NASA TECHNICAL MEMORANDUM

NASA TM X-71529

NASA TM X-71529

STRESS-RUPTURE STRENGTH AND MICROSTRUCTURAL STABILITY OF W-HF-C WIRE REINFORCED SUPERALLOY COMPOSITES

by Donald W. Petrasek and Robert A. Signorelli
Lewis Research Center
Cleveland, Ohio 44135

TECHNICAL PAPER proposed for presentation at
Composite Reliability Conference sponsored by the American Society for Testing and Materials in cooperation with the American Institute of Aeronautics and Astronautics, American Society of Mechanical Engineers, American Institute of Mining, Metallurgical and Petroleum Engineers
Las Vegas, Nevada, April 15-16, 1974.

STRESS-RUPTURE STRENGTH AND MICROSTRUCTURAL STABILITY OF W-HF-C WIRE REINFORCED SUPERALLOY COMPOSITES

by Donald W. Petrasek and Robert A. Signorelli

Lewis Research Center

ABSTRACT

W-Hf-C/superalloy composites were found to be potentially useful for turbine blade applications on the basis of stress-rupture strength. The 100- and 1000-hour rupture strengths obtained for 70 volume percent fiber composites tested at 1090° C (2000° F) were 420 and 280 MN/m^2 (61 000 and 41 000 psi). The investigation indicated that with better quality fibers, composites having 100- and 1000-hour rupture strengths of 570 and 370 MN/m^2 (82 000 and 54 000 psi) may be obtained. Metallographic studies indicated sufficient fiber-matrix compatibility for long time applications at 1090° C (2000° F) for 1000 hours or more.

E-7931

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 deficiences of high temperature materials can be overcome by design. Superalloys have been used as turbine blades with operating temperatures of 1260° C (2300° F)

and above 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 they have sufficient strength, solid uncooled blades can be used for latter stages which are currently cooled, thereby achieving increased performance. Cooled blades made from improved materials could be operated at higher temperatures with less cooling air then is required for todays 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.

Tungsten alloy/superalloy composites 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 MN/m^2 (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 ThO_2/superalloy composite having a

fiber content of 70 percent by volume. The horizontal band on the figure represents a range of strength-to-density values that might be required for turbine blades for an advanced turbojet engine. Superalloys are currently limited to 980° C (1800° F) while the composite can be used at the same strength-to-density level at 1090° C (2000° F), 110° C (200° F) higher than superalloys. The fiber composite contains 70 volume percent fiber and as a result is quite dense. It should be noted, however, that this volume percent fiber content 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 using improved high strength fibers of W-Hf-C. Composites consisting of a nickel base alloy containing 25 percent tungsten, 15 percent chromium, 2 percent titanium and 2 percent aluminum and reinforced with up to 60 volume percent W-Hf-C fibers were fabricated and evaluated. The composite specimens and W-Hf-C fiber

were evaluated in stress-rupture at 1090° C (2000° F). A metallographic and electron beam probe analysis was conducted to determine the extent of reaction between the W-Hf-C fiber and nickel-base alloy for exposure times up to 300 hours at 1093° C (2000° F).

MATERIALS, APPARATUS AND PROCEDURE

Fiber and Matrix Material

The fabrication history of the W-Hf-C fibers used in this investigation is reported in Reference 7. The fiber was developed as part of a continuing program support by NASA Lewis Research Center to provide improved property fibers for fiber composite use. The fiber was experimentally developed and not optimized. The diameter of the fiber used was 0.038 cm (0.015 in.) and 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, with the balance made up of tungsten. 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 cm (3 in.) compared to fully straightened tungsten (218 CS) fibers which have a radius of curvature of 100 cm (40 in.).

The composition of the nickel base alloy matrix material was selected based upon its compatibility with the fiber 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 powder was vacuum cast and atomized into fine powder ranging in size from -325 to +500 mesh.

Composite Specimen Fabrication

Composites containing the tungsten alloy wire and the nickel alloy were made by a slip casting process described in detail in Reference 1. The metal powder slip consisted of the nickel alloy powder and a solution of ammonium salt of alginic acid in water. Composite specimens were made by inserting continuous length tungsten alloy wires into an inconel tube and infiltrating the wire bundle with the metal slip. After slip casting, each specimen was removed from its tube, dried in air and then sintered at 820° C (1500° F) for one hour in hydrogen. The specimens were then reinserted into the inconel tubes, sealed at both ends and isostatically hot pressed at 820° C (1500° F) for 1 hour and then at 1090° C (2000° F) for 1 hour using helium pressurized to 140 MN/m^2 (20 000 psi). Fully densified specimens of over 99 percent theoretical density were produced and machined into specimens to be tested in stress-rupture.

Testing Procedure

Stress-rupture tests on the wire material were conducted at 1090° and 1200° C (2000° and 2200° F) in a measured vacuum of $7\times10^{-3} N/m^2$ (5×10^{-5} torr) using a stress-rupture apparatus specifically designed for the testing of small diameter fibers. A detailed description of this apparatus may be found in Reference 8.

Stress-rupture tests at 1090° C (2000° F) were conducted on composite test specimens using conventional creep machines and a helium atmosphere.

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 volume percent 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 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 factor 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 viewed in an electron microscope and photographs were taken at magnification factors 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 and to try to identify these elements and the extent to which they

diffused. The probe was also operated to scan for secondary electron backscatter images and X-ray fluorescense images for the elements Al, Cr, Hf, Ni, Ti, and W.

RESULTS

Fiber Material

Stress-Rupture Properties - The stress-rupture properties of the W-Hf-C fibers used in this investigation are listed in Table 1. 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 exists from ingot to ingot and from spool to spool. Figure 1 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 stress to cause rupture in 100 and 1000 hours at 1090° C (2000° F) was 1060 and 890 MN/m^2 (154 000 and 129 000 psi). The 1200° C (2200° F) test results indicated 100- and 1000-hour rupture strengths for the fiber of 715 and 590 MN/m^2 (103 000 and 85 000 psi). Figure 2 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 1 and no trend as a function of rupture time is observed.

Microstructure - Figure 3 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 3(a) is a transverse section at the edge of the fiber and Figure 3(b) is a transverse section near the center of the fiber. Similar structures and particle distribution and size are seen for both sections.

Figures 4(a) and (b) are electron micrographs of a fiber which failed in stress-rupture in 586.7 hours. An edge section is shown in Figure 4(a) and sections near the center of the wire are shown in Figures 4(b) and (c). The edge and center sections in Figures 4(a) and (b) appear to have larger particles than that observed for the preceding fiber specimen which was exposed for only 12.9 hours. There were areas in the center portion of the fiber however, such as in figure 4(c) which had an equivalent structure to that observed for the short time exposure.

The fiber specimen of Figure 3 (short time exposure) failed in stress-rupture with a very ductile fracture while the fiber specimen shown in Figure 4 (long time exposure) failed in a much less ductile manner. The electron micrograph study indicated that some particle coarsening occurred for the fiber exposed to the long time period and that this particle coarsening may have resulted in a less ductile material. Particle coarsening rates were calculated for 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 due to 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

Stress-Rupture Properties - 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 and reported in Reference 1. The specimens were tested in stress-rupture at 1090° and 1200° C (2000° and 2200° F) in a helium atmosphere. The 100-hour rupture strength for the nickel alloy was found to be 23 MN/m^2 (3200 psi) at 1090° C (2000° F).

Composite Material

Stress-Rupture Properties - The stress-rupture properties obtained for the composites are given in Table 2. The composite specimens had fiber contents ranging from about 15 to 60 volume percent and were tested at stress levels from 138 to 379 MN/m^2 (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 2. Figure 5 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-square fit of the data indicates that the stress for rupture in 100 hours is approximately 600 MN/m^2 (87 000 psi) while that for 1000 hours is 400 MN/m^2 (59 000 psi). Approximately 57 percent of the stress-rupture strength of the W-Hf-C fiber was retained in the composite for rupture in 100 hours and 45 percent for rupture in 1000 hours compared with as-received fibers tested in vacuum. The strength retension 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 to the composite. Pull-out of the fibers occurred because of misalignment of these fibers to the tensile axis of the specimen. The magnitude of these strength losses resulting from fiber pull-out and fabrication defects will be treated in more detail in the discussion section of this report.

Fiber-Matrix Reaction - The depth of reaction between the matrix and fiber was measured for each specimen tested in stress-rupture. The results of the measurements are listed in Table 2 and plotted as a function of rupture time in Figure 6. A least-square fit of the W-Hf-C fiber data is shown and also data for W-2 percent ThO_2 fibers in the same matrix material as reported in Reference 5. The depth of reaction after 100 hours exposure was 0.0046 cm (0.0018 in.). The degree of reaction between the matrix and the W-Hf-C fiber was similar to that observed for composites containing W-2 percent ThO_2 fibers and having the same matrix composition. Replica electron micrographs of composite specimens indicated that large HfC particles and large grains are formed in the fiber-matrix diffusion zone.

Electron Microprobe Study - Backscatter electron and x-ray images of composite specimens indicated a detectable chromium concentration in the tungsten alloy fiber reaction zone; however, no detectable nickel concentration was observed. X-ray raster micrographs for aluminum and titanium did not show any detectable concentrations of these elements in the diffusion zone of the fiber. X-ray raster micrographs for hafnium indicated that diffusion of hafnium into the matrix increases with time at temperature.

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 were generally much greater than indicated by the electron probe study for the above elements.

The most significant finding of the electron probe study was the results obtained for the hafnium and carbon traces. Figures 7 and 8 are plots of concentration versus distance from the fiber-matrix interface for carbon and hafnium. The profiles show the variation in relative concentration of either carbon or hafnium rather than the actual concentration of each element. Specimens which were exposed at 1090° C (2000° F) for three different time periods are plotted in the figures. Figure 7 shows that the concentration gradient for carbon between the fiber and matrix decreases with time of exposure indicating diffusion of carbon from the fiber into the matrix. Figure 8 shows a similar trend, hafnium diffusing into the matrix. The hafnium and carbon composition of the fiber implies that some excess carbon is 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 micrographs and electron probe traces thus indicate that HfC decomposition and diffusion of free carbon and hafnium into the matrix occurs. The nickel alloy matrix contained 0.0032 weight percent carbon while the fiber had a carbon content of almost 10 times that amount, 0.03 weight percent. The results imply that it may be beneficial to add hafnium to the matrix or to increase the carbon content in an attempt to inhibit the loss of HfC particles in the fiber.

DISCUSSION

Current Composite Stress-Rupture Properties

The stress-rupture properties of composites containing varying volume fiber contents can be determined through the use of the plot

shown in Figure 5. 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 5, 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 MN/m^2 (61 000 psi), that is, 0.70 × 600 MN/m^2 (0.70 × 87 000 psi). The foregoing method was also 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 MN/m^2 (41 000 psi). Prior to this investigation the strongest refractory metal fiber reinforced superalloy composite was the W-2 percent ThO_2 fiber composite having a 100-hour specific strength of 2100 m (83 000 in.) and a 1000-hour specific strength of 1600 m (63 000 in.). The results of this investigation indicate that W-Hf-C/superalloy composites show an improvement in specific strength over W-2 percent ThO_2 fiber composites. The 100-hour specific rupture strength for the W-Hf-C composite is 2650 m (104 000 in.) and the 1000-hour specific rupture strength is 1800 m (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 three and one half 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 lower than those which can be achieved

with this system. Higher stress-rupture strengths than those obtained were expected based on 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-2 percent ThO_2 fibers. The rupture strengths for W-2 percent ThO_2 fibers contained in the composite were 75 percent for rupture in 100-hours and 59 percent for rupture in 1000-hours compared with as-received fibers tested in vacuum (Ref. 5).

Further, composite rupture strength for 218 CS tungsten and W-2 percent ThO_2 reinforced superalloy composites had been related to depth of reaction (Ref. 1). Since the degree of reaction with the matrix was similar for W-Hf-C and W-2 percent ThO_2 fibers it would be expected that their strength retentions would be similar. Based on depth of reaction the W-Hf-C fibers contained in the composite would be expected to have a 100-hour and 1000-hour rupture strength of 800 and 520 MN/m^2 (116 000 and 76 000 psi) rather than the values of 600 and 400 MN/m^2 (87 000 and 59 000 psi) obtained in this investigation. As was noted in the RESULTS section of this report, the W-Hf-C fibers were bent and misaligned to the tensile axis of the test specimens and fiber ends were present on the surface of the test section due to the machining process employed 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.

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 fiber splits and the width and depth of the fiber split 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 surface fiber area exposed to the matrix due to the splits.

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-free 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 would found to be 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 that the surface fibers do not contribute to composite strength.

The specimens selected for the above appraisal are listed in Table 3. 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 volume percent fiber content shown in the table is that volume fiber content 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 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 3 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 5. Figure 9 is a plot of the stress carried by the fibers versus time to rupture at 1090° C (2000° F) for the specimens listed in Table 3. A least-square fit of the data indicates a 100-hour rupture strength of 810 MN/m^2 (117 000 psi) and a 1000-hour rupture strength of 530 MN/m^2 (77 000 psi). The rupture strength of W-Hf-C fibers contained in the composite based on these results and assumptions was 76 percent for rupture in 100-hours and 60 percent for rupture in 1000-hours compared to as-received fibers tested in vacuum; this is in agreement with the retention values obtained for W-2 percent ThO_2 fibers contained in the same matrix (Ref. 5). The above strengths obtained 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 10 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 MN/m^2 (82 000 psi), i.e., 0.7 times 810 MN/m^2 (0.7 times 117 000 psi). The W-Hf-C composite is seen to be the strongest fiber composite system and represents a significant improvement over W-2 percent ThO_2 fiber composites. A 65 percent improvement in the 100-hour rupture strength is obtained when W-Hf-C fibers are used compared to W-2 percent ThO_2 fibers (570 versus 340 MN/m^2 (82 000 versus 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 also shown in Figure 10. A 45 percent improvement in the 1000-hour rupture strength is obtained for W-Hf-C composites compared with W-2 percent ThO_2 fiber composites (370 versus 255 MN/m^2 (54 000 versus 37 000 psi)). The W-Hf-C composite is about nine times as strong as conventional superalloys at this temperature and time to rupture.

Prior to this investigation the strongest refractory fiber/superalloy composite was the W-2 percent ThO_2 fiber composite having a 100-hour specific strength of 2100 m (83 000 in.) and a 1000-hour specific strength of 1600 m (63 000 in.). The results of this investigation indicate that W-Hf-C/superalloy composites show an improvement in specific strength over W-2 percent ThO_2 fiber composites. The 100-hour specific rupture strength for the W-Hf-C composite is 3500 (140 000 in.) and the 1000-hour specific rupture strength is 2300 m (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 properties 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 m (92 000 in.) at 1090° C (2000° F). The strongest conventional superalloys have a specific 1000-hour rupture strength of 2350 m (92 000 in.) at 930° C (1700° F). The W-Hf-C composite based on this comparison has a 1650° 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 is 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 values 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 properties of the composite. Blades with varying fiber content can be fabricated 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 found to be 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 MN/m^2 (15 000 to 20 000 psi). The stresses generated in rotating blades are density dependent. The stress/density value at blade midspan for a typical superalloy with a density of 8.3 grams/cc (0.30 $lb/in.^3$) would range from 1200 to 1700 m (47 000 to 67 000 in.). It was assumed that a blade material must have a stress/density value of 1525 m (60 000 in.), near the middle of the range indicated above, for rupture in 1000 hours. The fiber content necessary for a 1000-hour rupture strength/density value of 1525 m (60 000 in.) for the W-Hf-C/superalloy composite 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/cc (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/cc (0.325 $lb/in.^3$). The W-Hf-C/superalloy blade density is only 22 percent higher than the value noted above. The matrix material used in this investigation had a high density, 9.15 grams/cc (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 compared to that of the nickel base alloy used in this investigation and having a density of 8.09 grams/cc (0.292 $lb/in.^3$) was used as a matrix material for composite property studies in 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/cc (0.363 lb/in.^{3}) which is only 11 percent higher than the value for the superalloys (8.97 grams/cc (0.325 lb/in.^{3})) reported above. Superalloys are currently limited to 980° C (1800° F) while the composite can be used at the same strength-to-density level at 1090° C (2000° F), 110° C (200° F) higher than superalloys.

SUMMARY OF RESULTS

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

1. Composites were fabricated having high stress-rupture properties at 1090° C (2000° F) compared to superalloys. The 100-hour stress-rupture strength obtained for 70 volume percent fiber composites at 1090° C (2000° F) was 420 MN/m^2 (61 000 psi) as compared with 80 MN/m^2 (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 MN/m^2 (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 had a 100-hour specific rupture strength of 2650 m (104 000 in.) and an extrapolated 1000-hour specific rupture strength at 1090° C (2000° F) of 1780 m (70 000 in.). The 100-hour specific rupture strength for the composite is over twice that for conventional case superalloys.

The 1000-hour specific rupture strength for the composite is over three and one half 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 are higher than data reported in the literature for any other composite.

4. The 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 nickel base alloy matrix was 0.0046 cm (0.0018 in.) after 100-hour 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 was found to occur for composites exposed at 1090° C (2000° F).

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 based on 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 MN/m^2 (82 000 psi). The 1000-hour rupture strength of the composite at the same temperature was 370 MN/m^2 (54 000 psi).

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

3. The W-Hf-C 70 volume percent fiber composite has 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 is varied along the blade span so as to tailor strength to that needed.

REFERENCES

1. Petrasek, D. W., Signorelli, R. A., and Weeton, J. W., "Refractory Metal Fiber Nickel Base Alloy Composites for Use at High Temperatures," NASA Report TN D-4787, National Aeronautics and Space Administration, Washington, D. C., Sept. 1968.

2. Glenny, R. J. E., Proceedings of the Royal Society of London, Series A, Vol. 319, No. 1536, Oct. 6, 1970, pp. 33-44.

3. Kovtov, V. F., Fonshtein, N. M and Shvarts, V. I., Metallovendenie i Termicheskaia Obrabotka Metallov, No 8, 1971, pp. 20-22.

4. Morris, A. W. H. and Burwood-Smith A., Fibre Science Technology, Vol. 3, No. 1, 1970, pp. 53-78.

5. Petrasek D. W. and Signorelli R. A., "Preliminary Evaluation of Tungsten Alloy Fiber/Nickel-Base Alloy Composites for Turbojet Engine Applications," NASA Report TN D-5575, National Aeronautics and Space Administration, Washington, D. C., Feb. 1970.

6. Signorelli, R. A., "Review of Status and Potential of Tungsten-Wire--Superalloy Composites for Advanced Gas Turbine Engine Blades, "NASA Report TM X-2599, National Aeronautics and Space Administration, Washington, D. C., Sept. 1972.

7. King, G. W., "Development of Wire Drawing Processess for Refractory Metal Fibert," NASA Report CR-120925, Westinghouse Electric Corporation, Bloomfield, N. J., Jan. 1972.

8. McDanels, D. L. and Signorelli, R. A., "Stress-Rupture Properties of Tungsten Wire from 1200° to 2500° F." NASA Report TN D-3467, National Aeronautics and Space Administration, Washington, D. C., July 1966.

9. Klopp, W. D. and Witzke, W. R., "Mechanical Properties of Arc-Melted Tungsten-Rhenium-Hafnium-Carbon Alloys," NASA Report TN D-5348, National Aeronautics and Space Administration, Washington, D. C., July 1969.

10. Petrasek, D. W., "High-Temperature Strength of Refractory-Metal Wires and Consideration for Composite Applications," NASA Report TN D-6881, National Aeronautics and Space Administration, Washington, D. C., Aug. 1972.

11. McDanels, D. L., Singorelli, R. A., and Weeton, J. W., "Analysis of Stress-Ruature and Creep Properties of Tungsten-Fiber-Reinforced Copper Composites," NASA Report TN D-4173, National Aeronautics and Space Administration, Washington, D. C., Sept. 1967.

E-1931

TABLE 1--STRESS-RUPTURE PROPERTIES OF W-Hf-C FIBER

Ingot number	Spool number	Test temperature		Stress		Life, hr	% Reduction in area
		deg C	deg F	MN/m^2	psi		
4027	1	1090	2000	[a]1300	189 000	4.4	44.2
				[a]1290	187 000	10.3	58.4
				[a]1230	178 000	21.1	23.2
				[a]1210	175 000	19.1	35.0
				[a]1150	167 000	61.5	44.5
				[a]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	Test stopped before fracture
		1200	2200	[a]918	133 000	28.3	15.3
				[a]841	122 000	42.9	21.9
				[a]765	111 000	104.3	11.5
				[a]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			1100	160 000	65.4	46.7
				758	110 000	586.7	10.7
	4			1100	160 000	113.8	24.2
				965	140 000	140.5	24.3
	5			1100	160 000	129.9	14.4
				965	140 000	211.6	10.3
4034T"B"	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
	2			1170	170 000	46.7	32.6
				1100	160 000	114.7	23.7
				1030	150 000	152.8	12.9
	3			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			1100	160 000	47.1	27.2
	5			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			1100	160 000	65.0	23.7
	13			1100	160 000	57.7	19.0
	14			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]Data from Ref. 12.

TABLE 2--STRESS-RUPTURE PROPERTIES FOR W-Hf-C WIRE-NICKEL ALLOY COMPOSITES

Specimen number	Specimen diameter,		Composite stress,		Fiber stress,		Rupture life, hr	Volume percent fiber	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	0.0003	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 3--STRESS RUPTURE PROPERTIES FOR W-Hf-C NICKEL ALLOY COMPOSITES CONTAINING MORE THAN 15% SPLIT FREE FIBERS AND NEGLECTING SURFACE FIBERS

Test Temperature - 1093° C (2000° F)

Specimen number	Composite stress,		Effective volume percent fiber content	Calculated fiber stress,		Rupture life, hr	Percent split free fibers
	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

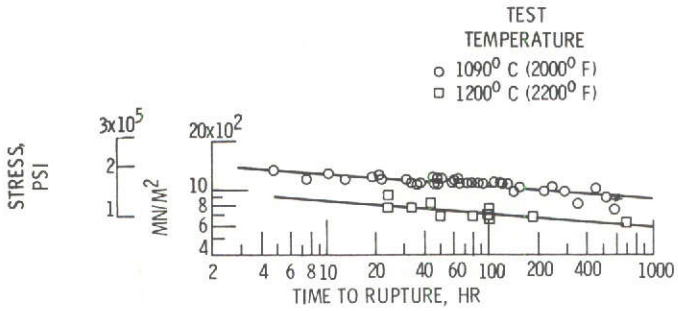


Figure 1. - Time to rupture as function of stress for W-Hf-C wire at 1090° C (2000° F) and 1200° C (2200° F).

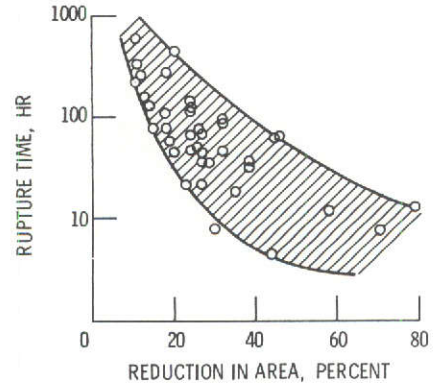
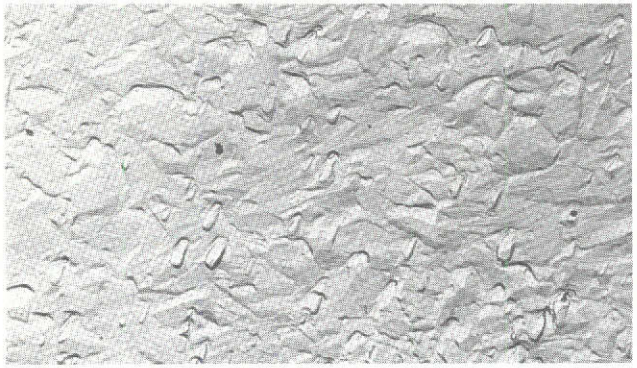


Figure 2. - Reduction in area as function of rupture time for W-Hf-C wire at 1090° C (2000° F).

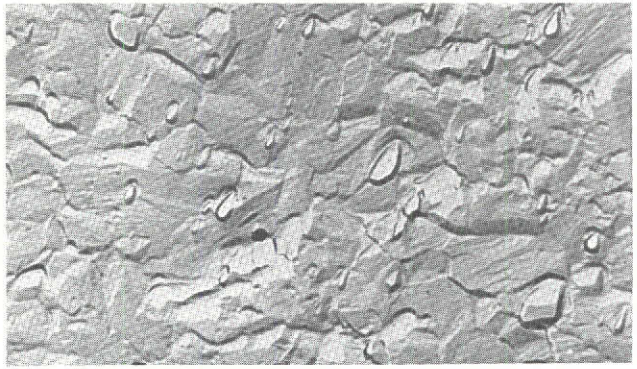
(a) EDGE SECTION. 1 μm

(b) CENTER SECTION. 1 μm

Figure 3. - Replica electron micrographs of W-Hf-C wire tested at 1093° C (2000° F). Stress, 1170 MN/m^2 (170 000 psi); time to rupture, 12.9 hours. X28 000.

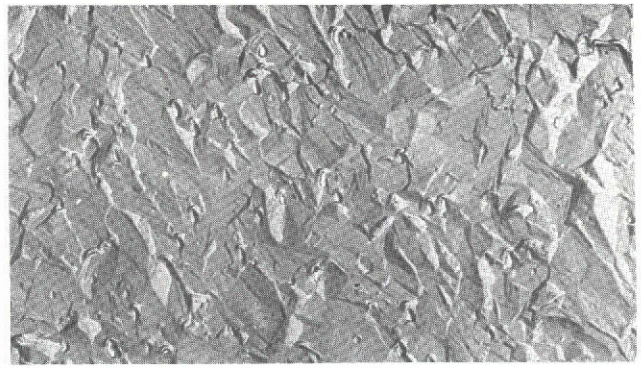


(a) EDGE SECTION. 1 μm



(b) CENTER SECTION, AREA 1. 1 μm

Figure 4. - Replica electron micrographs of W-Hf-C wire tested at 1093° C (2000° F). Stress, 758 MN/m^2 (110 000 psi); time to rupture, 586.7 hours. X28 000.



(c) CENTER SECTION, AREA 2. 1 μm

Figure 4. Concluded.

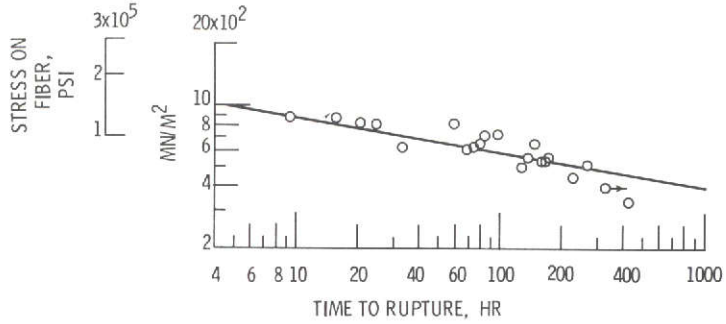


Figure 5. - Stress on fiber as function of rupture time for W-Hf-C composites at 1090° C (2000° F).

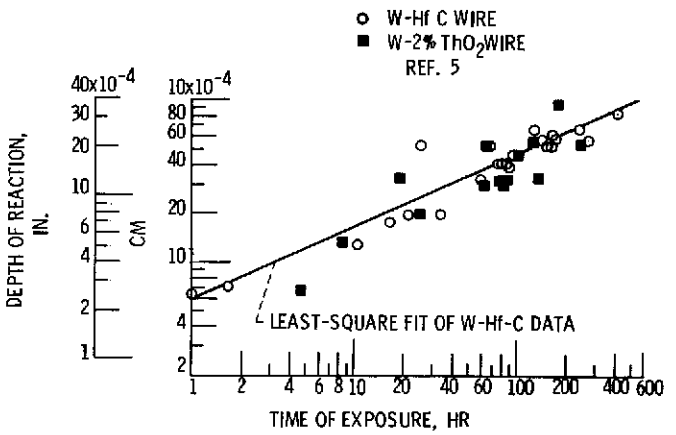


Figure 6. - Depth of reaction as function of time of exposure for W-Hf-C composites at 1090° C (2000° F).

E-7931

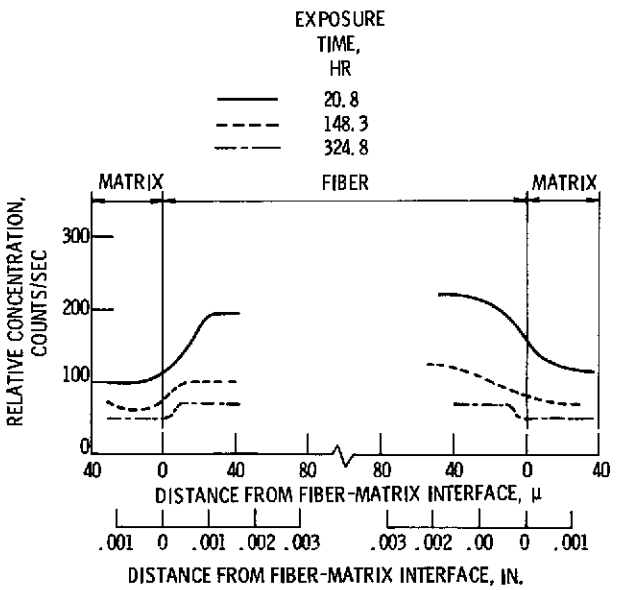


Figure 7. - Variation in concentration of carbon in fiber and matrix for various exposure times at 1090° C (2000° F).

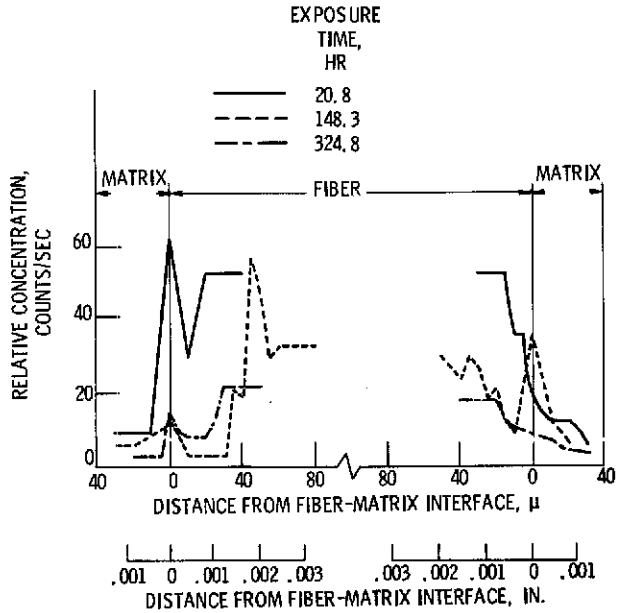


Figure 8. - Variation in concentration of hafnium in fiber and matrix for various exposure times at 1090° C (2000° F).

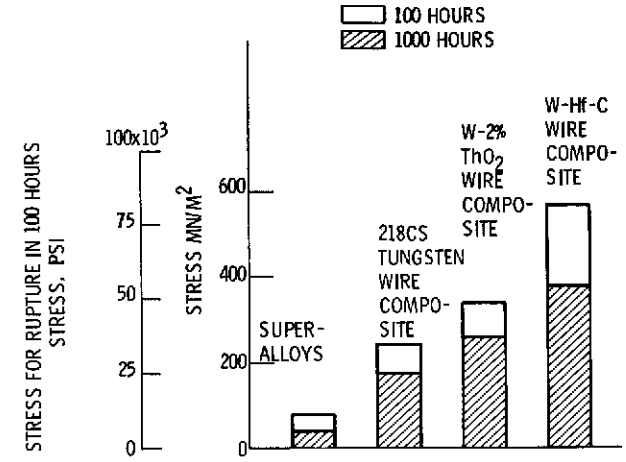


Figure 10. - A 100-hour and 100-hour rupture strength for refractory wire - nickel base alloy composites at 1090° C (2000° F). Fiber content - 70 volume percent.

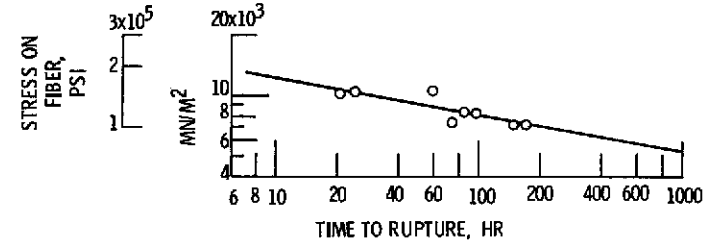


Figure 9. - Stress on fiber as function of time to rupture for W-Hf-C composite (neglecting surface fibers at 1090° C (2000° F). Specimens contain more than 15% split free fibers.