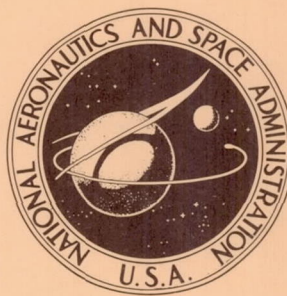


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STRESS-RUPTURE AND TENSILE PROPERTIES  
OF REFRACTORY-METAL WIRES AT 2000°  
AND 2200° F (1093° AND 1204° C)

*by Donald W. Petrusek and Robert A. Signorelli*

*Lewis Research Center*

*Cleveland, Ohio*

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NATIONAL AERONAUTICS AND SPACE ADMINISTRATION

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#### ABSTRACT

Tensile and stress-rupture tests were conducted on tungsten-, molybdenum-, and columbium-base alloy wires at room temperature, 2000<sup>o</sup> F (1093<sup>o</sup> C), and 2200<sup>o</sup> F (1204<sup>o</sup> C). Metallographic examinations were also made of the wire microstructure after testing. Ultimate tensile strength values of up to 240 000 psi (1655 MN/m<sup>2</sup>) for W - 5 percent Re - 2 percent ThO<sub>2</sub> wire tested at 2000<sup>o</sup> F (1093<sup>o</sup> C) and up to 160 000 psi (1103 MN/m<sup>2</sup>) for W - 3 percent Re wire tested at 2200<sup>o</sup> F (1204<sup>o</sup> C) were obtained. The best strength values obtained for a 100-hour rupture life were 96 000 psi (662 MN/m<sup>2</sup>) at 200<sup>o</sup> F (1093<sup>o</sup> C) and 69 000 psi (476 MN/m<sup>2</sup>) at 2200<sup>o</sup> F (1204<sup>o</sup> C), for W - 2 percent ThO<sub>2</sub> wire. The properties obtained suggested that the wires studied showed promise as potential fiber reinforcement in the 2000<sup>o</sup> to 2200<sup>o</sup> F (1093<sup>o</sup> to 1204<sup>o</sup> C) temperature range.



# STRESS-RUPTURE AND TENSILE PROPERTIES OF REFRACTORY-METAL

WIRES AT 2000<sup>0</sup> AND 2200<sup>0</sup> F (1093<sup>0</sup> and 1204<sup>0</sup> C)

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## SUMMARY

Tensile and stress-rupture tests were conducted on refractory-metal alloy wires at room temperature, 2000<sup>0</sup> F (1093<sup>0</sup> C) and 2200<sup>0</sup> F (1204<sup>0</sup> C). Wires of alloys molybdenum - 1.25 percent titanium - 0.30 percent zirconium - 0.15 percent carbon (TZC), molybdenum - 0.5 percent titanium - 0.08 percent zirconium - 0.015 percent carbon (TZM), molybdenum - 0.5 percent titanium (Mo - 0.5Ti), columbium - 20 percent tungsten - 1 percent zirconium (AS-30), columbium - 28 percent tantalum - 10 percent tungsten - 1 percent zirconium - 0.005 percent carbon (FS-85), doped tungsten, thoriated tungsten, tungsten-rhenium, and tungsten-rhenium-thoria were tested in the 5- to 15-mil- (0.013- to 0.038-cm-) diameter range. The wires were tested in a vacuum of  $1 \times 10^{-6}$  to  $5 \times 10^{-5}$  torr in tension and for rupture times up to 200 hours. The wire specimens were examined after fracture, and reduction-in-area measurements were made. Metallographic examinations were also made of the wire microstructure after testing.

Ultimate tensile strength values obtained were 240 000 psi (1655 MN/m<sup>2</sup>) for tungsten - 5 percent rhenium - 2 percent thoria (W - 5Re - 2ThO<sub>2</sub>) wire tested at 2000<sup>0</sup> F (1093<sup>0</sup> C) and 160 000 psi (1103 MN/m<sup>2</sup>) for tungsten - 3 percent rhenium (W - 3Re) wire at 2200<sup>0</sup> F (1204<sup>0</sup> C). Strength/density values for these materials were 350 000 inches (8.9 × 10<sup>3</sup> m) for W - 5Re - 2ThO<sub>2</sub> at 2000<sup>0</sup> F (1093<sup>0</sup> C) and 250 000 inches (6.4 × 10<sup>3</sup> m) for W - 3Re at 2200<sup>0</sup> F (1204<sup>0</sup> C). The best strength value obtained for a 100-hour rupture life was 96 000 psi (662 MN/m<sup>2</sup>) at 2000<sup>0</sup> F (1093<sup>0</sup> C) and 69 000 psi (476 MN/m<sup>2</sup>) at 2200<sup>0</sup> F (1204<sup>0</sup> C), for tungsten - 2 percent thoria (W - 2ThO<sub>2</sub>) wire.

The tensile and stress-rupture strengths of the wires investigated, with the exception of the AS-30 wire material, were superior to those reported for other forms of refractory metals. The superior properties obtained suggested that the wires studied showed promise as potential fiber reinforcement in the 2000<sup>0</sup> to 2200<sup>0</sup> F (1093<sup>0</sup> to 1204<sup>0</sup> C) temperature range. These results indicate that it may be possible to produce fiber-reinforced nickel or cobalt superalloys with over three times the tensile strength and up to five times the 100-hour rupture strength at 2000<sup>0</sup> F (1093<sup>0</sup> C) of the strongest superalloys.



## INTRODUCTION

The attractive high-strength potential of fiber composites is largely based on the properties of the fibers. As such, there is a need for fibers with improved properties and for fiber mechanical property data to aid in the selection and design of fiber-reinforced composite materials.

Refractory-metal alloy wires are of interest for fiber reinforcement of superalloy-type matrix materials for use between 2000<sup>o</sup> and 2200<sup>o</sup> F (1093<sup>o</sup> and 1204<sup>o</sup> C) because of their high strength at these temperatures. In previous work at the Lewis Research Center, composites of refractory-metal, fiber-reinforced, nickel-base alloys were produced that had stress-rupture properties superior to conventional superalloys at use temperatures of 2000<sup>o</sup> and 2200<sup>o</sup> F (1093<sup>o</sup> and 1204<sup>o</sup> C), reference 1. Stress for 1000-hour rupture values as great as four times that for the strongest conventional superalloys were obtained at 2000<sup>o</sup> F (1093<sup>o</sup> C). The refractory-metal alloy wires used in reference 1 had been developed for use as lamp filament materials and were not optimum for fiber reinforcement. Even stronger composites are possible when higher-strength fibers are used. The need for stronger refractory-metal alloy wire was recognized several years ago, and efforts have been sponsored by Lewis to fabricate stronger alloys into wire form. Data for these materials in fiber form would be useful in indicating the potential for such materials as reinforcing fibers.

Some work has been reported on the stress-rupture and creep-rupture properties of tungsten and tungsten alloy wire (refs. 2 to 5). Most of the data obtained, however, was on commercially drawn tungsten wire. This investigation was, therefore, conducted to evaluate the tensile and stress-rupture properties of these candidate materials for potential reinforcement of superalloys. Tensile and stress-rupture tests were conducted at 2000<sup>o</sup> and 2200<sup>o</sup> F (1093<sup>o</sup> and 1204<sup>o</sup> C). Wires of alloys TZC, TZM, Mo - 0.5 Ti, AS-30, FS-85, doped tungsten, thoria-tungsten, tungsten-rhenium, and tungsten-rhenium-thoria were tested in the 5- to 15-mil- (0.013- to 0.038-cm-) diameter range.

The wires were tested in a vacuum of  $1 \times 10^{-6}$  to  $5 \times 10^{-5}$  torr in tension and for rupture times up to 200 hours. The wire specimens were examined after fracture, and reduction-in-area measurements were made. Metallographic examinations were also made of the wire microstructure after testing to relate the observed structure to measured properties; however, the strengthening mechanisms were not investigated.

## MATERIALS, APPARATUS, AND PROCEDURE

### Wire Material

Wire compositions and diameters selected for study are listed in table I. All wire except the two tungsten-rhenium-thoria alloys was tested in the as-drawn, cleaned, and



straightened condition. The two tungsten-rhenium-thoria alloys also were tested after a thermal anneal treatment of 1 minute at  $3000^{\circ}\text{F}$  ( $1650^{\circ}\text{C}$ ). The  $\text{W} - 5\text{Re} - 2\text{ThO}_2$  alloy was also tested in a harder condition than that drawn commercially. The wire was drawn, without annealing treatments, from 0.024 to 0.008 inch diameter (0.061 to 0.020 cm). The commercially drawn condition is designated as process A in table I while the hard-drawn condition is designated as process B.

A chemical analysis of several of the wire materials was made. The chemical analysis results are listed in table II.

### Tensile Tests

The wire was tested in tension in a vacuum chamber at a vacuum of  $1 \times 10^{-6}$  to  $5 \times 10^{-5}$  torr using a constant-strain, screw-driven tensile machine. The strain rate used for all tests was 0.1 inch (0.25 cm) per minute. Tensile tests were conducted at room temperature,  $2000^{\circ}\text{F}$  ( $1093^{\circ}\text{C}$ ), and  $2200^{\circ}\text{F}$  ( $1204^{\circ}\text{C}$ ).

### Stress-Rupture Tests

The equipment used to conduct constant-load, stress-rupture tests is shown and described in detail in reference 5 and shown in figure 1. The wire was cut to 15 inch

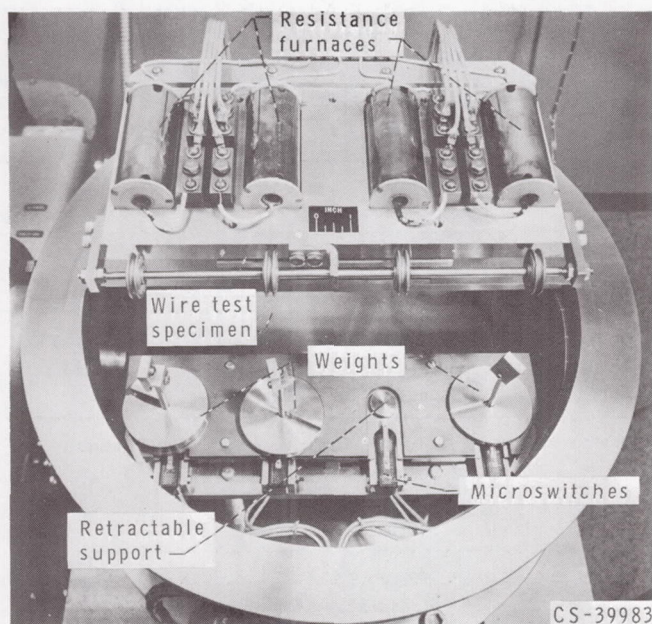


Figure 1. - Fiber stress-rupture testing apparatus.



(38 cm) lengths, and then clamped to a fixed mount, strung through the tantalum-wound resistance furnace, passed over a pulley, and attached to the appropriate weight. The weights were supported by the retractable supports while the furnaces and wire test specimens were heated to the test temperature and stabilized. Microswitches were actuated by the fallen weights as each specimen broke, disconnecting power to the furnace and recording the time to fracture. The entire assembly was covered by a cooled metal bell jar. Testing was conducted in a vacuum of  $1 \times 10^{-6}$  to  $5 \times 10^{-5}$  torr. Individual furnace temperatures, as well as wire test specimens, were monitored with platinum-platinum-13 percent rhodium thermocouples. The test temperature did not vary more than  $\pm 5^{\circ}$  F ( $\pm 3^{\circ}$  C) during the course of the test.

### Reduction-in-Area Measurements

After testing, the fracture area of each wire specimen was examined with a microscope at a magnification of  $\times 100$ . Reduction-in-area calculations were based on the difference between the known original wire area and the average of two observations, taken after testing, at a magnification of  $\times 100$ .

### Microstructural Analysis

After testing, the fracture edges of wire specimens were mounted in epoxy resin and metallographically polished using successively finer grit abrasive papers (to 600-grit size). The specimens were next polished with 3- and 0.5-micron diamonds, successively.

The specimens were then etched with the following solutions:

Wire material	Etch	Method of applying etch
Tungsten and tungsten-thoria	50 ml ammonium hydroxide and 10 ml hydrogen peroxide	Immersion for 1 minute
Tungsten-rhenium and tungsten-rhenium-thoria	100 ml water, 3 grams potassium ferricyanide, and 1 gram sodium hydroxide	Swabbing
Molybdenum alloys	100 ml water, 10 grams potassium ferricyanide, and 4 grams of sodium hydroxide	Swabbing
Columbium alloys	10 grams sodium hydroxide, 3 grams tartaric acid, 85 ml water, and 5 ml hydrogen peroxide	Swabbing



After etching, the samples were cleaned for several minutes in an ultrasonic bath of water and were given a final cleaning for 1 minute in an ultrasonic bath of ethyl alcohol. The specimens were then dried in a warm air blast, and the sample surface was further cleaned by dry-stripping with a replicating medium.

A two-step technique was used to replicate the wires. The samples were first replicated with 0.025 percent Mowital dissolved in chloroform and, after drying, 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 100-Å layer of carbon. The replica was cut into grid-size squares and placed into 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 a magnification of  $\times 16\ 600$ .

## RESULTS

### Tensile Properties

Room temperature. - The room-temperature tensile properties of the wire materials investigated are tabulated in table III. Most of the tungsten alloy wire materials were much stronger than the molybdenum or columbium alloy wire materials studied. The W - 5Re - 2ThO<sub>2</sub> wire in the hard-drawn condition was the strongest wire material studied at room temperature, having a tensile strength of 446 000 psi (3075 MN/m<sup>2</sup>). The molybdenum and columbium alloys had equivalent strength properties and were found to be much more ductile at room temperature than all but one of the tungsten alloys, W - 26Re.

Note that the strength of hard-drawn W - 5Re - 2ThO<sub>2</sub> wire was 50 percent greater than that obtained for commercially drawn wire. The ductility loss was slight, as noted in table III.

Elevated temperature. - The elevated-temperature tensile properties of the wires studied are tabulated in table IV. The tungsten-base alloys are much stronger than either the molybdenum- or columbium-base alloy wire materials at both 2000<sup>o</sup> and 2200<sup>o</sup> F (1093<sup>o</sup> and 1204<sup>o</sup> C). At 2000<sup>o</sup> F (1093<sup>o</sup> C) the tungsten alloys containing rhenium additions were the strongest wire materials studied. The hard-drawn W - 5Re - 2ThO<sub>2</sub> wire had a tensile strength of 243 000 psi (1675 MN/m<sup>2</sup>) at 2000<sup>o</sup> F (1093<sup>o</sup> C). Sufficient material was not available for testing this material at 2200<sup>o</sup> F (1204<sup>o</sup> C).

Wire ultimate tensile strength as a function of test temperature is shown in figure 2 for tungsten-base alloy wires. The largest dropoff in strength between room temperature and 2000<sup>o</sup> F (1093<sup>o</sup> C) was for the W - 218CS wire material. The wire materials containing tungsten-rhenium had the best percentage of strength retention between room temper-



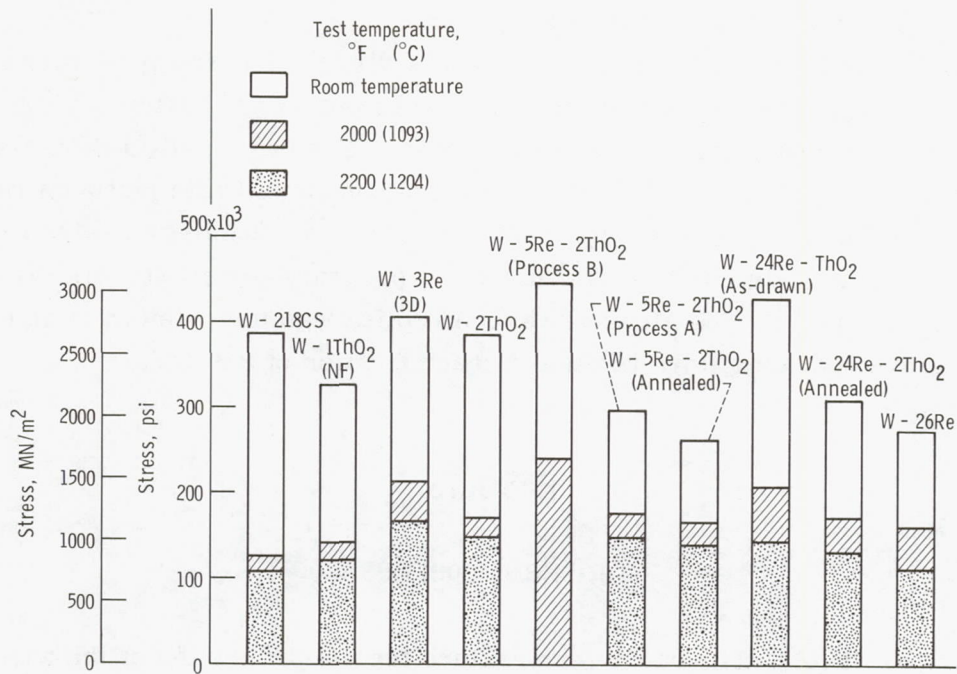


Figure 2. - Ultimate tensile strength of tungsten alloy wires.

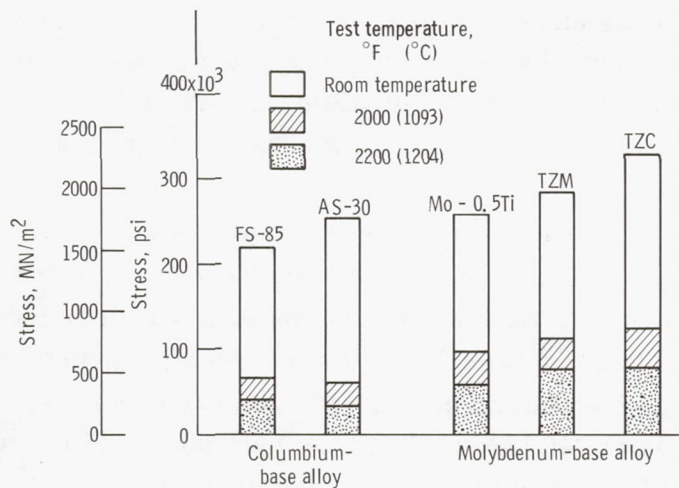


Figure 3. - Ultimate tensile strength of molybdenum alloy and columbium alloy wires.

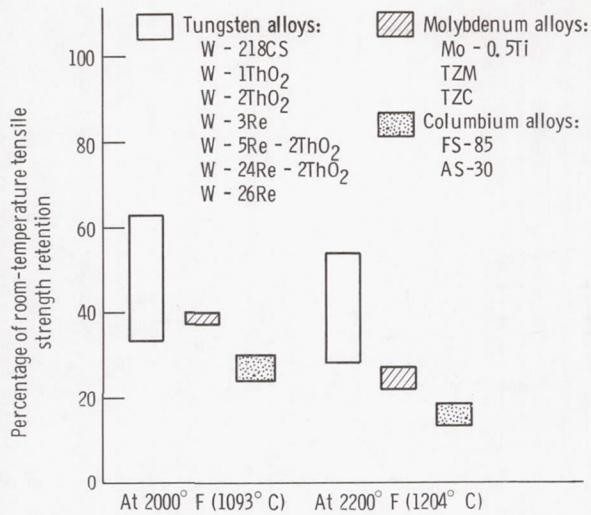


Figure 4. - Percentage of room-temperature tensile strength retention for wire.

ature and 2000° F (1093° C). However, these materials have the poorest strength retention between 2000° and 2200° F (1093° to 1204° C).

Figure 3 shows ultimate tensile strength as a function of test temperature for the molybdenum alloy and columbium alloy wires. The strongest molybdenum alloy is TZC followed by TZM and then Mo - 0.5Ti. The columbium-base alloy FS-85 is slightly stronger than AS-30 at elevated temperatures.

Figure 4 shows the percentage of tensile strength retention of the wire materials. The percentage of strength retention was calculated between room temperature and 2000° F (1093° C) and between room temperature and 2200° F (1204° C). The tungsten alloy wire materials show the best strength retention, while the columbium alloys show the poorest strength retention. The strength retention of the molybdenum alloys appears quite good up to 2000° F (1093° C), and competes with some of the tungsten alloys. At 2200° F (1204° C), however, the strength retention is lower than that of any of the tungsten alloys. The columbium-alloy strength retention does not compete with that of the other two materials.

## Stress-Rupture Properties

Stress-rupture strength. - Results of stress-rupture tests on the wire materials studied are tabulated in table V and plotted as stress to cause failure against rupture life in figures 5 to 8. The strongest wire material for rupture times greater than 50 hours at 2000° F (1093° C) was found to be W - 2ThO<sub>2</sub> followed by the hard-drawn tungsten-rhenium-thoria alloy and the W - 1ThO<sub>2</sub> wire. The TZM and TZC molybdenum alloys had



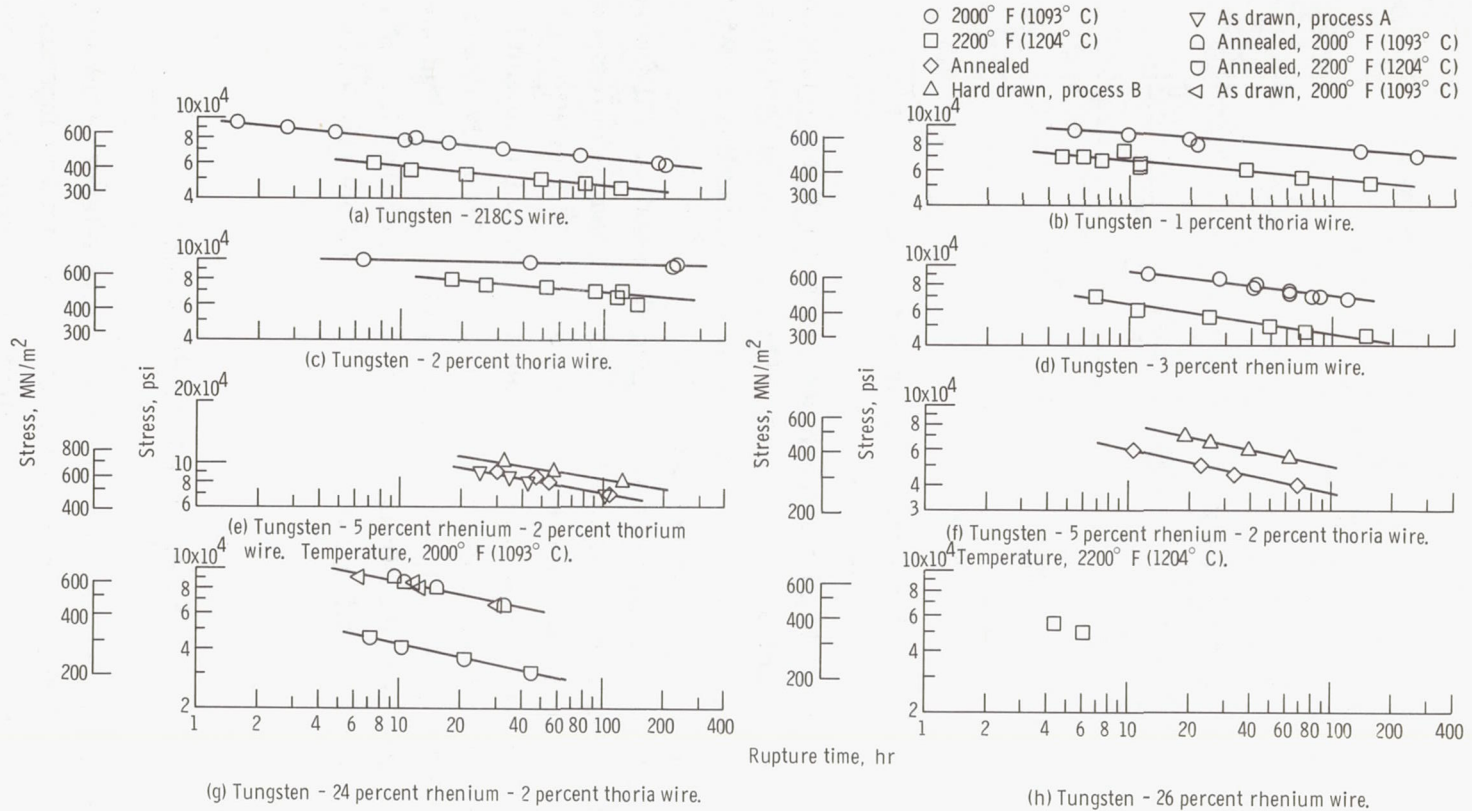


Figure 5. - Stress-rupture properties of tungsten alloy wire.

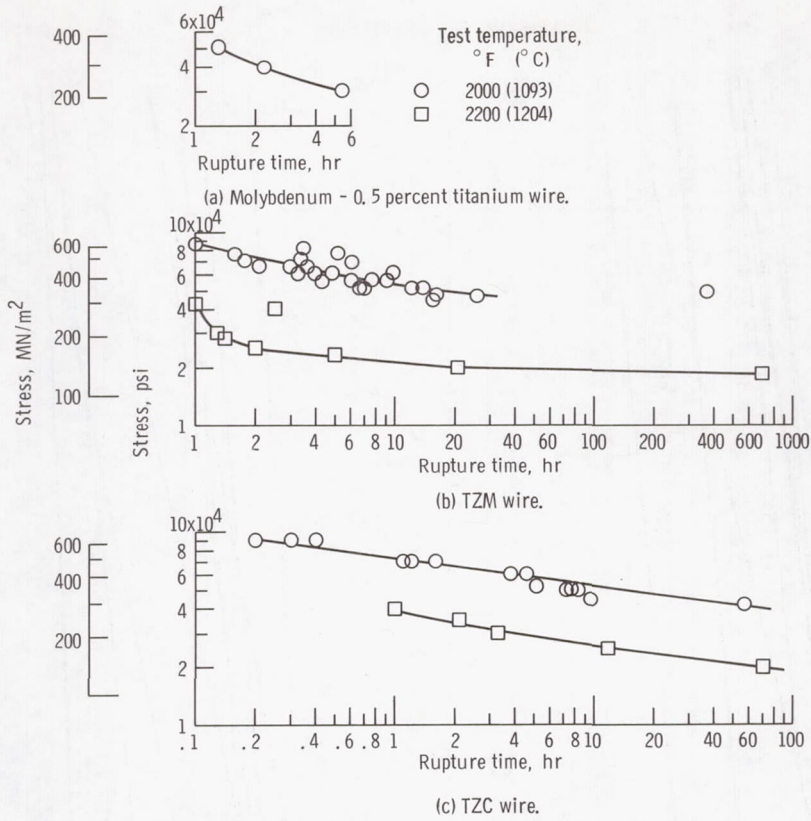


Figure 6. - Stress-rupture properties of molybdenum alloy wire.

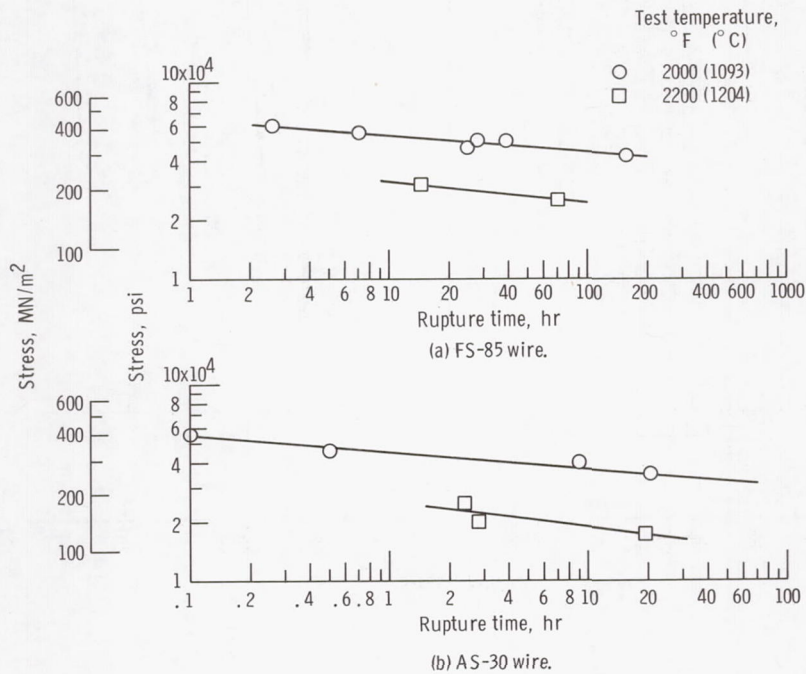


Figure 7. - Stress-rupture properties of columbium alloy wire.



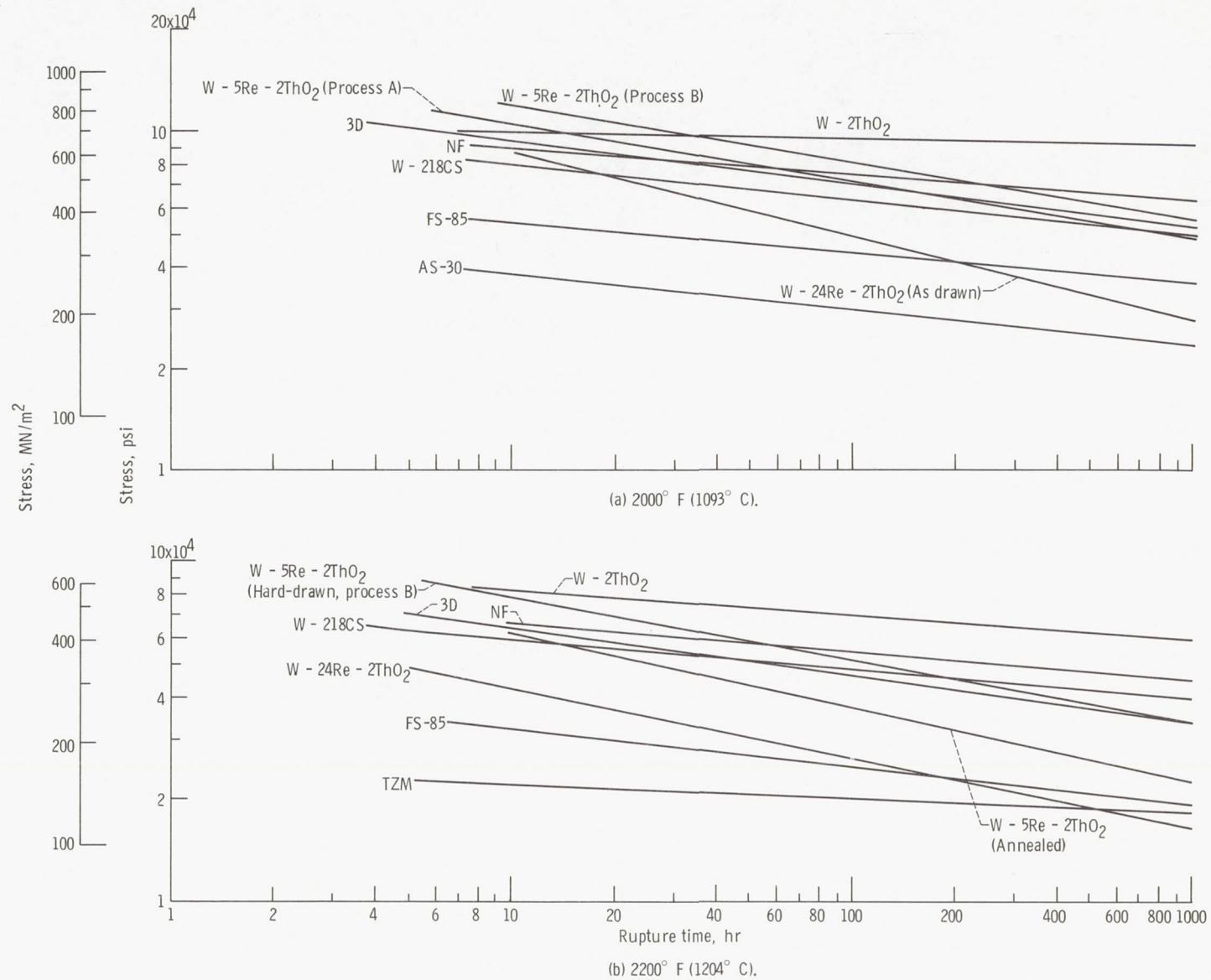


Figure 8. - Stress to cause rupture of wires at 2000° and 2200° F (1093° and 1204° C).

lower properties than the tungsten alloys. The two molybdenum alloys had equivalent stress-rupture properties at this temperature. The columbium alloy having the best stress-rupture properties was FS-85. At 2200<sup>o</sup> F (1204<sup>o</sup> C) the W - 2ThO<sub>2</sub> wire again was superior in stress-rupture strength to the other wire materials tested, followed by the W - 1ThO<sub>2</sub> wire.

The stress to cause rupture is plotted as a function of rupture life for the majority of the wires tested at 2000<sup>o</sup> F (1093<sup>o</sup> C) in figure 8(a) so that their relative stability with time can be compared. The slopes of the stress-rupture curves show the thoriated tungsten wire materials not containing rhenium to be the most stable and the strongest at this temperature, with the 2 percent thoriated wire having the best stability. The W - 218CS wire material also has a surprisingly shallow slope and is almost as stable as the 1 percent thoriated tungsten. The addition of 3 percent rhenium to the tungsten results in a less stable wire material, as shown in figure 8(a). The W - 5Re - 2ThO<sub>2</sub> wire has a steeper slope than does the 3 percent rhenium - tungsten wire material. The W - 24Re - 2ThO<sub>2</sub> wire has the steepest slope of any of the wire materials tested at this temperature. The addition of rhenium to tungsten thus appears to adversely affect the stability of the wire in stress rupture. For short-time applications, however, the wire materials containing rhenium are stronger than the other wire materials studied.

The same type of plot for the 2200<sup>o</sup> F (1204<sup>o</sup> C) stress-rupture tests is shown in figure 8(b). The TZM molybdenum alloy was the most stable of all the wires studied. The W - 2ThO<sub>2</sub> wire was most stable of the tungsten-base alloys at 2200<sup>o</sup> F (1204<sup>o</sup> C). The stability of the as-drawn tungsten-rhenium-thoria wire was slightly less than that in the hard-drawn condition and the same as that of the tungsten - high rhenium - thoria wire material.

Stress-rupture ductility. - The elevated-temperature ductility of the wire specimens tested in this investigation was determined by reduction-in-area measurements. The reduction-in-area results are presented in table V. Plots were made of reduction in area as a function of rupture time to determine if any trend exists between these two factors for the wire material tested. The majority of the data did not show a significant change in ductility as a function of rupture time but did show a significant change in comparison to tensile strength ductility. A noticeable change in ductility as a function of rupture time was observed for W - 218CS, W - 3Re, and FS-85 wire materials. A plot of reduction in area as a function of rupture time for W - 218CS wire tested at 2000<sup>o</sup> and 2200<sup>o</sup> F (1093<sup>o</sup> and 1204<sup>o</sup> C) is shown in figure 9(a). At 2000<sup>o</sup> F (1093<sup>o</sup> C) the ductility decreases with time, while at 2200<sup>o</sup> F (1204<sup>o</sup> C) no clear trend is evident other than that the ductility in rupture is much less than that in tension. Figure 9(b) is a plot of reduction in area as a function of rupture time for the W - 3Re wire tested at 2000<sup>o</sup> and 2200<sup>o</sup> F (1093<sup>o</sup> and 1204<sup>o</sup> C). Ductility decreases as a function of rupture time at both temperatures. The same type of plot is shown for FS-85 in figure 9(c) for specimens tested at 2000<sup>o</sup> F (1093<sup>o</sup> C). The ductility again decreases as a function of rupture time.



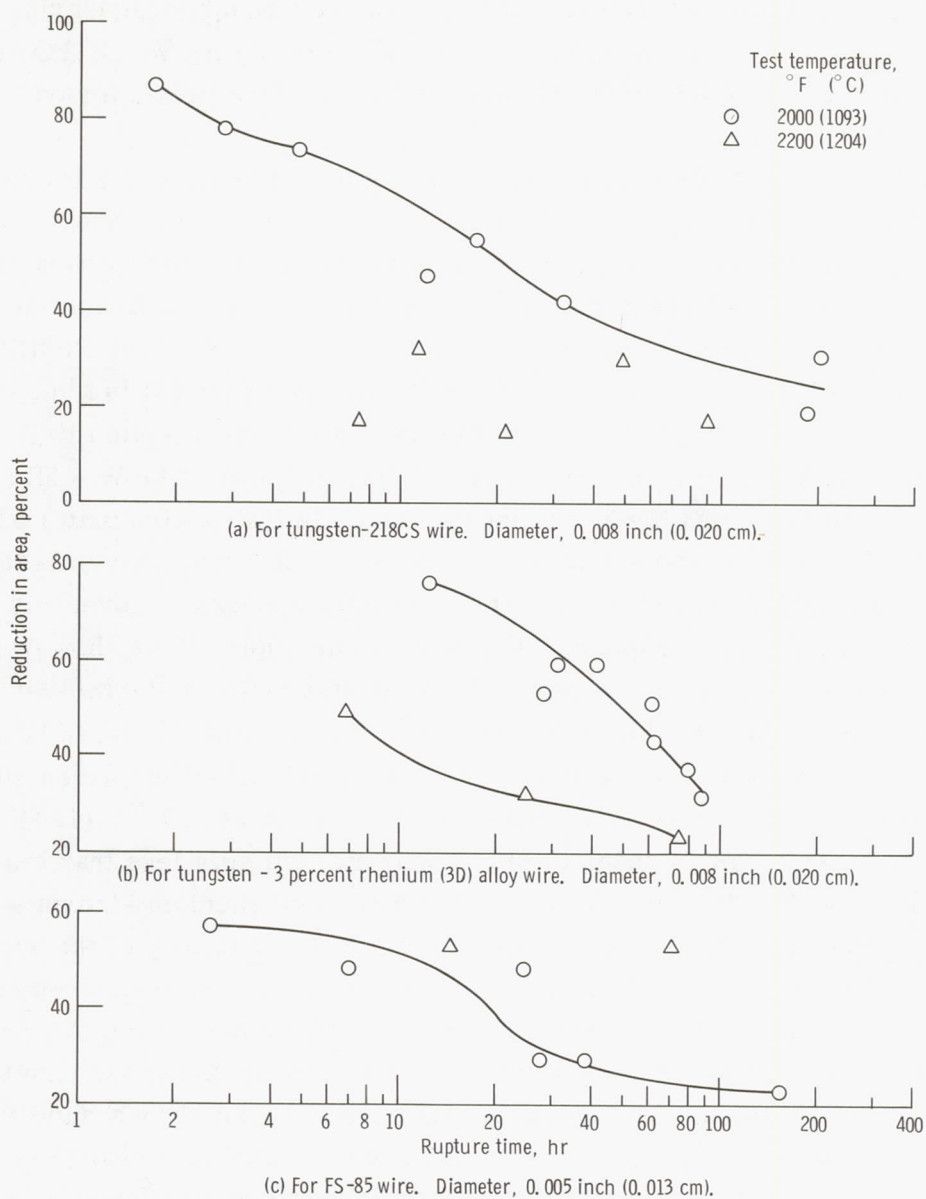


Figure 9. - Percentage of reduction in area as function of rupture time.

## MICROSTRUCTURAL STUDY

### Tungsten-Base Alloys

Tungsten - 218CS. - Figure 10(a) is an electron photomicrograph of a W - 218CS wire in the as-drawn condition. Heavily worked elongated grains measuring from 0.3 to 1.5 microns in width are visible. The grains increase slightly in width for specimens which failed for short times (1.6 hr) at 2000<sup>o</sup>F (1093<sup>o</sup>C), as shown in figure 10(b). The greatest

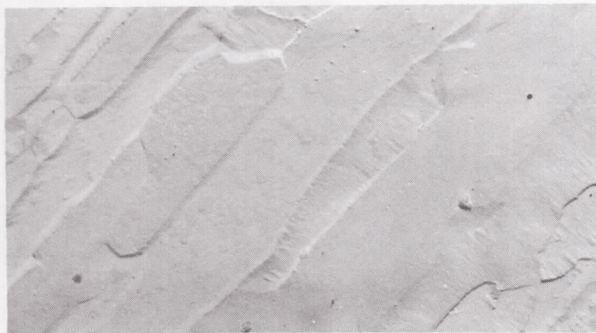
difference in microstructure between these two conditions, however, is the extensive small subgrain formation seen for the specimen tested at 2000<sup>o</sup> F (1093<sup>o</sup> C), as compared to the limited amount of subgrains for the specimen in the as-drawn condition. The microstructure of a specimen which failed after 200 hours at 2000<sup>o</sup> F (1093<sup>o</sup> C) (fig. 10(c)) shows an increase in grain boundary width and subgrain size. A specimen which failed in 7 hours at 2200<sup>o</sup> F (1204<sup>o</sup> C) (fig. 10(d)) had a microstructure containing many subgrains. A specimen exposed to the same temperature, 2200<sup>o</sup> F (1204<sup>o</sup> C), but which failed after 120 hours had a microstructure consisting of long, wide grains (fig. 10(e)).



(a) As drawn.



(b) After 1.6 hours at 2000<sup>o</sup> F (1093<sup>o</sup> C).



(c) After 200.7 hours at 2000<sup>o</sup> F (1093<sup>o</sup> C).



(d) After 7.4 hours at 2200<sup>o</sup> F (1204<sup>o</sup> C).



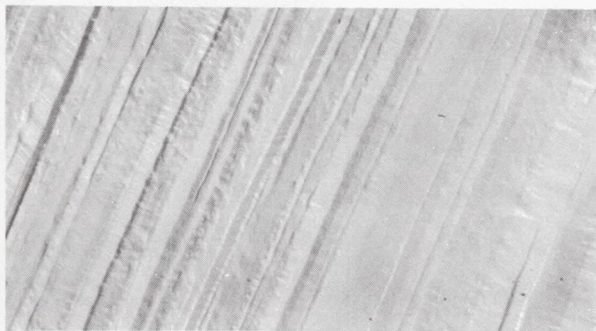
(e) After 120.7 hours at 2200<sup>o</sup> F (1204<sup>o</sup> C).

Figure 10. - Carbon replica of tungsten - 218CS wire. X16 600.



Tungsten - 3 percent rhenium. - The grain width of the as-drawn wire specimen shown in figure 11(a) was equivalent to that of the W - 218CS wire, varying from 0.3 to 1.5 microns. Figure 11(b) shows the microstructure of a W - 3Re wire which failed in stress rupture after 12 hours at 2000<sup>o</sup> F (1093<sup>o</sup> C). Little grain-size difference exists between this specimen and the one in the as-drawn condition. A specimen exposed for 120 hours at 2000<sup>o</sup> F (1093<sup>o</sup> C) (fig. 11(c)) shows extensive grain broadening, approximately three times that for the as-drawn condition. Subgrains also are present, ranging in width from 0.5 to 1.5 microns. The grain width in the specimen tested at 2200<sup>o</sup> F (1204<sup>o</sup> C) (fig. 11(d)) and exposed to temperature for 7 hours was similar to that tested at 2000<sup>o</sup> F (1093<sup>o</sup> C) for 120 hours. The grains, however, were not as distinct and showed much more substructure.

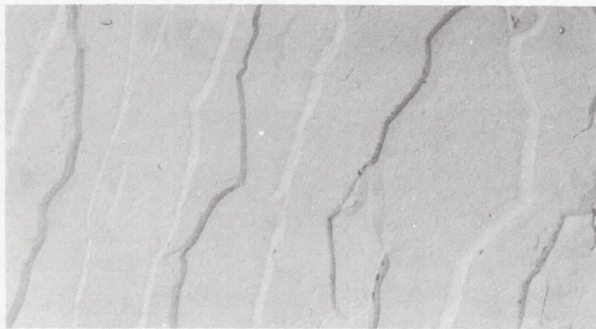
Tungsten - 1 percent thoria. - The grain width of the as-drawn W - 1ThO<sub>2</sub> wire was measured to be between 1 and 2 microns. The particle size of the thoria varied between 0.1 and 0.75 micron. The microstructure of the as-drawn wire is shown in figure 12(a). The microstructure of a specimen which failed after 5 hours at 2000<sup>o</sup> F (1093<sup>o</sup> C) is shown in figure 12(b). Grain broadening has occurred and the grains appear clean and free of subgrains. The microstructure of a specimen tested at the same temperature and which failed after 254 hours is shown in figure 12(c). The predominate feature of the



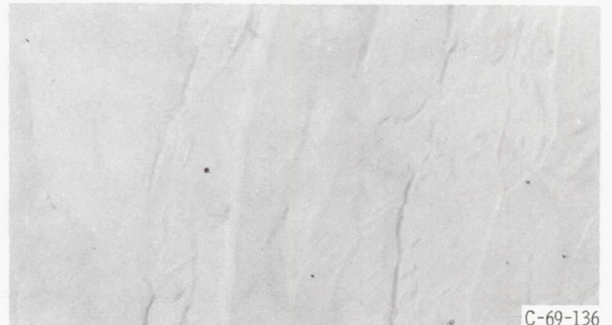
(a) As drawn.



(b) After 12.3 hours at 2000<sup>o</sup> F (1093<sup>o</sup> C).



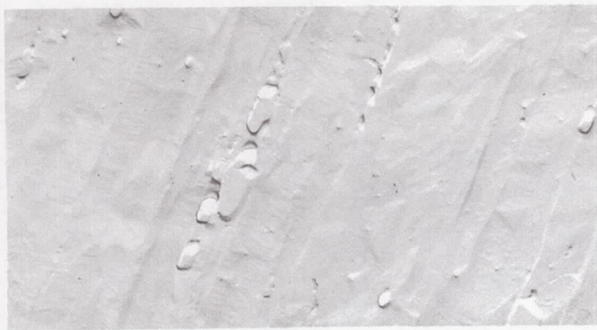
(c) After 119 hours at 2000<sup>o</sup> F (1093<sup>o</sup> C).



(d) After 6.8 hours at 2200<sup>o</sup> F (1204<sup>o</sup> C).

Figure 11. - Carbon replica of tungsten - 3 percent rhenium (3D) wire. X16 600.

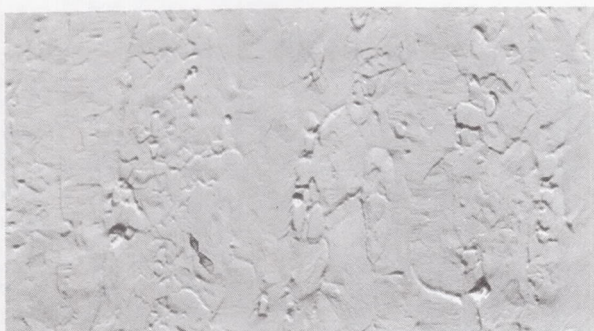




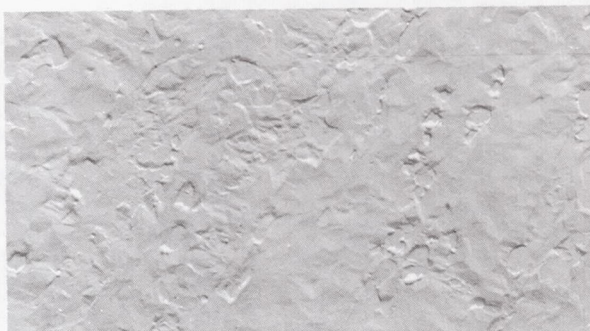
(a) As drawn.



(b) After 5.3 hours at 2000° F (1093° C).



(c) After 253.8 hours at 2000° F (1093° C).



(d) After 9.3 hours at 2200° F (1204° C).



(e) After 150.2 hours at 2200° F (1204° C).

Figure 12. - Carbon replica of tungsten - 1 percent thoria (NF) wire. X16 600.

microstructure is the formation of subgrains, which are clustered about the thoria particles. Subgrain formation was also observed for the specimen tested at 2200° F (1204° C) and which failed in 9 hours (fig. 12(d)). A specimen tested at the same temperature but which failed after 150 hours is shown in figure 12(e). Some subgrains appear to be annealed out, leaving only indications of subgrains. Grain broadening has also occurred, with the grain being 2 to 5 microns in width. No significant change in the thoria dispersion size was seen when comparing the as-drawn specimen with the specimen tested at 2200° F (1204° C) for 150 hours.

Tungsten - 2 percent thoria. - The microstructure of the wire in the as-drawn condition is shown in figure 13(a). The grain width of the as-drawn wire measured between



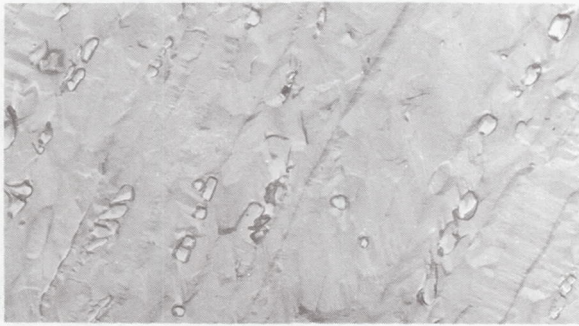
1 and 3 microns. The thoria particles ranged in size from 0.05 to 1 micron. Figure 13(b) is the microstructure of a wire exposed to 2000<sup>o</sup> F (1093<sup>o</sup> C) for 6.5 hours in stress rupture. The microstructure appears to be similar to that of the as-drawn condition. The microstructure of a wire exposed at the same temperature but for a period of 218.5 hours is shown in figure 13(c). Some subgrain formation was noted. Figures 13(d) and (e) show the microstructure of wires tested at 2200<sup>o</sup> F (1204<sup>o</sup> C) for times of 17.8 and 116 hours, respectively. Grain broadening occurred in both specimens; grain width was about double that of the wires tested at 2000<sup>o</sup> F (1093<sup>o</sup> C). Dispersion size remained constant throughout the temperature range studied.



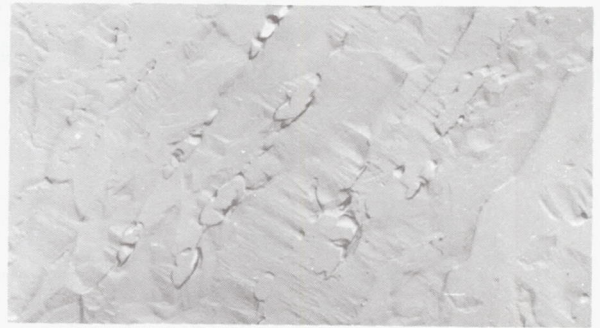
(a) As drawn.



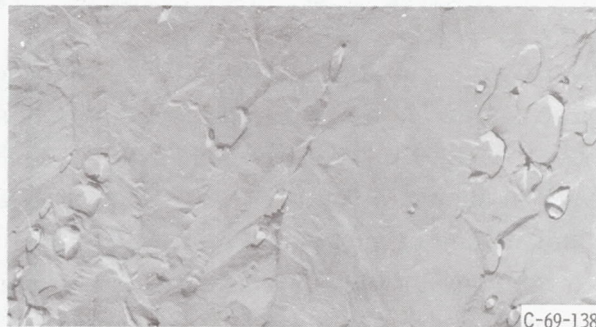
(b) After 6.5 hours at 2000<sup>o</sup> F (1093<sup>o</sup> C).



(c) After 218.5 hours at 2000<sup>o</sup> F (1093<sup>o</sup> C).



(d) After 17.8 hours at 2200<sup>o</sup> F (1204<sup>o</sup> C).



(e) After 116 hours at 2200<sup>o</sup> F (1204<sup>o</sup> C).

Figure 13. - Carbon replica of tungsten - 2 percent thoria wire. X16 600.

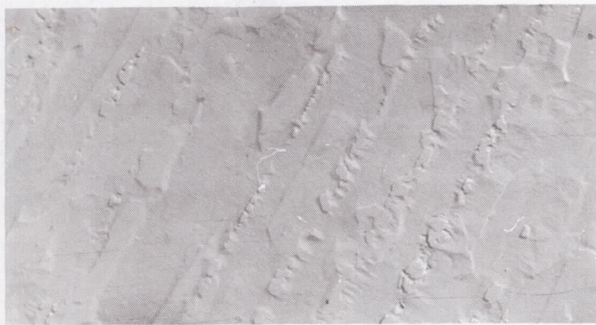




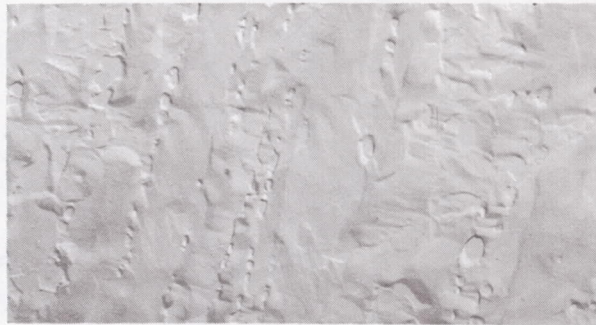
(a) As drawn.



(b) After 32.4 hours at 2000° F (1093° C).



(c) After 124.2 hours at 2000° F (1093° C).



(d) After 19 hours at 2200° F (1204° C).



(e) After 62.6 hours at 2200° F (1204° C).

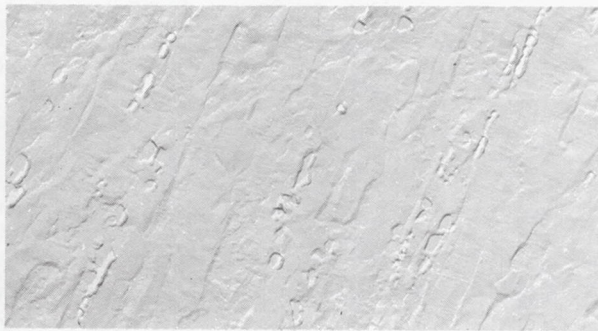
Figure 14. - Carbon replica of tungsten - 5 percent rhenium - 2 percent thoria wire. X16 600.

Tungsten - 5 percent rhenium - 2 percent thoria. - The as-drawn microstructure of the wire is shown in figure 14(a). The grain boundary width varies from 0.3 to 1.2 microns. Figure 14(b) shows the microstructure of a specimen which failed at 32 hours at 2000° F (1093° C). Extensive subgrain formation occurred, without much evidence of grain boundary widening. The long-time specimen which failed at 124 hours at 2000° F (1093° C) had a microstructure similar to the short-time run, as seen in figure 14(c). The microstructure of the specimen tested at 2200° F (1204° C) for 19 hours did not show extensive subgrain formation but appears to show an increase in the size of the dispersion (fig. 14(d)). The microstructure of the specimen tested for 63 hours at 2200° F (1204° C)

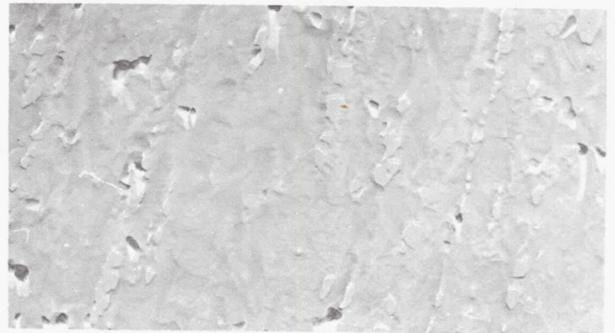


(fig. 14(e)) showed substantial evidence of extensive subgrain formation and had a larger size than those formed at 2000° F (1093° C).

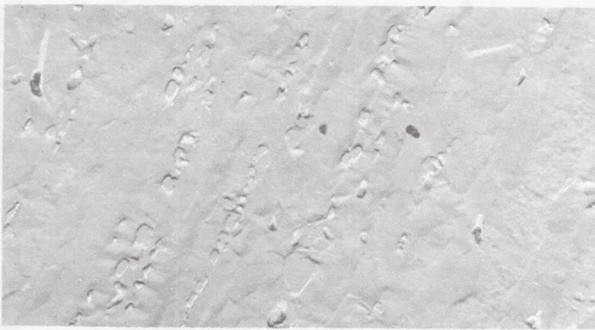
Tungsten - 24 percent rhenium - 2 percent thoria. - The as-drawn microstructure of this material is presented in figure 15(a). Stringering of the thoria particles is shown. Figure 15(b) is the microstructure of a specimen tested in stress rupture at 2000° F (1093° C) and which failed after 9.5 hours. The void areas shown are a result of the etching procedure. The particle content appears to be greater than that of the as-drawn specimen. It is believed that a phase has precipitated out of solution. The new phase formed is readily attacked by the etching solution and is much softer than the matrix



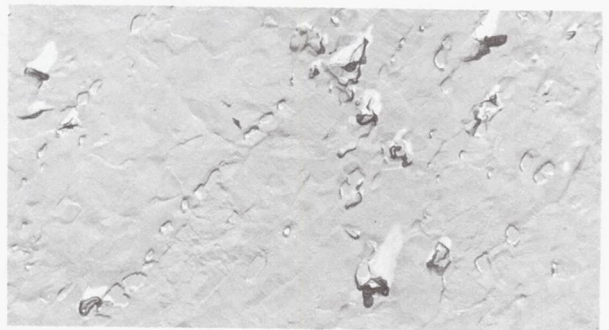
(a) As drawn.



(b) After 9.5 hours at 2000° F (1093° C).



(c) After 32.8 hours at 2000° F (1093° C).



(d) After 7.3 hours at 2200° F (1204° C).



(e) After 45.2 hours at 2200° F (1204° C).

Figure 15. - Carbon replica of tungsten - 24 percent rhenium - 2 percent thoria wire. X16 600.



material. The microstructure of a specimen tested in stress-rupture at the same temperature and which failed after 32.8 hours is presented in figure 15(c). Subgrain formation is shown. A specimen tested at 2200° F (1204° C) for 7.3 hours (fig. 15(d)) showed the formation of larger particles than observed at 2000° F (1093° C) and also grain boundary broadening. Figure 15(e) shows the microstructure of a specimen tested at 2200° F (1204° C) for 45.2 hours. Excessive grain growth has occurred, as well as agglomeration of the precipitated new phase.

## Molybdenum-Base Alloys

TZM. - The as-drawn microstructure of a TZM wire is shown in figure 16(a). The particle size of the precipitate formed (believed to be  $\text{Mo}_2\text{C}$  or  $\text{TiC}$ ) is approximately 0.1 micron. Figure 16(b) shows the microstructure of the wire after being stressed for 371 hours at 2000° F (1093° C). The particle size of the precipitates ranges from 0.1 to 0.3 micron and substantial grain growth has occurred as compared with the as-drawn condition. The microstructure of a specimen tested at 2200° F (1204° C) for 1 hour is shown in figure 16(c). The particle size of the precipitate is similar to that of the specimen tested at 2000° F (1093° C). The formation of subgrains is evident. Figure 16(d) shows



(a) As drawn.



(b) After 371 hours at 2000° F (1093° C).



(c) After 1 hour at 2200° F (1204° C).



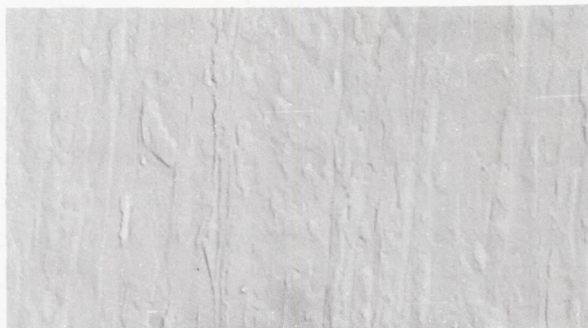
(d) After 698.2 hours at 2200° F (1204° C).

Figure 16. - Carbon replica of TZM wire. X16 600.

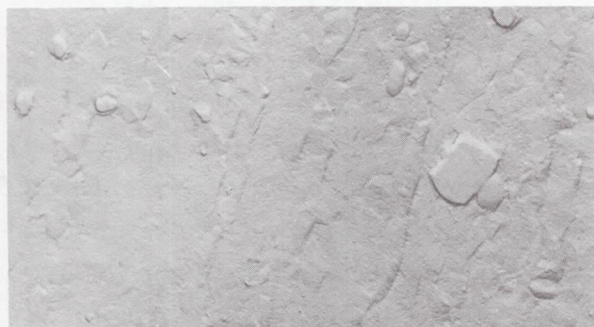




(a) As drawn.



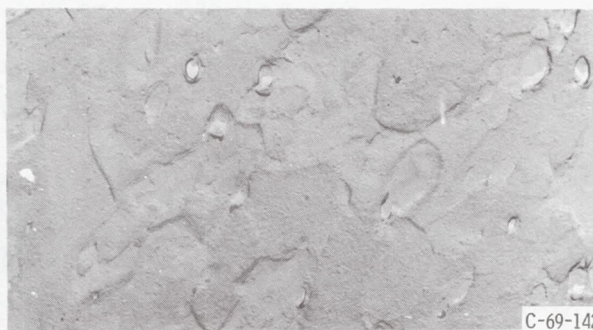
(b) After 0.3 hour at 2000° F (1093° C).



(c) After 56.6 hours at 2000° F (1093° C).



(d) After 0.1 hour at 2200° F (1204° C).



(e) After 70.7 hours at 2200° F (1204° C).

Figure 17. - Carbon replica of TZC wire. X16 600.

the microstructure of a specimen tested at 2200<sup>0</sup> F (1204<sup>0</sup> C) for 698 hours. Extensive subgrain boundary formation is observed.

TZC. - The microstructure of the as-drawn condition is shown in figure 17(a). The precipitate particle size ranged from 0.1 to 1 micron. Figure 17(b) shows the microstructure of a specimen tested in stress rupture at 2000<sup>0</sup> F (1093<sup>0</sup> C) for 0.3 hour. Some grain boundary widening has occurred as compared with the as-drawn condition. The microstructure of a specimen tested at 2000<sup>0</sup> F (1093<sup>0</sup> C) for 56.6 hours is shown in figure 17(c). Subgrain boundary formation is evident. Figures 17(d) and (e) show the

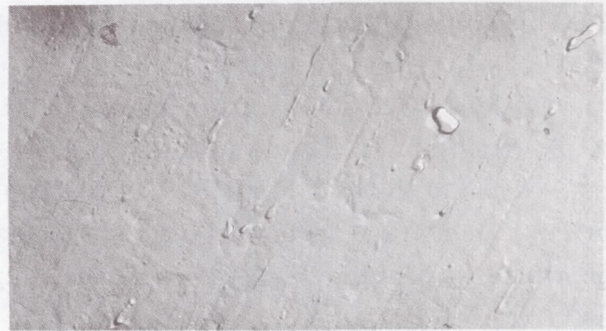
microstructures of specimens tested at 2200° F (1204° C) for 1 and 70.7 hours, respectively. Grain growth and particle coarsening appear to result.

### Columbium-Base Alloys

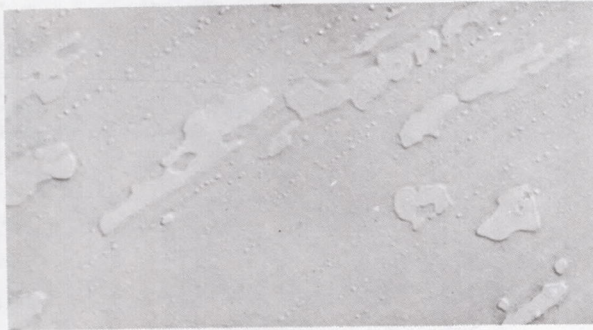
FS-85. - The as-drawn specimen (fig. 18(a)) shows fibrous grains ranging in width from 0.2 to 2 microns. Very few precipitates were present in the as-drawn specimen. Figure 18(b) shows the microstructure of a specimen tested at 2000° F (1093° C) for 2.6 hours. The grain size was between 0.6 and 3 microns, which is larger than that in



(a) As drawn.



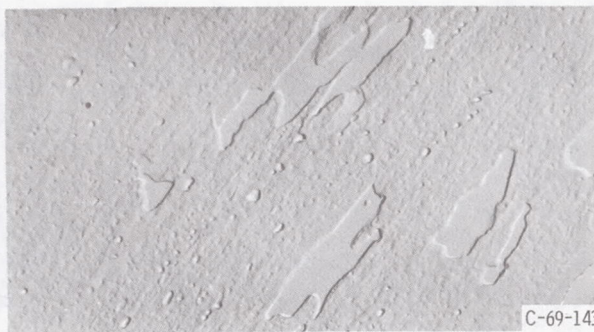
(b) After 2.6 hours at 2000° F (1093° C).



(c) After 152.7 hours at 2000° F (1093° C).



(d) After 14.4 hours at 2200° F (1204° C).



(e) After 70.7 hours at 2200° F (1204° C).

Figure 18. - Carbon replica of FS-85 wire. X16 600.



the as-drawn condition. An estimated 2 to 3 percent of the wire consisted of precipitates, believed to be oxides or nitrides. A higher percentage of precipitates was generally found at the edges of the wire than in the center. The precipitates ranged in size from 0.03 to more than 1 micron. A specimen exposed at 2000<sup>o</sup> F (1093<sup>o</sup> C) (fig. 18(c)) for 152.7 hours produced a much higher percentage of precipitates than the previous specimen. Two distinct types of precipitates were seen; small, rounded precipitates (300 to 1000 Å) and large and elongated precipitates (0.1 to 1 micron in width). Both precipitates appear to form in an orderly arrangement on the same planes. Figure 18(d) show the microstructure of a specimen tested at 2200<sup>o</sup> F (1204<sup>o</sup> C) for 14.4 hours. The microstructure appears to be similar to that of the specimen tested at 2000<sup>o</sup> F (1093<sup>o</sup> C) for 2.6 hours, except for a somewhat higher precipitate content. The specimen tested at 2200<sup>o</sup> F (1204<sup>o</sup> C) for 70.1 hours (fig. 18(e)) had a similar microstructure to that tested at 2000<sup>o</sup> F (1093<sup>o</sup> C) for 152.7 hours.

AS-30. - The microstructure of the as-drawn wire is shown in figure 19(a). The size of the precipitates visible in the microstructure ranged from 0.05 to 0.6 micron. A specimen tested at 2000<sup>o</sup> F (1093<sup>o</sup> C) for 0.1 hour showed grain growth and a larger amount of precipitates with a wider particle size range (0.03 to 0.8 micron) and more elongation particles than in the as-drawn condition (fig. 18(b)). The precipitates are believed to

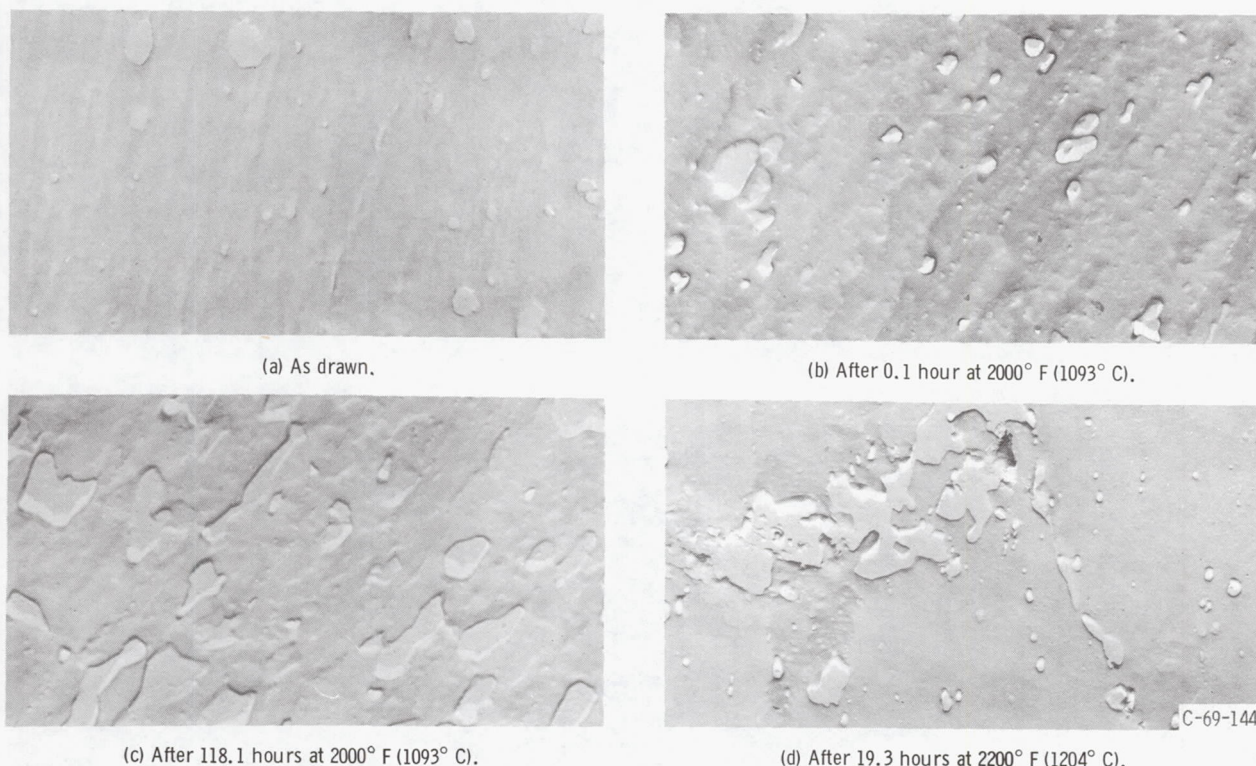


Figure 19. - Carbon replica of AS-30 wire. X16 600.



be either oxides or nitrides. Figure 19(c) shows the microstructure of a specimen tested at 2000° F (1093° C) for 118.1 hours. Large particles are formed, ranging in size from 0.1 to 0.8 micron. The volume percentage of the precipitate was quite high, estimated to be nearly 60 percent in some areas. A central band about 3 microns wide containing large particles was also observed. Figure 19(d) shows the microstructure of a specimen tested at 2200° F (1204° C) for 19.3 hours. The specimens contained a mixture of small and large precipitates, the large ones being predominant. While the edge of the wire specimen showed a fibrous grain structure, the center portion had large nonfibrous-looking grains (over 10 microns in width). In the center portion of the wire, the large particles were generally located in the grain boundaries and the small particles within the grain.

Table VI is a summary of the wire microstructure data as a function of exposure time and temperature.

## DISCUSSION

### Mechanical Properties

The ultimate tensile strength values obtained for the wire materials at room temperature, 2000° F (1093° C), and 2200° F (1204° C) were found to be much higher than those reported for other forms of the materials. The only exception was the 2000° and 2200° F (1093° and 1204° C) tensile strength of AS-30 wire, which was much lower than the tensile strength reported for the bulk material. For example, AS-30, warm-worked 90 percent and stress relieved at 2000° F (1093° C), was found to have a tensile strength of 85 000 psi (586 MN/m<sup>2</sup>) at 2000° F (1093° C) and 72 000 psi (496 MN/m<sup>2</sup>) at 2200° F (1204° C). The AS-30 wire material was found to have a tensile strength of 61 000 psi (421 MN/m<sup>2</sup>) at 2000° F (1093° C) and 33 000 psi (228 MN/m<sup>2</sup>) at 2200° F (1204° C). The tungsten-base alloys had the highest ultimate tensile strengths at all the temperatures investigated. Ultimate tensile strength values of up to 240 000 psi (1655 MN/m<sup>2</sup>) were obtained for wire tested at 2000° F (1093° C), and up to 160 000 psi (1103 MN/m<sup>2</sup>) at 2200° F (1204° C).

When density is taken into consideration, the molybdenum and columbium alloys compare more favorably with the tungsten-base alloys. Figures 20(a) and (b) give the specific ultimate tensile strength of the wire materials at 2000° and 2200° F (1093° and 1204° C). At 2000° F (1093° C), the W - 5Re - 2ThO<sub>2</sub> wire had the highest specific tensile strength, 350 000 inches (8.9×10<sup>3</sup> m), followed by TZC which had a specific tensile strength of about 340 000 inches (8.6×10<sup>3</sup> m). At 2200° F (1204° C) the W - 5Re - 2ThO<sub>2</sub> wire also has the highest specific tensile strength, about 250 000 inches (6.4×10<sup>3</sup> m), followed by



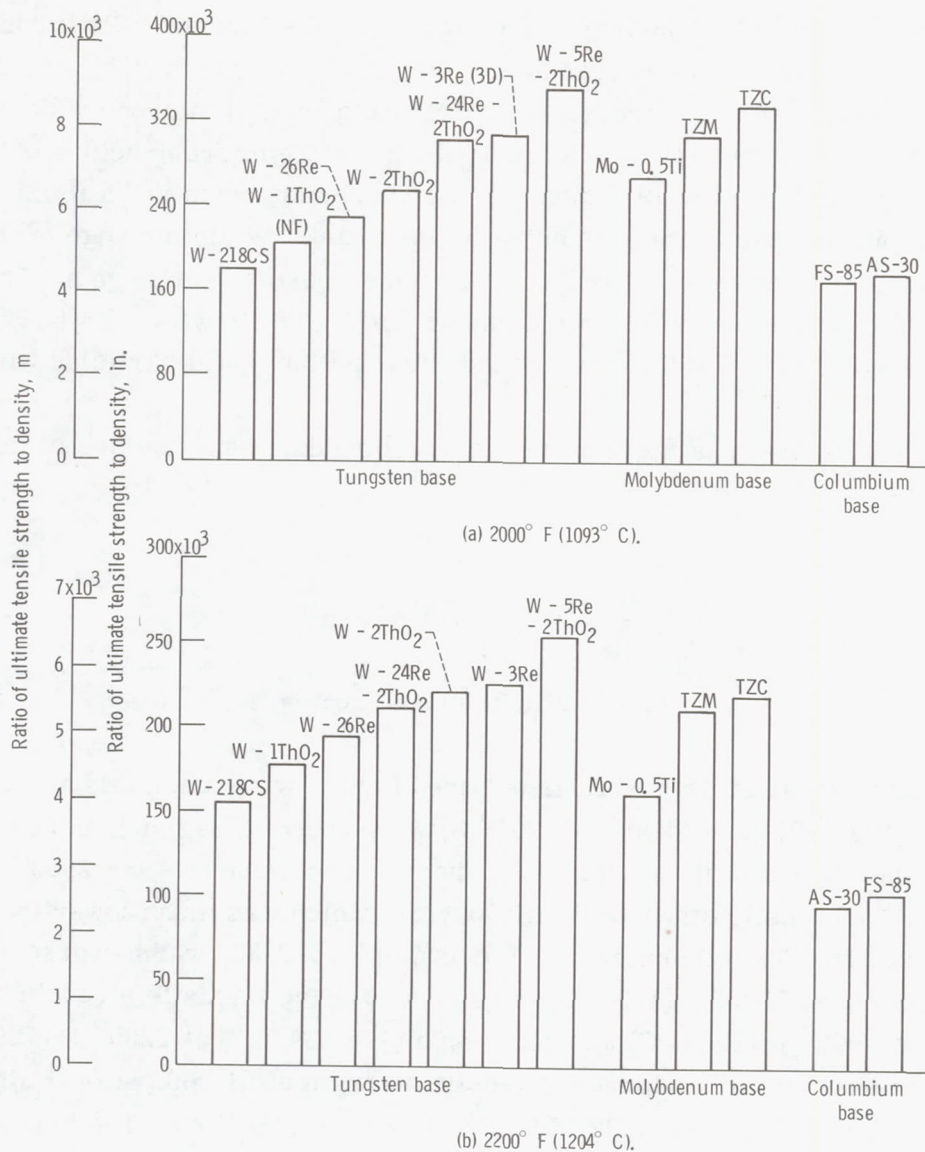


Figure 20. - Ratio of ultimate tensile strength to density for wires at elevated temperatures.

the W - 2ThO<sub>2</sub>, W - 3Re, and TZC wires. The results thus show that rhenium and thoria additions to tungsten enhance the tensile strength at these temperatures.

The stress-rupture values obtained for the wire materials indicate that the tungsten-base alloy wire materials had 100-hour rupture strengths as much as twice those of either the molybdenum- or columbium-base alloy materials. The W - 2ThO<sub>2</sub> wire material had the highest 100-hour stress-rupture strength at both temperatures (96 000 psi (662 MN/m<sup>2</sup>) at 2000° F (1093° C) and 69 000 psi (476 MN/m<sup>2</sup>) at 2200° F (1204° C)). If density is taken into consideration, the molybdenum- and columbium-base alloy wire

materials compare more favorably, as shown in figures 21(a) and (b). Figure 21(a) gives the ratio of the stress to cause rupture in 100 hours to density of the wire material (the specific 100-hour rupture strength of the wire material) for wires tested at 2000° F (1093° C). Figure 21(b) is the same type of plot for the wires tested at 2200° F (1204° C). Even when density is taken into account, however, the W - 2ThO<sub>2</sub> wire material is superior to the other wire materials investigated. The W - 2ThO<sub>2</sub> wire has specific 100-hour rupture strengths of 140 000 inches (3.6×10<sup>3</sup> m) at 2000° F (1093° C) and 100 000 inches (2.5×10<sup>3</sup> m) at 2200° F (1204° C).

As mentioned in the INTRODUCTION, it was not intended to relate variations in mechanical properties to strengthening mechanisms in this study. However, it can be observed that microstructural stability and superior properties typically were concurrent. Tungsten-base alloy wire materials having the most stable microstructures as a function of time at temperature also were, as expected, the most stable in stress rupture. The

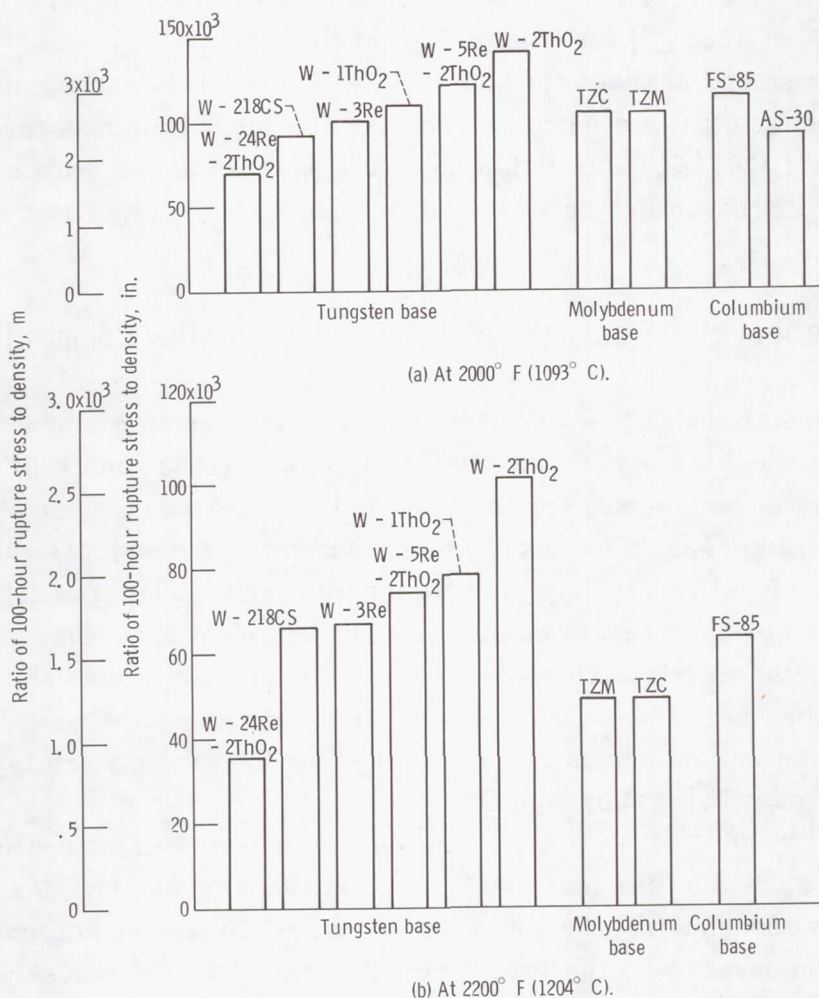


Figure 21. - Ratio of 100-hour rupture strength to density for wire materials.



tungsten wire materials containing thoria additions were found to have the most stable microstructure of the tungsten-base alloy wire materials investigated and also to be the most stable in stress-rupture. At 2000<sup>o</sup> F (1093<sup>o</sup> C), for example, tungsten wire material containing 1 and 2 percent thoria additions did not show any substantial difference in microstructure between short-time or long-time exposure. The W - 218CS and tungsten containing 3 percent rhenium additions, however, did show a substantial change in microstructure between short-time and long-time exposure at 2000<sup>o</sup> F (1093<sup>o</sup> C). Extensive grain growth occurred after long-time exposure for both wire materials.

Grain growth and particle coarsening appear to occur for wires of the molybdenum alloys TZM and TZC after long-time exposure at 2000<sup>o</sup> and 2200<sup>o</sup> F (1093<sup>o</sup> and 1204<sup>o</sup> C). While the tensile properties of these materials in wire form are much higher than the properties of the bulk material, the stress-rupture strengths are nearly equivalent. The results of the microstructural analysis of the molybdenum alloys suggest that a great deal of the stored energy from the wire-drawing process is annealed out on exposure at 2000<sup>o</sup> or 2200<sup>o</sup> F (1093<sup>o</sup> or 1204<sup>o</sup> C) for long periods of time.

The microstructural analysis for both the FS-85 and AS-30 columbium-base alloy wire materials showed that grain growth occurred for short-time exposure at 2000<sup>o</sup> and 2200<sup>o</sup> F (1093<sup>o</sup> and 1204<sup>o</sup> C). Long-time exposure at these temperatures indicated that the precipitation of a new phase occurs. Surface contamination was also indicated.

## Potential of Refractory-Metal Fiber in Superalloy Composites

Refractory-metal alloy wires are of interest for fiber reinforcement of superalloy-type matrix materials for use between 2000<sup>o</sup> and 2200<sup>o</sup> F (1093<sup>o</sup> and 1204<sup>o</sup> C). Previous experimental work at Lewis (ref. 1) has shown that composites of superalloys reinforced with available refractory-metal wires can be produced that have stress-rupture properties superior to conventional superalloys at use temperatures of 2000<sup>o</sup> and 2200<sup>o</sup> F (1093<sup>o</sup> and 1204<sup>o</sup> C). Composite strength was found to be dependent upon fiber properties and compatibility with the matrix. The stress-rupture properties of the composite could be approximated if the fiber stress-rupture properties were known. One of the objectives of this investigation was to determine the potential for the wire materials investigated as a reinforcing fiber for superalloy composites.

The potential 2000<sup>o</sup> F (1093<sup>o</sup> C) ultimate tensile strengths of superalloy composites containing wires studied in this investigation were calculated by using the rule of mixture relations. It was assumed that the composite contained 70 volume percent wire and that negligible reaction occurs with the wire during the fabrication or exposure for short times at 2000<sup>o</sup> F (1093<sup>o</sup> C). The strongest wire was selected from each wire alloy group. Figures 22(a) and (b) show the predicted 2000<sup>o</sup> F (1093<sup>o</sup> C) ultimate tensile strength and

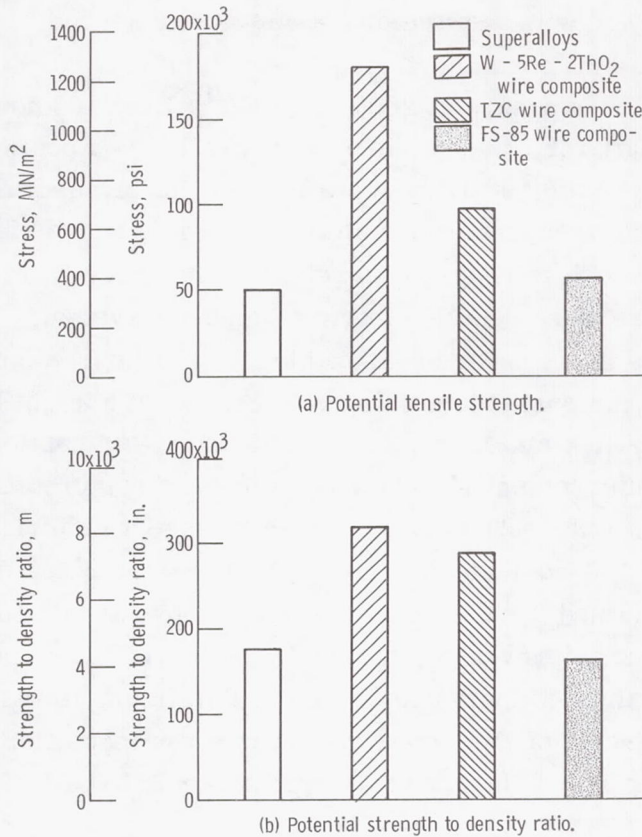


Figure 22. - Potential tensile strength and strength to density ratio of wire-superalloy composites at 2000° F (1093° C).

the specific ultimate tensile strength of composites compared with those of the strongest superalloys. The tungsten alloy wire composite appears to offer the most potential, even when density is taken into consideration. The potential specific ultimate tensile strength of the tungsten-rhenium-thoria wire-reinforced superalloy composite represents a use-temperature advantage of 200° F (93° C) over unreinforced superalloys. The molybdenum-wire-reinforced composite appears promising on a specific strength basis. Known reaction with nickel-base alloys (ref. 1), however, might preclude selection of this material for reinforcement applications unless protective diffusion barrier coatings are developed. The columbium alloy wire which was tested in this program did not have sufficient strength to warrant consideration for composite applications in tension at 2000° F (1093° C).

The potential 100-hour rupture strength of refractory-metal-alloy-wire - superalloy composites at 2000° F (1093° C) was also determined for composites containing 70 volume percent wire. The potential rupture strength of the composite was determined for two conditions. It was assumed that reaction with the wire and matrix does not occur, which



would represent a composite system in which the matrix is insoluble in the wire or in which the wires are coated with a diffusion barrier. The second condition assumed was that reaction between the matrix and wire does occur but that 80 percent of the wire properties are retained after exposure at 2000° F (1093° C) for 100 hours. The potential 100-hour rupture strength for wire-reinforced superalloy composites is given in figure 23. Also shown in figure 23 is the 100-hour rupture strength for superalloys and for a tungsten-base-alloy-wire - superalloy composite which was investigated in a previous program at Lewis and which is reported in reference 1. Ninety percent of the wire properties in stress rupture were retained in this composite system. The 80 percent value used in our calculations thus appears reasonable. A 100-hour rupture strength of 67 000 psi (462 MN/m<sup>2</sup>) for a composite containing W - 2ThO<sub>2</sub> wire might be obtained if reaction between the wire and matrix material could be avoided. Composites containing either TZC or FS-85 as a reinforcement would have a 100-hour rupture strength much lower than that obtained by using W - 2ThO<sub>2</sub> as the reinforcement material, even if reaction could be avoided. The 100-hour rupture strength for all the potential wire material would be greater than that obtained for the strongest superalloys at this temperature. If reaction with the wire cannot be prevented, the value for the 100-hour rupture life of the composites would be approximated by figure 23(b). In this figure it was assumed that reaction occurred but that 80 percent of the wire strength was retained after 100 hours exposure at 2000° F (1093° C). The W - 2ThO<sub>2</sub> wire composite would have a 100-hour rupture

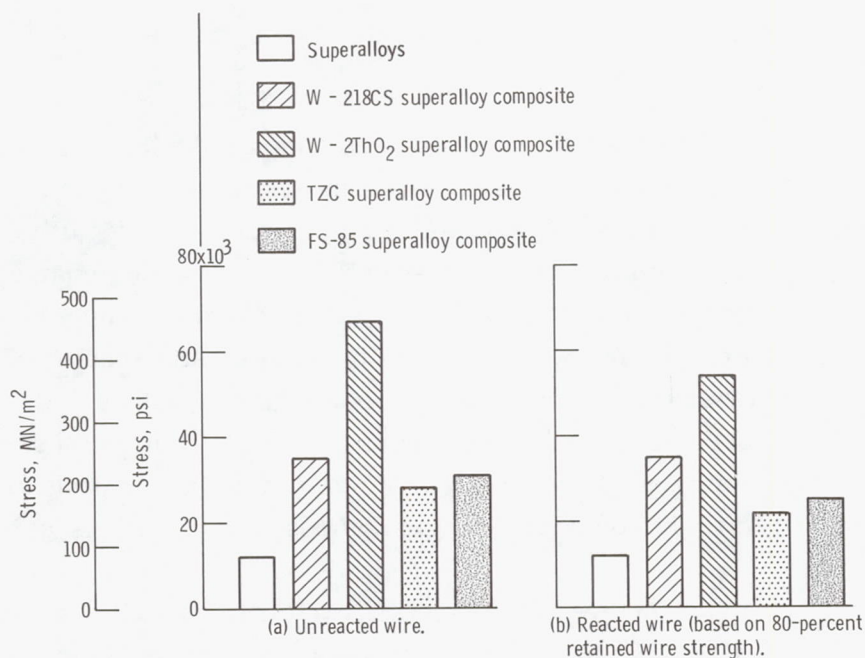


Figure 23. - Potential 100-hour rupture strength of wire-superalloy composites at 2000° F (1093° C). Fiber content, 70 volume percent.

strength of 54 000 psi ( $372 \text{ MN/m}^2$ ) compared to a value of 35 000 psi ( $241 \text{ MN/m}^2$ ) reported in reference 1 for a tungsten wire composite. Composites reinforced with TZC or FS-85 would have a 100-hour rupture strength lower than that obtainable for the tungsten wire composites but higher than that obtained for superalloys. A potential increase in the 100-hour rupture strength of over 50 percent exists for composites containing W -  $2\text{ThO}_2$  wire as compared with the composite produced in reference 1 which contained W - 218CS wire. The potential 100-hour rupture strength of the W -  $2\text{ThO}_2$  wire composite is also over four times that of the strongest superalloys at this temperature. The W -  $2\text{ThO}_2$  wire composite has a potential use-temperature advantage of approximately  $400^\circ \text{ F}$  ( $204^\circ \text{ C}$ ) over superalloys and  $80^\circ \text{ F}$  ( $27^\circ \text{ C}$ ) over the W - 218CS wire composite reported in reference 1.

The density of the composite materials, however, is much greater than that of superalloys and should also be taken into consideration. The potential 100-hour specific rupture strength for wire-superalloy composites is presented in figure 24. The two assumptions used in plotting figure 23, unreacted wire and reacted wire, were also used to construct figure 24. When density is taken into consideration, the molybdenum alloy wire, TZC, and the columbium alloy wire, FS-85, appear more promising. The specific 100-hour rupture strength for the W -  $2\text{ThO}_2$  - composite, if reaction is assumed, is 92 000 inches ( $2.3 \times 10^3 \text{ m}$ ) as compared to 58 000 inches ( $1.5 \times 10^3 \text{ m}$ ) for the W - 218CS wire - superalloy composite reported in reference 1 and 39 000 inches ( $1 \times 10^3 \text{ m}$ ) for the strongest superalloys. This represents a use-temperature advantage of  $200^\circ \text{ F}$  ( $93^\circ \text{ C}$ ) over the

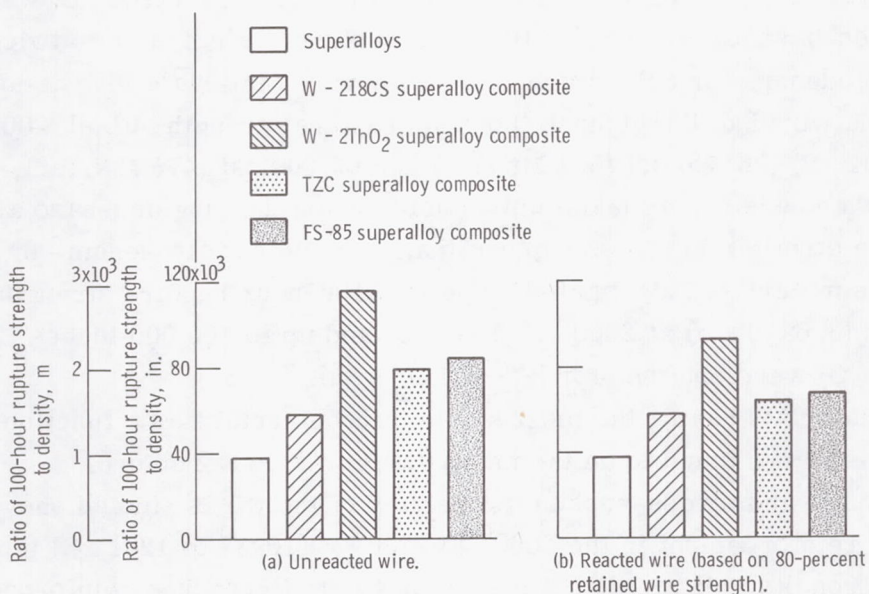


Figure 24. - Potential 100-hour rupture strength to density ratio of wire-superalloy composites at  $2000^\circ \text{ F}$  ( $1093^\circ \text{ C}$ ). Fiber content, 70 volume percent.



strongest superalloys. The TZC wire composites and the FS-85 wire composites compare more favorably with the W - 2ThO<sub>2</sub> wire composite when density is taken into account; however, they are weaker in specific stress-rupture strength (or stress-rupture strength as a function of density). The molybdenum and columbium alloy wire composites, however, offer potential improvement over superalloys at this temperature when compared on a stress/density basis.

## SUMMARY OF RESULTS

The tensile and stress-rupture properties of refractory-metal alloy wires of TZC, TZM, Mo - 0.5Ti, AS-30, FS-85, doped tungsten, thoria-tungsten, tungsten-rhenium, and tungsten-rhenium-thoria were determined for rupture times up to 200 hours and for room temperature, 2000<sup>o</sup> F (1093<sup>o</sup> C), and 2200<sup>o</sup> F (1204<sup>o</sup> C). The rupture properties were correlated to microstructure. The results obtained led to the following conclusions:

1. The ultimate tensile strength values obtained for the wire materials at room temperature, 2000<sup>o</sup> F (1093<sup>o</sup> C), and 2200<sup>o</sup> F (1204<sup>o</sup> C) were much higher than those reported for other forms of the materials, except for the AS-30 wire material.

2. The tungsten-base alloy wire materials studied had the highest ultimate tensile strengths at 2000<sup>o</sup> and 2200<sup>o</sup> F (1093<sup>o</sup> and 1204<sup>o</sup> C). Ultimate tensile strengths of 240 000 psi (1655 MN/m<sup>2</sup>) for W - 5Re - 2ThO<sub>2</sub> wire tested at 2000<sup>o</sup> F (1093<sup>o</sup> C) and 160 000 psi (1103 MN/m<sup>2</sup>) for W - 3Re wire tested at 2200<sup>o</sup> F (1204<sup>o</sup> C) were obtained.

3. Tungsten-base alloy wire had 100-hour rupture strengths up to twice those of either the molybdenum- or columbium-base alloy wire materials investigated. Tungsten-2 percent thoria wire had the highest 100-hour rupture strength at both 2000<sup>o</sup> and 2200<sup>o</sup> F (1093<sup>o</sup> and 1204<sup>o</sup> C) (96 000 psi (662 MN/m<sup>2</sup>) and 69 000 psi (476 MN/m<sup>2</sup>), respectively).

4. Even when density was taken into consideration the tungsten-base alloy wire materials were stronger in stress-rupture than either the molybdenum- or columbium-base alloy wire materials investigated. Specific 100-hour rupture strengths of up to 140 000 inches (3.6×10<sup>3</sup> m) at 2000<sup>o</sup> F (1093<sup>o</sup> C) and up to 100 000 inches (2.5×10<sup>3</sup> m) at 2200<sup>o</sup> F (1204<sup>o</sup> C) were obtained for W - 2ThO<sub>2</sub> wire.

5. Wire materials having the most stable microstructure as a function of time at temperature also were found to be the most stable in stress-rupture.

6. The tensile and stress-rupture properties of the wires studied showed promise as potential fiber reinforcement in the 2000<sup>o</sup> to 2200<sup>o</sup> F (1093<sup>o</sup> to 1204<sup>o</sup> C) temperature range. These results indicate that it may be possible to produce reinforced nickel or

cobalt superalloys with over three times the tensile strength and up to five times the 100-hour rupture strength at 2000<sup>0</sup> F of the strongest conventional superalloys.

Lewis Research Center,  
National Aeronautics and Space Administration,  
Cleveland, Ohio, January 3, 1969  
129-03-09-01-22.

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TABLE I. - COMPOSITION AND DIAMETER OF WIRE MATERIAL

Composition	Wire diameter		Density, g/cm <sup>3</sup>	Condition
	in.	cm		
Tungsten - base alloys				
W - 218CS	0.008	0.020	19.24	As drawn; cleaned and straightened ↓ Hard drawn (process B) Commercially drawn (process A) Annealed Commercially drawn Annealed
W - 1ThO <sub>2</sub> (NF)	.008	.020	19.10	
W - 3Re (3D)	.008	.020	19.35	
W - 2ThO <sub>2</sub>	.015	.038	18.91	
W - 5Re - 2ThO <sub>2</sub>	.008	.020	19.07	
W - 5Re - 2ThO <sub>2</sub>	↓	↓	19.07	
W - 5Re - 2ThO <sub>2</sub>	↓	↓	19.07	
W - 24Re - 2ThO <sub>2</sub>	↓	↓	19.40	
W - 24Re - 2ThO <sub>2</sub>	↓	↓	19.40	
W - 26Re	.015	.038	19.76	
Molybdenum - base alloys				
Mo - 0.5Ti	0.005	0.013	10.16	As drawn; cleaned and straightened
Mo - 0.5Ti - 0.08Zr - 0.015C (TZM)	.008	.020	10.16	As drawn; cleaned and straightened
Mo - 1.25Ti - 0.30Zr - 0.15C (TZC)	.005	.013	10.05	As drawn; cleaned and straightened
Columbium - base alloys				
Cb - 28Ta - 10W - 1Zr - 0.005C (FS-85)	0.005	0.013	10.60	As drawn; cleaned and straightened
Cb - 20W - 1Zr (AS-30)	.005	.013	9.60	As drawn; cleaned and straightened

TABLE II. - CHEMICAL ANALYSIS OF WIRE MATERIAL

Element	Tungsten-base alloy				
	W - 1ThO <sub>2</sub>	W - 2ThO <sub>2</sub>	W - 3Re	W - 5Re - 2ThO <sub>2</sub>	W - 24Re - 2ThO <sub>2</sub>
Composition, wt. %					
Thorium	0.95	1.6	----	1.78	1.7
Rhenium	----	---	2.79	4.89	22.54
Element	Molybdenum-base alloys		Columbium-base alloys		
	TZM	TZC	FS-85	AS-30	
Composition, wt. %					
Titanium	0.45	1.18	----	-----	
Zirconium	.085	.27	0.84	-----	
Carbon	.0312	.12	.031	-----	
Oxygen	.0196	.0331	.044	0.0588	
Nitrogen	.0055	.0063	.0047	.0106	
Tantalum	-----	-----	27.95	-----	
Tungsten	-----	-----	10.44	-----	

TABLE III. - ROOM-TEMPERATURE TENSILE PROPERTIES OF REFRACTORY WIRES

Wire material	Diameter		Ultimate tensile strength		Elongation, percent in 1 in. (2.5 cm)	Reduction in area, percent
	in.	cm	psi	MN/m <sup>2</sup>		
Tungsten base:						
W - 218CS	0.008	0.020	386×10 <sup>3</sup>	2661	4.5	39.9
W - 1ThO <sub>2</sub> (NF)	.008	.020	327	2251	6.3	18.5
W - 2ThO <sub>2</sub>	.015	.038	384	2648	5.5	14.2
W - 3Re (3D)	.008	.020	404	2785	5.7	45.6
W - 5Re - 2ThO <sub>2</sub> :						
Hard drawn (Process B)	.008	.020	446	3075	3.9	16.7
As drawn (Process A)	.008	.020	296	2041	5.8	20.3
Annealed	.008	.020	261	1797	6.3	18.4
W - 24Re - 2ThO <sub>2</sub> :						
As drawn	.008	.020	428	2951	3.0	27.8
Annealed	.008	.020	309	2127	6.1	38.0
W - 26Re	.015	.038	275	1896	26.4	52.0
Molybdenum base:						
Mo - 0.5Ti	0.005	0.013	260×10 <sup>3</sup>	1793	4.3	64.0
TZM	.008	.020	285	1965	4.4	56.3
TZC	.005	.013	329	2268	4.8	45.2
Columbium base:						
FS-85	0.005	0.013	219×10 <sup>3</sup>	1510	6.1	74.8
AS-30	.005	.013	255	1758	7.3	64.0



TABLE IV. - ELEVATED-TEMPERATURE TENSILE

## PROPERTIES OF WIRE MATERIALS

## (a) Tungsten-base alloys

Wire material	Diameter		Ultimate tensile strength		Reduction in area, percent
	in.	cm	psi	MN/m <sup>2</sup>	
Test temperature, 2000° F (1093° C)					
W - 218CS	0.008	0.020	126×10 <sup>3</sup>	869	92.0
W - 1ThO <sub>2</sub> (NF)	.008	.020	142	979	71.1
W - 3Re (3D)	.008	.020	214	1475	88.6
W - 2ThO <sub>2</sub>	.015	.038	173	1193	50.2
W - 5Re - 2ThO <sub>2</sub> :					
Hard drawn (Process B)	.008	.020	243	1675	7.3
As drawn (Process A)	.008	.020	176	1213	25.6
Annealed	.008	.020	166	1145	40.5
W - 24Re - 2ThO <sub>2</sub> :					
As drawn	.008	.020	211	1455	35.0
Annealed	.008	.020	174	1199	53.3
W - 26Re	.015	.038	164	1131	80.8
Test temperature, 2200° F (1204° C)					
W - 218CS	0.008	0.020	108×10 <sup>3</sup>	745	84.5
W - 1ThO <sub>2</sub> (NF)	.008	.020	122	841	70.8
W - 3Re (3D)	.008	.020	157	1082	91.7
W - 2ThO <sub>2</sub>	.015	.038	150	1034	51.0
W - 5Re - 2ThO <sub>2</sub> :					
As drawn	.008	.020	148	1020	35.0
Annealed	.008	.020	141	972	42.4
W - 24Re - 2ThO <sub>2</sub> :					
As drawn	.008	.020	147	1014	31.1
Annealed	.008	.020	131	903	39.9
W - 26Re	.015	.038	114	786	84.8

## (b) Molybdenum-base alloys

Wire material	Diameter		Ultimate tensile strength		Reduction in area, percent
	in.	cm	psi	MN/m <sup>2</sup>	
Test temperature, 2000° F (1093° C)					
Mo - 0.5Ti	0.005	0.013	98×10 <sup>3</sup>	676	83.6
TZM	.005	.013	113	779	67.1
TZC	.005	.013	125	862	87.0
Test temperature, 2200° F (1204° C)					
Mo - 0.5Ti	0.005	0.013	59×10 <sup>3</sup>	407	88.9
TZM	.005	.013	77	531	94.2
TZC	.005	.013	79	545	93.2

## (c) Columbium-base alloys

Wire material	Diameter		Ultimate tensile strength		Reduction in area, percent
	in.	cm	psi	MN/m <sup>2</sup>	
Test temperature, 2000° F (1093° C)					
FS-85	0.005	0.013	66×10 <sup>3</sup>	455	84.9
AS-30	.005	.013	61	421	94.5
Test temperature, 2200° F (1204° C)					
FS-85	0.005	0.013	40×10 <sup>3</sup>	276	95.2
AS-30	.005	.013	33	228	----

TABLE V. - STRESS-RUPTURE PROPERTIES OF WIRE MATERIALS

(a) Tungsten-base alloys

Wire material	Test temperature		Stress		Rupture time, hr	Reduction in area, percent			
	°F	°C	psi	MN/m <sup>2</sup>					
W - 218CS	2000	1093	95×10 <sup>3</sup>	655	1.6	86.8			
			90	621	2.8	77.4			
			85	586	4.8	73.7			
			80	552	11.9	68.4			
			78	538	10.5	64.0			
			75	517	17.2	54.4			
			70	483	31.9	41.8			
			65	448	77.0	25.6			
			60	414	188.3	19.0			
			58	400	200.7	29.8			
	2200	1204	60×10 <sup>3</sup>	414	7.4	16.7			
			55	379	11.3	31.9			
			53	365	21.3	14.4			
			50	345	49.3	29.8			
			47	324	80.1	16.7			
			45	310	120.7	----			
			W - 1ThO <sub>2</sub> (NF)	2000	1093	95×10 <sup>3</sup>	655	5.3	29.8
						90	621	9.9	25.6
85	586	19.4				9.7			
80	552	21.5				14.4			
75	517	136.2				39.9			
70	483	253.8				29.8			
2200	1204	75×10 <sup>3</sup>		517	9.3	47.4			
		70		483	5.9	14.4			
		70		483	4.6	25.6			
		68		469	7.2	16.7			
65	448	11.1	16.7						
		63	434	11.1	7.4				
		60	414	37.4	40.0				
		55	379	69.9	37.9				
		52	359	150.2	23.4				
		W - 3Re (3D)	2000	1093	90×10 <sup>3</sup>	621	12.3	80.8	
					85	586	27.9	78.6	
					80	552	42.0	62.5	
78	538				41.2	72.4			
75	517				60.9	59.3			
73	503				61.8	41.8			
70	483				79.0	36.0			
70	483				87.3	31.9			
68	469				119.0	36.0			
2200	1204				70×10 <sup>3</sup>	483	6.8	49.2	
			60	414	11.0	----			
			55	379	24.8	31.9			
			50	345	48.9	----			
			47	324	74.2	23.4			
			45	310	147.4	----			



TABLE V. - Continued. STRESS-RUPTURE PROPERTIES OF WIRE MATERIALS

(a) Concluded. Tungsten-base alloys

Wire material	Test temperature		Stress		Rupture time, hr	Reduction in area, percent	
	°F	°C	psi	MN/m <sup>2</sup>			
W - 2ThO <sub>2</sub>	2000	1093	100×10 <sup>3</sup>	689	6.5	45.0	
			97	669	43.3	26.2	
			95	655	228.1	42.2	
			93	641	218.5	38.2	
	2200	1204	80×10 <sup>3</sup>	552	17.8	15.4	
			75	517	26.1	14.2	
			73	503	51.7	20.2	
			70	483	89.6	29.3	
			70	483	120.6	49.1	
			65	448	116.0	20.4	
	2200	1204	60	414	146.6	17.8	
W - 5Re - 2ThO <sub>2</sub> , hard drawn (process B)	2000	1093	100×10 <sup>3</sup>	689	32.4	14.4	
			90	621	57.4	16.7	
			80	552	124.2	19.0	
	2200	1204	70×10 <sup>3</sup>	483	19.0	17.2	
			65	448	25.3	13.5	
			60	414	39.2	18.5	
			55	379	62.6	19.5	
	W - 5Re - 2ThO <sub>2</sub> , as drawn (process A)	2000	1093	90×10 <sup>3</sup>	621	24.8	15.5
85				586	34.8	13.8	
80				552	42.3	12.4	
70				483	103.0	16.0	
W - 5Re - 2ThO <sub>2</sub> , annealed	2000	1093	90×10 <sup>3</sup>	621	30.0	16.6	
			85	586	47.2	18.3	
			80	552	54.2	13.7	
			70	483	106.8	11.7	
	2200	1204	60×10 <sup>3</sup>	414	10.6	17.3	
			50	345	22.9	16.4	
			45	310	33.7	19.6	
			40	276	68.6	20.1	
W - 24Re - 2ThO <sub>2</sub> , as drawn	2000	1093	90×10 <sup>3</sup>	621	6.4	30.5	
			85	586	12.0	30.3	
			80	552	12.7	31.5	
			65	448	30.3	28.2	
W - 24Re - 2ThO <sub>2</sub> , annealed	2000	1093	90×10 <sup>3</sup>	621	9.5	33.0	
			85	586	10.7	29.0	
			80	552	15.3	29.0	
			65	448	32.8	31.7	
	2200	1204	45×10 <sup>3</sup>	310	7.3	33.4	
			40	276	10.3	32.1	
			35	241	21.0	32.1	
			30	207	45.2	36.2	
W - 26Re	2200	1204	55×10 <sup>3</sup>	379	4.3	78.2	
			50	345	6.0	73.8	

TABLE V. - Continued. STRESS-RUPTURE PROPERTIES OF WIRE MATERIALS

(b) Molybdenum-base alloys

Wire material	Test temperature		Stress		Rupture time, hr	Reduction in area, percent
	°F	°C	psi	MN/m <sup>2</sup>		
Mo - 0.5Ti	2000	1093	50×10 <sup>3</sup>	345	1.3	85.2
			40	276	2.2	70.6
			30	207	5.4	77.4
TZM	2000	1093	85×10 <sup>3</sup>	586	1.0	93.7
			80	552	3.5	94.3
			75	517	1.6	89.4
			75	517	5.2	91.7
			70	483	1.8	47.4
			70	483	2.0	16.7
			70	483	3.4	92.4
			68	469	6.2	95.5
			65	448	2.2	23.4
			65	448	3.0	21.2
			65	448	3.6	27.7
			65	448	3.7	----
			60	414	3.3	47.4
			60	414	4.0	31.9
			60	414	4.9	36.0
			60	414	9.9	23.4
			55	379	4.3	16.7
			55	379	6.0	56.1
			55	379	7.7	36.0
			55	379	9.2	23.4
			50	345	6.8	21.2
			50	345	7.1	21.2
			50	345	12.2	29.7
			50	345	13.9	19.0
			47	324	371.0	9.7
			46	317	16.2	94.9
			45	310	26.2	95.9
44	303	15.8	95.9			
	2200	1204	42×10 <sup>3</sup>	290	1.0	14.4
			40	276	2.5	16.7
			30	207	1.3	27.7
			28	193	1.4	97.3
			25	172	2.0	23.4
			23	159	5.0	98.1
			20	138	20.5	----
			18	124	698.2	20.0
			17	117	Test stopped at 700 hr	----



TABLE V. - Concluded. STRESS-RUPTURE PROPERTIES OF WIRE MATERIALS

(b) Concluded. Molybdenum-base alloys

Wire material	Test temperature		Stress		Rupture time, hr	Reduction in area, percent
	<sup>o</sup> F	<sup>o</sup> C	psi	MN/m <sup>2</sup>		
TZC	2000	1093	90×10 <sup>3</sup>	621	0.3	45.2
			90	621	.4	45.2
			90	621	.2	87.2
			90	621	.2	66.4
			70	483	1.6	32.7
			70	483	1.1	32.8
			70	483	1.2	60.0
			70	483	1.1	22.4
			60	414	4.6	95.2
			60	414	3.8	45.2
			52	359	5.1	32.7
			50	345	8.3	29.4
			50	345	7.7	26.0
			50	345	8.3	15.2
	50	345	7.3	22.5		
	45	310	9.5	22.5		
	42	290	56.6	32.7		
	2200	1204	40×10 <sup>3</sup>	276	1.0	22.5
			35	241	2.1	22.5
			30	207	3.3	19.2
25			172	11.7	22.4	
20			138	70.7	8.0	

(c) Columbium-base alloys

Wire material	Test temperature		Stress		Rupture time, hr	Reduction in area, percent	
	<sup>o</sup> F	<sup>o</sup> C	psi	MN/m <sup>2</sup>			
FS-85	2000	1093	60×10 <sup>3</sup>	414	2.6	56.4	
			55	379	7.0	48.2	
			50	345	27.7	29.6	
			50	345	38.3	29.6	
			46	317	24.7	48.2	
			42	290	152.7	22.4	
	2200	1204	30×10 <sup>3</sup>	207	14.4	53.6	
			25	172	70.1	53.6	
	AS-30	2000	1093	55×10 <sup>3</sup>	379	0.1	42.4
				46	317	.5	39.2
40				276	9.0	53.7	
35				241	20.4	45.2	
30				207	118.1	45.2	
2200		1204	25×10 <sup>3</sup>	172	2.4	64.0	
			20	138	2.8	51.2	
			15	103	19.3	70.8	

TABLE VI. - SUMMARY OF WIRE MICROSTRUCTURE DATA AS FUNCTION OF EXPOSURE TIME AND TEMPERATURE

Wire material	Condition				
	As drawn	Exposed at 2000 <sup>o</sup> F (1093 <sup>o</sup> C)		Exposed at 2200 <sup>o</sup> F (1204 <sup>o</sup> C)	
		Short time	Long time	Short time	Long time
W - 218CS	Grain width, 0.3 to 1.5 microns	Increase in grain width and formation of subgrains	Increase in grain width and subgrain size	Formation of many subgrains	Formation of long, wide grains
W - 3Re (3D)	Grain width, 0.3 to 1.5 microns	Grain width same as in the as-drawn condition	Grain width broadening to three times that of as-drawn condition; formation of subgrains 0.5 to 1.5 microns in width	Structure similar to long-time exposure at 2000 <sup>o</sup> F (1093 <sup>o</sup> C)	-----
W - 1ThO <sub>2</sub> (NF)	Grain width, 1 to 2 microns	Grain width broadening, free of subgrains	Formation of subgrains	Formation of subgrains	Grain width broadening, 2 to 5 microns; some subgrains annealed out
W - 2ThO <sub>2</sub>	Grain width, 1 to 3 microns	Structure similar to as-drawn condition	Formation of subgrains	Grain width broadening	Grain width broadening, 2 to 4 microns
W - 5Re - 2ThO <sub>2</sub>	Grain width, 0.3 to 1.2 microns	No evidence of grain width broadening; subgrain formation	Structure similar to short-time exposure	Increase in dispersion size; little subgrain formation	Extensive subgrain formation; subgrains larger than those formed at 2000 <sup>o</sup> F (1093 <sup>o</sup> C)
W - 24Re - 2ThO <sub>2</sub>	-----	Void areas formed; particle content greater than in as-drawn condition	Subgrain formation	Grain width broadening; particle size increase	Excessive grain growth; agglomeration of particles
TZM	Particle size, 0.1 micron	-----	Grain growth; particle size, 0.1 to 0.3 micron	Subgrain formation; particle size, 0.1 to 0.3 micron	Extensive subgrain formation
TZC	Particle size, 0.1 to 1 micron	Some grain width broadening	Subgrain formation	Grain growth and particle coarsening	Grain growth and particle coarsening
FS-85	Grain width, 0.2 to 2 microns	Grain width increase; grain width, 0.6 to 3 microns; higher particle content at edge of wire than in center	Higher particle content than for short-time exposure; two types of particles formed: small, rounded particles and long, elongated particles	Structure similar to short-time, 2000 <sup>o</sup> F (1093 <sup>o</sup> C) condition but having higher particle content	Structure similar to long-time, 2000 <sup>o</sup> F (1093 <sup>o</sup> C) condition
AS-30	Particle size, 0.05 to 0.6 microns	Grain growth occurs; higher percent of particles formed than in as-drawn condition	Larger particles are formed as compared with short-time condition; particle size, 0.1 to 0.8 micron; higher volume percentage of particles formed	Mixture of small and large particles, large particles predominate	-----



