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## EFFECT OF ROLLING ON THE HIGH TEMPERATURE TENSILE AND STRESS-RUPTURE PROPERTIES OF TUNGSTEN FIBER-SUPERALLOY COMPOSITES

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# EFFECT OF ROLLING ON THE HIGH TEMPERATURE TENSILE AND STRESS-RUPTURE PROPERTIES OF TUNGSTEN FIBER-SUPERALLOY COMPOSITES

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## ABSTRACT

An investigation was conducted to determine the effects of mechanical working on the 1093° C (2000° F) tensile and stress-rupture strength of tungsten alloy/superalloy composites. Hot pressed composites containing either conventional tungsten lamp filament wire or tungsten-1% ThO<sub>2</sub> wire and a nickel base alloy matrix were hot rolled at 1093° C (2000° F). The hot pressed and rolled composite specimens were then tested in tension and stress-rupture at 1093° C (2000° F). Rolling decreased the degree of fiber-matrix reaction as a function of time of exposure at 1093° C (2000° F). The stress-rupture properties of the rolled composites were superior to hot pressed composites containing equivalent diameter fibers. Rolling did not appreciably affect the 1093° C (2000° F) ultimate tensile strength of the composites.

## 1. INTRODUCTION

Tungsten alloy (fiber)/superalloy (matrix) composites potentially useful in advanced turbine blade and vane applications have been developed at the NASA Lewis Research Center. They exhibit high stress-rupture strength and can have adequate impact resistance. For example, their 100 hour stress-rupture strength at 1093° C (2000° F) is four times that of the strongest conventional superalloys (338 MN/m<sup>2</sup> (49,000 psi) versus 83 MN/m<sup>2</sup> (12,000 psi)) (1). Furthermore, their high temperature impact strength was found to be very good (2). Unfortunately, their room temperature impact strength was low due to poor bonding of the hot pressed superalloy powder particles of which the matrix is composed. Hot rolling following the initial hot pressing procedure improved their room temperature impact strength by improving the interparticle bond strengths.

Thus hot pressed and rolled composites, that had four times the impact strength of hot pressed composites at room temperature, were found to have sufficient impact strength for consideration in turbine blade applications. However, the effect of rolling on composite stress rupture strength, fiber-matrix compatibility and other factors had not been determined. A study of these potentially important effects was therefore deemed warranted.

Hot rolling of refractory fiber-superalloy composites has been utilized by other investigators to fabricate such composites (3 to 7), but very few have attempted to isolate the effects of working on the strength properties of the composites. It was shown in ref. 3 that the tensile strength of rolled composites was lower than that of hot pressed tungsten/superalloy composites. In the

study of ref. 3, composite bars containing continuous fibers were rolled into sheet by rolling parallel to the fiber direction. Metallographic and radiographic studies revealed that the tungsten fibers fragmented into short lengths when the rolling direction was parallel to the fibers, lowering composite strength.

Tungsten/copper composites were reduced up to 95 percent by rolling parallel to the fiber direction without fiber cracking (unpublished studies at the Lewis Research Center). Tungsten/superalloy composites were also reduced by rolling with no apparent fiber cracking when appropriate steps were taken to minimize reaction and recrystallization of the fibers. The success in these previous mechanical working attempts served to provide encouragement for further study of the effects of rolling.

The object of the present investigation was to determine the effects of hot rolling on the 1093° C (2000° F) tensile and stress-rupture strength of tungsten/superalloy composites. Rolling effects on fiber-matrix reactions were also examined as related to exposure time at 1093° C (2000° F).

## 2. MATERIALS APPARATUS AND PROCEDURE

### 2.1 MATERIAL

Two commercial lamp filament wires, 218 tungsten and tungsten-1% ThO<sub>2</sub> were used in this investigation. The wire was used in the as-drawn, cleaned and straightened condition. The wire diameters were 0.051 cm (0.020 in) for the tungsten-1% ThO<sub>2</sub> and 0.038 cm (0.015 in) for the 218 wire materials.

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 (8). The nominal composition of the nickel alloy was 56 percent nickel, 25 percent tungsten, 15 percent chromium, 2 percent aluminum, and 2 percent titanium. The nickel alloy was vacuum cast and atomized into fine

powder with a particle size range of -325 to +500 mesh. A chemical analysis of the powder is given in ref. 8.

### 2.2 COMPOSITE FABRICATION

Composites containing the tungsten alloy fiber and the nickel alloy were fabricated by a slip casting process, as described in ref. 8. The metal powder slip consisted of the nickel alloy powder and a solution of ammonium salt of alginate acid in water. The composition, viscosity, pH, and density of the metal slip are listed in ref. 8.

Composite specimens were prepared by inserting continuous length tungsten alloy fibers into a nickel tube containing a wire screen at the bottom and several layers of filter paper. The nickel tube was then placed on a vibrating table, and slip was poured into the fiber bundle while the tube was vibrating. As the nickel alloy powder settled to the bottom of the bundle, excess liquid media were syphoned off the top, and more slip was added. This process was continued until the nickel alloy powder level reached the top of the fiber bundle. The vibrator was then turned off, and a vacuum was applied to the tube to remove any additional liquid media left in the casting. The casting was removed from the tube and dried in air for approximately 24 hours at 60° C (140° F). The castings were sintered at 816° 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 castings were inserted in close fitting nickel tubes having an outside diameter of 2.06 cm (0.81 in) and a wall thickness of 0.08 cm (0.03 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 canned castings were then densified by isostatically hot pressing the tubes with helium at 137.9 MN/m<sup>2</sup> (20,000 psi), first at 816° C (1500° F) for 1 hour and then at 1093° C (2000° C)

for 1 hour. Composites rods were made with fiber contents ranging from 50 to 70 volume percent. The diameter of the pressed rods varied from 1.85 to 1.88 cm (0.73 to 0.74 in).

### 2.3 ROLLING PROCEDURE

The composite rods were held at a temperature of 1093° C (2000° F) for 1 hour and were then rolled on a two high mill at a surface speed of 0.41 m/sec (80 feet/minute). Twenty centimeters diameter by thirty centimeters (eight inch diameter by twelve inch) face rod rolls containing seven grooves ranging in diameter from 1.75 to 0.89 cm (0.69 to 0.35 in) were used. Approximately 5 percent reduction in area per pass was given each specimen. After each pass the specimen was returned to the furnace and held at 1093° C (2000° F) for a minimum of 15 minutes. The specimens were reduced a total of 75 to 80 percent.

### 2.4 TESTING PROCEDURE

The as-fabricated and as-rolled rods were centerless ground into button head type specimens having a test section 2.54 cm (1.00 in) long and a 0.287 cm (0.113 in) diameter.

The composite specimens were tested in tension at 1093° C (2000° F). The tests were conducted in a capsule evacuated to  $1.3 \times 10^{-3}$  N/m<sup>2</sup> ( $1 \times 10^{-5}$  torr). An Instron tensile testing machine was used at a crosshead speed of 0.25 cm/min (0.1 inch per minute). Stress-rupture tests on composite specimens were conducted in conventional constant-load creep machines, and a helium atmosphere was used to minimize oxidation. Tests were conducted at 1093° C (2000° F).

### 2.5 METALLOGRAPHIC STUDY

Specimens and rods were examined metallographically to determine the depth of the reaction zone between the nickel alloy matrix and the fiber 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 either 150X or 300X. The depth of the reaction zone is defined as the distance from the fiber-matrix interface to the interface in the fiber where a microstructural change is observed. The cross-sectional area and the volume percent fiber content for all composite specimens were determined by sectioning the specimen transversely in an area immediately adjacent to the fracture. The sections were mounted, polished and photographed at a magnification of 25X. A wire count was obtained from the photographs and the volume percent fiber content was calculated from the known wire cross sectional area and the composite specimen cross sectional area.

## 3. RESULTS

### 3.1 ROLLING STUDY

The composite rods were successfully reduced 75 to 80 percent in area by rolling. Figure 1 shows the composite rods in several stages of reduction. The rods shown below the initial hot pressed billet were reduced 32, 59, and 78 percent in area respectively. The composite rod reduced 78 percent in area contained flashing and had an elliptical rather than circular cross section. Radiographs taken of the rolled rods indicated that the fibers did not fracture during rolling. The radiographs however failed to give concrete evidence that the fibers did not contain microcracks from working but did tend to verify that the fibers were macroscopically continuous. Continuous fibers were leached out of the matrix from rolled rods to verify continuity. The rods were electrolytically leached with a solution of 20 volume percent phosphoric acid and 80 volume percent water at a current density of 0.2 to 0.4 amps/cm<sup>2</sup>. The leached fibers contained areas where inadvertent sharp bends had occurred due to the rolling procedure used. The fibers were reduced in area by the same amount observed for the entire composite.

**Microstructure.** Figure 2(a) is a transverse section of a hot pressed composite containing tungsten-1% ThO<sub>2</sub> fibers and figure 2(b) is a transverse section of a composite containing 218 tungsten fibers. The depth of reaction between the matrix and fibers was determined to be 0.0012 cm (0.0005 in). Distinct matrix grain boundaries are observed for the hot pressed composites shown in figure 2.

A transverse and longitudinal section of a composite containing tungsten-1% ThO<sub>2</sub> fibers and reduced in area by rolling 77 percent is shown in figure 3. The wire is elliptical in shape and has a cross sectional area equivalent to a fiber having a diameter of 0.024 cm (0.0094 in). The matrix grain boundaries are less distinct than in the hot pressed condition. The longitudinal section also shows that the matrix particles have been elongated. The depth of reaction between the fiber and matrix is approximately one-half that for the hot pressed condition (0.00066 cm (0.00026 in)). The initial reaction zone formed during hot pressing is reduced in thickness during rolling. If no further reaction took place during rolling the reaction zone thickness would have been 0.00046 cm (0.00018 in). Some reaction with the fibers thus occurred during rolling.

Figure 4 is a transverse and longitudinal section of a composite containing 218 tungsten fibers which were reduced by rolling 78 percent. The fiber is elliptical in shape and has a cross sectional area equivalent to a fiber having a diameter of 0.017 cm (0.0067 in). The matrix microstructure is similar to that observed for the composites containing the tungsten-1% ThO<sub>2</sub> fibers. The depth of reaction with the fiber after rolling is similar also, 0.00066 cm (0.00026 in).

### 3.2 TENSILE TESTS

**Fibers.** Tensile tests were conducted on individual 218 tungsten fibers which were leached out of a composite rolled to a reduction in area of 78 percent. These tensile tests were conducted

at room temperature and 1093° C (2000° F). The room temperature tensile strength obtained was 2320 MN/m<sup>2</sup> (337,000 psi) while the tensile strength at 1093° C (2000° F) was 786 MN/m<sup>2</sup> (114,000 psi). The reduction in area at fracture was measured for the rolled fiber and was found to be 28 percent at room temperature and 55 percent at 1093° C (2000° F). The fiber was thus ductile at both temperatures.

The 218 tungsten fibers contained in the hot pressed composites were found to have a room temperature tensile strength of 2280 MN/m<sup>2</sup> (331,000 psi) and a 1093° C (2000° F) tensile strength of 765 MN/m<sup>2</sup> (111,000 psi). The small diameter rolled fiber thus had similar room temperature and elevated temperature strength compared to that of the larger diameter fiber which had not been rolled. The rolled fiber tensile strength values obtained may be low because of possible contamination during the leaching process and the presence of sharp bends formed during the rolling process.

**Composites.** Hot pressed and hot pressed and rolled composites were tested in tension at 1093° C (2000° F). The tensile strength results obtained are shown in Table I and plotted in figure 5. The dashed and solid curve shown in the figure are least square fits of the data obtained for hot pressed composites. The tensile strength values obtained for the rolled composites were approximately the same as those for hot pressed specimens. The percent elongation values appear to be greater for rolled composites as compared to the hot pressed composites.

The highest tensile strength value obtained was for a composite containing 79.5 volume percent 218 tungsten fibers which had a strength of 669 MN/m<sup>2</sup> (97,000 psi) at 1093° C (2000° F). The highest tensile strength value reported for conventional superalloys at this temperature is 345 MN/m<sup>2</sup> (50,000 psi).

### 3.3 STRESS-RUPTURE TESTS

Hot pressed specimens. The stress-rupture properties of hot pressed composites tested at  $1093^{\circ}\text{C}$  ( $2000^{\circ}\text{F}$ ) and a stress of  $240\text{ MN/m}^2$  ( $35,000\text{ psi}$ ) are shown in Table II. The composites contained 218 tungsten fibers having a diameter of  $0.038\text{ cm}$  ( $0.015\text{ in}$ ). Figure 6 is a plot of fiber content contained in the composite as a function of time to rupture. Extrapolation of the curve to rupture in 100 hours indicates that a fiber content of 70 volume percent is required for rupture to occur at a stress of  $240\text{ MN/m}^2$  ( $35,000\text{ psi}$ ). This is in agreement with previous work (8).

Rolled composite specimens. The stress-rupture strengths and depths of fiber reaction obtained for the rolled composites tested at  $1093^{\circ}\text{C}$  ( $2000^{\circ}\text{F}$ ) are given in Table III.

## 4. DISCUSSION

### 4.1 218 TUNGSTEN FIBER COMPOSITES

Fiber-matrix reaction. The most significant result of rolling was its effect in decreasing fiber-matrix reaction. Reaction depth is plotted against exposure time (rupture life) for the rolled composites in figure 7. Also shown in figure 7 are data from ref. 8 on hot pressed composites containing either  $0.020$  or  $0.038\text{ cm}$  ( $0.008$  or  $0.015\text{ in}$ ) diameter 218 tungsten fibers. The rolled composite contained fibers having a smaller diameter than the fibers contained in the hot pressed composites shown in figure 7. Smaller diameter fibers would be expected to be reacted more than larger diameter fibers because of their smaller radius of curvature. The smaller diameter hot pressed fiber, for example, showed a slightly greater reaction with the matrix than the larger diameter fiber. The least squares fit of the rolled composite data indicates that the rolled fiber had a lower level of reactivity with the matrix than fibers contained in hot pressed composites even though the rolled fiber diameter was less than the fibers contained in hot pressed composites.

Surface and grain boundary diffusion are believed to be the rate controlling mechanisms governing fiber-matrix reaction. Results reported for hot pressed tungsten/superalloy composites in ref. 8 indicate that the rate of fiber reaction was influenced by the porosity of the matrix material. The greater the matrix porosity the larger the reaction with the fiber which indicates that surface and grain boundary diffusion are the rate controlling mechanisms governing reaction. This conclusion is further substantiated by results reported in ref. 10. Diffusion rates of tungsten in single crystal and polycrystal nickel were measured at temperatures between  $1100$  and  $1275^{\circ}\text{C}$  ( $2012$  and  $2327^{\circ}\text{F}$ ). Radioautographs taken on sectioned polycrystalline nickel-tungsten diffusion couples clearly showed that grain boundary diffusion took place. The data for the grain boundary diffusion through polycrystalline nickel indicated a rate of diffusion higher by a factor of 3 than volume diffusion at temperatures above  $1100^{\circ}\text{C}$  ( $2012^{\circ}\text{F}$ ). The hot pressed composites studied in this investigation were not fully densified and contained some microporosity. Impact strength results reported in ref. 2 indicate that impurities may be present in the matrix grain boundaries. Both of these factors (matrix porosity and grain boundary impurities) would enhance surface and grain boundary diffusion. Decreased fiber-matrix reaction is believed to be due to the effect rolling has on surface and grain boundary diffusion. Rolling would inhibit surface and grain boundary diffusion by the elimination of matrix porosity and by the elimination of a continuous oxide or other impurity network in the matrix grain boundaries.

Stress-rupture strength. The effect of rolling on the stress-rupture properties of the composites may be determined by comparing the stress-rupture properties of the rolled composites with that of hot pressed composites containing the same cross sectional area fiber so that the effect of the fiber's surface-to-volume ratio is eliminated.

Differences in fiber surface-to-volume ratio would affect composite strength. If, for example, the depth of penetration into a 0.020 cm (0.008 in) diameter fiber was 0.005 cm (0.002 in) after exposure for 100 hours at 1093<sup>o</sup> C (2000<sup>o</sup> F), then 75 percent of the fiber would be reacted. A fiber having a diameter of 0.038 cm (0.015 in) exposed for the same length of time and having the same depth of penetration would only be reacted 46 percent. Reaction lowers the strength of the fiber. The greater the degree of reaction, the lower the strength of the fiber. The differences between fiber surface-to-volume ratios thus must be taken into consideration.

The rolled composite contained fibers having a cross sectional area equivalent to a 0.017 cm (0.0067 in) diameter fiber. Data do not exist for a hot pressed composite containing fibers having a diameter of 0.017 cm (0.0067 in). The stress-rupture strength of a hot pressed composite containing equivalent diameter fibers compared to the rolled composite was calculated using data obtained in ref. 8 for hot pressed composites containing 0.020 cm (0.008 in) diameter fibers. It was assumed that the 0.017 cm (0.0067 in) diameter fiber had stress-rupture properties equivalent to the 0.020 cm (0.008 in) diameter fiber and that the rate of reaction with the matrix was similar. The percent of the hot pressed 0.017 cm (0.0067 in) diameter fiber which was reacted was then calculated for various exposure times and plotted as shown in figure 8. Actual data obtained in ref. 8 for hot pressed composites containing 0.020 cm (0.008 in) diameter fibers and for rolled composites containing 0.017 cm (0.0067 in) equivalent diameter fibers are also shown and plotted in figure 8. Times for similar percent fiber reactions to occur were then graphically determined. For example, 40 percent of a 0.017 cm (0.0067 in) diameter fiber in a hot pressed composite was reacted after 16 hours exposure as compared to 30 hours exposure for the same percent reaction for a 0.020 cm (0.008 in) diameter fiber.

The stress-rupture strength of the smaller diameter fiber was then assumed to be equivalent to the stress-rupture strength of the larger diameter fiber having an equivalent reaction percent. The stress to cause rupture in 16 hours for the smaller diameter fiber in the hot pressed composite, for example, was equivalent to the stress to cause rupture in 30 hours for the larger diameter fiber. A stress rupture curve was then constructed for a hot pressed composite containing 0.017 cm (0.0067 in) diameter fibers by the approach just described. A similar type calculation was made for the rolled composite containing 0.017 cm (0.0067 in) diameter fiber. This calculation would be the expected rolled fiber strength if the properties of the rolled fiber were similar to that obtained for 0.020 cm (0.008 in) diameter fiber in the hot pressed composite. The calculated properties just described and the actual rolled composite data are plotted in figure 9. The least squares fit of the rolled composite data indicate that the rolled composite is stronger in stress-rupture than a hot pressed composite containing equivalent diameter fibers. The plot also indicates, however, that the rolled composite strengths are lower than those obtained from the calculated curve for rolled composites which implies that the rolled unreacted portion of the fiber is weaker in stress-rupture than the unreacted portion of 0.020 cm (0.008 in) diameter fiber. Sharp bends formed in the fiber as a result of the rolling procedure used are believed to partially account for the lower than expected rolled fiber strength.

#### 4.2 TUNGSTEN-1% ThO<sub>2</sub> FIBER COMPOSITES

Fiber-matrix reaction. Figure 10 is a plot of the reaction depth against time of exposure (rupture life) for composites containing tungsten-1% ThO<sub>2</sub> fibers and tested at 1093<sup>o</sup> C (2000<sup>o</sup> F). The dashed curve shown is a least squares fit of the rolled composite data, while the solid curve is a least squares fit of data obtained in ref. 8 for hot pressed composites containing fibers having a diameter of 0.051 cm (0.020 in). The

reaction depth measured for the rolled fiber is seen to be much less than that with fibers contained in composites that were hot pressed only, which is consistent with the results obtained using 218 tungsten fibers. The reaction depth for the rolled fiber was only 53 percent of that obtained for the hot pressed fiber after exposure for 100 hours. The depth of reaction for rolled 218 tungsten fibers was 72 percent of that obtained for the hot pressed fibers. Rolling was thus more effective in lowering reaction for composites containing tungsten-1% ThO<sub>2</sub> fibers in comparison with composites containing 218 tungsten fibers.

The results obtained in this investigation were for composites reduced 80 percent in area by rolling. It was of interest to determine if such a severe degree of working was necessary or if less severe working could be used to obtain the same degree of fiber-matrix reaction. A specimen containing tungsten-1% ThO<sub>2</sub> wire which had been reduced approximately 30 percent in area by rolling was annealed for 170 hours at 1093° C (2000° F). The depth of the fiber reaction zone was measured after exposure and was found to be 0.0043 cm (0.0017 in). The reaction with the fiber for the specimen reduced 30 percent was thus similar to that for specimens reduced 80 percent in area. Large deformations are thus not necessary for lowering fiber reaction.

Stress-rupture strength. The least squares fit of the stress to cause rupture in 100 hours for the rolled fiber indicated that the rolled fiber was slightly stronger than the hot pressed large diameter fiber (340 versus 330 MN/m<sup>2</sup> (50,000 versus 48,000 psi)). Because of the smaller fiber cross section and higher surface-to-volume ratio 52 percent of the rolled fiber was reacted as compared with 45 percent reacted area for the hot pressed large diameter fiber after exposure for 100 hours. If it is assumed that the reacted portion of both fibers contributes the same stress carrying capabilities then the unreacted portion of the rolled fiber must be slightly stronger than

the unreacted portion of the hot pressed large diameter fiber.

The results obtained for tungsten-1% ThO<sub>2</sub> rolled composites suggest that composite properties can be further improved if the starting fiber diameter prior to rolling is greater than 0.051 cm (0.020 in) diameter. Calculations were made to determine the stress-rupture strength of a rolled composite which contained fibers having a starting diameter such that after rolling the fiber diameter was 0.051 cm (0.020 in). The calculated values are plotted in figure 11. Also shown in figure 11 are the values for the hot pressed composite and for the rolled composite studied in this investigation. The calculated rolled composite containing 0.051 cm (0.020 in) diameter fibers is observed to be much stronger than the hot pressed composite having the same diameter fibers. The stress to cause rupture in 100 hours is approximately 35 percent higher for the rolled composite as compared with the hot pressed composite.

## 5. CONCLUDING REMARKS

The results obtained in this investigation have shown that rolling following the initial hot pressing procedure can be beneficial to the stress-rupture properties of tungsten alloy/superalloy composites. Mechanical working by rolling these composites resulted in decreasing the reaction which occurs between the fiber and the matrix material. Because of the reduced fiber-matrix reaction, smaller diameter fibers may be used in the rolled composite to obtain the same stress-rupture strengths as obtained in composites using larger diameter fibers which were only hot pressed and not rolled. Thinner sheet material could thus be fabricated from rolled composites as compared to hot pressed composites without a sacrifice in stress-rupture strength. The stress-rupture strength of hot pressed tungsten/superalloy composites can be improved by rolling. A hot pressed and rolled composite containing the same diameter fiber as



that contained in a hot pressed composite would be expected to be stronger in stress-rupture at 1093<sup>o</sup> C (2000<sup>o</sup> F) than the hot pressed composite. The results obtained in this investigation also suggest that it may be advantageous to fabricate such composites by diffusion bonding of nickel base superalloys in sheet form with refractory metal fibers rather than by the hot pressing powder technique presently employed.

Previous work, ref. 2, has shown that hot pressed and rolled tungsten alloy/superalloy composites had improved impact resistance when compared with composites in the hot pressed condition. The results obtained in the current investigation have shown that improved impact resistance could be obtained without sacrifice in the composite strength properties.

Thermal fatigue of composites is a serious problem that can arise from strain incompatibility. When the composite is subjected to thermal cycles, thermal stresses are generated in the composite due to thermal gradients and to differences in expansivity between the fiber and matrix which leads to matrix cracking. Matrix cracks can reduce composite strength and can expose the fibers to oxidation. A highly densified wrought matrix is more likely to deform without cracking than a less dense as-pressed powder matrix. Although the thermal fatigue resistance of the composites mentioned above has not been determined, rolling may also be beneficial with respect to their resistance to thermal fatigue.

## 6. SUMMARY OF RESULTS

Hot pressed composites of tungsten alloy (fiber)/superalloy (matrix) were mechanically worked by rod rolling and tested in tension and stress rupture at 1093<sup>o</sup> C (2000<sup>o</sup> F) to determine the effect of working on these properties. Reductions in area of up to 80 percent were successfully obtained by rolling such composites at 1093<sup>o</sup> C (2000<sup>o</sup> F). The fibers contained in the composite were also successfully reduced the same amount

without any cracking occurring. The following major results were obtained.

1. Rolling can enhance composite stress-rupture strength. The stress rupture properties of the rolled composites were superior to hot pressed composites containing equivalent diameter fibers. The improved stress rupture strength for the rolled composites was due to reduced fiber reaction.
2. Rolling decreased the degree of fiber-matrix reaction as a function of time of exposure at 1093<sup>o</sup> C (2000<sup>o</sup> F). A decrease in the reaction between the fiber and matrix of up to 50 percent was noted for rolled composites after exposure at 1093<sup>o</sup> C (2000<sup>o</sup> F) for 100 hours as compared with hot pressed composites which had not been rolled. This decrease in fiber reaction occurred for composites reduced in area as little as 30 percent.
3. Rolling did not appreciably affect the 1093<sup>o</sup> C (2000<sup>o</sup> F) ultimate tensile strength of the composites. The 1093<sup>o</sup> C (2000<sup>o</sup> F) ultimate tensile strengths of rolled composites were similar to those obtained for composites which had not been rolled.

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**TABLE I**  
**ULTIMATE TENSILE STRENGTH DATA FOR COMPOSITES**  
**TESTED AT 1093<sup>o</sup> C (2000<sup>o</sup> F)**

Fiber Material	Condition of Composite	Tensile Strength		Vol. % Fiber	% Elongation in 2.5 cm (1 in.)
		MN/m <sup>2</sup>	psi		
218 Tungsten 0.038 cm (0.015 in.) diameter	Hot pressed	321	46,500	40.3	3.2
		341	49,500	45.2	---
		430	62,000	54.7	4.2
		540	78,000	60.8	4.2
		670	97,000	79.5	---
		424	61,500	51.2	---
218 Tungsten 0.017 cm (0.0067 in.) equivalent diameter	Hot pressed and rolled	480	69,000	56.8	6.5
		500	73,000	59.0	8.9
		490	71,000	61.8	7.2
Tungsten-1% ThO <sub>2</sub> 0.051 cm (0.020 in.) diameter	Hot pressed	460	67,000	46.2	4.2
		470	68,000	50.9	4.2
		493	71,500	52.8	4.6
		520	76,000	54.0	4.8
		420	61,000	55.0	---
		530	77,000	55.3	5.4
		570	83,000	60.3	4.9
		540	78,000	64.4	---
		631	91,500	67.5	5.5
Tungsten-1% ThO <sub>2</sub> 0.024 cm (0.0094 in.) equivalent diameter	Hot pressed and rolled	550	80,000	46.8	6.6
		600	87,000	63.9	5.1
		580	84,000	64.3	5.1

**TABLE II**

**STRESS-RUPTURE LIVES OF HOT PRESSED  
COMPOSITES CONTAINING 218 TUNGSTEN  
FIBERS AND TESTED AT 240 MN/m<sup>2</sup>  
(35,000 psi) AND 1093° C (2000° F)**

Life (hrs)	Fiber Content %	Calculated Stress on Fiber	
		MN/m <sup>2</sup>	psi
1.7	48.2	500	73,000
4.4	49.1	490	71,000
5.5	50.0	480	70,000
33.4	62.7	390	56,000

\* Fiber diameter - 0.038 cm (0.015 in.)

**TABLE III**

**STRESS-RUPTURE PROPERTIES OF MECHANICALLY WORKED COMPOSITES  
TESTED AT 1093° C (2000° F)**

Fiber Material	Composite Stress		Rupture Life hrs.	Calculated Stress on Fiber		Vol. % Fiber	Fiber- matrix cm	Reaction Depth in.	
	MN/m <sup>2</sup>	psi		MN/m <sup>2</sup>	psi				
218 Tungsten 0.017 cm (0.0067 in.) equivalent diameter	140	20,000	150.6	220	32,000	61.6	0.00269	0.00106	
			300.4	210	31,000	65.3	.00462	.00182	
			378.5	230	33,000	60.5	.00356	.00140	
	170	25,000	33.0	330	48,000	52.4	.00132	.00052	
			52.4	350	50,000	49.7	.00203	.00080	
	200	30,000	22.9	400	58,000	51.9	.00198	.00078	
			41.8	360	52,000	58.7	.00132	.00052	
			83.1	340	49,000	61.8	.00236	.00093	
	240	35,000	26.3	360	52,000	66.9	.00231	.00091	
			32.7	390	57,000	61.3	.00132	.00052	
	Tungsten-1% ThO <sub>2</sub> 0.024 cm (0.0094 in.) equivalent diameter	140	20,000	82.6	300	43,000	46.3	.00165	.00065
				243.6	280	41,000	48.8	.00330	.00130
170		25,000	114.3	280	41,000	61.2	.00462	.00182	
			171.1	260	38,000	66.6	.00462	.00182	
200		30,000	83.2	350	51,000	59.4	.00462	.00182	
			97.6	380	55,000	54.6	.00396	.00156	
240		35,000	147.8	520	76,000	45.9	.00305	.00120	
			51.3	480	70,000	49.9	.00330	.00130	
			266.5	370	54,000	65.5	.00462	.00182	

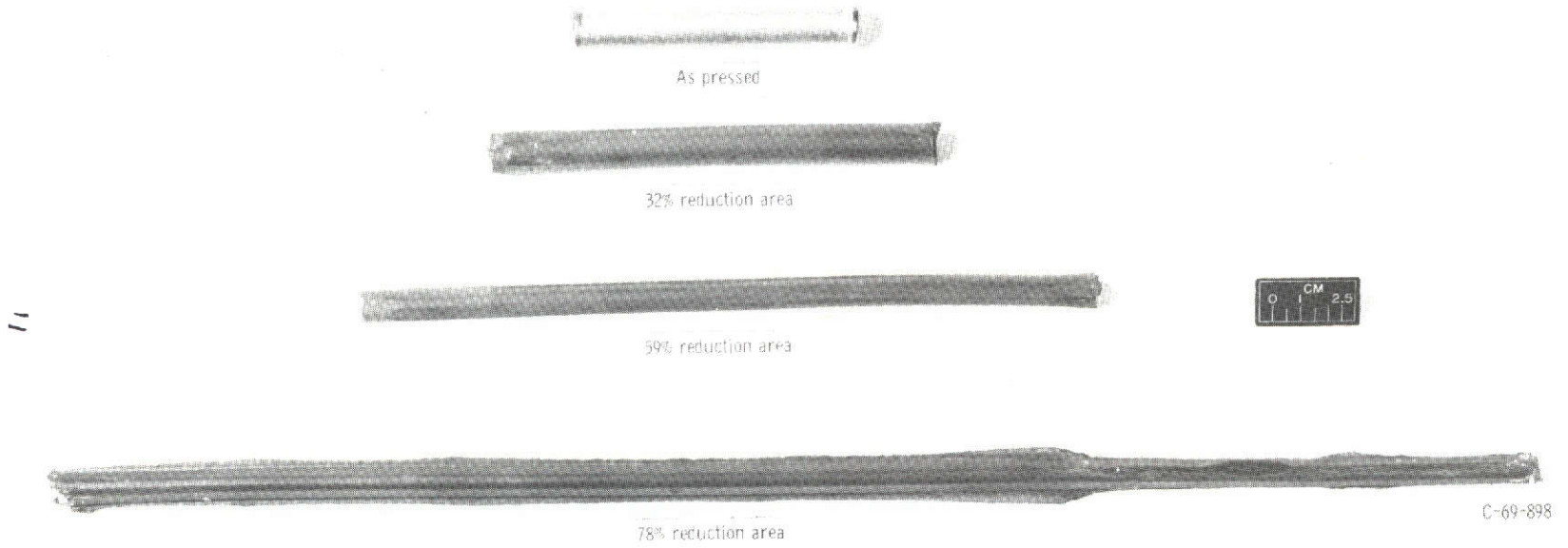
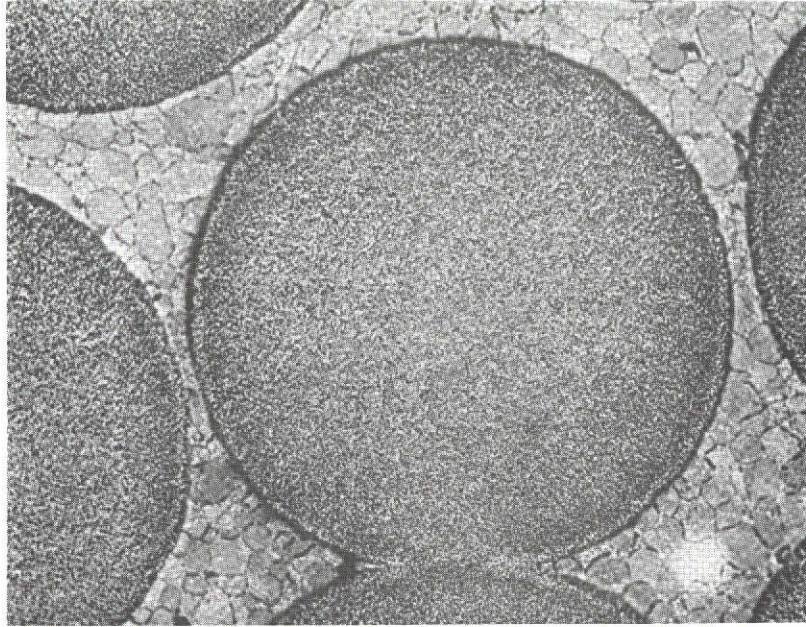
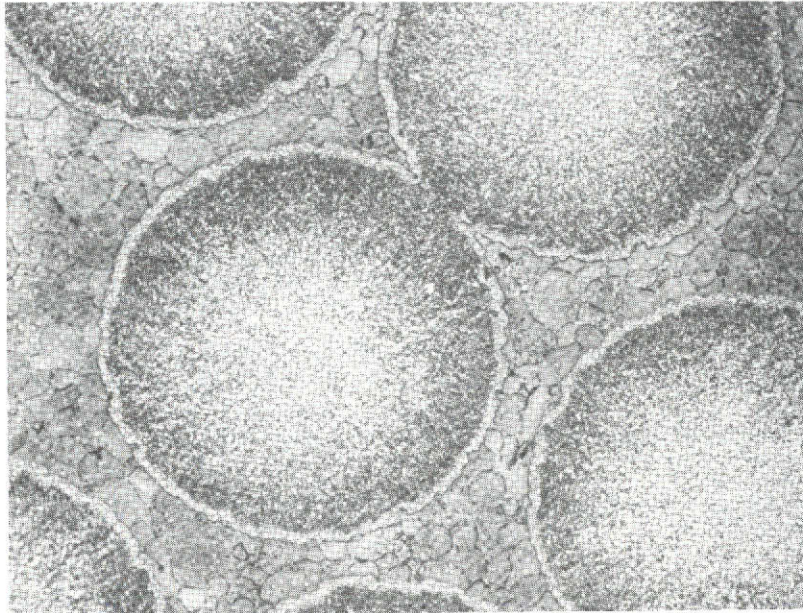


Figure 1. - Typical rolling rods in the hot pressed condition and after various reductions by hot rolling.

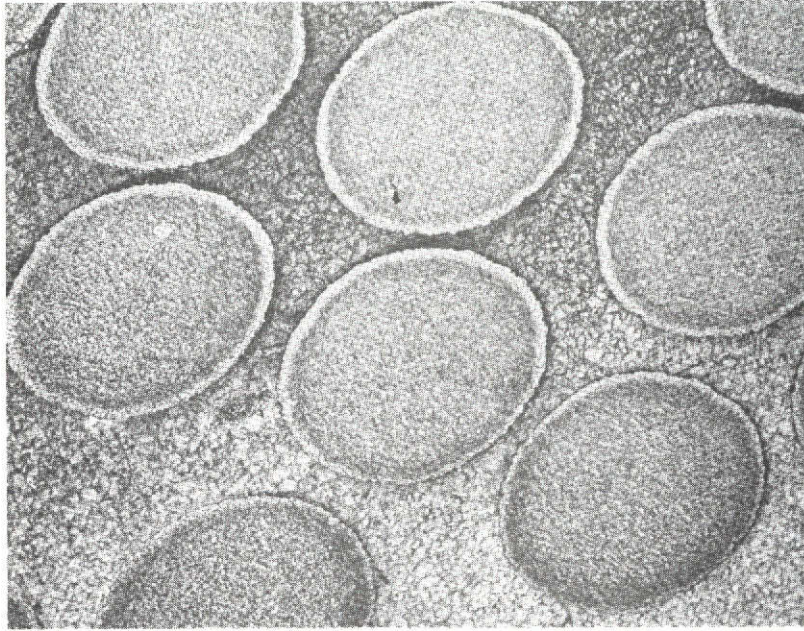


(a) Fiber material-tungsten- 1% ThO<sub>2</sub> 0.051 cm (0.020 in.) diameter. X150.

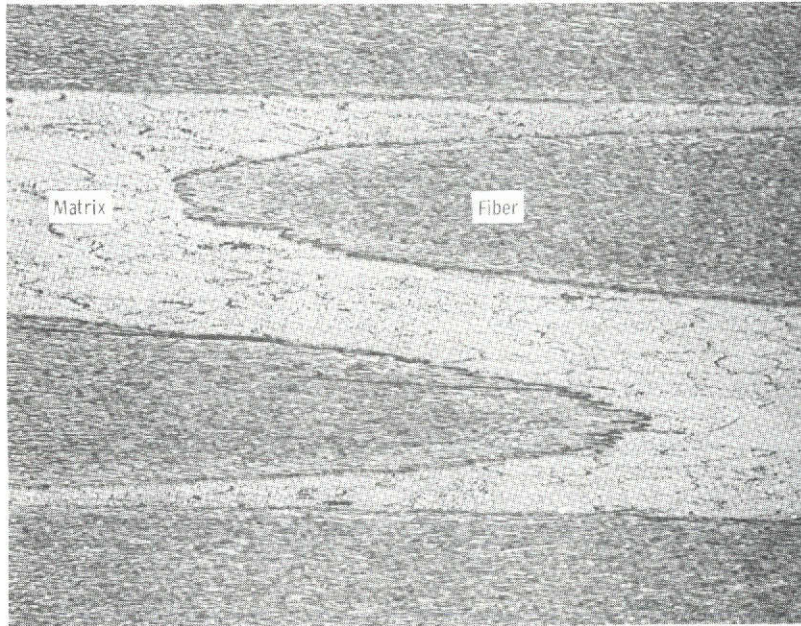


(b) Fiber material-218 tungsten 0.038 cm (0.015 in.) diameter. X150.

Figure 2. - Transverse section of refractory fiber-superalloy composites in the hot pressed condition.

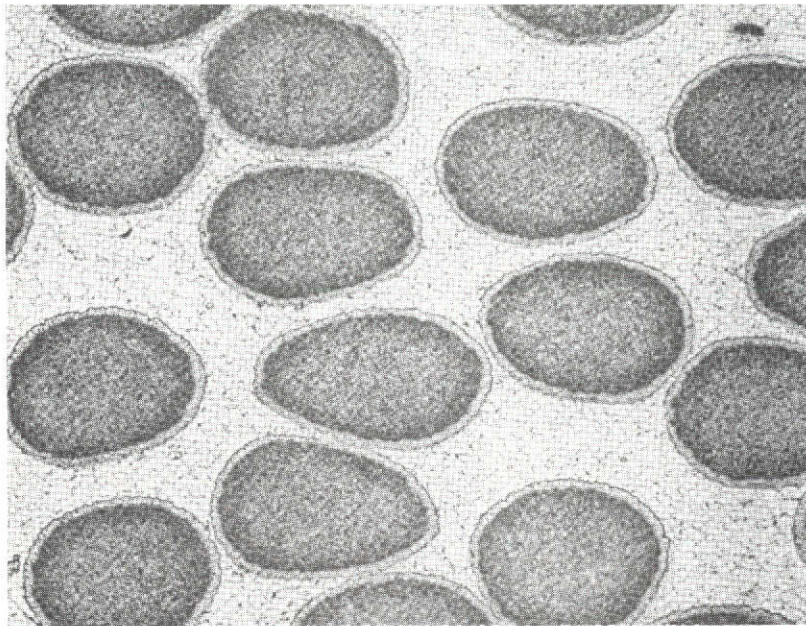


(a) Transverse section.

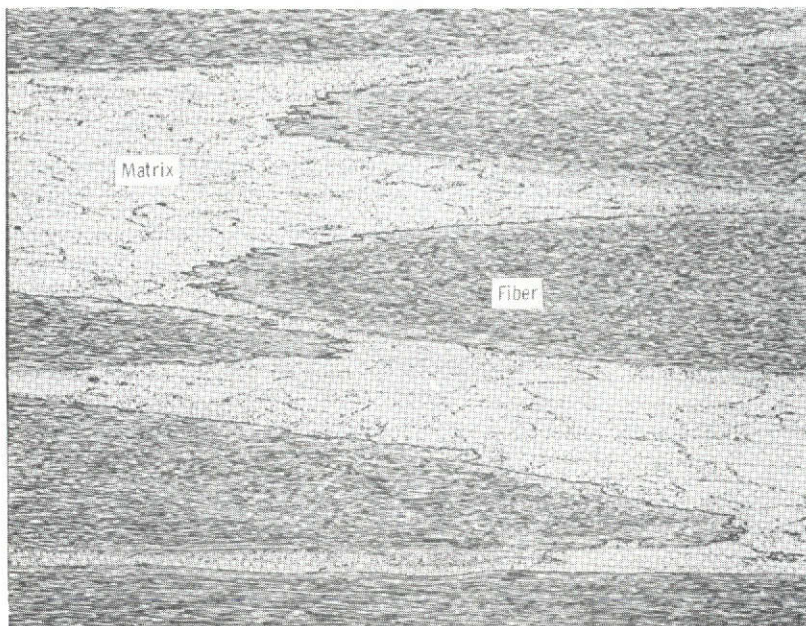


(b) Longitudinal section.

Figure 3. - Microstructure of rolled tungsten-1% ThO<sub>2</sub> fiber-superalloy composite, X150.



(a) Transverse section.



(b) Longitudinal section.

Figure 4. - Microstructure of rolled 218 tungsten fiber-superalloy composite, X150.

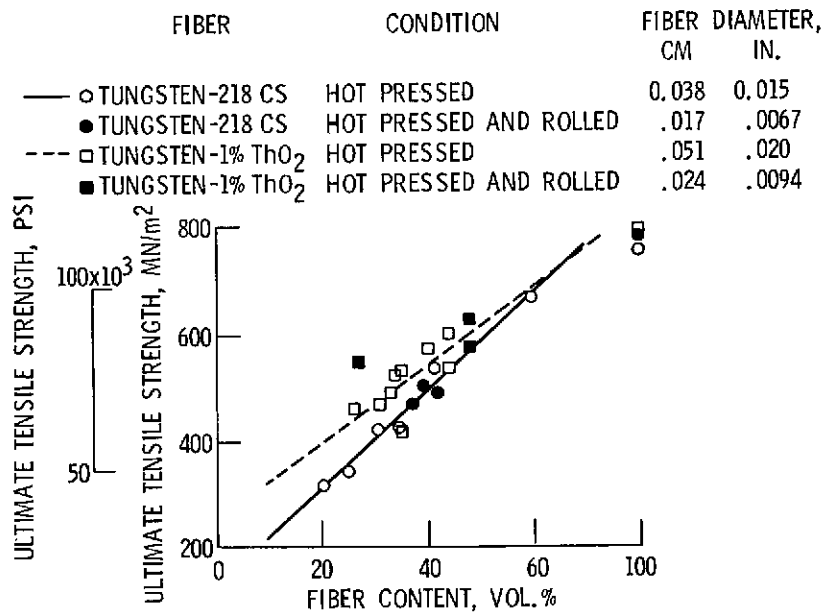


Figure 5. - Ultimate tensile strength of refractory fiber-superalloy composites at  $1093^{\circ}\text{C}$  ( $2000^{\circ}\text{F}$ ).

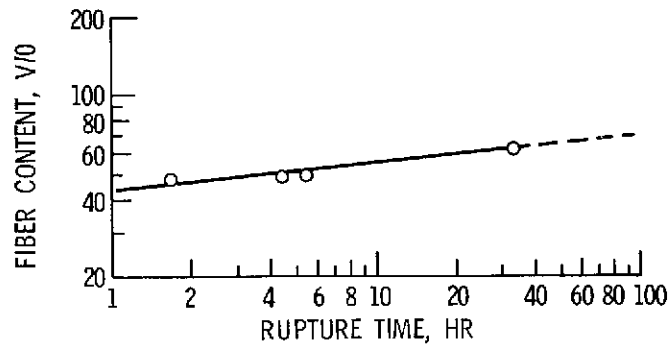


Figure 6. - Fiber content versus rupture time for hot pressed 218 tungsten fiber-superalloy composites at  $240\text{ MN/m}^2$  ( $35\ 000\text{ psi}$ ) and  $1093^{\circ}\text{C}$  ( $2000^{\circ}\text{F}$ ). Fiber diameter,  $0.038\text{ cm}$  ( $0.015\text{ in.}$ ).



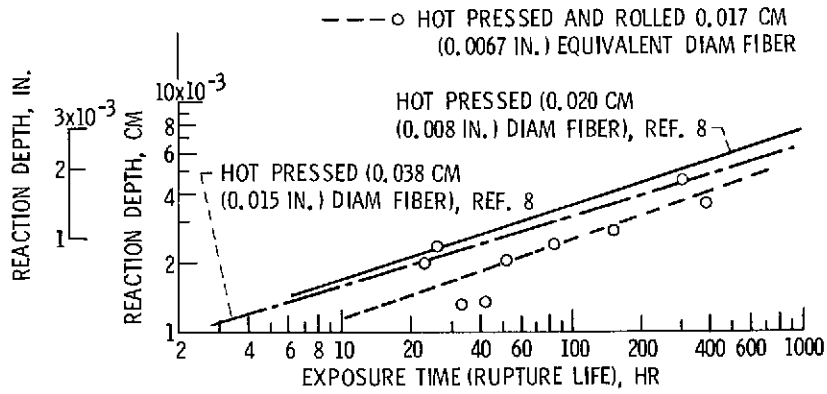


Figure 7. - Effect of rolling on fiber reaction depth for 218 tungsten fibers in superalloy composites. Test temperature, 1093° C (2000° F).

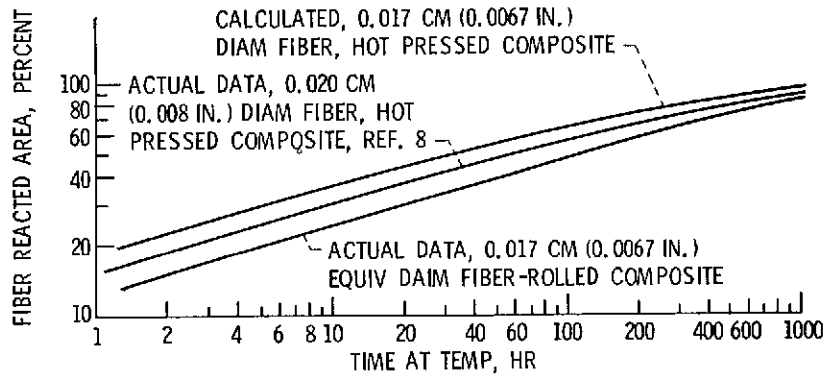


Figure 8. - Effect of rolling on the area of fiber reacted for 218 tungsten fibers in superalloy composites. Temperature, 1093° C (2000° F).

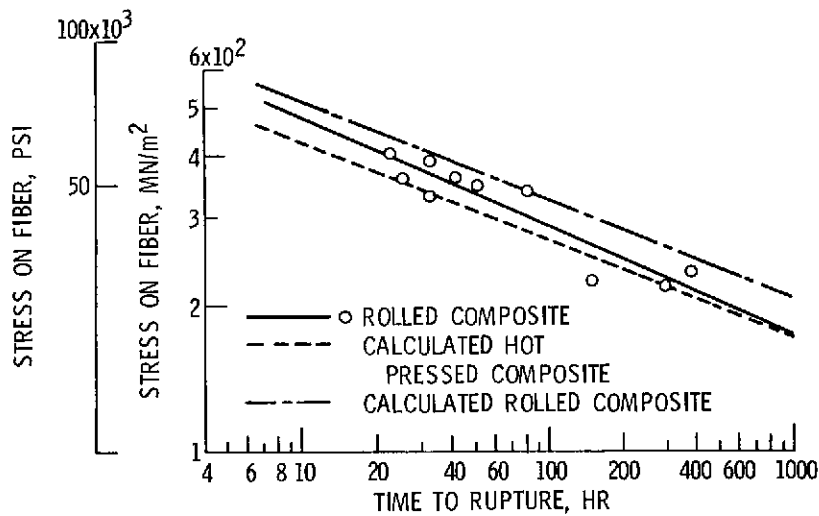


Figure 9. - Effect of rolling on the 1093° C (2000° F) rupture strength of 218 tungsten fibers in superalloy composites.

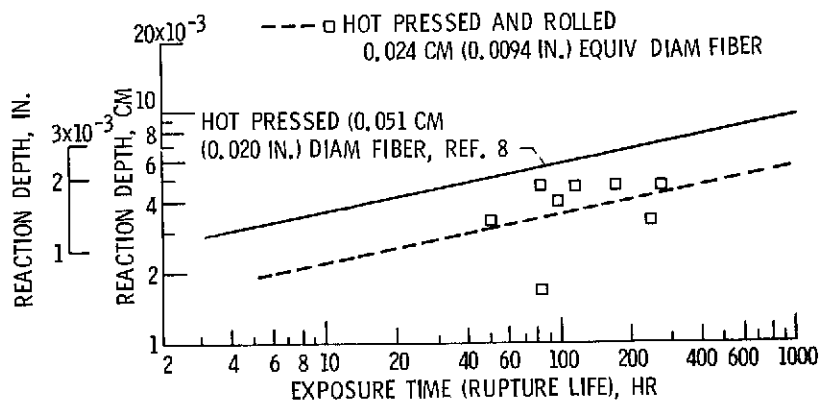


Figure 10. - Effect of rolling on fiber reaction depth for tungsten-1% ThO<sub>2</sub> fibers in superalloy composites. Test temperature, 1093° C (2000° F).

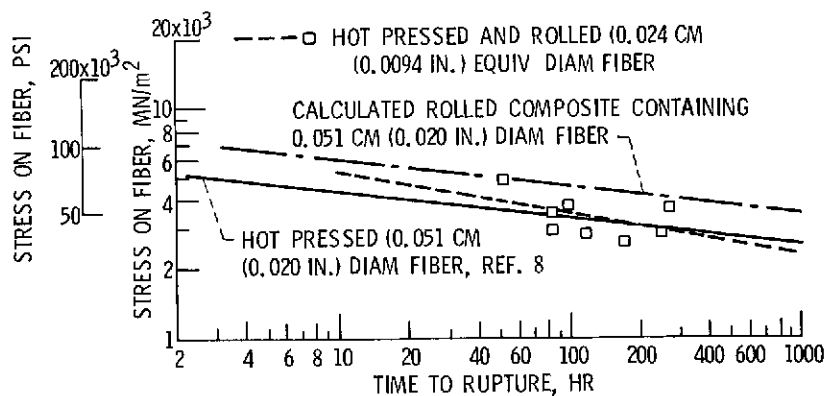


Figure 11. - Effect of rolling on the 1093° C (2000° F) rupture strength of tungsten-1% ThO<sub>2</sub> fibers in superalloy composites.