber - superalloy composites. A determination of these properties would permit a discussion of the potential of such materials for use in turbojet engine components. In this program, additional work was conducted by using improved high-strength fibers of tungsten-2-percent thoria and tungsten-5-percent rhenium-2-percent thoria rather than the conventional lamp filament wires used previously. Composites of a nickel-base alloy reinforced with tungsten-2-percent thoria or tungsten-5-percent rhenium-2-percent thoria wire were fabricated and contained up to 70 volume percent wire. The composite specimens were evaluated in stress-rupture tests at 2000° and 2200° F (1093° and 1204° C). Exploratory studies were also made of the oxidation resistance and impact resistance of refractory metal fiber - superalloy composites. Conventional lamp filament, tungsten-1-percent thoria, and 218 CS tungsten wire were used as the reinforcement material for these latter studies. Oxidation tests were conducted in static air at 2000° F (1093° C). Izod or Charpy impact tests were conducted on the composite specimens at temperatures from 70° to 2000° F (21° to 1093° C).

MATERIALS, APPARATUS, AND PROCEDURE

Wire Material

Two commercial lamp filament wires, 218 CS tungsten and tungsten-1-percent thoria, and two experimental alloy wires supplied by contractors, tungsten-5-percent rhenium-2-percent thoria and tungsten-2-percent thoria, were used in this investigation. The wire was in the as-drawn, cleaned, and straightened condition. The wire diameters were 0.020 inch (0.051 cm) for the tungsten-5-percent rhenium-2-percent thoria and tungsten-1-percent thoria, 0.015 and 0.010 inch (0.038 and 0.025 cm) for the tungsten-2-percent thoria, and 0.015 inch (0.038 cm) for the 218 CS tungsten.

Matrix Material

The composition of the nickel-base matrix material was selected on the basis of its compatibility with tungsten fibers, as determined in a prior investigation (ref. 1). The nominal composition of the nickel alloy was 56 percent nickel, 25 percent tungsten, 15 percent chromium, 2 percent aluminum, and 2 percent titanium. The nickel alloy was vacuum cast and atomized into fine powder with a particle range of -325 to 500 mesh. A chemical analysis of the powder is given in table I. Vacuum-cast stress-rupture specimens for the alloy were obtained from the master melt used for making the powder.

TABLE I. - CHEMICAL ANALYSIS IN WEIGHT PERCENT OF NICKEL ALLOY METAL POWDER

Aluminum	Carbon	Chromium	Phophorus	Sulfur	Titanium	Tungsten	Nitrogen	Oxygen	Hydrogen	Nickel
1.96	0.0032	15.19	0.0006	0.001	1.84	24.61	0.01	0.0063	0.0020	Balance

Composite Specimen Fabrication

Composites containing the tungsten alloy wire materials and the nickel alloy were fabricated by a slip casting process, as described in reference 1. The metal powder slip consisted of the nickel alloy powder and a solution of ammonium salt of alginic acid in water. The composition, viscosity, pH, and density of the metal slip are listed in table II.

TABLE II. - METAL POWDER SLIP COMPOSITIONS AND PROPERTIES

Composition, wt. %			Viscosity at infinite shear		Slip density, g/cm^3	Percent theoretical density of slip casting	pH
Metal powder	Water	Binder material	cP	$(N)(sec)/m^2$			
89.90	10.00	0.10	3000	3.0	5.2	57	7.4

Composite specimens were prepared by inserting continuous-length tungsten alloy wires into a nickel tube containing a wire screen at the bottom and several layers of filter paper, as shown in figure 1. The nickel tube was connected to a rubber hose that was attached to a mechanical pump. The nickel tube was then placed on a vibrating table, and slip was poured into the wire bundle while the tube was vibrated. As the nickel alloy powder settled to the bottom of the bundle, excess liquid media were syphoned off the top, and more slip was added. This process was continued until the nickel alloy powder level reached the top of the wire bundle. The vibrator was then turned off, and a vacuum was applied to the tube to remove any additional liquid media left in the casting. The specimen was removed from the tube and dried in air for approximately 24 hours at 140° F (60° C). The specimens were then sintered at 1500° F (816° C) for 1 hour in dry hydrogen to volatilize the binder material and to reduce any nickel or chromium oxide present on the surface of the powders. After sintering, the specimens were inserted in closely fitting Inconel tubes with a wall thickness of 0.014 inch (0.036 cm). Nickel plugs were inserted in the top and bottom of each tube, and the tube was electron beam welded in a vacuum. The sealed tubes were leak tested in helium. The composite specimens were densified by isostatically hot pressing the tubes with helium at 20 000 psi (137.89 MN/m^2), first at 1500° F (816° C) for 1 hour and then at 2000° F (1093° C) for 1 hour. Composite specimens were made with fiber contents ranging from 25 to 70 volume percent. Fully densified specimens of over 99 percent theoretical density were produced and machined into test specimens.

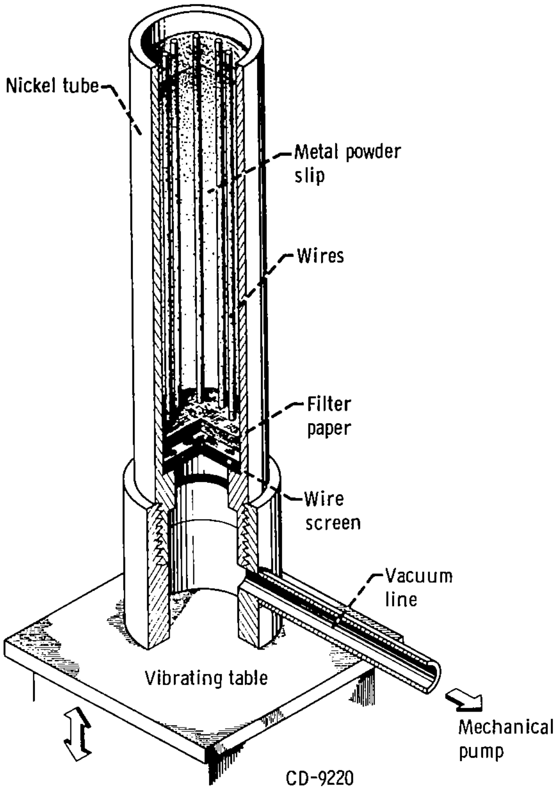


Figure 1. - Slip casting apparatus.

Testing Procedure

Tensile tests. - The wire material was tested in tension at 70°, 2000°, and 2200° F (21°, 1093°, and 1204° C). The tests were conducted in a capsule evacuated to 1×10^{-5} torr (1.3×10^{-3} N/m^2). A constant-strain-rate, screw-driven crosshead, universal tensile testing machine was used at a crosshead speed of 0.1 inch per minute (0.25 cm/min).

Stress-rupture tests. - Stress-rupture tests of the wire materials were conducted in an apparatus specifically designed for the simultaneous testing of up to four wires. A detailed description of this apparatus is given in reference 2. The inside of the chamber is shown in figure 2. In this testing unit, the wire was strung through a tantalum-wound resistance furnace and around a pulley and attached to a weight pan. The chamber was closed and the system evacuated to a measured vacuum of approximately 5×10^{-5} to 1×10^{-6} torr (6.7×10^{-3} to 1.3×10^{-4} N/m^2). The furnaces were turned on and allowed to stabilize at the desired test temperature. After temperature stabilization, the weights were applied to the specimens by lowering a retractable support.

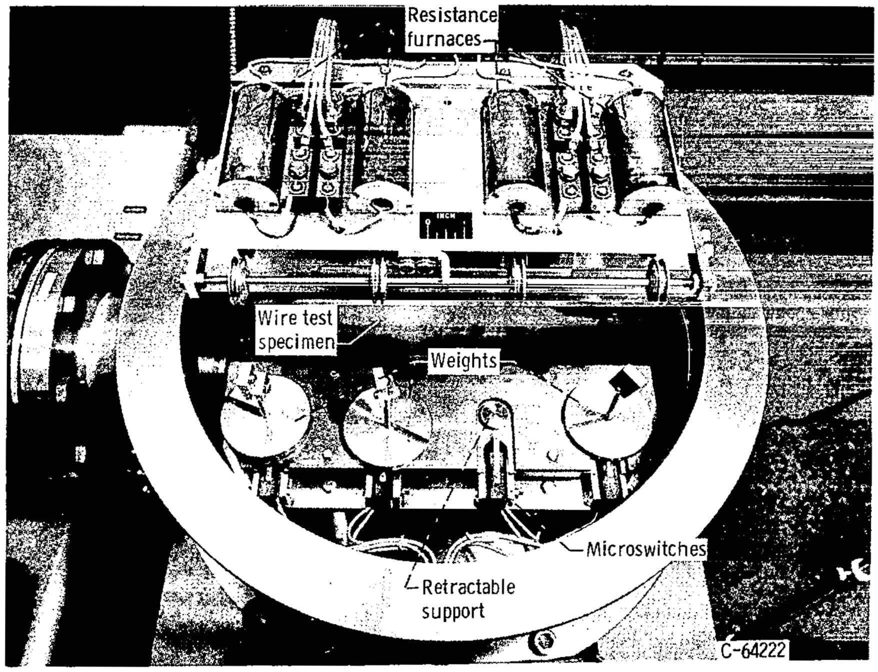


Figure 2. - Fiber stress-rupture testing apparatus.

Stress-rupture tests on composite test specimens were conducted in conventional creep machines, and a helium atmosphere was used to minimize oxidation. Tests were conducted at 2000° F (1093° C) and in some cases at 2200° F (1204° C).

Oxidation Study

An exploratory study was made of the oxidation resistance of refractory metal fiber - superalloy composites at 2000° F (1093° C). Conventional lamp filament wire, 218 CS tungsten and tungsten-1-percent thoria, was used in the composites. The matrix material had the same composition as that used for the stress-rupture study. The composite oxidation specimens were fabricated by the same procedure as that for the stress-rupture specimens. The as-pressed composite specimens were thus completely encased with a 0.014-inch (0.036-cm) cladding of Inconel. Three different types of oxidation specimens were obtained from the as-pressed cylindrical rods by grinding away all or some of the Inconel cladding. The three types were machined as follows:

Type 1: Both ends and the cylindrical surface were ground to leave a 0.006-inch (0.015-cm) layer of Inconel on all the exterior surfaces of the specimen.

Type 2: The same procedure as that for type 1 was followed except that both ends of the specimen were further ground to remove the 0.006-inch (0.015-cm) layer of Inconel. The cross-sectional ends of all wires were thus exposed.

Type 3: All the Inconel cladding was removed from both ends and the cylindrical surface. The cross-sectional ends of all wires and the longitudinal section of wires lying in the cylindrical surface of the specimen were exposed.

The oxidation specimens were approximately 1/4 inch (0.625 cm) in diameter by 1/2 inch (1.25 cm) in length with fiber contents ranging from 40 to 70 volume percent. The surfaces of the specimens were ground to a finish of 16 microinches (40×10^{-6} m). The composite specimens were suspended from a platinum wire tied to a ceramic rod. This rod was placed across the top of a zirconia crucible so that the specimen was suspended in it. The crucible lip was grooved to prevent rolling of the ceramic rod and contact between specimen and crucible wall. The composite specimens were exposed to a static air atmosphere in an electric box furnace. In all cases, the specimens were placed in the furnace where the temperature was 2000° F (1093° C). Specimens were oxidized for 5, 50, and 100 hours and were then examined metallographically.

Impact Resistance Studies

Impact tests were made on 218 CS tungsten and tungsten-1-percent thoria wire-reinforced nickel alloy composite specimens with fiber contents ranging from 50 to 60 volume percent. A standard Charpy tester and a low-capacity Izod impact tester were used. The low-capacity Izod impact tester is described in more detail in reference 3. Charpy specimens were machined to ASTM specification E23-64, whereas Izod specimens were machined to one-half the size of the ASTM specifications. The Charpy specimens were bars 0.394 by 0.394 by 2.165 inches (1.00 by 1.00 by 5.499 cm), and the Izod specimens were bars 0.1875 by 0.1875 by 1.5 inches (0.4762 by 0.4762 by 3.8 cm). Standard unnotched bar-type specimens and specimens in the "V" notched condition were studied. The testing was conducted in accordance with ASTM specification E23-64. Charpy impact tests were conducted at 2000° F (1093° C), whereas miniature Izod impact specimens were tested at temperatures from 70° to 300° F (21° to 149° C). Charpy impact specimens were heated to 2000° F (1093° C) in a resistance-wound furnace with an argon atmosphere. The specimens were held at temperature for 10 minutes and were then transferred to the Charpy impact tester and tested within 5 seconds. The Izod impact specimens were heated by propane torches. When the specimens reached the desired test temperature, they were impact tested.

The small specimens and low capacity of the miniature Izod tests were used to better differentiate between relatively small impact values. The very large capacity

Charpy was preferred for the high values obtained above the brittle transition temperature of the tungsten alloy wires.

Metallographic Studies

Stress-rupture specimens were examined metallographically to determine the depth of the reaction zone between the matrix material and fiber as a function of time at temperature and to determine the volume percent fiber content of the specimens. The depth of reaction was measured optically on transverse sections of composite specimens by using a Filar eyepiece at a magnification of 500. The depth of the reaction zone is defined as the distance from the fiber-matrix interface to the interface in the fiber where a microstructural change is observed. The cross-sectional area and the volume percent fiber content for all composite specimens were determined by sectioning the specimen transversely in an area immediately adjacent to the fracture. The sections were mounted, polished, and photographed at a magnification of 25. A wire count was obtained from the photographs, and the volume percent fiber contents were calculated.

TABLE III. - TENSILE STRENGTH OF WIRE MATERIAL

Wire material	Wire diameter		Tensile strength at test temperature					
	in.	cm.	70° F (21° C)		2000° F (1093° C)		2200° F (1204° C)	
			psi	MN/m^2	psi	MN/m^2	psi	MN/m^2
Tungsten-2-percent thoria-5-percent rhenium	0.020	0.051	310 000	2137	185 000	1276	147 000	1014
Tungsten-2-percent thoria	0.010	0.025	399 000	2751	147 000	1014	132 000	910
	0.015	0.038	384 000	2648	173 000	1193	150 000	1034
218 CS tungsten	0.015	0.038	346 000	2386	111 000	765	94 000	648
Tungsten-1-percent thoria	0.020	0.051	335 000	2310	116 000	800	107 000	738

TABLE IV. - STRESS-RUPTURE PROPERTIES OF WIRE MATERIALS TESTED

(a) Tungsten-5-percent rhenium-2-percent thoria wire (diam, 0.020 in. or 0.051 cm)

Stress		Life, hr	Reduction in area, percent
psi	MN/m^2		
Test temperature, 2000° F (1093° C)			
100 000	690	18.4	28.6
90 000	621	41.2	26.9
90 000	621	41.2	31.9
85 000	586	85.3	48.1
80 000	552	144.3	47.4
Test temperature, 2200° F (1204° C)			
80 000	552	4.6	33.6
80 000	552	5.2	27.7
60 000	414	17.1	30.3
50 000	345	55.3	----
40 000	276	174.1	----

(b) Tungsten-2-percent thoria wire (diam, 0.010 in. or 0.025 cm)

Stress		Life, hr	Reduction in area, percent
psi	MN/m^2		
Test temperature, 2000° F (1093° C)			
120 000	827	14.0	----
110 000	758	24.0	29.4
107 000	738	36.5	11.6
105 000	724	61.0	13.5
100 000	690	[a]328.9	----
Test temperature, 2200° F (1204° C)			
90 000	621	13.4	----
80 000	552	34.1	15.4
75 000	517	66.4	13.5
70 000	483	118.7	29.4

[a]Test stopped.

[b]Test stopped after 282.5 hr.

(c) Tungsten-2-percent thoria wire (diam, 0.015 in. or 0.038 cm)

Stress		Life, hr	Reduction in area, percent
psi	MN/m^2		
Test temperature, 2000° F (1093° C)			
100 000	690	6.5	45.0
97 000	669	43.3	26.2
95 000	655	228.1	42.2
93 000	641	218.5	38.2
Test temperature, 2200° F (1204° C)			
80 000	552	17.8	15.4
75 000	517	26.1	14.2
73 000	503	51.7	----
70 000	483	89.6	29.3
70 000	483	120.6	49.1
65 000	448	116.0	20.4
60 000	414	146.6	17.8

(d) Tungsten-218CS wire (diam, 0.015 in. or 0.038 cm)

Stress		Life, hr	Reduction in area, percent
psi	MN/m^2		
Test temperature, 2000° F (1093° C)			
55 000	379	86.8	2.8
60 000	414	44.2	8.0
70 000	483	9.5	45.2
80 000	552	4.0	83.5
Test temperature, 2200° F (1204° C)			
40 000	276	105.9	5.5
43 000	296	36.5	9.2
45 000	310	29.3	10.4

TABLE IV. - Concluded. STRESS-RUPTURE PROPERTIES OF WIRE MATERIALS TESTED

(e) Tungsten-1-percent thoria wire (diam, 0.020 in. or 0.051 cm)

Stress		Life, hr	Reduction in area, percent
psi	MN/m^2		
Test temperature, 2000° F (1093° C)			
90 000	621	5.1	65.7
85 000	586	7.7	56.5
83 000	572	23.4	56.5
80 000	552	133.5	68.7
75 000	517	(b)	
Test temperature, 2200° F (1204° C)			
45 000	310	279.1	7.0
50 000	345	79.3	13.5
55 000	379	33.9	20.0
55 000	379	12.2	32.7
60 000	414	19.5	36.0
60 000	414	21.3	28.6

[a]Test stopped.
[b]Test stopped after 282.5 hr.

RESULTS

Wire Properties

The tensile strengths of the wire materials investigated are given in table III. The 2000° and 2200° F (1093° and 1204° C) tensile strengths of the two experimental alloy wires were considerably higher than the two commercially available wires. The tungsten-rhenium-thoria wire had the lowest strength at 70° F (21° C); however, the 2000° F (1093° C) tensile strength was better than that of the other wire materials investigated. This wire was the strongest material tested in tension at 2000° F (1093° C), and it also compared favorably at 2200° F (1204° C).

The stress-rupture properties of the wire materials are listed in table IV. The stress-rupture properties for the 218 CS tungsten and tungsten-1-percent thoria wire were determined in a previous program (ref. 1). Plots of stress against time to rupture for the wire materials tested at 2000° and 2200° F (1093° and 1204° C) are presented in figure 3. The two experimental alloy wire materials were stronger in stress-rupture than the conventional lamp filament wire materials. The strongest wire in stress-rupture was the tungsten-2-percent thoria material.

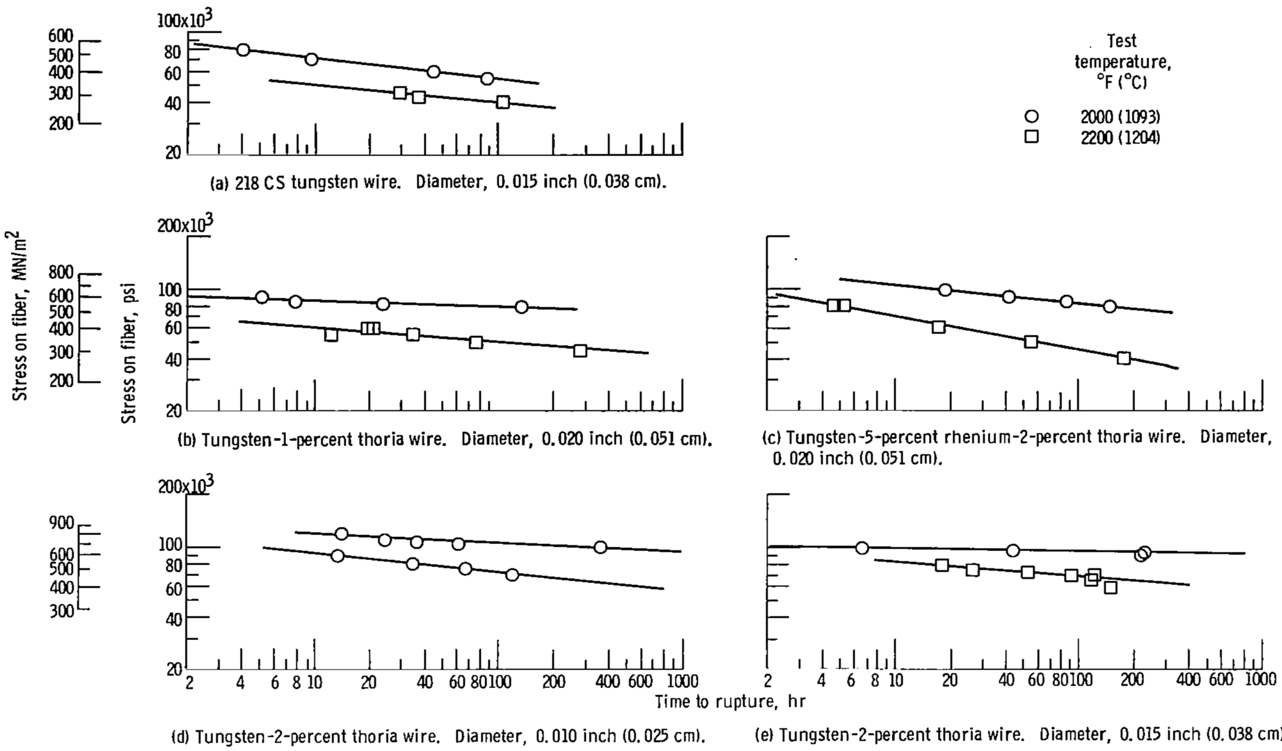


Figure 3. - Fiber stress as function of rupture time for wire materials tested.

TABLE V. - STRESS-RUPTURE PROPERTIES OF NICKEL-BASE ALLOY MATRIX MATERIAL

Stress		Life, hr	Reduction in area, percent
psi	MN/m^2		
Test temperature, 2000° F (1093° C)			
3000	20.7	108.6	29.0
3000	20.7	125.0	35.9
3500	24.1	63.6	33.4
4000	27.6	32.2	37.3
5000	34.5	8.8	42.4
Test temperature, 2200° F (1204° C)			
1000	6.9	116.7	12.5
1500	10.3	17.8	11.7

Matrix Properties

Vacuum-cast stress-rupture specimens for the nickel-base alloy matrix were obtained from the master melt used for making the powder. The specimens were stress-rupture tested at 2000° and 2200° F (1093° and 1204° C) in a helium atmosphere. The stress-rupture data were obtained in a previous program (ref. 1), and are given in table V. A plot of stress against time to rupture for the material tested at 2000° and 2200° F (1093° and 1204° C) is shown in figure 4. The 100-hour rupture strengths at 2000° and 2200° F (1093° and 1204° C) were 3200 and 1100 psi (22 and 76 MN/m^2), respectively.

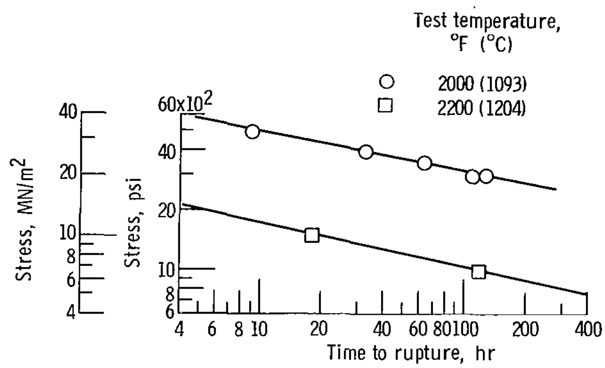


Figure 4. - Stress as function of rupture life for nickel-base alloy matrix.

Composite Stress-Rupture Properties

The stress-rupture properties obtained for the composites are given in tables VI to VIII. The composite specimens had fiber contents ranging from 25 to 70 volume percent and were tested at stress levels from 30 000 to 55 000 psi (207 and 379 MN/m^2). Determination of the composite stress-rupture strength for a specific life at a specific fiber content necessitated a determination of the fiber strength contribution in the composite. The stress on the fiber was calculated by neglecting the stress on the matrix and by dividing the composite specimen load by the area of fiber contained in the composite. The fiber was assumed to carry the major portion of the load during stress-rupture, and the matrix contribution was assumed to be negligible, which is in accordance with the analysis of the stress-rupture properties of composites reported in reference 4. The stress-carrying capability of the wire in the nickel alloy matrix material was then evaluated and a determination of the best wire-matrix combination made. The calculated stress on the fiber is given in tables VI to VIII. Figure 5(a) is a plot of the stress on tungsten-5-percent rhenium-2-percent thoria wire contained in the nickel alloy matrix

TABLE VI. - STRESS-RUPTURE DATA FOR NICKEL ALLOY TUNGSTEN-5-PERCENT RHENIUM-2-PERCENT THORIA WIRE COMPOSITES

[Wire diameter, 0.020 in. (0.051 cm).]

Stress		Life, hr	Fiber, vol. %	Stress on fiber		Penetration	
psi	MN/m^2			psi	MN/m^2	in.	cm
Test temperature, 2000° F (1093° C)							
30 000	207	37.0	36.4	82 400	568	0.00085	0.00216
35 000	241	21.4	40.8	85 800	592	0.00080	0.00203
		48.3	49.0	71 400	492	.00085	.00216
		72.4	60.3	58 100	401	-------	-------
		90.5	56.5	62 000	427	.00167	.00424
		92.4	55.6	63 000	434	.00167	.00424
40 000	276	3.2	41.8	95 800	655	0.00040	0.00102
		25.1	47.1	85 000	586	.00083	.00211
		91.2	67.5	59 300	409	.00125	.00318
45 000	310	6.9	44.0	102 400	706	0.00041	0.00104
		7.3	50.2	89 600	618	.00085	.00211
Test temperature, 2200° F (1204° C)							
15 000	103	20.4	67.8	22 200	153	0.00500	0.01270

as a function of rupture time at 2000° F (1093° C). A least-squares fit was obtained for the data. The stress on the fiber for rupture in 100 hours is 63 000 psi (434 MN/m^2), whereas that for a 1000-hour rupture life if 42 000 psi (290 MN/m^2). Figure 5(b) is a plot of the stress on the 0.015-inch- (0.038-cm-) diameter tungsten-2-percent thoria wire as a function of rupture time for the wire contained in the nickel-base matrix material. A least-squares fit was also made of these data. The stress on the wire for rupture in 100 hours at 2000° F (1093° C) is approximately 70 000 psi (483 MN/m^2), whereas that for 1000-hour rupture is 53 000 psi (365 MN/m^2). Limited data at 2200° F (1204° C) show a 100-hour rupture strength for the wire of 18 000 psi (124 MN/m^2). Figure 5(c) is a plot of fiber stress against rupture life for 0.010-inch- (0.025-cm-) diameter tungsten-2-percent thoria wire in the nickel-base matrix material tested at 2000° F (1093° C). The 100-hour rupture strength for this wire material was 56 000 psi (386 MN/m^2), and the 1000-hour rupture strength was 38 000 psi (262 MN/m^2). The plots in figure 5 thus indicate that the 0.015-inch- (0.038-cm-) diameter tungsten-2-percent thoria wire was the strongest wire material in stress-rupture. The plots can also be used to determine the stress-rupture properties of composites containing vary-

TABLE VII. - STRESS-RUPTURE DATA FOR NICKEL ALLOY

TUNGSTEN-2-PERCENT THORIA WIRE COMPOSITES

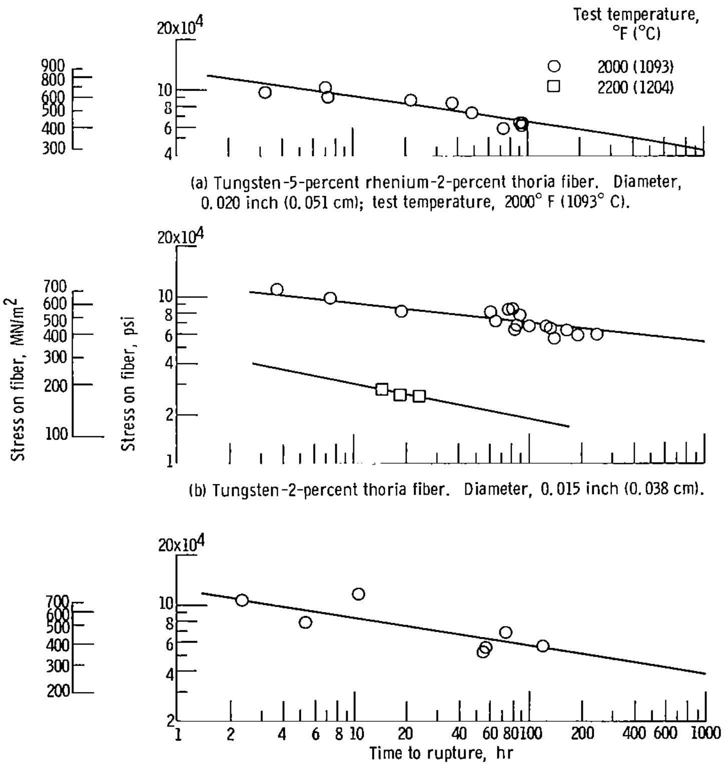
[Wire diameter, 0.015 in. (0.038 cm).]

Stress		Life, hr	Fiber, vol. %	Stress on fiber		Penetration	
psi	MN/m^2			psi	MN/m^2	in.	cm
Test temperature, 2000° F (1093° C)							
30 000	207	7.5	31.1	96 500	665	0.00053	0.00135
		86.3	44.9	66 800	461	.00156	.00396
		83.7	48.5	62 000	427	.00117	.00297
35 000	241	140.2	63.8	54 900	379	-------	-------
		180.6	60.8	57 700	399	0.00374	0.00950
		244.0	59.0	59 300	409	.00208	.00528
40 000	276	0.1	24.7	161 800	1116	0.00041	0.00104
		88.9	52.3	76 400	527	.00132	.00335
		101.5	61.3	65 300	450	-------	-------
		126.4	61.5	65 100	449	.00213	.00541
		3.7	36.0	111 200	767	.00027	.00686
45 000	310	65.4	64.4	70 000	483	0.00208	0.00528
		134.1	70.8	63 600	439	.00132	.00335
		167.2	62.0	72 500	500	.00213	.00541
50 000	345	0.2	40.6	123 200	849	0.00026	0.00066
		77.4	60.9	82 100	566	.00125	.00318
		81.0	60.0	83 300	574	.00125	.00318
55 000	379	0.3	48.5	113 500	783	0.00026	0.00066
		.8	38.3	143 500	989	.00052	.00132
		18.7	67.8	81 200	560	.00130	.00330
		61.1	69.1	79 700	550	.00117	.00297
Test temperature, 2200° F (1204° C)							
15 000	103	14.8	53.5	28 100	194	0.00416	0.01057
		18.4	58.6	25 600	177	.00500	.01270
		23.8	59.3	25 300	174	.00390	.00991

TABLE VIII. - STRESS-RUPTURE DATA FOR NICKEL ALLOY TUNGSTEN-2-PERCENT THORIA WIRE COMPOSITES AT 2000° F (1093° C)

[Wire diameter, 0.010 in. (0.025 cm).]

Stress		Life, hr	Fiber, vol. %	Stress on fiber		Penetration	
psi	MN/m^2			psi	MN/m^2	in.	cm
30 000	207	54.7	57.9	52 000	359	0.00096	0.00244
		72.9	44.7	67 100	463	.00093	.00236
35 000	241	2.3	33.2	105 500	727	0.00025	0.00064
		5.2	45.5	77 000	531	.00031	.00079
		57.4	63.1	55 500	383	.00080	.00203
		120.3	62.9	55 700	384	.00160	.00406
40 000	276	10.5	34.9	114 800	792	0.00025	0.00064



(c) Tungsten-2-percent thoria fiber. Diameter, 0.010 inch (0.025 cm); test temperature, 2000° F (1093° C).

Figure 5. - Stress on fiber as function of rupture time for nickel alloy wire composites.

ing volume fractions of fibers. The stress on the fibers to cause rupture in a specific time is multiplied by the volume fraction of fiber contained in the composite. From the data shown in figure 5(b), for example, a composite containing the large-diameter tungsten-2-percent thoria wire and having a fiber content of 70 volume percent would be expected to have a 100-hour stress-rupture strength at 2000° F (1093° C) of 49 000 psi (338 MN/m^2), that is, 0.70×70 000 psi (0.70×483 MN/m^2). Table VII shows that a specimen containing 70.8 volume percent fiber stressed at 45 000 psi (310 MN/m^2) had a rupture life of 134 hours and that a specimen containing 69.1 volume percent fiber stressed at 55 000 psi (379 MN/m^2) had a rupture life of 61 hours. The calculated 100-hour rupture strength of 49 000 psi (338 MN/m^2) for a composite containing 70 volume percent fiber thus appears to be valid. The foregoing method was used to calculated the 100- and 1000-hour rupture strengths at 2000° F (1093° C) for composites containing 70 volume percent fiber of the wire materials investigated. The results are plotted in figure 6. The large-diameter tungsten-2-percent thoria wire composites are the strongest composite system for rupture in 100 and 1000 hours.

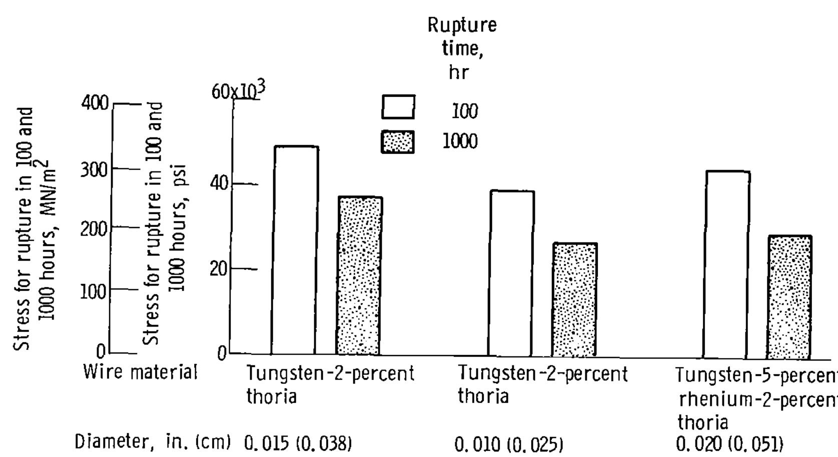
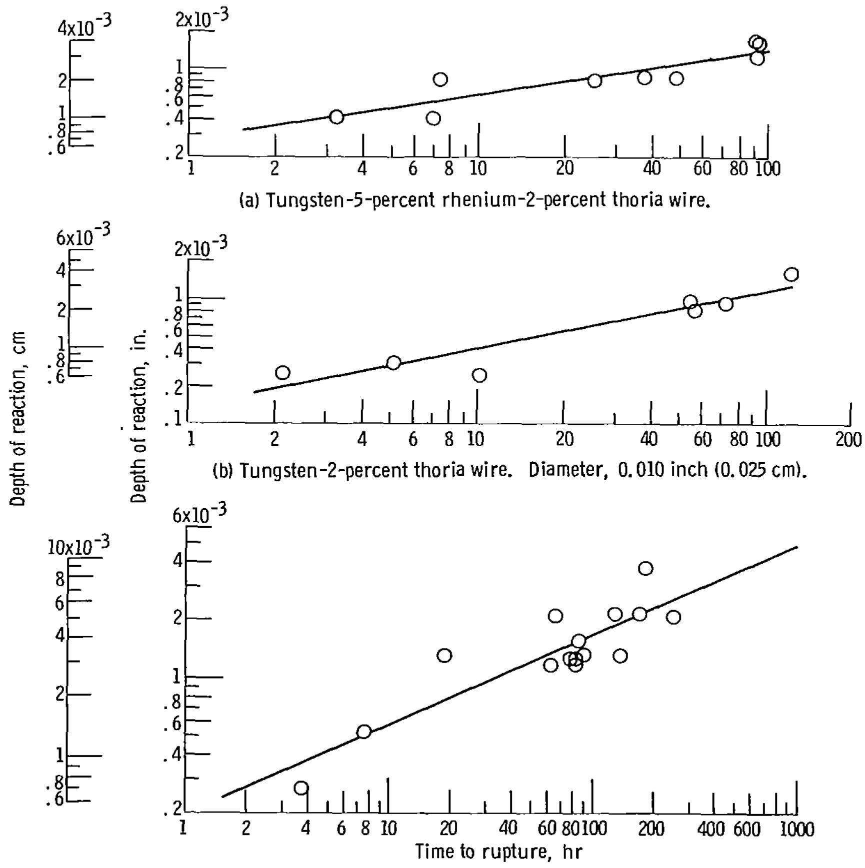


Figure 6. - 100- and 1000-hour stress-rupture strengths of nickel-base tungsten alloy wire composite at 2000° F (1093° C). Fiber content, 70 volume percent.

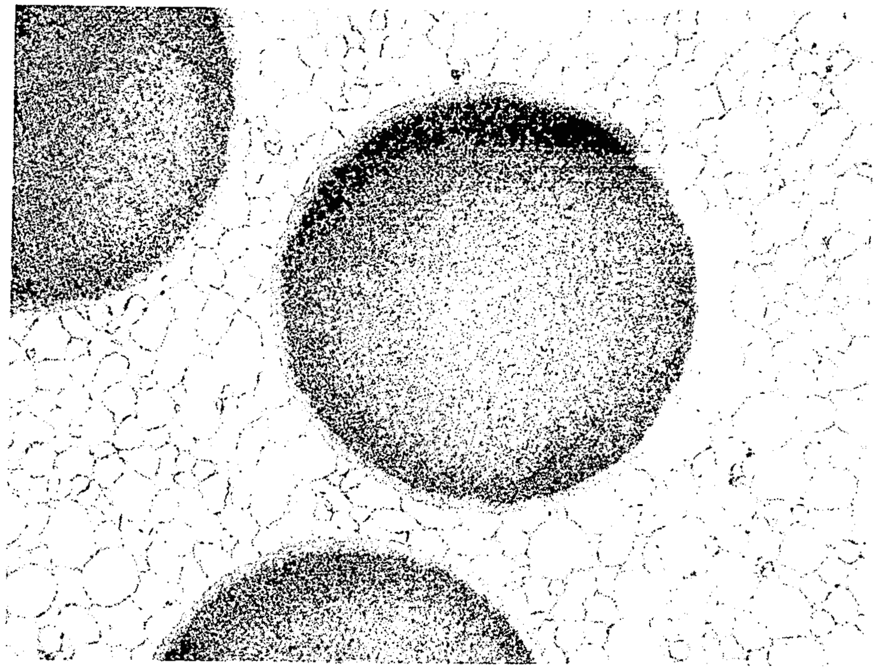
Composite Compatibility Studies

The properties reported for the different combinations of wire and nickel-base alloy used may be related to the degree of reactivity between the matrix and wire reinforcement as well as to the initial properties of these composite components. Generally, the smaller the depth of penetration in the fiber the higher the composite properties. This simple gage of composite strength must be qualified for varying wire size and for variations in the properties of the reacted zone. The depth of the reaction between the matrix and the fiber was measured for each specimen tested in stress rupture. The results of the measurements are listed in tables VI to VIII and plotted as a function of rupture time in figure 7. A least-squares fit was made of the plotted data. The depth of reaction after a 100-hour exposure was 0.00142 inch (0.00361 cm) for the tungsten-5-percent rhenium-2-percent thoria wire, 0.00116 inch (0.00295 cm) for the 0.01-inch-(0.025-cm-) diameter tungsten-2-percent thoria wire, and 0.00165 inch (0.00419 cm) for the 0.015-inch- (0.038-cm-) diameter tungsten-2-percent thoria wire. The area fraction of fiber not reacted was calculated to be 74 percent for the tungsten-rhenium-thoria wire, 58 percent for the small-diameter tungsten-thoria wire, and 64 percent for the large-diameter tungsten-thoria wire. The depth of the fiber-matrix reaction zone was equivalent for the tungsten-rhenium-thoria wire material and for the large-diameter tungsten-thoria wire material. The tungsten-thoria wire, however, is much stronger than the

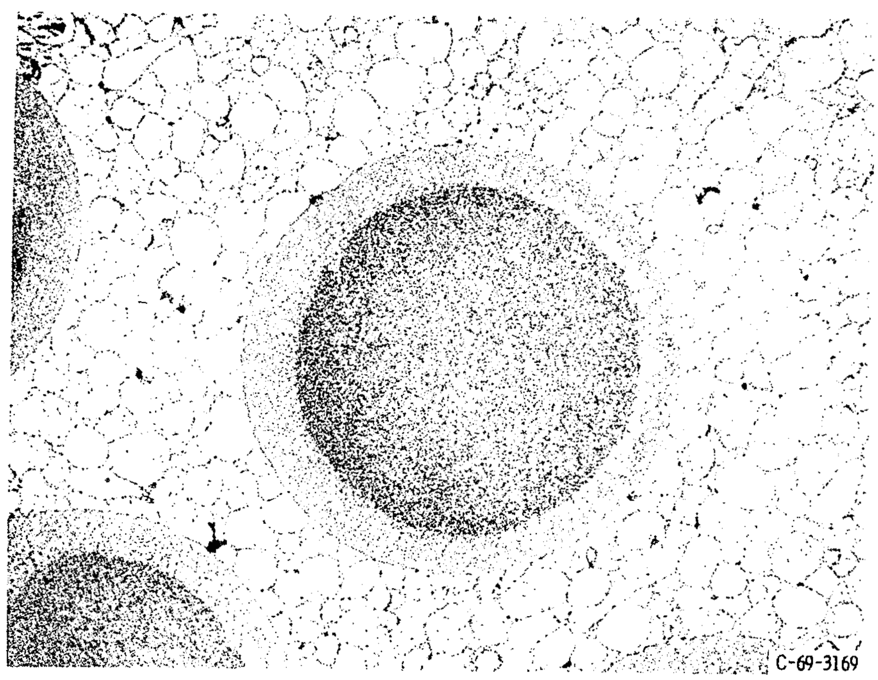


(c) Tungsten-2-percent thoria wire. Diameter, 0.015 inch (0.038 cm).

Figure 7. - Depth of reaction as function of time to rupture for nickel-base alloy composites at 2000° F (1093° C).

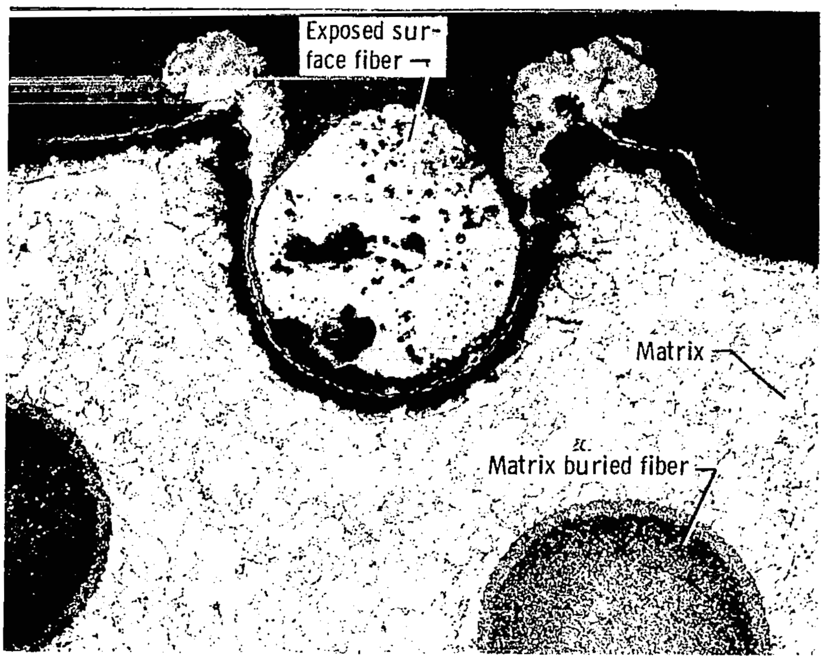


(a) Exposed for 0.8 hour.

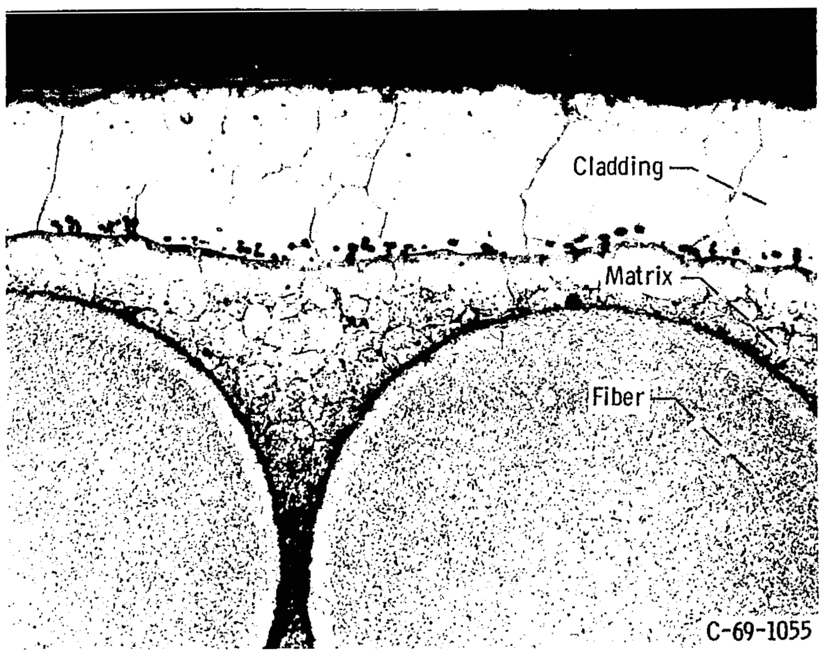


(b) Exposed for 86.3 hours.

Figure 8. - Comparison of fiber-matrix reaction at 2000° F (1093° C) for tungsten-2-percent thoria nickel alloy composite. Magnification, X150.



(a) Fiber, 0.015-inch-(0.038-cm-) diameter 218 CS tungsten. Test duration, 5 hours. Magnification, X100.



(b) Fiber, 0.020-inch-(0.051-cm) diameter tungsten-1-percent thoria. Test duration, 50 hours. Magnification, X150.

Figure 9. - Transverse section of oxidized tungsten alloy fiber - nickel alloy composite. Test temperature, 2000° F (1093° C); atmosphere, air.

rhenium-containing wire material, the result being a higher composite strength. The small-diameter tungsten-thoria wire formed the smallest reaction zone with the matrix material. The area fraction reacted, however, was the largest of the three wires investigated because of its higher surface-to-area ratio.

Typical reactions that occurred between the matrix material and tungsten-2-percent thoria wire at 2000° F (1093° C) are shown in figure 8. This figure is a cross section of a composite that failed at 0.8 hour in a stress-rupture test at 55 000 psi (379 MN/m^2). The microstructure of a composite that failed at 86.3 hours in a stress-rupture test at 30 000 psi (207 MN/m^2) is shown in figure 8(b).

Oxidation Studies

Specimens in which the fibers were completely encased in a 0.006-inch (0.015-cm) layer of Inconel, type 1 specimens, exhibited good oxidation resistance at 2000° F (1093° C) for times to 300 hours. Specimens in which the ends were sectioned to expose all the refractory metal wires to the oxidizing environment, type 2 specimens, exhibited excessive oxidation in air at 2000° F (1093° C). The tungsten alloy wires oxidized at their normal high rate. Specimens in which the container material was completely removed to expose all the refractory metal wires in the longitudinal direction and surface wires in the transverse direction, type 3 specimens, also exhibited excessive oxidation at 2000° F (1093° C). The majority of the oxidation, however, occurred in the longitudinal direction. In the transverse direction, only the wires exposed to the atmosphere were oxidized, as shown in figure 9(a). The matrix material protected the internal fibers from oxidation in the transverse direction. Specimens completely encased in a 0.006-inch (0.015-cm) layer of Inconel, type 1 specimens, exhibited good oxidation resistance at 2000° F (1093° C) for exposure times over 300 hours. A cross-sectional view of such a specimen tested in air at 2000° F (1093° C) for 50 hours is shown in figure 9(b). The Inconel cladding was oxidized, and a coherent oxide scale formed with the Inconel. Oxidation had not progressed to the composite, and the surface wires were not affected by the oxidation of the cladding. The reaction zone formed with the wire after exposure for 50 hours at 2000° F (1093° C), as shown in figure 5(b), resulted from alloying of the matrix material with the wire.

Impact Studies

The notched Izod impact test specimens yielded the following results: at 70° F (21° C), 4.7 inch-pounds (0.53 J); at 250° F (121° C), 4.7 inch-pounds (0.53 J); and at

300° F (149° C), 26.7 inch-pounds (3.02 J). The unnotched Izod specimen results were as follows: at 70° F (21° C), 3.25 inch-pounds (0.37 J); at 185° F (85° C), 7.1 inch-pounds (0.80 J); and at 300° F (149° C), greater than 26.7 inch-pounds (3.02 J). Two Charpy tests were conducted at 2000° F (1093° C). An unnotched specimen had a measured impact strength of 29 foot-pounds (39.3 J) and a notched specimen had an impact strength of 27.5 foot-pounds (37.3 J). The low impact strength of the composites below 300° F (149° C) may be attributed to the brittleness of the tungsten wires below their transition temperature, as indicated in previous work (ref. 5). Above 300° F (149° C), the impact resistance of the composite compares favorably with that of the superalloys.

DISCUSSION

Composite Stress-Rupture Strength

The large-diameter, 0.015 inch (0.038 cm), tungsten-2-percent thoria wire composites were the strongest composite system of those investigated for rupture in 100 and 1000 hours at 2000° F (1093° C). Figure 10 compares the 100- and 1000-hour stress-rupture strengths obtained for composites containing 70 volume percent tungsten-2-percent thoria wire and filament wire of tungsten-1-percent thoria. The 100- and 1000-hour rupture strengths of conventional cast nickel alloys at 2000° F (1093° C) are

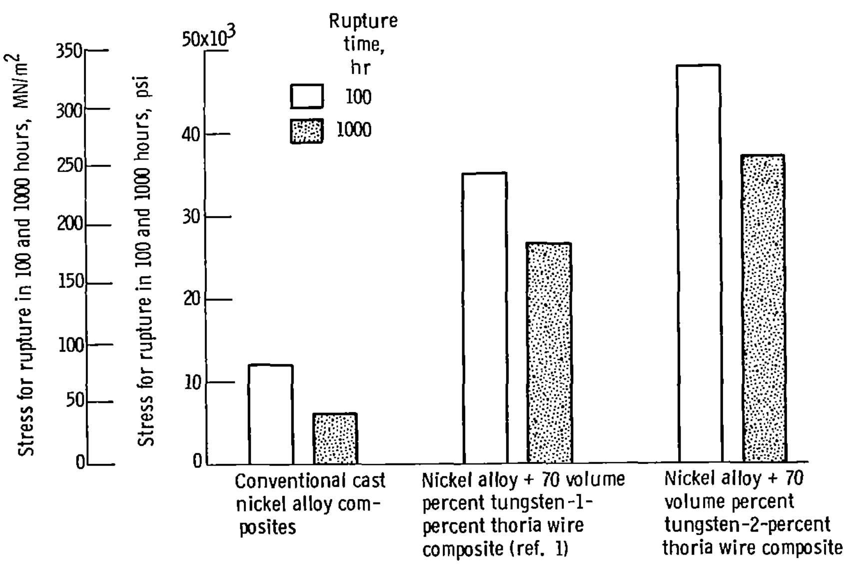


Figure 10. - 100- and 1000-hour rupture strengths of cast nickel alloy and tungsten-thoria wire-reinforced nickel alloy composites tested at 2000° F (1093° C).

also shown. A 40-percent improvement in 100-hour rupture strength was obtained with tungsten-2-percent thoria as the reinforcement material compared with tungsten-1-percent thoria wire. Both composite systems were fabricated by the same process and contained a matrix material with the same composition. A similar improvement was obtained for a 1000-hour rupture strength with tungsten-2-percent thoria wire used as the reinforcement material rather than tungsten-1-percent thoria wire. The tungsten-2-percent thoria wire composite had a 100-hour rupture strength four times that of the strongest conventional cast superalloys at 2000° F (1093° C) and a 1000-hour rupture strength six times that of the superalloys.

The density of the composite material was much greater than that of cast nickel alloys and must be taken into consideration. The tensile stresses in turbine blades, for example, are a result of centrifugal loading; therefore, the density of the material is important. Tungsten has a density about 2.3 times that of most nickel-base alloys, and thus a composite containing 70 volume percent tungsten fibers has a density approximately 1.9 times that of most nickel-base alloys. A comparison of the specific strength properties of the composite and conventional superalloys is therefore significant. A plot comparing the 100- and 1000-hour specific rupture strengths of cast nickel alloys and tungsten-thoria wire-reinforced nickel alloy composites at 2000° F (1093° C) is shown in figure 11. Even when density is taken into consideration, the composite containing tungsten-2-percent thoria wire is much stronger than the conventional cast superalloys. The tungsten-2-percent thoria wire composite has a 100-hour specific rupture strength of 83 000 inches (2108 m), over two times that of the superalloys, at 2000° F (1093° C), and

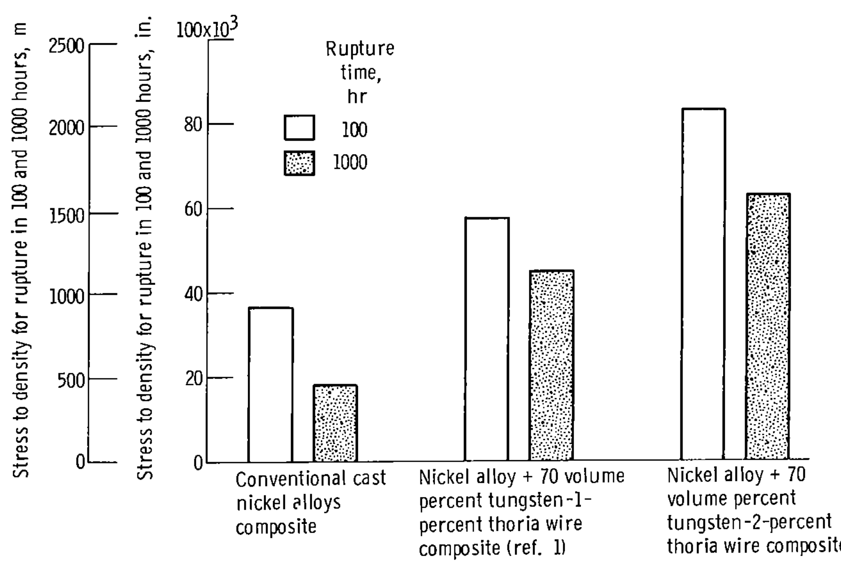


Figure 11. - 100- and 1000-hour specific rupture strengths of cast nickel alloy and tungsten-thoria wire-reinforced nickel alloy composites tested at 2000° F (1093° C).

a 1000-hour specific rupture strength of 63 000 inches (1600 m), over three times that of the superalloys. A comparison of the stress-density properties of superalloys and composite materials indicates the promise of composite materials for turbine bucket use. Standard nickel alloys used as turbine buckets have a stress-density value of about 60 000 inches (1524 m) for a 1000-hour rupture life at 1800° (982° C). The fiber composite has similar stress-density values at 2000° F (1093° C). Based on this comparison of strength, the fiber composite has a 200° F (93° C) use temperature advantage over conventional superalloys.

The density of the 70 volume percent fiber-reinforced composite is about twice that of superalloys. This high density would result in a weight penalty for an exact geometric copy of existing turbine buckets, despite the strength-density advantage of the composite material. However, it may be possible to take advantage of the strength-density property of the fiber composite to avoid a weight penalty. Modification of the bucket configuration with little change in the external airfoil shape could result in significant weight reduction. The weight can be reduced by varying the fiber content along the bucket length to match the stress pattern. Also, the airfoil can be made hollow, and the internal and external taper can be reduced to make use of the improved strength and stiffness. The modified design could result in a weight advantage over conventional materials rather than in a weight penalty.

Oxidation and Impact Properties

Stress-rupture strength alone does not satisfy the material requirements for turbine bucket applications. The material must be able to resist the oxidation and impact imposed by engine operating conditions as well as those by other failure mechanisms. Studies of oxidation resistance and impact failure are in progress at the Lewis Research Center and encouraging preliminary results have been obtained.

The results of the preliminary oxidation study indicated that advantage can be taken of the better oxidation properties of the matrix material or of a cladding material on the outer surface of the composite specimen. The tungsten alloy fiber oxidizes at its normal high rate when exposed to the oxidizing environment. A few thousands of an inch of matrix or cladding material, however, protected the fiber from oxidation for exposure times over 300 hours at 2000° F (1093° C).

The best superalloys may require additional oxidation protection for some turbine applications. Progress has been made in developing coatings to provide this protection, and some of the promising coatings currently available may be ideal cladding materials for fiber composites. These coatings are expected to be even more effective than the Inconel cladding used on the oxidation test specimens.

An exploratory study was made to determine the impact resistance of refractory metal fiber - superalloy composites. The impact resistance necessary for turbine buckets is not clearly related to laboratory impact test data. However, cast nickel alloys in the unnotched condition, which were run successfully in turbojet engine tests, have been reported to have low-capacity Izod impact strengths of less than 15 inch-pounds (1.69 J) at 70° and 1650° F (21° and 899° C) (refs. 6 and 7). The impact strength of the composites compared favorably with superalloys at test temperatures of 300° F (149° C) and above. Greater impact strength of composites at 250° F (121° C) and below is desirable and probably can be obtained. Impact resistance at 70° F (21° C) may be increased by decreasing the fiber content and by improving the ductility of the matrix. The volume fiber content along the length of a bucket can be varied because of the normal variation of stress along the length of turbine buckets. The effect of varying the fiber content and matrix ductility has not been investigated. The matrix composition of this study was selected on the basis of matrix-fiber compatibility, not on impact properties. Improvement of impact strength at 70° F (21° C) should be investigated with both these variables included. However, the impact strength already demonstrated probably does not prevent the use of this composite in rotating turbine buckets. This optimistic view is based on the variation in impact resistance permitted by the normal variation in operating conditions of a turbojet engine. The operating time at bucket temperatures of 250° F (121° C) and below occurs only during starting and stopping the engines and amounts to only seconds per flight. Bucket stresses are very low during starts and stops since engine rotational speeds are low. Thus, the limited total time at temperature and the low stress reduce the probability of impact failure. Furthermore, since the aircraft will be on the ground when the engine is started or stopped, the potential danger is maintenance cost rather than safety hazard. The composite material is sufficiently impact resistant at 70° F (21° C) for handling in routine maintenance. Increased impact strength of composites is expected from the research indicated earlier; however, the impact values already obtained are sufficient to warrant consideration of the composite for use in turbojet engine buckets.

SUMMARY OF RESULTS

The potential of tungsten-2-percent thoria, tungsten-5-percent rhenium-2-percent thoria, 218CS tungsten, and tungsten-1-percent thoria wire-reinforced nickel-base superalloy composites for turbine bucket applications was evaluated. The evaluation was based on the following laboratory test data: stress-rupture properties at 2000° and 2200° F (1093° and 1204° C), oxidation resistance at 2000° F (1093° C), and impact resistance from 70° to 2000° F (21° to 1093° C). The following results were obtained:

1. Composites were fabricated with excellent stress-rupture properties at 2000° F (1093° C). The 100-hour stress-rupture strength obtained for 70 volume percent fiber composites at 2000° F (1093° C) was 49 000 psi (338 MN/m^2) as compared with 11 500 psi (79 MN/m^2) for the best cast nickel alloys. The 1000-hour rupture strength obtainable (by extrapolation) for the composite was 37 000 psi (255 MN/m^2) as compared with 6000 psi (41 MN/m^2) for cast superalloys at 2000° F (1093° C).

2. The high density of the tungsten alloy wire reduced the strength advantage of the composite in comparison with that of lower density materials. However, the 70 volume percent tungsten-2-percent thoria wire-reinforced composite had a 100-hour specific rupture strength of 83 000 inches (2108 m) at 2000° F (1093° C) and an extrapolated 1000-hour specific rupture strength of 63 000 inches (1600 m). These values are over two to three times the respective values for conventional superalloys. This strength advantage represents a potential use temperature increase for turbine buckets of 200° F (93° C) above that possible with uncooled superalloy materials.

3. The strength of the composites was related to the strength of the fiber and the degree to which fiber-matrix reaction could be controlled. The depth of the fiber-matrix reaction zone was equivalent for the tungsten-5-percent rhenium-2-percent thoria and the large-diameter tungsten-2-percent thoria wire materials when exposed to 2000° F (1093° C). Both wire materials retained 75 percent of their strength for rupture in 100 hours at 2000° F (1093° C). The tungsten-2-percent thoria wire, however, was much stronger than the tungsten-5-percent rhenium-2-percent thoria wire, which resulted in the highest composite strength.

4. Advantage can be taken of the good oxidation resistance of the superalloy matrix material by surrounding the fibers with the matrix material or a cladding material so that they are not exposed to an oxidizing environment. The tungsten alloy fiber oxidized at its normal high rate when exposed to a flowing gas furnace oxidizing environment. A few thousandths of an inch of oxidation resistant material, however, was sufficient to protect the fibers from the same environment at 2000° F (1093° C) for times up to 300 hours in noncyclic furnace tests.

5. Above 300° F (149° C), the impact resistance of the composite compared favorably with that of superalloys. Lower impact strength values were obtained at temperatures below 300° F (149° C), which is below the ductile-brittle transition temperature of the tungsten wire.

Lewis Research Center,
National Aeronautics and Space Administration,
Cleveland, Ohio, September 25, 1969,
129-03.

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