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STRESS-RUPTURE STRENGTH AND MICROSTRUCTURAL STABILITY OF TUNGSTEN-HAFNIUM-CARBON-WIRE-REINFORCED SUPERALLOY COMPOSITES

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16. Abstract

Tungsten-hafnium-carbon - superalloy composites were found to be potentially useful for turbine blade applications on the basis of stress-rupture strength. The 100- and 1000-hr rupture strengths calculated for 70 vol. % fiber composites based on test data at 1090° C (2000° F) were 420 and 280 MN/m^2 (61 000 and 41 000 psi), respectively. The investigation indicated that, with better quality fibers, composites having 100- and 1000-hr rupture strengths of 570 and 370 MN/m^2 (82 000 and 54 000 psi), respectively, may be obtained. Metallographic studies indicated sufficient fiber-matrix compatibility for long-time applications at 1090° C (2000° F) for 1000 hr or more.

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SUMMARY

The potential of tungsten-hafnium-carbon- (W-Hf-C-) fiber-reinforced nickel-base superalloy composites for turbine blade applications was evaluated on the basis of stress-rupture strength. Composites were produced with stress-rupture properties superior to those of conventional cast superalloys at 1090° C (2000° F). The 100-hour stress-rupture strength for a 70-volume-percent-fiber-content composite was 420 meganewtons per square meter (61 000 psi) as compared with 80 meganewtons per square meter (11 500 psi) for the strongest superalloys. The 1000-hour stress-rupture strength obtained for the composite was 280 meganewtons per square meter (41 000 psi) as compared with 40 meganewtons per square meter (6000 psi) for superalloys. The results also implied that with an improved specimen fabrication technique, or with the use of straight and split-free fibers rather than the bent and split W-Hf-C fibers used in this investigation, stronger composites could be obtained. Composites having 100- and 1000-hour rupture strengths of 570 and 370 meganewtons per square meter (82 000 and 54 000 psi), respectively, using W-Hf-C wire should be possible. The composites were also much stronger than superalloys when density was taken into consideration. Their 100-hour specific rupture strength at 1090° C (2000° F) was over $2\frac{1}{2}$ times that of the superalloys, and the 1000-hour specific rupture strength was over $3\frac{1}{2}$ times that of the superalloys. The 70-volume-percent-W-Hf-C-fiber composite has a potential 165° C (300° F) use temperature advantage over the strongest conventional superalloys based on the 1000-hour specific rupture strength.

Metallographic studies indicated that particle coarsening occurred in the hafnium carbide (HfC) dispersion-strengthened fibers after long-time exposure at 1090° C (2000° F), and the depth of reaction between the fibers and the nickel-base-alloy matrix was 0.0046 centimeter (0.0018 in.) after 100 hours at 1090° C (2000° F). Decomposition of HfC particles in fibers contained in the composite and diffusion of carbon and hafnium into the matrix occurred for composites exposed at 1090° C (2000° F).

The results indicate that W-Hf-C - superalloy composites are potentially useful for turbine blade applications on the basis of stress-rupture strength. Turbine blades of W-Hf-C - superalloy composites could permit a 110° C (200° F) increase in engine operating temperature compared with superalloy blades without causing a severe weight penalty.

INTRODUCTION

The developers of gas turbines have recognized for years the many benefits to be realized by going to higher engine operating temperatures. The use of superalloys for turbine blade materials for reasonable life times is currently limited to a material temperature of approximately 980° to 1010° C (1800° to 1850° F) because of the stresses imposed on the blade. It seems unlikely that further substantial high-temperature strength improvements will be made with superalloys by conventional metallurgical techniques. To some extent the deficiencies of high-temperature materials can be overcome by design. Superalloys have been used in turbine blades with operating gas temperatures of 1260° C (2300° F) or more by cooling the blades with bleed air from the compressor. However, further increases in performance for turbines will require materials with increased operating temperature capability because further gains possible from cooling of presently available materials are limited. Therefore, an important objective of materials research for turbine engines is to develop materials that will permit higher operating temperatures.

Both solid and cooled blade designs are attractive for higher gas temperature turbines. If solid uncooled blades had sufficient strength, they could be used for later stages which are currently cooled and thereby improve performance. Cooled blades made from improved materials could be operated at higher temperatures with less cooling air than is required for today's materials. One of the materials under study for use in higher temperature turbine blades is a composite consisting of a superalloy matrix reinforced by refractory-metal fibers.

Composites of tungsten alloys and superalloys which have been investigated at a number of laboratories (refs. 1 to 5) have the potential of combining the high-temperature strength of a refractory metal with the oxidation resistance, toughness, and ductility of a superalloy. Previous work at the Lewis Research Center demonstrated that 70-volume-percent-fiber composites could be produced to have 100- and 1000-hour rupture strengths at 1090° C (2000° F) of 338 and 255 meganewtons per square meter (49 000 and 37 000 psi) (ref. 5). Figure 1 is a plot of the 1000-hour stress-rupture strength divided by the material density as a function of temperature for superalloys and for a tungsten-2-percent-thoria ($W\text{-}2ThO_2$) - superalloy composite having a fiber content of 70 percent by volume. The horizontal band in the figure represents a range of strength-density ratios that might be required for turbine blades for an advanced turbojet engine. Superalloys are currently limited to about 980° C (1800° F), while the composite can be used at the same strength-density ratio at 1090° C (2000° F). The fiber composite contains 70 volume percent fiber and as a result is quite dense. It should be noted, however, that this fiber concentration would be present only in the critical section of the turbine blade (that section of the blade where the combination of stress and temperature is most severe), as described in reference 6. The average fiber content of

a blade would be expected to be less than half this value, or less than 35 volume percent. A potential further improvement in composite strength and lowering of fiber content for turbine blade application can be achieved through the use of stronger tungsten alloy fibers than were used in the past. Improved high-strength tungsten alloy fibers have been made available as part of a continuing contract effort by the Lewis Research Center to obtain higher strength fiber materials.

The object of the present investigation was to determine the potential for turbine blade application of superalloy composites in terms of the 1090° C (2000° F) stress-rupture strength that can be obtained by using improved high-strength fibers of tungsten-hafnium-carbon (W-Hf-C). Composites consisting of a nickel- (Ni-) base alloy containing 25 percent W, 15 percent chromium (Cr), 2 percent titanium (Ti), and 2 percent aluminum (Al) and reinforced with up to 60 volume percent W-Hf-C fibers were fabricated and evaluated. The composite specimens and W-Hf-C fibers were evaluated in stress-rupture at 1090° C (2000° F). Metallographic and electron beam probe analyses were conducted to determine the extent of reaction between the W-Hf-C fibers and the nickel-base alloy for exposure times up to 300 hours at 1093° C (2000° F).

MATERIALS, APPARATUS, AND PROCEDURE

Fiber Material

The fabrication history of the W-Hf-C fiber used in this investigation is reported in reference 7. The fiber was developed as part of a continuing program by the NASA Lewis Research Center to provide fibers with improved properties for use in composites. The fiber was experimentally developed and not optimized. The diameter of the fiber used was 0.038 centimeter (0.015 in.), and the fiber was received in the as-drawn, cleaned, and straightened condition. The nominal chemical composition in weight percent of the fiber was 0.03 C, 0.37 Hf, and the balance W. The fiber contained a high percentage of longitudinal splits and was not fully straightened. The radius of curvature for the W-Hf-C fiber was calculated to be 7 centimeters (3 in.), compared with 100 centimeters (40 in.) for fully straightened 218CS tungsten fibers.

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 in weight percent of the nickel alloy was 56 Ni, 25 W, 15 Cr, 2 Al, and 2 Ti. The nickel alloy was vacuum cast and atomized into a fine powder with a

particle size 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 in making the powder.

Composite Specimen Fabrication

Composites containing the tungsten alloy fibers 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 the ammonium salt of alginic acid in water. The composition, viscosity, pH, and density of the metal slip are listed in table II.

Composite specimens were prepared by inserting continuous-length W-Hf-C fibers into a nickel alloy tube containing a wire screen at the bottom and several layers of filter paper, as shown in figure 2. The nickel alloy tube was connected to a rubber hose that was attached to a mechanical pump. The nickel alloy tube was then placed on a vibrating table, and slip was poured into the fiber bundle while the tube was vibrated. As the nickel alloy powder settled to the bottom of the bundle, excess liquid was siphoned 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 stopped, and a vacuum was applied to the tube to remove any liquid remaining in the casting. The specimen was removed from the tube and dried in air for approximately 24 hours at 60° C (140° F). The specimens were then sintered at 820° C (1500° F) 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 into closely fitting Inconel tubes with a wall thickness of 0.036 centimeter (0.014 in.). 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 140 meganewtons per square meter (20 000 psi), first at 820° C (1500° F) for 1 hour and then at 1090° C (2000° F) for 1 hour. Composite specimens were made with fiber contents ranging from 15 to 60 volume percent. Fully densified specimens of over 99 percent theoretical density were produced and machined into test specimens.

Specimen Configuration

The as-pressed specimens were machined to the specimen dimensions shown in figure 3 by centerless grinding. Button-head specimens were machined to have a test-section length of 2.54 centimeters (1.00 in.). The test-section diameters varied from

0.3099 to 0.3282 centimeter (0.1220 to 0.1292 in.). The machined test section contained exposed surface wires of W-Hf-C.

Stress-Rupture Tests

Stress-rupture tests on single fibers of W-Hf-C were conducted in a stress-rupture apparatus specifically designed for the testing of up to four fibers simultaneously. A detailed description of this apparatus may be found in reference 8. A photograph of the inside of the chamber is shown in figure 4. 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 was evacuated to a measured vacuum of approximately 7×10^{-3} to 1×10^{-4} newton per square meter (5×10^{-5} to 1×10^{-6} torr). The furnaces were then turned on and allowed to stabilize at the desired test temperature. After stabilization the weights were applied to the wire specimens by lowering the retractable support. Tests were conducted at 1090° and 1200° C (2000° and 2200° F) for periods up to 600 hours.

Stress-rupture tests on composite specimens were conducted in conventional creep machines, in a helium atmosphere to limit oxidation. Tests were conducted at 1090° C (2000° F) for rupture times up to 400 hours.

Metallographic Study

Stress-rupture specimens were examined metallographically to determine the depth of the reaction zone between the nickel alloy matrix and the W-Hf-C wire as a function of time and temperature and to determine the fiber content of the specimens. The depth of reaction was measured optically on transverse sections of composite specimens at a magnification of 150. 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 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.

Replica electron micrographs were taken of transverse sections of wire and composite specimens in an area adjacent to the fracture edge. A two-step technique was used to replicate the specimens. The specimens were first replicated with a solution of 0.025 percent Mowital dissolved in chloroform and, after drying, they were reinforced with 1.5 percent Parlodion in amyl acetate. The two plastic layers were then dry-

stripped with pressure-sensitive cellulose tape, shadowed with platinum-carbon, and reinforced with a 0.01-micrometer layer of carbon. The replica was cut into grid size squares and placed in an amyl acetate solution to remove the pressure-sensitive cellulose tape and to dissolve the Parlodion. The Mowital-carbon replica was then viewed in an electron microscope, and photographs were taken at magnifications of 8000 and 28 000.

Electron Microprobe Studies

Electron microprobe studies were conducted on transverse sections of composite specimens. These studies were made to determine whether there was elemental diffusion between the W-Hf-C wire and the matrix, to identify these elements, and to determine the extent to which they diffused. A continuous traverse was made across specimen cross sections, at a rate of 10 micrometers per minute, in order to determine whether the wires contained traces of Al, Cr, C, Hf, Ti, Ni, and W. The probe was also operated to scan for secondary electron backscatter images and X-ray fluorescence images of the elements Al, Cr, Hf, Ni, Ti, and W. Photographs were taken of all images on the probe display screen at a magnification of 500.

RESULTS

Fiber Material

Stress-rupture properties. - The stress-rupture properties of the W-Hf-C fibers used in this investigation are listed in table III. The fibers tested were taken from several ingots and spools. The T and N designations for the ingots refer to the tail and nose sections of extruded ingots from which the fibers were drawn. Variations in time to rupture at a specific stress exist from ingot to ingot and from spool to spool. Figure 5 is a plot of the time to rupture as a function of the stress on the W-Hf-C fibers tested at 1090° and 1200° C (2000° and 2200° F). The curves are fitted to the data by least squares. The stresses to cause rupture in 100 and 1000 hours at 1090° C (2000° F) were 1060 and 890 meganewtons per square meter (154 000 and 129 000 psi), respectively. The 1200° C (2200° F) test results indicated 100- and 1000-hour rupture strengths for the fiber of 715 and 590 meganewtons per square meter (103 000 and 85 000 psi), respectively. Figure 6 is a plot of the reduction in area at fracture as a function of the time to rupture for fibers tested at 1090° C (2000° F). The plot shows a steady decrease in ductility at fracture with increasing time to rupture. At 1200° C

(2200° F) the reduction in area at fracture is relatively constant at about 10 to 20 percent, as shown in table III, and no trend as a function of rupture time is observed.

Microstructure. - Figure 7 shows electron micrographs of a W-Hf-C fiber tested at 1090° C (2000° F). The fiber failed in stress-rupture after 12.9 hours. Figure 7(a) is a transverse section at the edge of the fiber, and figure 7(b) is a transverse section near the center of the fiber. Similar structures and particle distribution and size are seen for both sections.

Electron micrographs of a fiber which failed in stress-rupture in 586.7 hours are shown in figure 8. An edge section is shown in figure 8(a), and sections near the center of the wire are shown in figures 8(b) and (c). The edge and center sections in figures 8(a) and (b) appear to have larger particles than those in the fiber specimen shown in figure 7, which was exposed for only 12.9 hours. There were areas in the center portion of the fiber, however, such as that shown in figure 8(c), which had a structure equivalent to that observed for the short-time exposure (fig. 7).

The fiber specimen of figure 7 (short-time exposure) failed in stress-rupture with a very ductile fracture, while the fiber specimen shown in figure 8 (long-time exposure) failed in a much less ductile manner. The electron micrograph study indicated that some particle coarsening occurred in the fiber exposed for the long time and that this particle coarsening may have resulted in a less ductile material. Particle coarsening rates were calculated for hafnium carbide (HfC) particles contained in a tungsten alloy in reference 9. These calculated results indicated that the HfC particles should be very stable at 1090° C (2000° F). Results obtained in this investigation indicate that HfC particles contained in the tungsten alloy fiber are not stable for long-time exposure at 1090° C (2000° F). The difference in stability of the HfC particles observed in this investigation as compared to that calculated from reference 9 may be related to stored energy in the fiber due to the large amount of cold-working employed in the wire drawing process. The recrystallization temperature of this material could be lowered because of the large amount of cold work given the fiber. In a previous investigation (ref. 10) grain broadening was observed for W-Hf-C fibers exposed at 1090° C (2000° F) for long time periods. Particle growth could be accelerated as grain boundaries sweep over the particles.

Matrix Material

Vacuum-cast stress-rupture specimens for the nickel-base alloy matrix were obtained from the master melt for making the powder and tested in a past program; the results are reported in reference 1. The specimens were tested in stress-rupture at 1090° and 1200° C (2000° and 2200° F) in a helium atmosphere. A plot of stress against time to rupture for the material tested is shown in figure 9. The 100-hour rupture

strength for the nickel alloy was found to be 23 meganewtons per square meter (3200 psi) at 1090° C (2000° F).

Composite Material

Stress-rupture properties. - The stress-rupture properties obtained for the composites are given in table IV. The composite specimens had fiber contents ranging from about 15 to 60 volume percent and were tested at stress levels from 138 to 379 meganewtons per square meter (20 000 to 55 000 psi). 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 entire load during the stress-rupture test, 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 11. The stress-carrying capability of the fiber in the nickel alloy matrix material was thus calculated and is given in table IV. Figure 10 is a plot of the stress on the W-Hf-C fiber contained in the nickel alloy matrix as a function of time to rupture at 1090° C (2000° F). The least-squares fit of the data indicates that the stress for rupture in 100 hours is approximately 600 meganewtons per square meter (87 000 psi), while that for rupture in 1000 hours is approximately 400 meganewtons per square meter (59 000 psi). Compared with as-received fibers tested in vacuum, approximately 57 percent of the stress-rupture strength of the W-Hf-C fiber was retained in the composite specimens that ruptured in 100 hours and 45 percent in those that ruptured in 1000 hours. The strength retention for the W-Hf-C fiber was expected to be low because of the manner in which the specimens were fabricated and the presence of fiber splits. Because of wire bend and the method of fabricating specimens, fiber ends were present on the surface of the specimen test section. The fibers on the periphery of the test section pulled out during the test and did not contribute their full strength on the composite. Fibers pulled out because of misalinement to the tensile axis of the specimen. The magnitude of the strength losses resulting from fiber pullout and fabrication defects is treated in more detail in the section DISCUSSION.

Fiber-matrix reaction. - The depth of reaction between the matrix and the fibers was measured for each specimen tested in stress-rupture. The results of the measurements are listed in table IV and plotted as a function of rupture time in figure 11. A least-squares fit of the W-Hf-C fiber data is shown and also data from reference 5 for W-2ThO_2 fibers in the same matrix material. The depth of reaction after 100 hours of exposure was 0.0046 centimeter (0.0018 in.). The degree of reaction between the

matrix and the W-Hf-C fibers was similar to that observed for composites containing W-2ThO_2 fibers and having the same matrix composition.

Microstructure study. - Replica electron micrographs of a composite specimen tested at 400 meganewtons per square meter (30 000 psi) are shown in figure 12. Figure 12(a) shows the microstructure of the specimen at the fiber-matrix interface. Particles believed to be HfC and large grains are formed at the edge of the fiber near the interface. Figure 12(b) shows the structure of the diffusion zone away from the interface. The structure is similar to that formed near the fiber-matrix interface. A section near the center of the wire is shown in figure 12(c). Large particles and grains are not observed as is the case near the edge of the fiber and in the diffusion zone. The same section is shown at a higher magnification in figure 12(d). Some particle coarsening may have taken place since the particles shown are larger than those of figure 7 (short-time exposure for fiber not contained in composite).

Electron microprobe study. - Backscatter electron and X-ray images of a composite specimen exposed for 324.8 hours at 1090° C (2000° F) are shown in figure 13. Figure 13(a) shows the electron backscatter image. The fiber-matrix interface and alloyed fiber zone can be seen. The depth of this zone was 0.0079 centimeter (0.0031 in.). Figures 13(b), (e), and (f) are X-ray raster micrograph images for nickel, tungsten, and chromium. A detectable chromium concentration was observed in the tungsten alloy fiber reaction zone; however, no detectable nickel concentration was observed. X-ray raster micrographs for aluminum and titanium (figs. 13(c) and (d)) did not show any detectable concentrations of these elements in the diffusion zone of the fiber. X-ray raster micrograph hafnium images were also obtained. Figure 14 shows hafnium images for composite specimens exposed to 1090° C (2000° F) for various time periods. Figure 14 indicates that diffusion of hafnium out into the matrix increases with time at temperature. Figure 14(c), for example, shows a buildup of a high hafnium content at the fiber-matrix interface after exposure for 148.3 hours, while figure 14(d) shows some high hafnium content areas in the matrix after exposure for 324.8 hours.

The results of the electron probe study indicated some loss of titanium, aluminum, nickel, and chromium from the matrix to the tungsten fibers, but the depth of penetration into the fibers was relatively small. The depth of penetration zones measured optically was generally much greater than that indicated by the electron probe study for these elements.

The most significant finding of the electron probe study was the results obtained for the hafnium and carbon traces. Figures 15 and 16 are plots of concentration as a function of distance from the fiber-matrix interface for carbon and hafnium, respectively. The profiles show the variation in relative concentration of either carbon or hafnium rather than the actual concentration of each element. Data for specimens which were exposed at 1090° C (2000° F) for three different time periods are plotted in the figures. Figure 15 shows that the concentration gradient for carbon between the fiber and the

matrix decreases with time of exposure, which indicates diffusion of carbon from the fiber into the matrix. Figure 16 shows a similar trend for hafnium, which also diffused into the matrix. The hafnium and carbon composition of the fiber implies that some excess carbon was available as an interstitial in the tungsten but that all of the hafnium was available for the formation of HfC particles in the tungsten. The X-ray raster micrograph images and electron probe traces thus indicate HfC decomposition and diffusion of free carbon and hafnium into the matrix. The nickel alloy matrix contained 0.0032 weight percent carbon, while the fiber had a carbon content almost 10 times that amount, 0.03 weight percent. The results imply that it may be beneficial to add hafnium to the matrix or increase its carbon content in an attempt to inhibit the loss of HfC in the fiber.

DISCUSSION

Current Composite Stress-Rupture Properties

The stress-rupture properties of composites containing varying concentrations of fiber can be determined through the use of the plot shown in figure 10. The stress on the fiber to cause rupture in a specific time to rupture (determined from the curve presented in fig. 10) is multiplied by the volume fraction of fiber content in the specimen. From the data shown in figure 10, for example, a composite containing 70 volume percent W-Hf-C fibers would be expected to have a 100-hour stress-rupture strength at 1090° C (2000° F) of 420 meganewtons per square meter (61 000 psi), that is, 0.70×600 meganewtons per square meter ($0.70 \times 87\ 000$ psi). The foregoing method also was used to calculate the 1000-hour rupture strength of a composite containing 70 volume percent W-Hf-C fibers. The 1000-hour rupture strength for the composite was calculated to be 285 meganewtons per square meter (41 000 psi).

Figure 17 compares the 100- and 1000-hour rupture strengths of various refractory-fiber - nickel-base-alloy composites containing 70 volume percent fibers with those of conventional superalloys at 1090° C (2000° F). The W-Hf-C-fiber composite is the strongest composite system investigated to date and represents an improvement over W-2ThO_2-fiber composites. The 100-hour rupture strength of the W-Hf-C composite is 25 percent greater than that for the W-2ThO_2 composite, and the 1000-hour rupture strength of the W-Hf-C composite is about 10 percent greater than that of the W-2ThO_2 composite. The W-Hf-C composite is over five times as strong as conventional superalloys for rupture in 100 hours and over seven times as strong for rupture in 1000 hours at this temperature.

The density of the composite material is greater than that of superalloys 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. The

W-Hf-C fiber has a density of 19.3 grams per cubic centimeter (0.697 lb/in.^{3}), while the nickel-base matrix material used in this investigation has a density of 9.15 grams per cubic centimeter (0.330 lb/in.^{3}). A composite containing 70 volume percent fibers has a density approximately 1.9 times that of most superalloys. A comparison of the specific strength properties of composites and conventional superalloys is therefore significant. A plot comparing the 100- and 1000-hour specific rupture strengths of conventional superalloys and of refractory-fiber - superalloy composites containing 70 volume percent fibers is presented in figure 18. Even when density is taken into account, all the composite materials are stronger than superalloys. Prior to this investigation the strongest refractory-superalloy composite was the W-2ThO_2-fiber composite, which has a 100-hour specific strength of 2100 meters (83 000 in.) and a 1000-hour specific strength of 1600 meters (63 000 in.). The results of this investigation indicate that W-Hf-C - superalloy composites show an improvement in specific strength over W-2ThO_2-fiber composites. The 100-hour specific rupture strength for the W-Hf-C composite is 2650 meters (104 000 in.), and the 1000-hour specific rupture strength is 1800 meters (70 000 in.). The 100-hour specific rupture strength for the composite is over twice that for conventional cast superalloys. The 1000-hour specific rupture strength for the composite is over $3\frac{1}{2}$ times that for conventional cast superalloys.

Potential Composite Stress-Rupture Properties

The stress-rupture strengths obtained for the W-Hf-C-fiber composites studied in this investigation are believed to be lower than those which can be achieved with this system. Higher stress-rupture strengths than those obtained were expected because of the compatibility of the W-Hf-C fiber with the matrix. The results obtained in this investigation showed that the degree of fiber-matrix reaction for the W-Hf-C composites was similar to that observed for composites containing W-2ThO_2 fibers. The rupture strengths for W-2ThO_2 fibers contained in the composite for 100- and 1000-hour lives, respectively, were 75 and 59 percent of the rupture strengths of as-received fibers tested in vacuum (ref. 5). Further, composite rupture strengths for 218CS-tungsten - and W-2ThO_2-reinforced superalloy composites have been related to depth of reaction (ref. 1). Since the degree of reaction with the matrix was similar for W-Hf-C and W-2ThO_2 fibers, it would be expected that their strength retentions would also be similar. Based on depth of reaction, the W-Hf-C fibers contained in the composite would be expected to have 100- and 1000-hour rupture strengths of 800 and 520 meganewtons per square meter (116 000 and 76 000 psi), respectively, rather than the values of 600 and 400 meganewtons per square meter (87 000 and 59 000 psi) obtained in this investigation. As noted in the section RESULTS, the W-Hf-C fibers were bent and misalined to the tensile axis of the test specimens, and fiber ends were present on the surface of the test

section because of the machining process as well as the fabrication method used to obtain specimens. The fibers on the surface of the test section pulled out of the matrix during the test and did not contribute their full strength to the composite. Another factor lowering the strength contribution of the W-Hf-C fibers was the presence of wire splits. Figure 19 shows a transverse section of an as-pressed composite specimen. The majority of fibers contained in the composite exhibit fiber splits which resulted from the fiber drawing process prior to composite fabrication. The number of fibers containing splits and the width and depth of the fiber splits varied for the composite specimens tested. The area of fiber reacted as a function of time at temperature is increased because of the increase in fiber surface area exposed to the matrix due to the split, as indicated in figure 19. Since increased fiber reaction lowers composite strength, elimination of fiber splits would increase fiber and composite strength. Specimens containing split-free fibers or moderately split fibers would be expected to be stronger than specimens containing severely split fibers. The majority of specimens tested contained only split fibers. Some specimens did, however, contain some split-free fibers, and these specimens were stronger than the specimens containing only split fibers. A more realistic appraisal of the potential of W-Hf-C-fiber composites can be gained by considering only those specimens containing some split-free fibers and by taking into account the fact that the surface fibers do not contribute to composite strength.

The specimens selected for this appraisal are listed in table V. It was assumed that the surface fibers did not contribute to composite strength. The stress carried by the remaining fibers was found by dividing the load placed on the specimen by the area occupied by the fibers since it was assumed that the fibers carry all of the load. The effective fiber content shown in the table is that volume of fiber which carries the load and thus does not include the surface fibers. The number of split-free fibers was determined for each specimen, and this value was divided by the number of load carrying fibers to arrive at the percent of split-free fibers present in each specimen. Only composite specimens containing 15 or more percent split-free fibers were considered, so that the effect of split fibers on the stress-rupture strength of the fibers would be reduced. The majority of composite specimens listed in table V contained approximately 25 percent split-free fibers. One specimen contained 92 percent split-free fibers. This specimen had the largest positive deviation from the least-squares fit of the rupture data, as shown in figure 10. Figure 20 is a plot of the stress carried by the fibers as a function of time to rupture at 1090° C (2000° F) for the specimens listed in table V. A least-squares fit of the data indicates a 100-hour rupture strength of 810 meganewtons per square meter (117 000 psi) and a 1000-hour rupture strength of 530 meganewtons per square meter (77 000 psi). The rupture strength of W-Hf-C fibers contained in the composite based on these results and assumptions was 76 percent of as-received fiber rupture strength from vacuum tests for rupture in 100 hours and 60 percent for rupture in 1000 hours; these values are in agreement with the retention values obtained for

W-2ThO_2 fibers contained in the same matrix (ref. 5). These strengths for W-Hf-C fibers are assumed to be more truly representative of the potential of this fiber and are the strength values which will be used for the following comparisons.

Figure 21(a) compares the calculated 100-hour rupture strengths of various refractory-fiber - nickel-base alloy composites containing 70 volume percent fiber with those of conventional superalloys at 1090° C (2000° F). The 100-hour rupture strength for the W-Hf-C composite containing 70 volume percent fiber was calculated to be 570 meganewtons per square meter (82 000 psi). Based on the assumption cited previously, that is equivalent to 0.7 × 810 meganewtons per square meter (0.7 × 117 000 psi). The W-Hf-C composite is the strongest fiber composite system and represents a significant improvement over W-2ThO_2-fiber composites. A 65-percent improvement in the 100-hour rupture strength is obtained when W-Hf-C fibers are used instead of W-2ThO_2 fibers (570 compared with 340 MN/m^2 (82 000 compared with 49 000 psi)). The W-Hf-C composite is almost seven times as strong as conventional superalloys at this temperature and is stronger than most refractory-metal alloys.

A similar type comparison was made for the 1000-hour rupture strength of these materials and is shown in figure 21(b). A 45-percent improvement in the 1000-hour rupture strength is obtained for W-Hf-C composites compared with W-2ThO_2-fiber composites (370 compared with 255 MN/m^2 (54 000 compared with 37 000 psi)). The W-Hf-C composite is about nine times as strong as conventional superalloys at this temperature and time to rupture.

Bar graphs comparing the 100- and 1000-hour specific rupture strength properties of conventional superalloys and of refractory-fiber - superalloy composites containing 70 volume percent fibers and tested at 1090° C (2000° F) are shown in figure 22. Prior to this investigation the strongest refractory-fiber - superalloy composite was the W-2ThO_2-fiber composite having a 100-hour specific strength of 2100 meters (83 000 in.) and a 1000-hour specific strength of 1600 meters (63 000 in.). The results of this investigation indicate that W-Hf-C - superalloy composites show an improvement in specific strength over W-2ThO_2-fiber composites. The 100-hour specific rupture strength for the W-Hf-C composite is 3500 meters (140 000 in.), and the 1000-hour specific rupture strength is 2300 meters (92 000 in.). The W-Hf-C composite is over three times as strong on a specific strength basis as superalloys for rupture in 100 hours and over four times as strong for rupture in 1000 hours.

A comparison of the stress-density ratios of superalloys and composite materials indicates the potential of composite materials for turbine blade use. The 70-volume-percent-W-Hf-C-fiber composite has a calculated specific 1000-hour rupture strength of 2350 meters (92 000 in.) at 1090° C (2000° F). The strongest conventional superalloys have a specific 1000-hour rupture strength of 2350 meters (92 000 in.) at 930° C (1700° F). The W-Hf-C composite based on this comparison has a 165° C (300° F) use temperature advantage over the strongest conventional superalloys.

Potential Application

Fiber-reinforced composite materials have been the subject of intensive research because they offer the potential for substantially improved properties compared to currently used materials. Their use could permit increased performance in many engineering systems. One of the systems that is limited by the capability of current materials is the turbojet engine. Designers would prefer to increase operating temperatures of such engines to increase efficiency and reduce pollution. Increased strength at elevated temperatures may be achieved with W-Hf-C-fiber - superalloy composites compared with superalloys. This increased strength in turn would permit an increase in turbine operating temperature.

A persistent concern held about tungsten-fiber - superalloy composites has been what their response to thermal fatigue will be. Composite thermal fatigue properties are being studied at this laboratory. As yet, insufficient data have been obtained to permit definitive conclusions to be drawn.

The other major concern is that there is a large weight penalty associated with their use despite their superior strength-density ratios compared with superalloys. However, the turbine blade weight for a solid blade of tungsten-fiber - superalloy composite need not greatly exceed that for a similar blade made from a conventional superalloy if reasonable measures are taken in design and fabrication of the composite. Two variables can be used to overcome the high density of the refractory-alloy fiber. The fiber content can be varied along the blade span so as to tailor strength to that needed, and the blade airfoil thickness near the base can be slightly reduced compared with a superalloy blade because of the improved strength-density ratios of the composite. Blades with varying fiber content can be fabricated by using conventional diffusion bonding techniques. Fiber-free superalloy foil and monolayer superalloy matrix composite tape, each cut to the appropriate contours, can be stacked and bonded in closed dies.

Fiber content variation or selective reinforcement can reduce the average fiber content significantly. Sample blade density calculations made to illustrate the effectiveness of selective reinforcement are presented in reference 6. The average fiber content of the blade was less than one-half the maximum fiber content at any one cross section of the blade.

Midspan stresses in a typical solid superalloy blade range from 103 to 138 meganewtons per square meter (15 000 to 20 000 psi). The stresses generated in rotating blades are density dependent. The stress-density ratios at blade midspan for a typical superalloy with a density of 8.3 grams per cubic centimeter (0.30 lb/in.^{3}) would range from 1200 to 1700 meters (47 000 to 67 000 in.). It was assumed that a blade material must have a stress-density ratio of 1525 meters (60 000 in.), near the middle of this range, for rupture in 1000 hours. The fiber content necessary for a 1000-hour rupture-strength - density ratio of 1525 meters (60 000 in.) for the W-Hf-C - superalloy com-

posite described in this investigation would be 36 percent, the maximum fiber content necessary at any one cross section of the blade. The average fiber content of the blade would be less than 18 percent. A W-Hf-C-fiber - superalloy composite having a fiber content of 18 percent and a matrix similar to that used in this study would have a density of 10.9 grams per cubic centimeter (0.396 lb/in.^{3}). High-strength superalloys which have about a 980° C (1800° F) use temperature limit as turbine blades have densities as high as 8.97 grams per cubic centimeter (0.325 lb/in.^{3}). The W-Hf-C - superalloy blade density is only 22 percent higher than this value. The matrix material used in this investigation had a high density, 9.15 grams per cubic centimeter (0.33 lb/in.^{3}). The composite density can also be lowered by using lower density matrix materials. A nickel-base alloy having the same compatibility with tungsten fibers as that of the nickel-base alloy used in this investigation and having a density of 8.09 grams per cubic centimeter (0.292 lb/in.^{3}) was used as a matrix material for the composite property studies of reference 1. A W-Hf-C-fiber composite using this material as a matrix for a turbine blade application would have a density of 10.0 grams per cubic centimeter (0.363 lb/in.^{3}), which is only 11 percent higher than the value for the superalloys (8.97 g/cm^3 (0.325 lb/in.^{3})). Superalloys are currently limited to 980° C (1800° F), while composites with the same strength-density ratio can be used at 1090° C (2000° F), 110° C (200° F) higher than superalloys.

CONCLUDING REMARKS

Considerable scatter in rupture properties was obtained for composite specimens and was related to fiber defects. Much of the fiber used for reinforcement contained axial splits, and all the fibers were bent because of residual curvature from drawing and spooling. However, straight, defect-free, stronger fibers are believed to be obtainable by optimizing the fiber drawing process. When composite data were corrected to account for fiber imperfections, the resulting composite properties approached those predicted from defect-free fiber data. For example,

(1) The 100-hour stress-rupture strength calculated for a 70-volume-percent-fiber composite at 1090° C (2000° F) was 570 meganewtons per square meter (82 000 psi). The 1000-hour rupture strength of the composite at the same temperature was 370 meganewtons per square meter (54 000 psi).

(2) The 70-volume-percent-fiber composite would have a 100-hour specific rupture strength of 3550 meters (140 000 in.) and a 1000-hour specific rupture strength of 2350 meters (92 000 in.) at 1090° C (2000° F).

(3) The 70-volume-percent-W-Hf-C-fiber composite would have a 165° C (300° F) use temperature advantage over the strongest conventional superalloys based on the 1000-hour specific rupture strength.

(4) Turbine blades of W-Hf-C - superalloy composites would offer a 110° C (200° F) increase in engine operating temperature without a severe weight penalty if the fiber content were varied along the blade span so as to tailor strength to that needed.

SUMMARY OF RESULTS

The potential for turbine blades of superalloy composites using improved high-strength fibers of tungsten-hafnium-carbon (W-Hf-C) was determined in terms of the 1090° C (2000° F) stress-rupture strength. The following results were obtained:

1. Composites containing up to 60 volume percent fibers were fabricated having high stress-rupture properties at 1090° C (2000° F) compared with superalloys. The 100-hour stress-rupture strength calculated for 70-volume-percent-fiber composites at 1090° C (2000° F) was 420 meganewtons per square meter (61 000 psi) as compared with 80 meganewtons per square meter (11 500 psi) for the strongest cast nickel alloys. The 1000-hour stress-rupture strength obtainable (by extrapolation from data obtained up to 400 hr) for the composite was 285 meganewtons per square meter (41 000 psi).

2. The high density of the tungsten alloy fiber reduced the strength advantage of the composite in comparison with that of lower density materials. However, the 70-volume-percent-W-Hf-C-fiber-reinforced composite would have a 100-hour specific rupture strength of 2650 meters (104 000 in.) and an extrapolated 1000-hour specific rupture strength at 1090° C (2000° F) of 1780 meters (70 000 in.). The 100-hour specific rupture strength for the composite was over twice that for conventional cast superalloys. The 1000-hour specific rupture strength for the composite was over $3\frac{1}{2}$ times that for conventional cast superalloys.

3. The W-Hf-C - superalloy composite stress-rupture data at 1090° C (2000° F) reported in this investigation were higher than data reported in the literature for any other composite.

4. The hafnium carbide (HfC) dispersion-strengthened fiber exhibited particle coarsening after long-time exposure at 1090° C (2000° F).

5. The depth of reaction between the W-Hf-C fiber and the nickel-base alloy matrix was 0.0046 centimeter (0.0018 in.) after 100 hours of exposure at 1090° C (2000° F).

6. Decomposition of HfC particles in fibers contained in the composite and diffusion of carbon and hafnium into the matrix occurred for composites exposed at 1090° C (2000° F).

Lewis Research Center,
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TABLE I. - CHEMICAL ANALYSIS OF NICKEL ALLOY METAL POWDER

[Concentrations in wt. %.]

Aluminum	Carbon	Chromium	Phosphorus	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

TABLE II. - METAL POWDER SLIP COMPOSITION AND PROPERTIES

Composition, wt. %			Viscosity of infinite shear		Slip density, g/cm^3	Percent of 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

TABLE III. - STRESS-RUPTURE PROPERTIES OF TUNGSTEN-HAFNIUM-CARBON FIBER

Ingot (a)	Spool	Test temperature		Stress		Life, hr	Reduction in area, percent
		°C	°F	MN/m^2	psi		
4027	1	1090	2000	[b]1300	189 000	4.4	44.2
				[b]1290	187 000	10.3	58.4
				[b]1230	178 000	21.1	23.2
				[b]1210	175 000	19.1	35.0
				[b]1150	167 000	61.5	44.5
				[b]1110	161 000	108.3	18.0
				1040	150 000	247.9	11.9
				1000	145 000	449.7	20.6
				986	143 000	286.1	18.1
				896	130 000	520.6	(c)
		1200	2200	[b]918	133 000	28.3	15.3
				[b]841	122 000	42.9	21.9
				[b]765	111 000	104.3	11.5
				[b]689	100 000	188.4	28.5
				620	90 000	705.6	14.4
4034T	1	1090	2000	1170	170 000	64.0	27.2
				1100	160 000	92.3	31.7
	3	1090	2000	1100	160 000	65.4	46.7
				758	110 000	586.7	10.7
	4	1090	2000	1100	160 000	113.8	24.2
				965	140 000	140.5	24.3
	5	1090	2000	1100	160 000	129.9	14.4
				965	140 000	211.6	10.3
4034T[d]	2	1090	2000	1170	170 000	22.5	27.2
				1100	160 000	85.2	31.7
				827	120 000	345.6	11.6
4034N	1	1090	2000	1100	160 000	78.6	15.4
				1170	170 000	44.6	20.2
4034N	2	1090	2000	1170	170 000	46.7	32.6
				1100	160 000	114.7	23.7
				1030	150 000	152.8	12.9
	3	1090	2000	1170	170 000	49.2	26.4
				1100	160 000	78.0	17.8
4035T	3	1090	2000	1170	170 000	31.2	38.7
				1100	160 000	33.6	38.6
	4	1090	2000	1100	160 000	47.1	27.2
	5	1090	2000	1170	170 000	7.5	70.1
				1100	160 000	34.8	27.2
		1200	2200	758	110 000	33.1	16.6
				689	100 000	49.7	16.6
	10	1090	2000	1170	170 000	12.9	78.8
				1100	160 000	72.2	26.0
	6	1200	2200	689	100 000	79.0	22.5
				655	95 000	99.1	12.9
4035N	10	1090	2000	1100	160 000	78.6	----
	12	1090	2000	1100	160 000	65.0	23.7
	13	1090	2000	1100	160 000	57.7	19.0
	14	1090	2000	1100	160 000	48.5	23.7
		1200	2200	758	110 000	23.6	25.0
				689	100 000	97.7	17.9
4037N	1	1090	2000	1170	170 000	7.5	30.0
				1100	160 000	37.2	28.9

[a]Designations T and N refer, respectively, to tail and nose sections of ingot.
[b]Data from ref. 10.
[c]Test stopped before fracture.
[d]Bottom half of tail section.

TABLE IV. - STRESS RUPTURE PROPERTIES FOR TUNGSTEN-HAFNIUM-CARBON-WIRE - NICKEL-ALLOY COMPOSITES

Specimen	Specimen diameter		Composite stress		Fiber stress		Rupture life, hr	Fiber content, vol. %	Reaction zone depth	
	cm	in.	MN/m^2	psi	MN/m^2	psi			cm	in.
570	0.3203	0.1261	220	32 000	1180	172 000	0.7	18.8	0.0008	0.0003
569	.3155	.1242	138	20 000	885	128 000	9.3	15.6	.0013	.0005
619	.3139	.1236	310	45 000	875	127 000	15.7	35.5	.0018	.0007
587	.3137	.1235	345	50 000	825	120 000	20.8	41.7	.0020	.0008
584	.3254	.1281	345	50 000	825	120 000	24.6	41.8	.0053	.0021
590	.3124	.1230	172	25 000	615	89 000	32.8	28.1	.0020	.0008
597	.3139	.1236	379	55 000	820	119 000	59.0	46.4	.0033	.0013
617	.3150	.1240	276	40 000	605	88 000	68.0	45.6	.0053	.0021
592	.3152	.1241	207	30 000	620	90 000	73.7	33.3	.0041	.0016
616	.3150	.1240	276	40 000	655	95 000	78.7	42.3	.0041	.0016
591	.3152	.1241	207	30 000	715	104 000	84.2	28.8	.0041	.0016
582	.3162	.1245	310	45 000	725	105 000	95.7	42.7	.0046	.0018
625	.3162	.1245	207	30 000	495	72 000	128.2	41.9	.0066	.0026
631	.3132	.1233	310	45 000	560	81 000	137.7	55.5	.0058	.0023
578	.3254	.1281	345	50 000	660	96 000	148.3	52.1	.0053	.0021
589	.3099	.1220	310	45 000	525	76 000	159.2	59.1	.0053	.0021
581	.3127	.1231	241	35 000	525	76 000	165.2	46.2	.0061	.0024
580	.3142	.1237	310	45 000	570	83 000	170.1	54.5	.0058	.0023
620	.3152	.1241	207	30 000	455	66 000	230.6	45.1	.0066	.0026
613	.3157	.1243	207	30 000	515	75 000	268.7	39.9	.0056	.0022
557	.3282	.1292	207	30 000	410	59 000	[a]324.8	51.2	.0079	.0031
598	.3117	.1227	172	25 000	345	50 000	413.6	50.2	.0084	.0033

[a]Test stopped before fracture.

TABLE V. - STRESS-RUPTURE PROPERTIES FOR TUNGSTEN-HAFNIUM-CARBON-WIRE - NICKEL-ALLOY COMPOSITES CONTAINING MORE THAN 15 PERCENT SPLIT-FREE FIBERS WITH SURFACE FIBERS NEGLECTED

[Test temperature, 1090° C (2000° F).]

Specimen	Composite stress		Effective fiber content, vol. %	Calculated fiber stress		Rupture life, hr	Percent of fibers which are split-free
	MN/m^2	psi		MN/m^2	psi		
587	345	50 000	33.9	1110	147 000	20.8	39
584	345	50 000	32.9	1050	152 000	24.6	25
597	379	55 000	35.3	1080	156 000	59.0	92
592	207	30 000	27.7	745	108 000	73.7	26
591	207	30 000	24.9	830	120 000	84.2	24
582	310	45 000	37.7	820	119 000	95.7	54
578	345	50 000	46.6	740	107 000	148.3	15
580	310	45 000	42.6	730	106 000	170.1	24

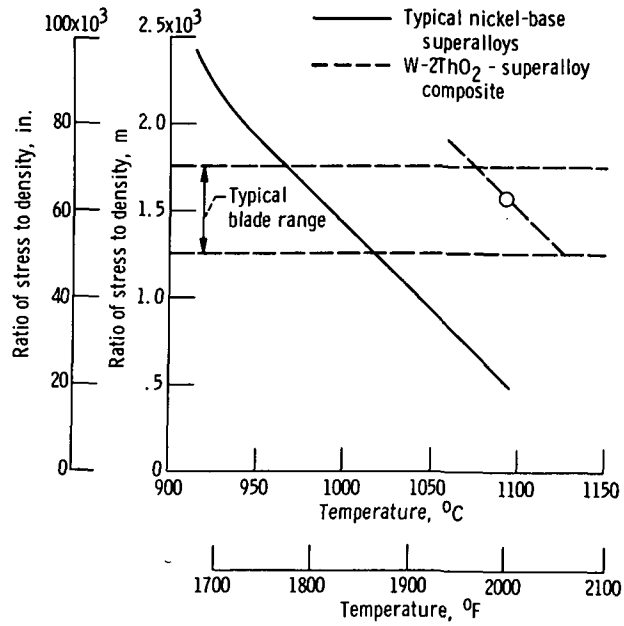


Figure 1. - Potential turbine blade use temperatures for 1000-hour life.

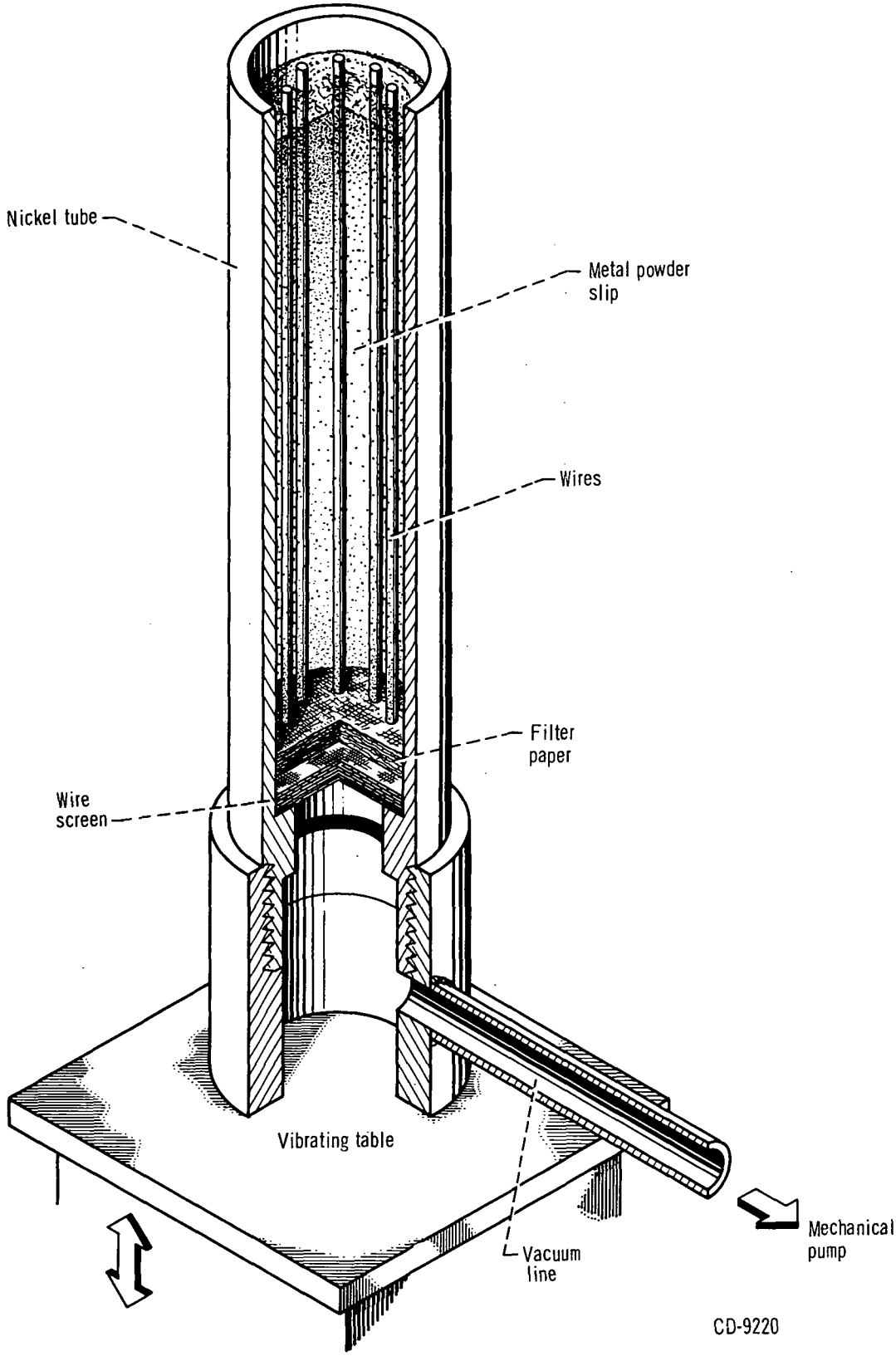


Figure 2. - Slip casting apparatus.

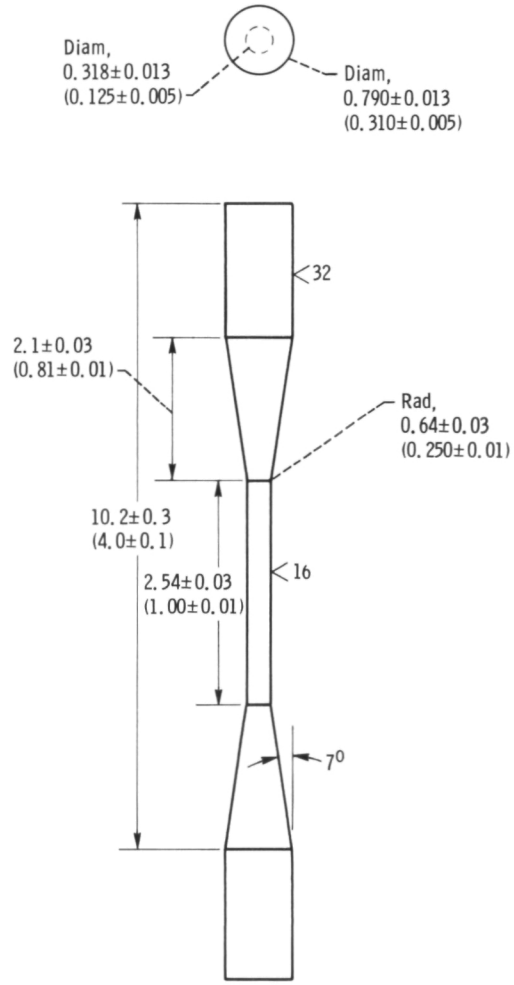


Figure 3. - Sketch of stress-rupture specimen. (Dimensions are in centimeters (in.).)

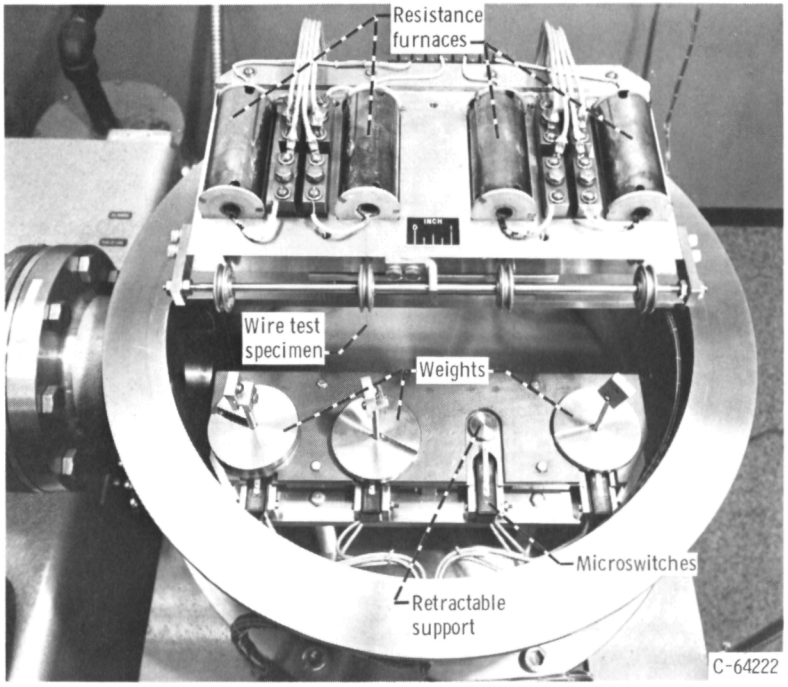


Figure 4. - Fiber stress-rupture testing apparatus.

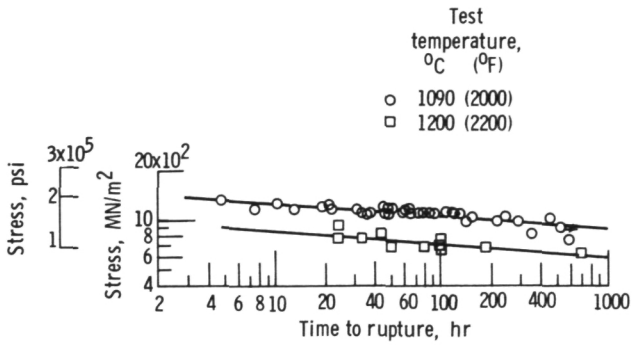


Figure 5. - Time to rupture as function of stress for W-Hf-C wire at 1090° and 1200° C (2000° and 2200° F).

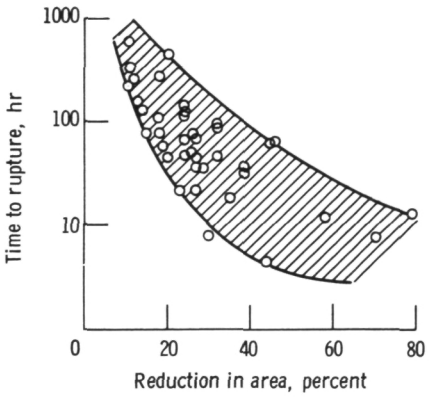
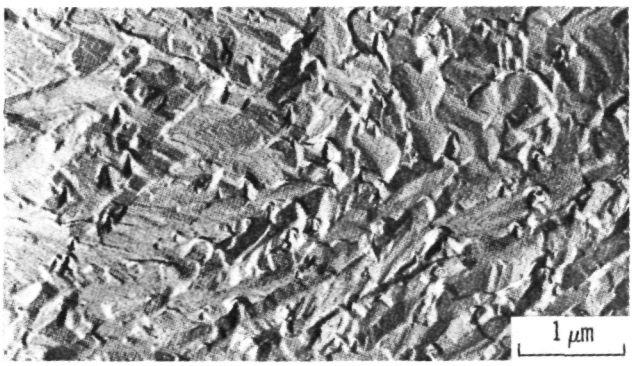
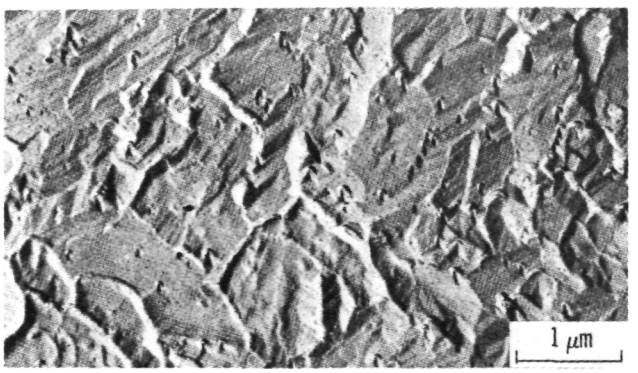


Figure 6. - Reduction in area as function of time to rupture for W-Hf-C wire at 1090° C (2000° F),



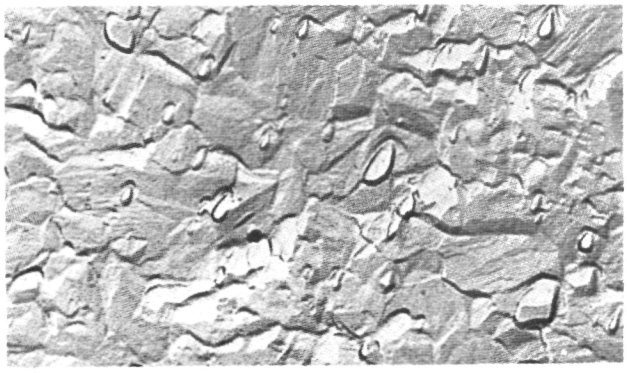
(a) Edge section.



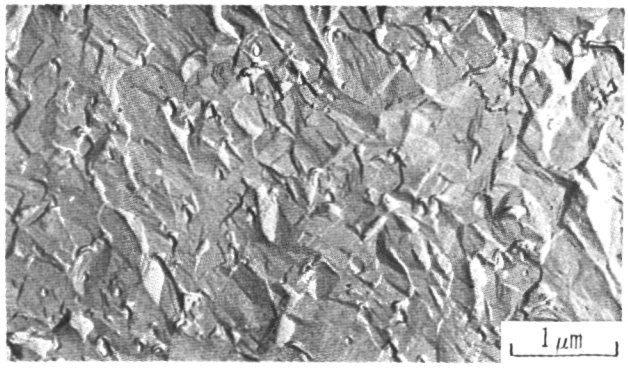
(b) Center section.

Figure 7. - Replica electron micrographs of W-Hf-C wire tested at 1090° C (2000° F). Stress, 1170 meganewtons per square meter (170 000 psi); time to rupture, 12.9 hours. X28 000.

(a) Edge section.



(b) Center section, area 1.



(c) Center section, area 2.

Figure 8. - Replica electron micrographs of W-Hf-C wire tested at 1090° C (2000° F). Stress, 758 meganewtons per square meter (110 000 psi); time to rupture, 586.7 hours. X28 000.

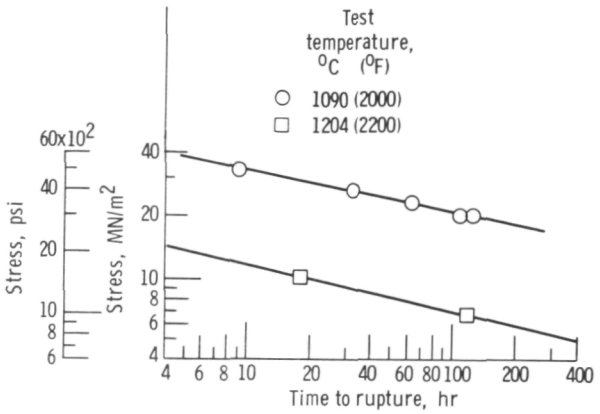


Figure 9. - Stress as function of rupture life for nickel-base alloy matrix (ref. 1).

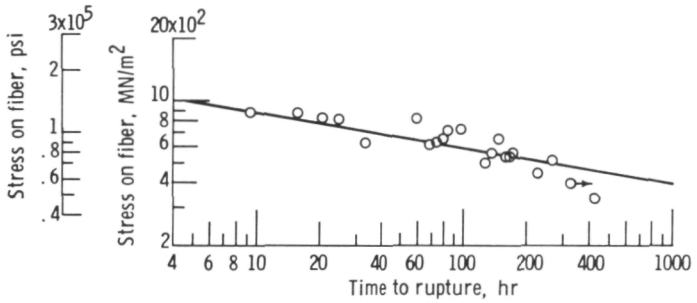


Figure 10. - Stress on fiber as function of rupture time for W-Hf-C composites at 1090° C (2000° F).

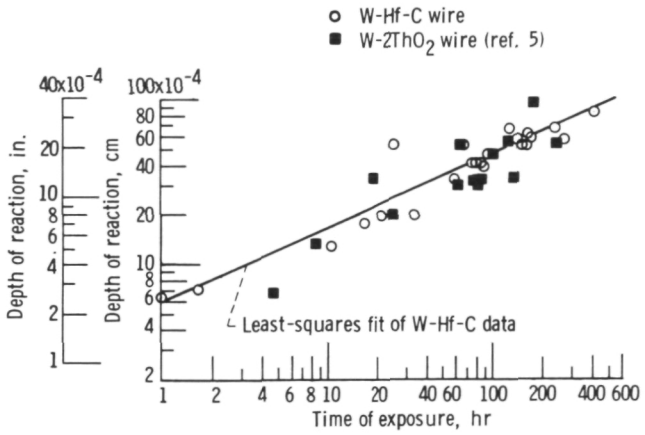
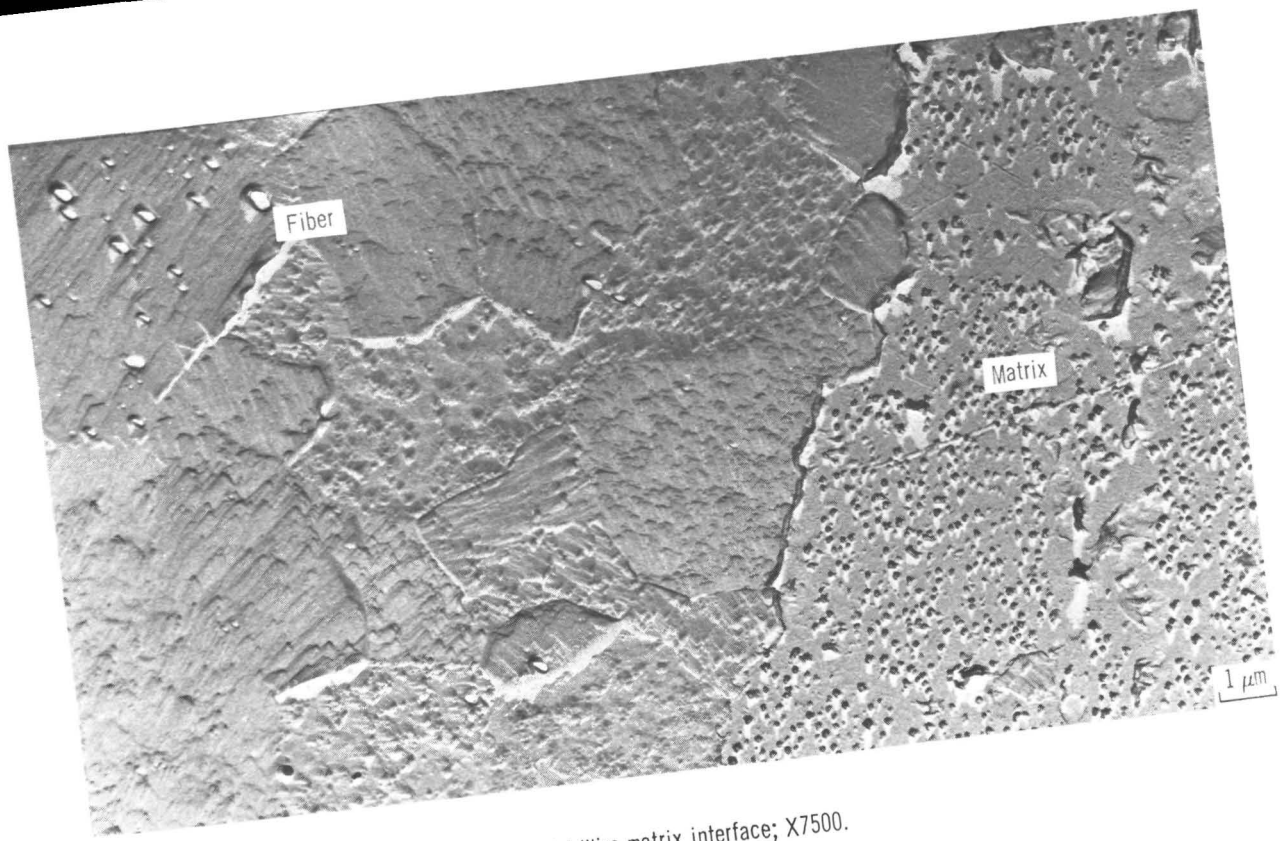
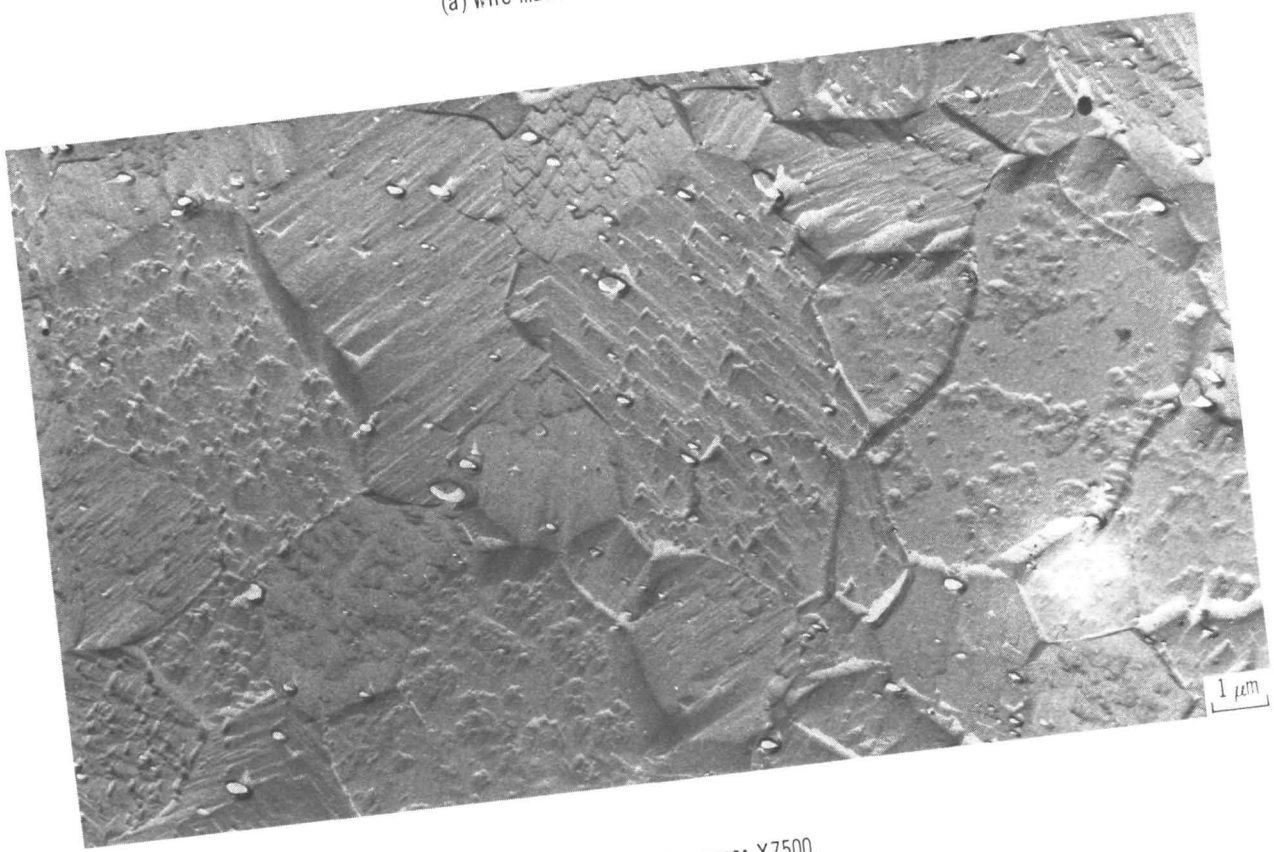


Figure 11. - Depth of reaction as function of time of exposure for W-Hf-C composites at 1090° C (2000° F).

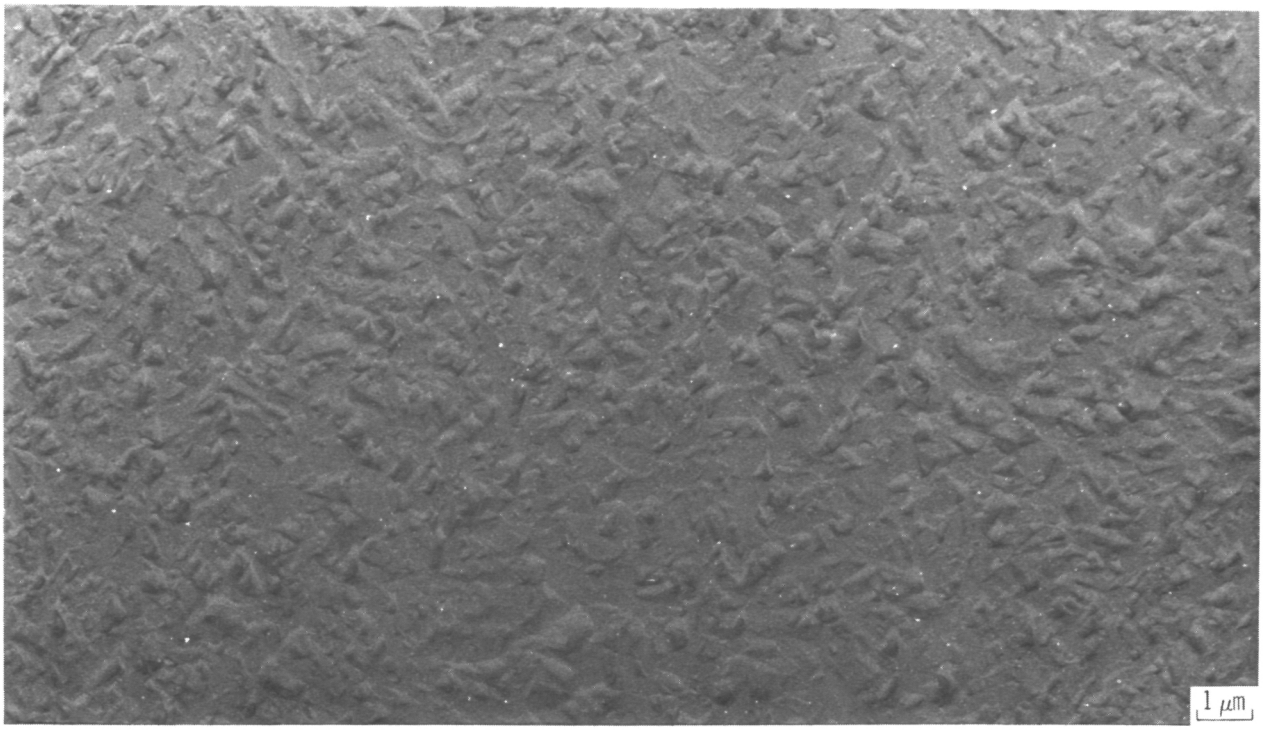


(a) Wire-matrix interface; X7500.

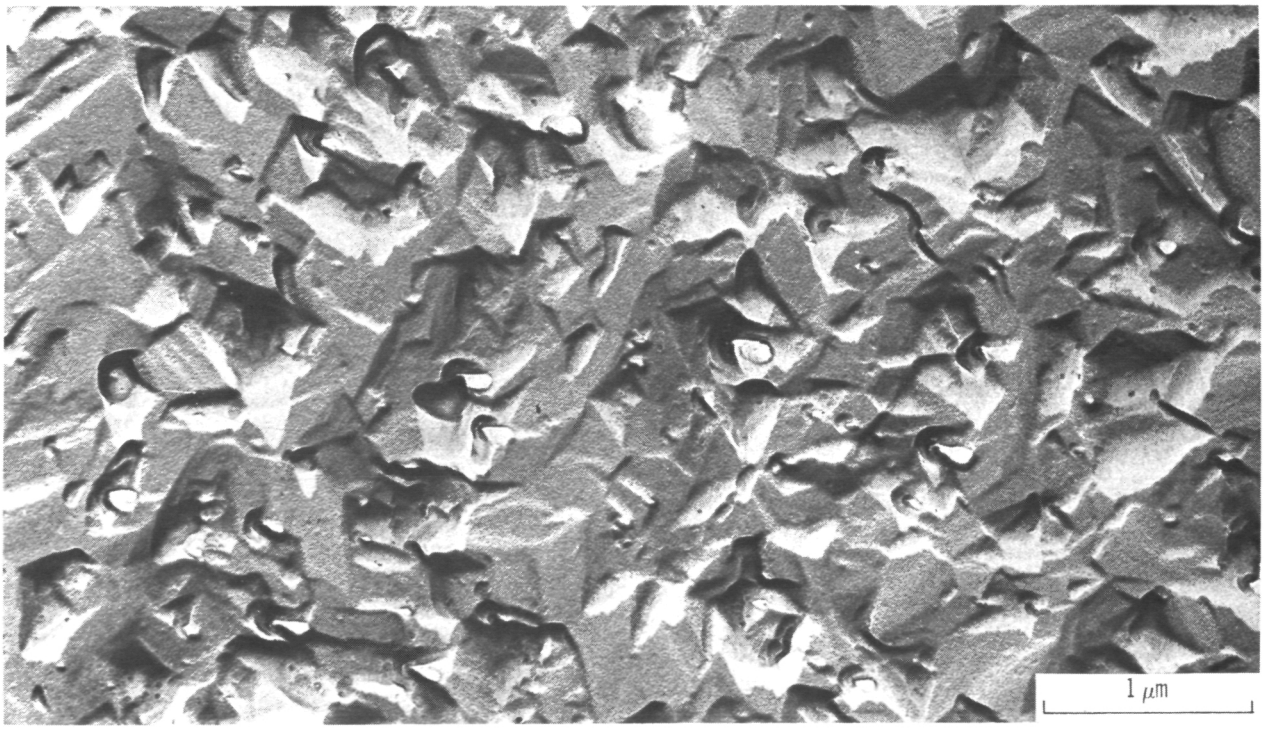


(b) Wire diffusion zone; X7500.

Figure 12. - Replica electron micrographs of W-Hf-C-wire - superalloy composite tested at 1090° C (2000° F). Stress, 207 meganewtons per square meter (30 000 psi); test stopped after 324.8 hours.

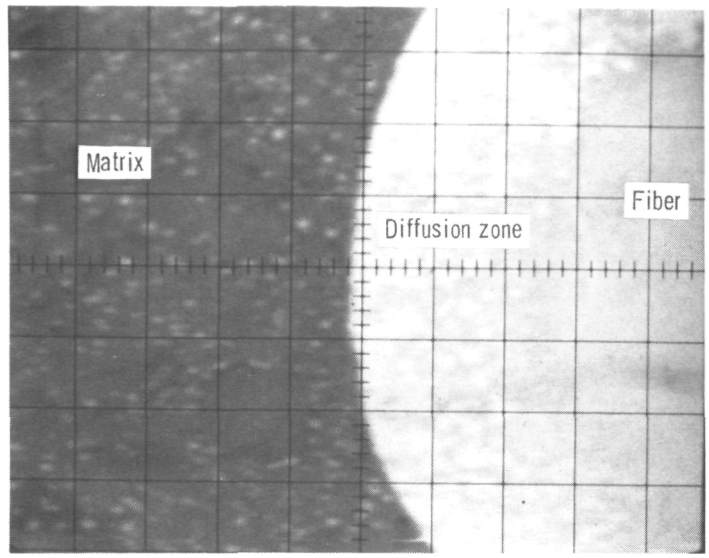


(c) Center of wire; X7500.

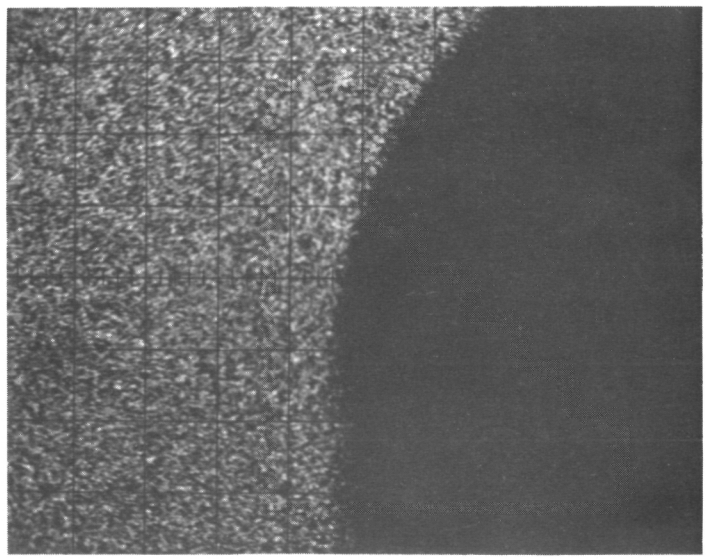


(d) Center of wire; X28 000.

Figure 12. - Concluded.

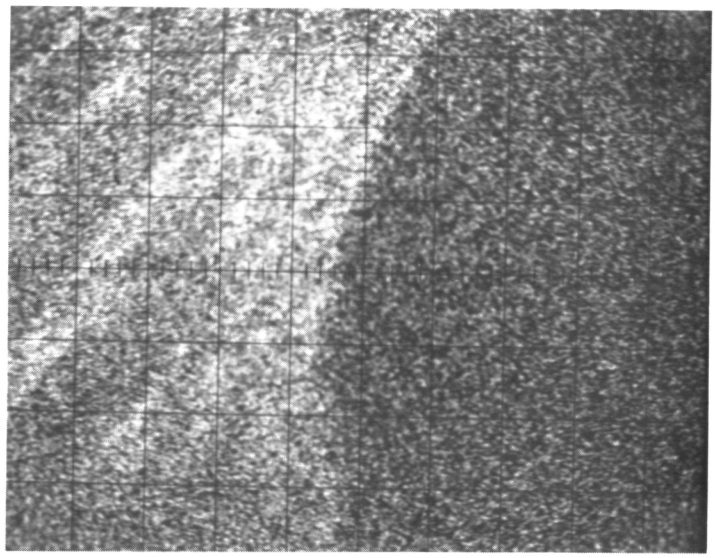


(a) Electron backscatter image.

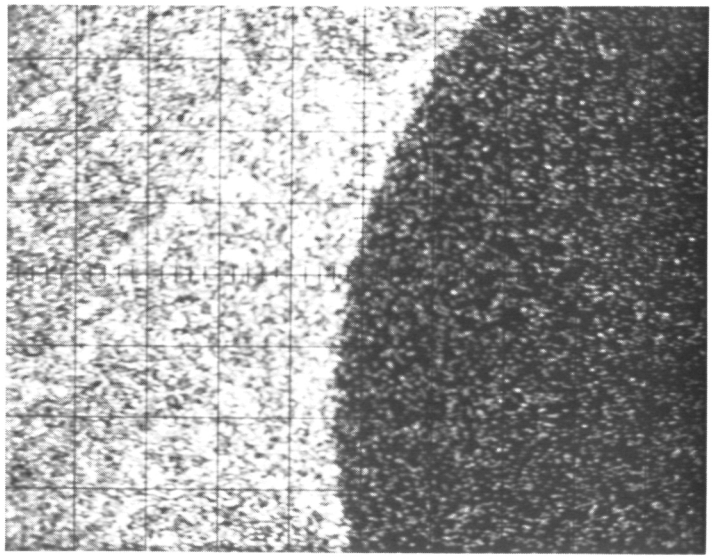


(b) X-ray raster micrograph nickel image.

Figure 13. - Backscatter electron and X-ray raster micrograph images of W-Hf-C wire - superalloy composite tested at 1090° C (2000° F). Stress, 207 meganewtons per square meter (30 000 psi); test stopped after 324.8 hours. X500.

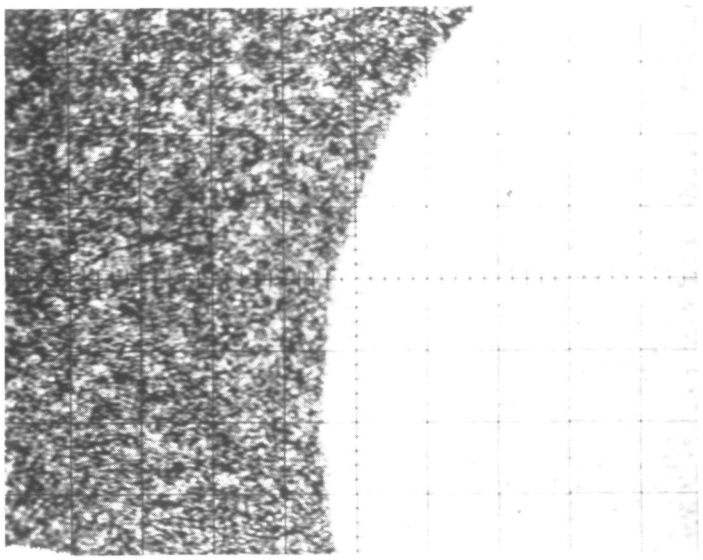


(c) X-ray raster micrograph aluminum image.

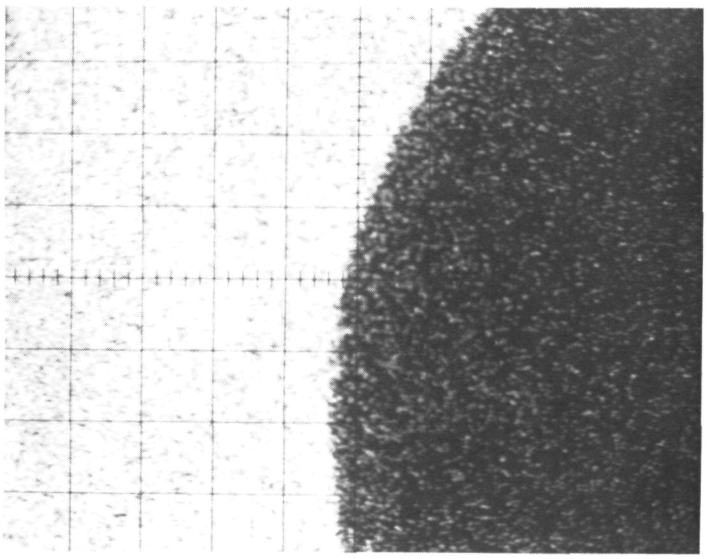


(d) X-ray raster micrograph titanium image.

Figure 13. - Continued.

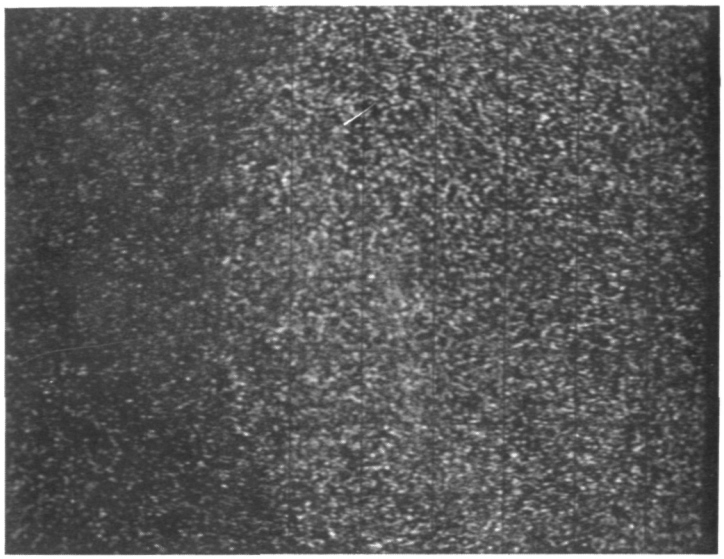


(e) X-ray raster micrograph tungsten image.

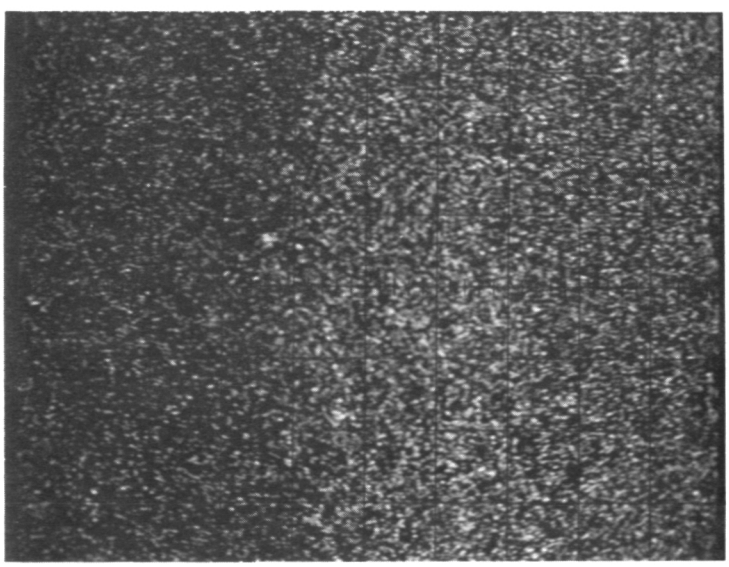


(f) X-ray raster micrograph chromium image.

Figure 13. - Concluded.

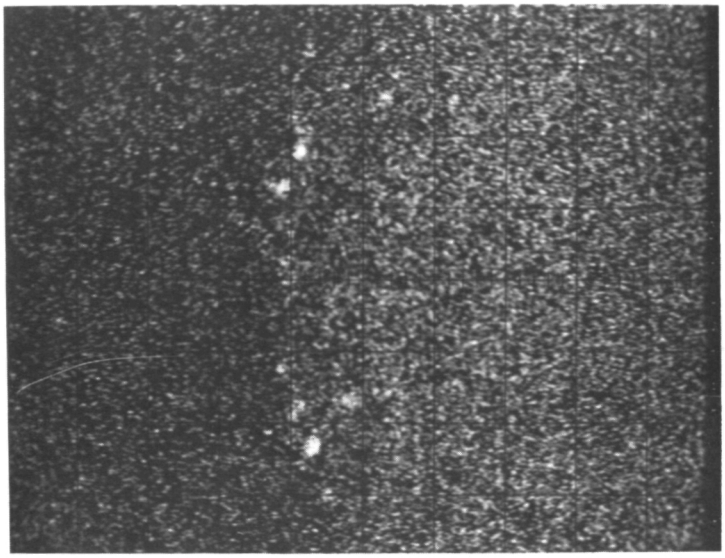


(a) Time of exposure, 0.7 hour.

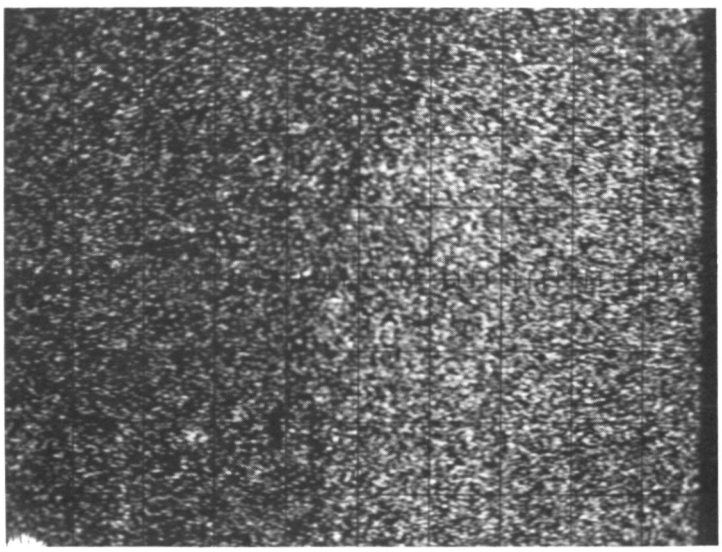


(b) Time of exposure, 59.0 hours.

Figure 14. - X-ray raster micrograph hafnium images for W-Hf-C-wire - superalloy composites for various times of exposure at 1090° C (2000° F). X500.



(c) Time of exposure, 148.3 hours.



(d) Time of exposure, 324.8 hours.

Figure 14. - Concluded.

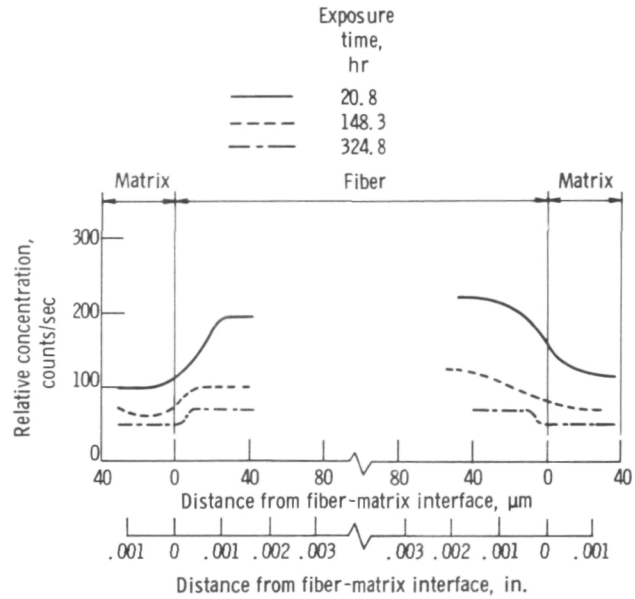


Figure 15. - Variation in concentration of carbon in fiber and matrix for various exposure times at 1090° C (2000° F).

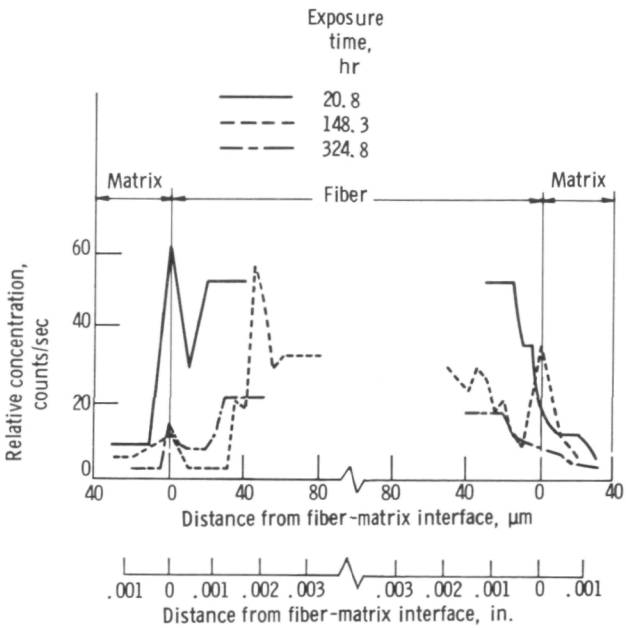


Figure 16. - Variation in concentration of hafnium in fiber and matrix for various exposure times at 1090° C (2000° F).

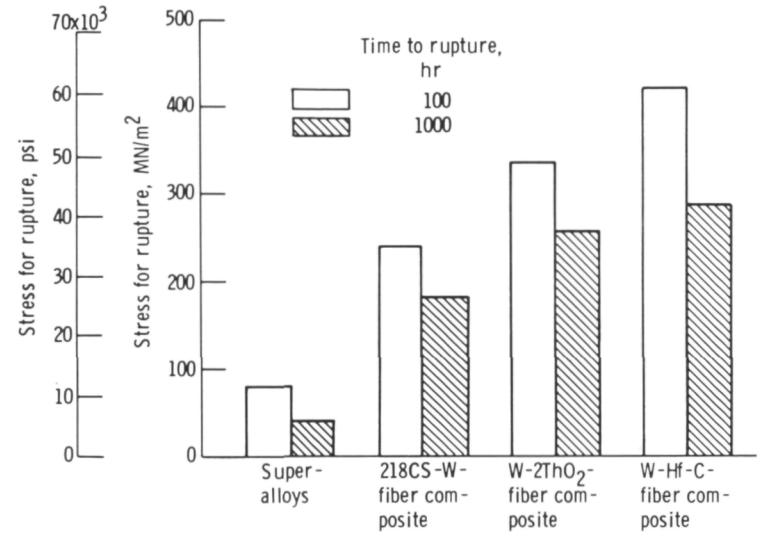


Figure 17. - Stress for rupture in 100 and 1000 hours for refractory-fiber - nickel-base-alloy composites at 1090° C (2000° F). Fiber content, 70 volume percent.

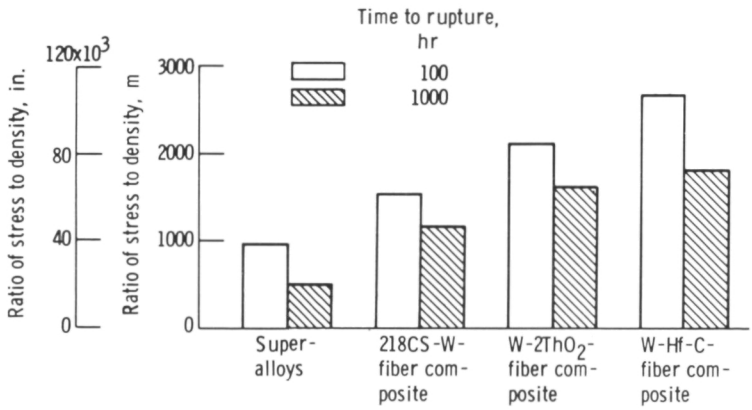
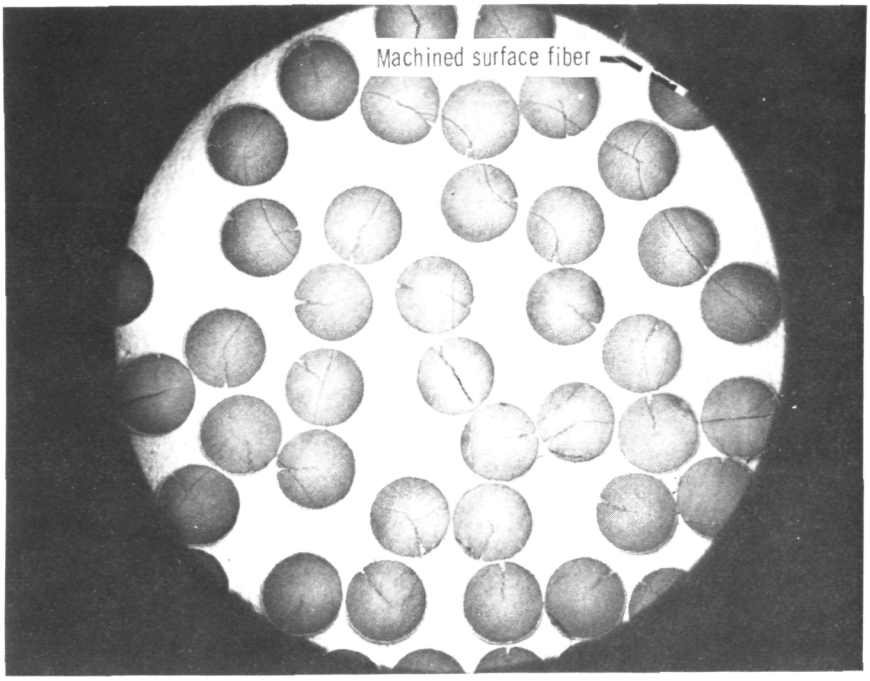
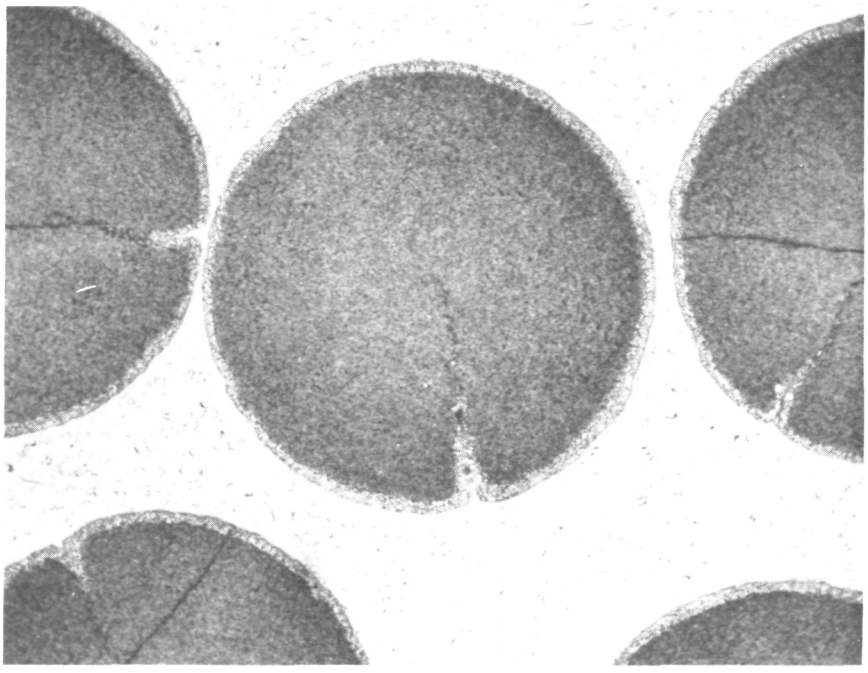


Figure 18. - Specific strength for rupture in 100 and 1000 hours for refractory-fiber - nickel-base-alloy composites at 1090° C (2000° F). Fiber content, 70 volume percent.



(a) Complete section; X25.



(b) Partial section; X150.

Figure 19. - Transverse section of as-fabricated W-Hf-C-wire - superalloy composite.

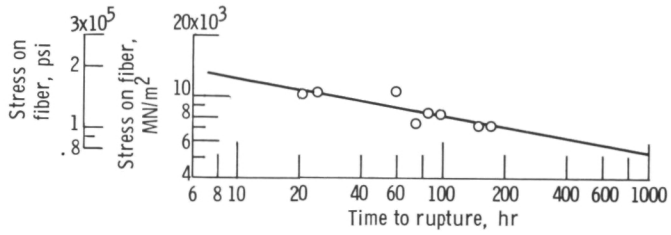
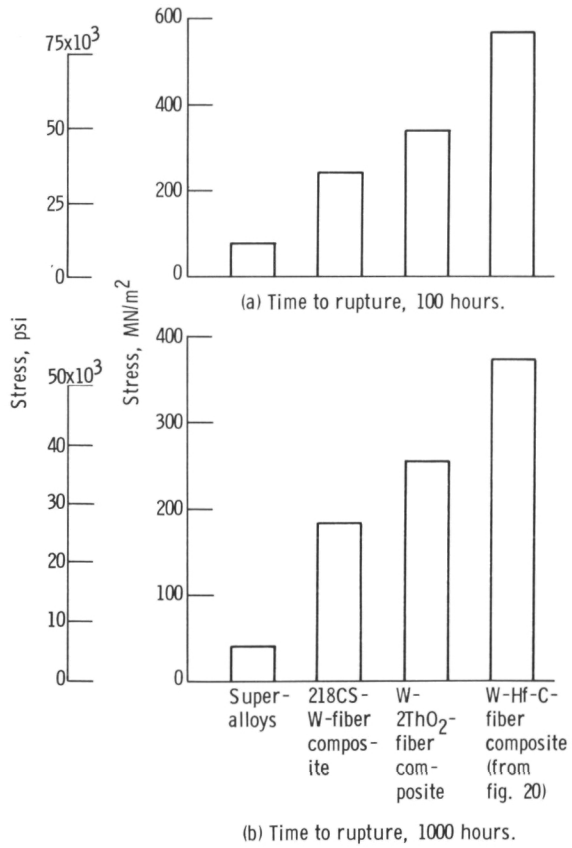
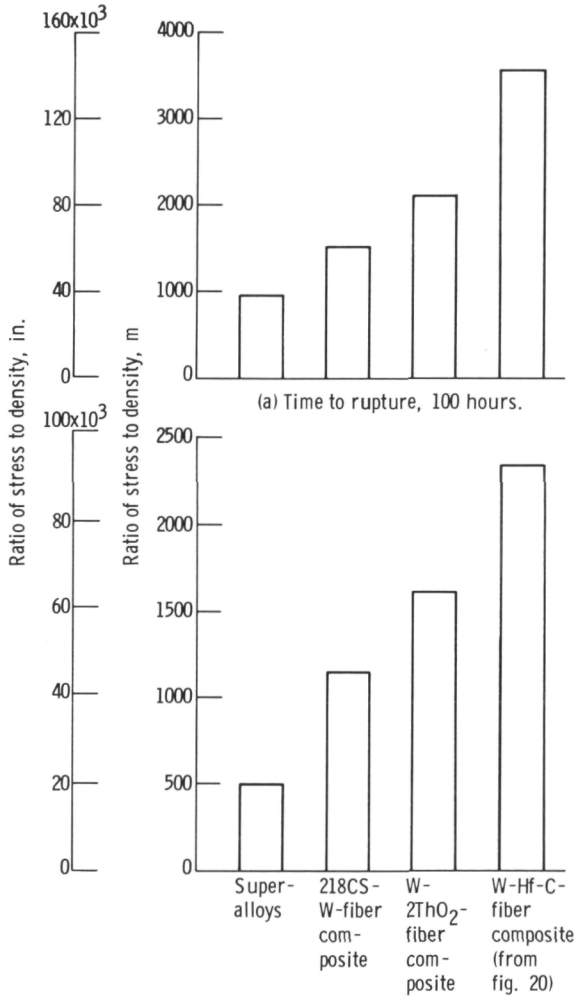


Figure 20. - Stress on fiber as function of time to rupture for W-Hf-C composite (with surface fibers neglected) at 1090° C (2000° F). Specimens contained more than 15 percent split-free fibers.



(b) Time to rupture, 1000 hours.

Figure 21. - Rupture stress for refractory-fiber - nickel-base-alloy composites at 1090° C (2000° F) compared with data from figure 20. Fiber content, 70 volume percent.



(b) Time to rupture, 1000 hours.

Figure 22. - Specific rupture strength for refractory-fiber - nickel-base-alloy composites at 1090° C (2000° F) compared with data from figure 20. Fiber content, 70 volume percent.

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