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A DIRECTIONALLY SOLIDIFIED IRON-CHROMIUM-ALUMINUM-TANTALUM CARBIDE EUTECTIC ALLOY

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16. Abstract

A eutectic alloy, Fe-13.6Cr-3.7Al+9TaC, was directionally solidified in a high gradient furnace, producing a microstructure of alined TaC fibers in an oxidation resistant α-iron matrix. Tensile and stress rupture properties, thermal cycling resistance, and microstructures were evaluated. The alloy displays at 1000° C an ultimate tensile strength of 58 MPa and a 100-hour rupture life at a stress of 21 MPa. Thermal cycling to 1100° C induces faceting in the TaC fibers.

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A DIRECTIONALLY SOLIDIFIED IRON-CHROMIUM-ALUMINUM-TANTALUM CARBIDE EUTECTIC ALLOY

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SUMMARY

An iron-base eutectic alloy containing about 13.6 percent chromium, 3.7 percent aluminum and 9 percent tantalum carbide by weight was directionally solidified and tested to determine its potential as a gas turbine material. The directionally solidified alloy contains alined tantalum carbide fibers with $\langle 100 \rangle$ parallel to the direction of solidification in an α-iron matrix with $\langle 110 \rangle$ parallel to the direction of solidification. The alloy displays ultimate tensile strengths of 602 megapascals at room temperature, 58 megapascals at 1000° C, and 34 megapascals at 1100° C. Its estimated rupture stress for a 100-hour life is 21 megapascals at 1000° C and 11 megapascals at 1100° C. The alloy displays excellent oxidation-erosion resistance, but during thermal cycling to 1100° C in a burner rig, faceting of the tantalum carbide fibers occurs, and the specimens distorted because of the loads imposed by the test apparatus. Because of its relatively low high-temperature strength, the alloy is unsuitable for use in gas turbine vanes, which at present experience stresses between 35 and 70 megapascals at 1100° C.

INTRODUCTION

Stator vanes undergo the highest metal temperatures in aircraft gas turbines and must withstand stresses between 35 and 70 megapascals in an environment that can cause rapid deterioration by oxidation and erosion. The vanes are often made of cobalt-base alloys and protected by coatings. The service life of the stator vanes could be extended if a strong alloy with greater resistance to the environment could be found. Iron-base alloys containing chromium and aluminum are among the most oxidation resistant materials (ref. 1). These alloys have been used commercially for many years as electrical resistance heating elements. Often they contain about 1 weight percent of yttrium to enhance the stability of the oxides (refs. 2 to 4). Their high temperature strength, however, is generally low, and a strengthening mechanism must be introduced before they can be considered for use in the structural parts of gas turbine engines. Some possible strengthening methods are oxide dispersion and fiber or laminate reinforcement.

The objective of the program described in this report was to determine the possibility of strengthening iron-chromium-aluminum alloys by reinforcing them with alined tantalum carbide fibers. Tantalum carbide fibers have been successfully introduced into nickel and cobalt alloys by directional solidification, creating high-strength, high-temperature alloys, such as the Co+TaC and Ni+TaC in-situ composites (refs. 5 to 7).

The iron-chromium-aluminum+tantalum carbide alloy described here was directionally solidified to produce an iron-based matrix containing fibers of tantalum carbide. The composition of the matrix used is essentially that of Walter and Cline (ref. 8). Limited tensile and stress-rupture tests were performed at 1000° and 1100° C on as-solidified specimens. Thermal cycling tests and microstructures are also described.

EXPERIMENTAL PROCEDURE

Preparation of Melts

All materials were prepared from elemental constituents, except for tantalum carbide. The purity of each of the charge constituents was better than 99.8 percent. The 1500-gram mastermelts were produced in 400 cubic-centimeter zirconia crucibles. The melt chamber of the vacuum furnace was evacuated to about 2 pascals (15 μm Hg) and back filled with argon to suppress any boiling in the melt. The initial charge in the crucible was iron and tantalum carbide to which chromium and aluminum were added just before pouring at about 1650° C. The zirconia shell molds, embedded in fire-clay grog, had been preheated to about 875° C. One melt, which contained no tantalum carbide, was cast into button-head shaped tensile bars. The other melts were cast into 10-millimeter-diameter remelt bars about 140 millimeters long.

Directional Solidification

The directional solidification was performed in a radiofrequency, induction heated Bridgman furnace (fig. 1). The remelt ingots were contained in 12.5-millimeter-inside-diameter, high-purity alumina crucibles, which were heated by radiation from a graphite susceptor. Argon flowing through the apparatus at 0.5 cubic meter per hour protected the melt from contamination. The surface temperature of the melt was determined by optical pyrometry. A thermal gradient, previously determined to be about 250° C per centimeter at the liquid-solid interface (ref. 9), was generated by a water spray at the base of the furnace setup. The solidified bars were withdrawn from the furnace at controlled rates by a screw mechanism.

Microstructural Analysis

Each directionally solidified bar was surface ground along its length to provide a flat surface about 7 millimeters wide. This surface was then hand polished and examined by optical metallography to determine the quality of the alined structure and to determine what portions of the bars were suitable for machining into specimens for mechanical testing. Specimens subjected to metallographic examination for other purposes were given a final polish with alumina slurries. Since the carbide structure was clearly evident in the polished material, no etching was necessary. However, the specimens that were to undergo scanning electron microscopy were etched electrolytically in a solution of 30 percent hydrochloric acid, 7.5 percent nitric acid, 7.5 percent acetic acid, and 55 percent water by volume.

The texture of specimens was determined with a pole figure goniometer, using nickel-filtered copper radiation. The intensity of the {200} reflections of the tantalum carbide fibers and of the {110} reflections of the α-iron matrix were recorded. Specimens were prepared by sectioning perpendicular to the axis of solidification and etching the cut surface in a mixture of hydrochloric acid and hydrogen peroxide.

Mechanical Tests

The as-cast carbide-free specimens and the stress-rupture specimens from bars directionally solidified at the rate of 20 millimeters per hour conformed to figure 2(a); all other test specimens were machined according to figure 2(b).

Tensile tests were conducted in air at room temperature, 1000° and 1100° C, mostly at crosshead speeds of 0.5 millimeter per minute. On some tests a speed of 1.2 millimeters per minute was used.

Stress-rupture tests were performed in air at 1000°, 1100°, and 1200° C. The specimens were heated to the test temperature in 3 to 5 hours and soaked an additional 1 to 2 hours before loading.

Thermal Cycling

Thermal cycling was performed in a burner rig in which typical gas-turbine engine environments can be simulated (ref. 10). The test specimens were heated in a Mach 0.3, 1650° C blast of combustion products from JP-5 grade jet fuel and air. Cooling was accomplished by a Mach 0.7 blast of room-temperature air.

The thermal cycling specimens were 12.5-millimeter-diameter, 75-millimeter long bars in which a 40-millimeter exposed test section had been directionally solidified at 10 millimeters per hour. The specimens had well alined tantalum carbide fibers as determined by the examination of a ground and polished 7-millimeter-wide, longitudinal flat.

Eight specimens were exposed simultaneously in the burner rig. The specimens were secured in a holder which rotated at 450 rpm. The centerline of the specimens was 21 millimeters from the centerline of the holder and parallel to the centerline of the holder. The specimens were secured by setscrews.

In each cycle the specimens were heated for 2 minutes and reached a metal temperature of 1100° C. They were cooled to 425° C in 1 minute. The burner rig was shut down after 300, 600, 1200, and 1800 cycles to permit the specimens to be examined and weighed. Changes in the microstructure were determined after 1800 cycles.

RESULTS AND DISCUSSION

Selection of Composition

As a result of a survey of the literature and binary phase diagrams (refs. 1 to 4, 8, and 11), compositions in the iron-chromium-aluminum system were chosen for the matrix. Tantalum carbide was chosen as the reinforcement, because of previous work (refs. 5 to 7) in which fibers were grown successfully in various matrices. From preliminary button and full-scale melts, it was determined that the combination of an iron-base matrix alloyed by weight with 15 percent chromium, 4 percent aluminum, and 0.5 percent tantalum, and reinforced with 8 or 9 percent tantalum carbide could be directionally solidified as a fiber-reinforced ductile alloy. This results in an overall composition which contains (by weight percent) 13.6 chromium, 3.7 aluminum, 9 tantalum carbide, and the balance, iron. The as-cast structure for this alloy is shown in figure 3.

Solidification Rates

Metallography showed that full alinement was produced at withdrawal rates of 5 and 10 millimeters per hour and that cellular structures could begin to appear at 20 millimeters per hour. A typical alined structure of the alloy is shown in figure 4. The test sections of most specimens were solidified at the rate of 10 millimeters per hour, while portions used for gripping were solidified more rapidly. Tensile and stress-rupture specimens usually had a 45-millimeter gage section solidified at 10 millimeters per hour (note exceptions in table II) and 15-millimeter grip lengths at each end solidified at 15 millimeters per hour or faster. All specimens tested in this program failed within the gage sections.

Microstructure

The material directionally solidified in this program contained tantalum carbide fibers that varied in cross section from round to polygonal to ribbon-like (fig. 5). Conventional pole figures from the {110} reflections of α-iron matrix show a very strong ⟨110⟩ orientation (fig. 6(a)) parallel to the direction of solidification. Similarly a pole figure using the {200} reflections of tantalum carbide revealed a strong ⟨100⟩ orientation parallel to the direction of solidification (fig. 6(b)). These findings differ from those reported by Walter and Cline who found a ⟨110⟩ orientation in the tantalum carbide (ref. 8).

Mechanical Testing

Tensile test results obtained on the directionally solidified alloy and on as-cast carbide-free Fe-Cr-Al are presented in table I and compared with the tensile properties of an oxide dispersion strengthened(Fe-15Cr-6Al-2Y, with 4 vol % oxide) alloy (ref. 12). The directionally solidified, carbide-reinforced material had an ultimate strength at room temperature of 602 megapascals (which is an advantage of 117 megapascals over the as-cast carbide-free alloy) and an ultimate strength of 58 megapascals at 1000° C (an advantage of 47 megapascals). On the other hand, the oxide dispersion strengthened Fe-Cr-Al displayed an ultimate tensile strength of 38 megapascals with 7.7 percent elongation at 1093° C; and the directionally solidified, carbide-strengthened alloy, 35 megapascals with 55 percent elongation at 1100° C (ref. 12).

Again, in stress rupture, the oxide dispersion strengthened alloy (ref. 12) is stronger than the directionally solidified, carbide-reinforced alloy as shown in table II and plotted in figure 7. In the longitudinal direction the estimated stress for a 100-hour life at 1000° C is 21 megapascals and at 1100° C only 11 megapascals in the directionally solidified, tantalum carbide-containing alloy. By comparison, a 100-hour life can be obtained in the oxide dispersion strengthened alloy with about 36 megapascals at 1000° C and 28 megapascals at 1100° C. Elongation in the latter, however, is lower. But, the fiber-reinforced, directionally solidified alloy is much stronger than the as-cast matrix without tantalum carbide, which, when stressed to only 5.5 megapascals at 1100° C, fails on loading.

The changes brought about by loading at room and at elevated temperatures are evident in the microstructure. The as-directionally solidified fibers (fig. 4) break into short segments under tensile loading at room temperature (fig. 8). The failures in tensile and stress rupture at 1000° C and higher are accompanied by extensive void formation in the matrix and spheroidization of broken fibers (figs. 9 to 11).

Thermal Cycling

Thermal cycling at 1100° C was terminated at 1800 cycles, when the specimens had severely distorted under the combined effect of centrifugal and air-blast loadings (fig. 12). Examination of the microstructure showed that faceting of the fibers had occurred (fig. 13, cf. fig. 4). Faceting of tantalum carbide fibers had previously been observed by Dunlevey and Wallace (ref. 13) after a cobalt alloy had been subjected to as few as 200 2-minute cycles.

The thermal cycling resulted in weight gains of 1.04, 1.47, 1.98, and 2.41 milligrams per square centimeter of exposed surface at 300, 600, 1200, and 1800 cycles, respectively. These changes are quite small and demonstrate that the excellent oxidation-erosion resistance of the Fe-Cr-Al matrix appears not to have been degraded by the presence of tantalum carbide fibers.

CONCLUDING REMARKS

By directional solidification, highly oxidation resistant iron-chromium-aluminum alloys containing alined tantalum carbide fiber have been produced. The fibers greatly strengthen the iron base matrix, but the strength of the alloy is inferior to that produced in similar matrices by oxide dispersion strengthening. The expectation that directionally solidified Fe-13.6Cr-3.7Al+9TaC would be useful in vane applications for gas turbine engines where stresses of 35 to 70 MPa at 1000 to 1100° C are encountered, is unfulfilled.

SUMMARY OF RESULTS

Iron alloys containing chromium and aluminum additions have excellent oxidation resistance, but they have very low strength at elevated temperatures. Therefore, a program was undertaken to determine whether the high-temperature strength could be increased sufficiently to make iron-base alloys suitable for gas turbine vane applications. This involved producing by directional solidification an alloy having an α-iron matrix containing by weight 15 percent chromium and 4 percent aluminum reinforced with 8 to 9 weight percent tantalum carbide fibers. The following results were obtained:

1. When directionally solidified at rates of 10 millimeters per hour or less, with a gradient of 250° C per centimeter, the tantalum carbide was alined in fibers and ribbons in a matrix of α-iron. Strong orientations of $\langle 100 \rangle$ for the tantalum carbide and of $\langle 110 \rangle$ for the matrix were obtained parallel to the direction of solidification.

2. The directionally solidified alloy had ultimate tensile strengths of 602 megapascals at room temperature, 58 megapascals at 1000° C, and 34 megapascals at 1100° C.

3. The directionally solidified alloy had a 100-hour stress-rupture life for 21 megapascals at 1000° C and 11 megapascals at 1100° C. This is not adequate for anticipated vane stresses of 35 to 70 megapascals at 1100° C.

4. The alloy offers excellent resistance to oxidation and erosion in burner rig tests. However, the thermal cycling causes faceting of the tantalum carbide fibers.

Lewis Research Center,
National Aeronautics and Space Administration,
Cleveland, Ohio, October 20, 1976,
505-01.

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TABLE I. - TENSILE TESTS

Type of alloy	Nominal composition, wt %	Temperature, °C	Ultimate tensile strength, MPa	0.2% yield strength, MPa	Elongation, %
Directionally solidified	Fe-13.6Cr-3.7Al+9TaC	25	602	566	16
		1000	58	58	11
		1100	34	34	55
As cast	Fe-15Cr-4Al	25	481	348	35
		1000	11.4	11.0	84
Oxide dispersion strengthened[a]	Fe-15Cr-6Al-2Y(4 vol % oxide)	25	625	410	15
		1093	38	34	7.5

[a]Cross-rolled sheet, properties are averaged for specimens cut parallel to direction of final rolling pass (ref. 12).

TABLE II. - STRESS RUPTURE TESTS

Type of alloy	Nominal composition, wt %	Solidification rate, mm/hr	Temperature, °C	Stress, MPa	Life, hr	Elongation, %
Directionally solidified	Fe-13.6Cr-3.7Al+9TaC	10	1000	22.0	19.7	21
		↓	1000	51.8	.1	29
			1100	11.0	86.2	--
		↓	1100	27.5	.1	20
		20	1100	20.7	.4	35
		20	1200	20.7	.04	31
		20	1200	27.5	.02	37
As cast	Fe-15Cr-4Al	--	1000	11	(a)	--
		--	1100	5.5	(a)	--
Oxide dispersion strengthened[b]	Fe-15Cr-6Al-2Y(4 vol % oxide)	--	1093	31.0	3.2	15.8
		--	↓	31.0	58.5	8
		--		29.3	116.1	8.5
		--	↓	29.3	139.7	9.5

[a]Failed on loading.
[b]Cross-rolled sheet, properties are averaged for specimens cut parallel to direction of final rolling pass (ref. 12).

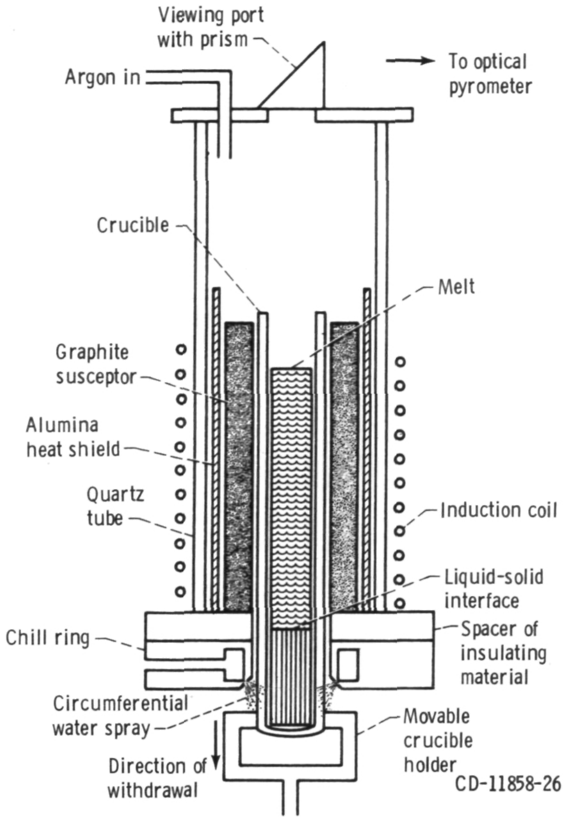


Figure 1. - Schematic representation of directional solidification apparatus.

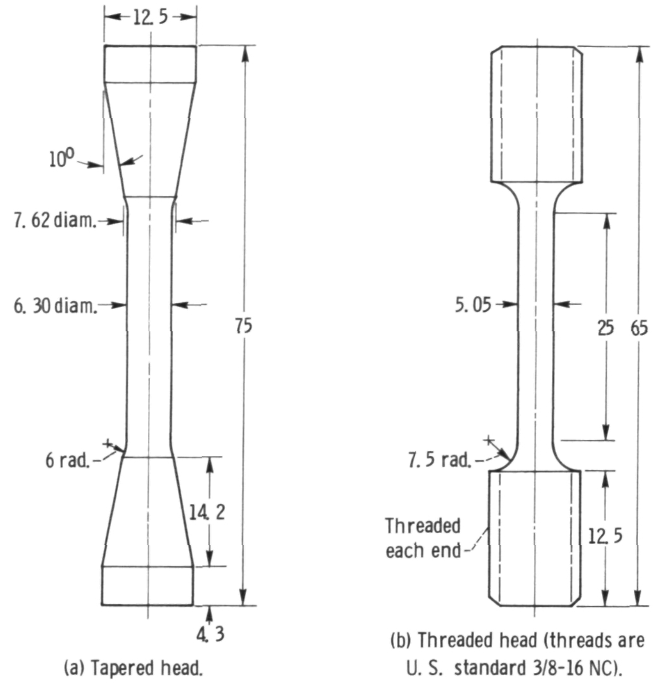


Figure 2. - Specimens used for tensile and stress rupture tests (all dimension are in mm).

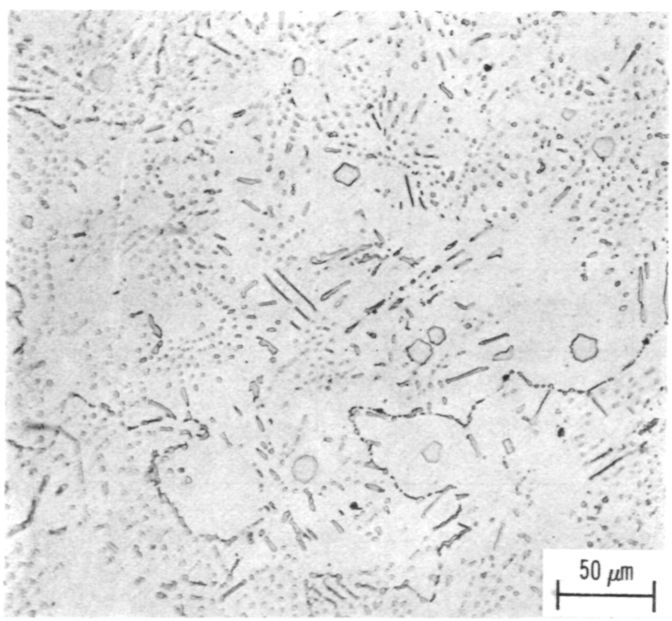
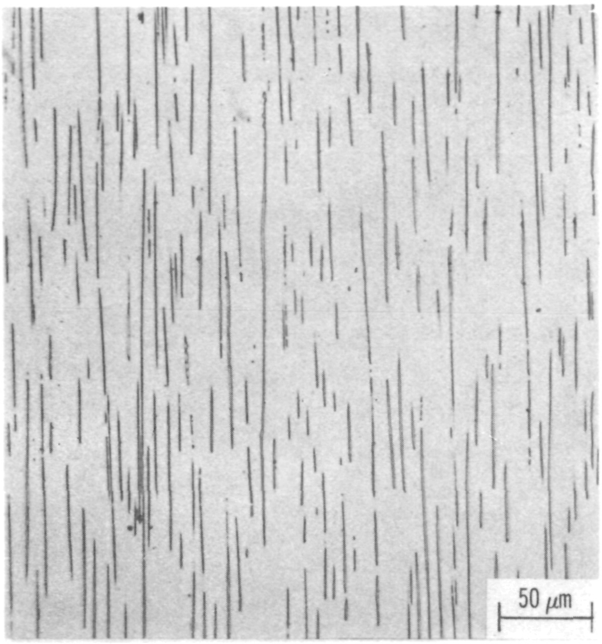
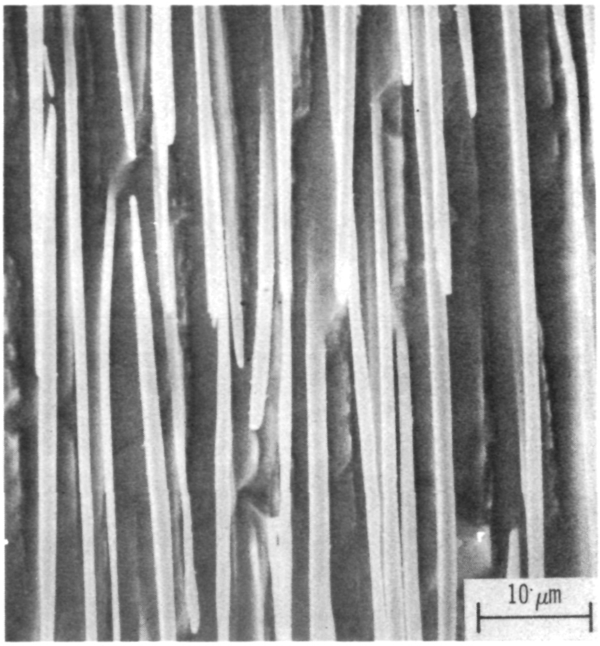


Figure 3. - Microstructure of as-cast Fe-13.6Cr-3.7Al+9TaC alloy. Etched.

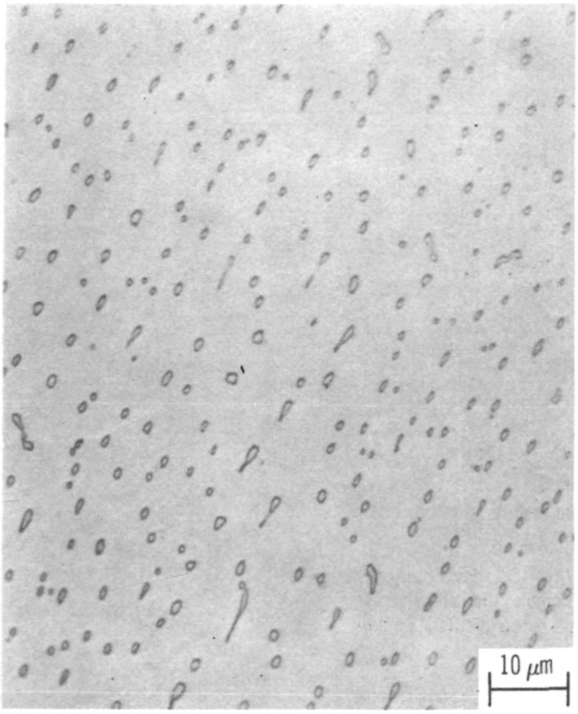


(a) Optical micrograph, unetched.

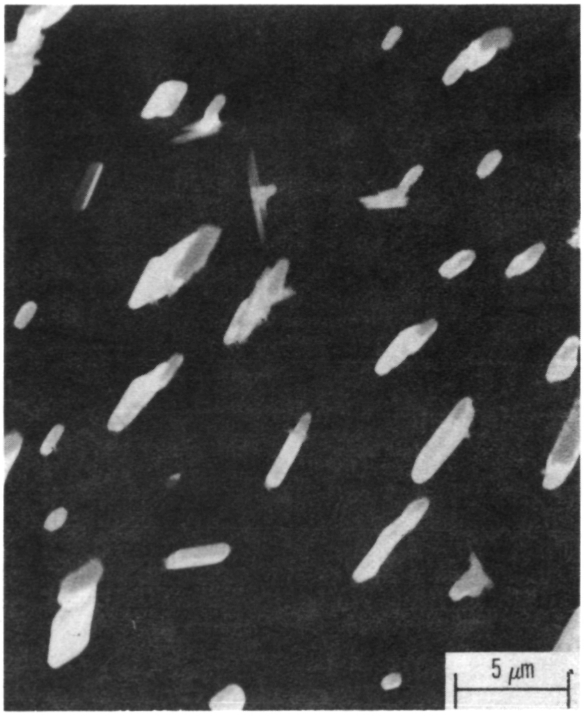


(b) Scanning electron micrograph, etched.

Figure 4. - Longitudinal sections of an Fe-13.6Cr-3.7Al+9TaC alloy specimen directionally solidified at 10 millimeters per hour.

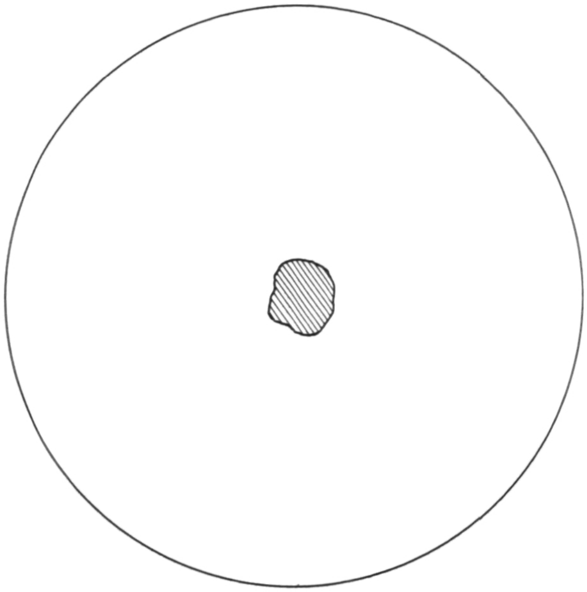


(a) Optical micrograph, unetched.

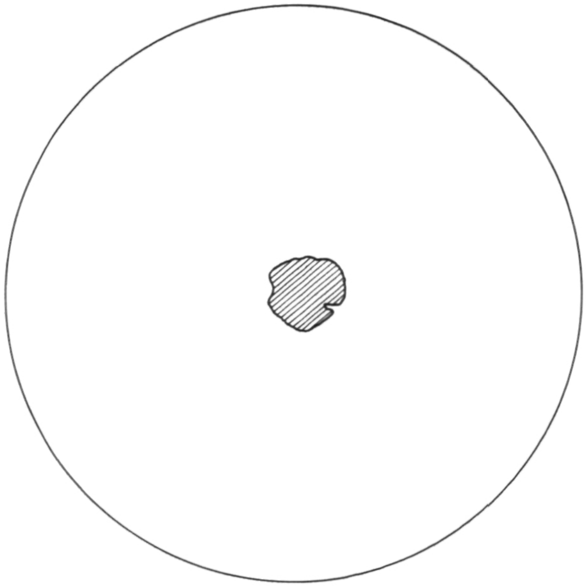


(b) Scanning electronmicrograph, etched.

Figure 5. - Cross sections of Fe-13.6Cr-3.7Al+9TaC alloy directionally solidified at 10 millimeters per hour.



(a) Reflection of {110} planes of alpha-iron matrix showing ⟨110⟩ texture.



(b) Reflection of {200} planes of tantalum carbide showing ⟨100⟩ texture.

Figure 6. - Pole figures of Fe-13.6Cr-3.7Al-9TaC alloy directionally solidified at 10 millimeters per hour. Shading indicates intense reflections. Direction of solidification is perpendicular to plane of figure.

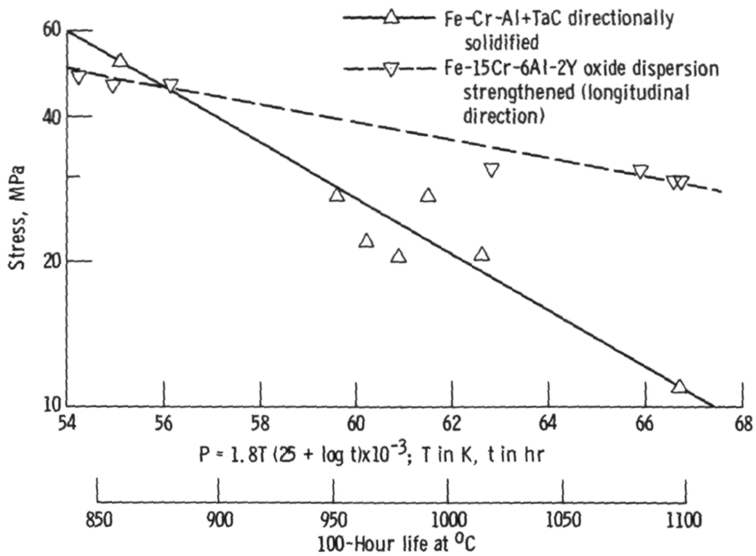


Figure 7. - Larson-Miller plot of stress rupture data for directionally solidified Fe-Cr-Al+TaC alloy and 4 volume percent oxide dispersion strengthened Fe-15Cr-6Al-2Y alloy (ref. 12).

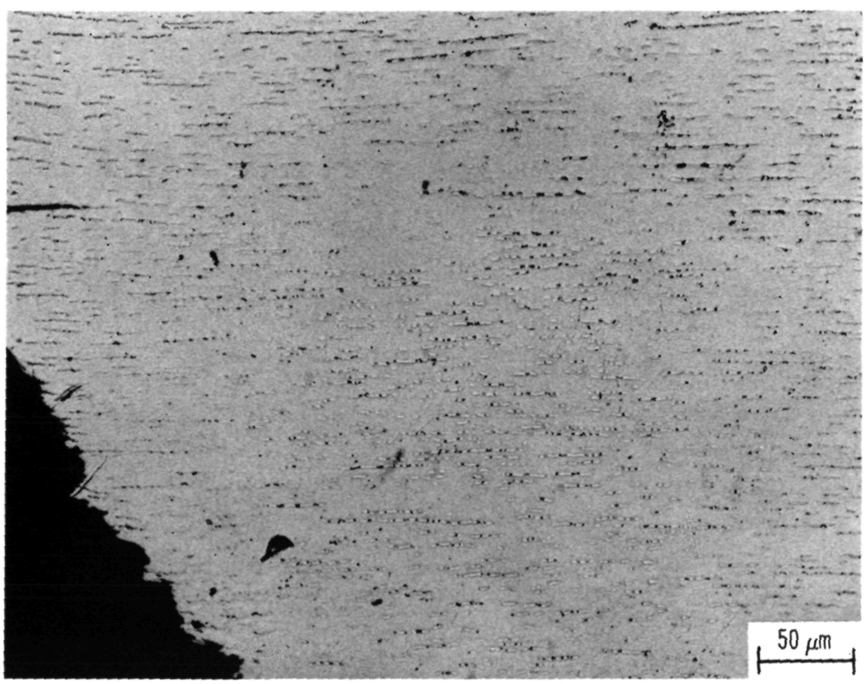


Figure 8. - Longitudinal section at failure of tensile specimen from Fe-13.6Cr-3.7Al+9TaC alloy directionally solidified at 10 millimeters per hour. Tested at room temperature; unetched.

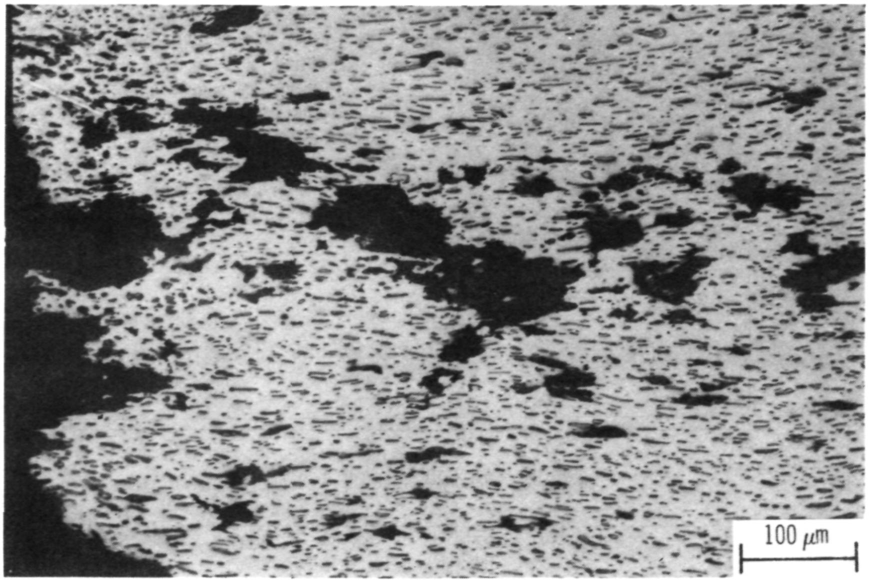
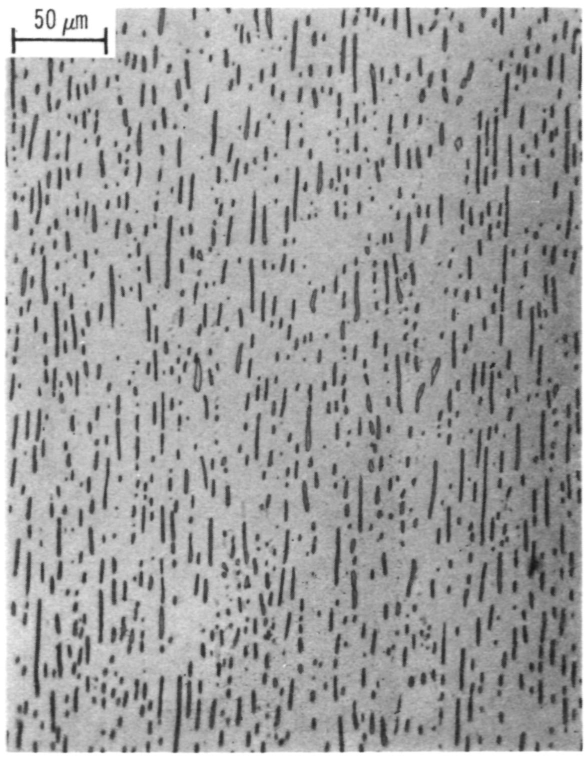


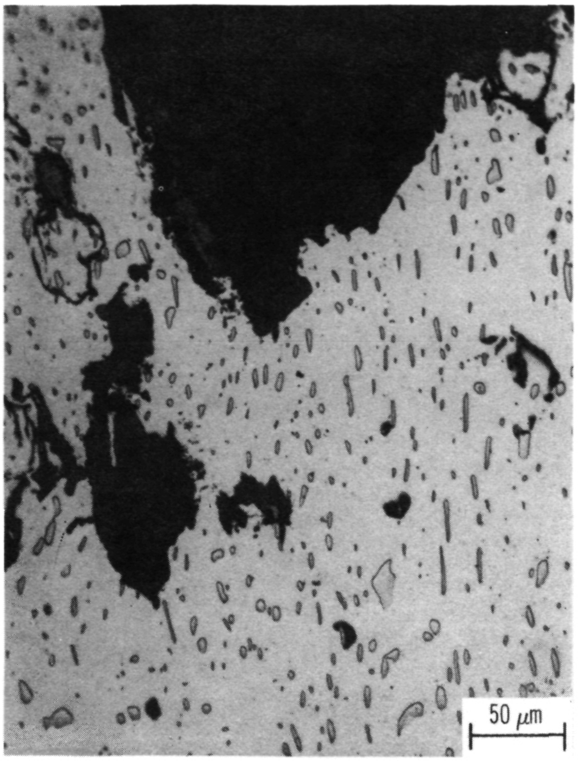
Figure 9. - Longitudinal section at failure of tensile specimen from Fe-13.6Cr-3.7Al+9TaC alloy directionally solidified at 10 millimeters per hour. Tested at 1100° C; unetched.

(a) At fracture surface.

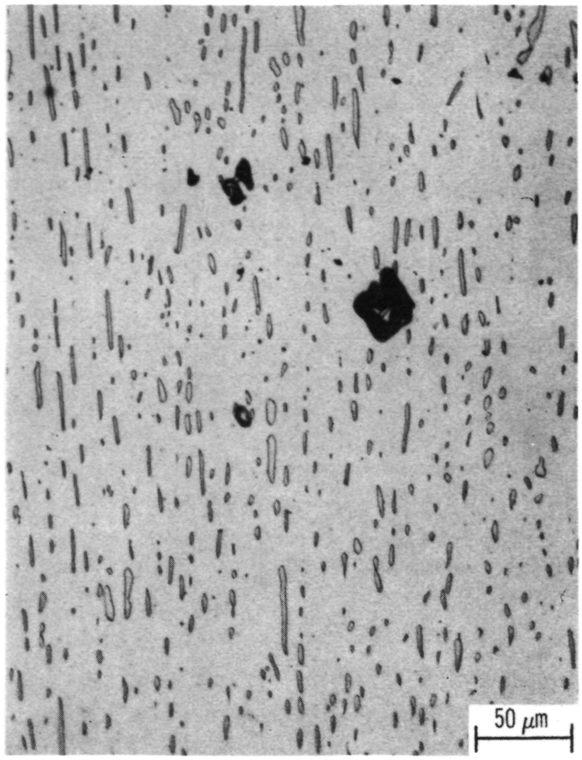


(b) At 1 centimeter from fracture surface.

Figure 10. - Longitudinal sections of stress rupture specimen failed at 1000° C in 19.7 hours under stress of 22 megapascals. Fe-13.6Cr-3.7Al+9TaC alloy directionally solidified at 10 millimeters per hour; unetched.



(a) At fracture surface.



(b) At 1 centimeter from fracture surface.

Figure 11. - Longitudinal sections of stress rupture specimen failed at 1100° C in 86.2 hours under stress of 11 megapascals. Fe-13.6Cr-3.7Al+9TaC alloy directionally solidified at 10 millimeters per hour; unetched.

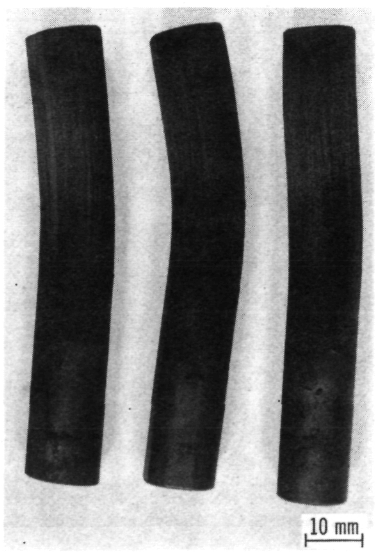
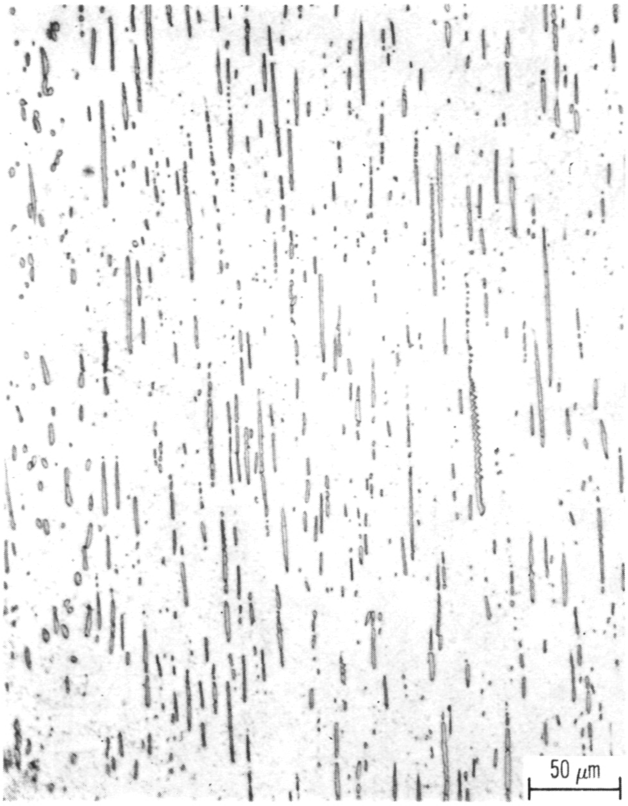
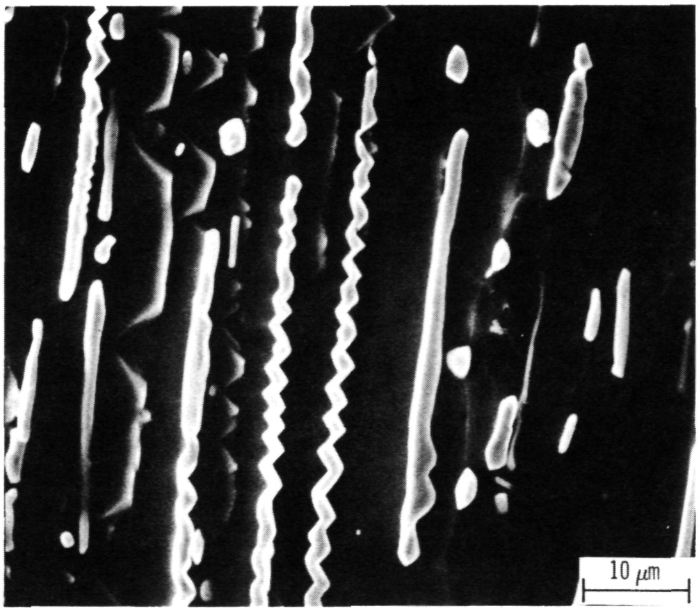


Figure 12. - Appearance of specimens of Fe-13.6Cr-3.7Al+9TaC alloy, directionally solidified at 10 millimeters per hour, after 3-minute thermal cycles between 1100° and 425° C.



(a) Optical micrograph, unetched.



(b) Scanning electron micrograph, etched.

Figure 13. - Longitudinal sections of an Fe-13.6Cr-3.7Al+9Ta alloy specimen direction- ally solidified at 10 millimeters per hour. Specimen was subjected to 1800 3-minute thermal cycles between 1100° and 425° C.

NASA-Langley, 1977 E-8890

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