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Work performed for
U.S. DEPARTMENT OF ENERGY
Nuclear Energy
Reactor Systems Development and Technology
Washington, D.C. 20545
Under Interagency Agreement DE-AI03-86SF16310

Prepared for
The Materials Research Society Fall Meeting
Boston, Massachusetts, December 1-5, 1993

TENSILE AND STRESS-RUPTURE BEHAVIOR OF HAFNIUM CARBIDE DISPERSED MOLYBDENUM AND TUNGSTEN BASE ALLOY WIRES

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ABSTRACT

The tensile strain rate sensitivity and the stress-rupture strength of Mo-base and W-base alloy wires, 380 μm in diameter, were determined over the temperature range from 1200 to 1600 K. Three molybdenum alloy wires; Mo + 1.1 wt% hafnium carbide (MoHfC), Mo + 25 wt% W + 1.1 wt% hafnium carbide (MoHfC+25W) and Mo + 45 wt% W + 1.1 wt% hafnium carbide (MoHfC+45W), and a W + 0.4 wt% hafnium carbide (WHfC) tungsten alloy wire were evaluated.

The tensile strength of all wires studied was found to have a positive strain rate sensitivity. The strain rate dependency increased with increasing temperature and is associated with grain broadening of the initial fibrous structures. The hafnium carbide dispersed W-base and Mo-base alloys have superior tensile and stress-rupture properties than those without HfC. On a density compensated basis the MoHfC wires exhibit superior tensile and stress-rupture strengths to the WHfC wires up to approximately 1400 K. Addition of tungsten in the Mo-alloy wires was found to increase the long-term stress-rupture strength at temperatures above 1400 K. Theoretical calculations indicate that the strength and ductility advantage of the HfC dispersed alloy wires is due to the resistance to recrystallization imparted by the dispersoid.

INTRODUCTION

High temperature applications such as space power conversion have generated great interest in fiber reinforced metallic composites. Refractory metals and alloys reinforced with refractory metal alloy fibers have been shown to be applicable for extremely high temperature ranges [1]. The useful temperature depends upon the combination of fiber and matrix. The tungsten (W) fiber reinforced niobium alloy composite was reported to have high tensile and creep strength in the temperature range of 1400 to 1500 K [1]. The performance of a composite is usually dependent upon the fiber component. The major portion of the tensile and creep strength of the composite is associated with the properties of the fiber. The use of a strong and stiff fiber is desired for a high strength composite material.

Wires of the hafnium carbide dispersion strengthened W and W-Re alloys, ranging in diameter from 200 to 380 μm , have recently been shown to possess superior tensile and stress-rupture strengths, compared to the potassium bubble dispersed or the thoria dispersed W wires [2,3]. The fine hafnium carbide dispersoids were reported to be more effective than bubble or thoria dispersoids [3,4] in preserving the heavily unidirectionally elongated fibrous grain structure of the W alloy wires. The effectiveness of the hafnium carbide on the mechanical properties of other

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refractory metal base alloy wires, such as molybdenum (Mo), is of interest. Molybdenum (Mo) with its lower density than W appears to be attractive for making lower weight composites.

Houck [5,6] has studied the mechanical properties of Mo alloys, such as TZC (Mo + 1.25% Ti + 0.30% Zr + 0.15% C), TZM (Mo + 0.5% Ti + 0.08% Zr + 0.015% C), Mo + 0.5% Ti (all percentages in this report are in weight percent) and Mo + 0.5% Zr, and reported that the recrystallization temperature for these alloys varies from 1200 to 1700 K, depending on the alloying element. The TZC and TZM alloys possess a relatively high tensile strength, but the alloying elements do not appear stable at the higher testing temperatures. The stress-rupture strength of these alloys generally are much lower than the W-base alloys. The present study focussed on the determination of the tensile and stress-rupture properties of the hafnium carbide dispersed Mo-based alloy wires and on the comparison with hafnium carbide dispersed W alloy wires. The study also evaluated the effect of HfC dispersoids in stabilizing the fibrous microstructures in the wires in the temperature range of 1200 to 1600 K.

EXPERIMENTAL PROCEDURE

Materials

The chemical compositions of the hafnium carbide dispersed Mo and W alloy wires examined in this study are given in Table I. MoHfC wires, Mo with hafnium carbide, were the HfC dispersed simple Mo-base alloys, and MoHfC+45W and MoHfC+25W wires were the HfC dispersed and alloyed with 49.5% W and 30.4% W, respectively. The Mo-base wires were fabricated by powder metallurgy techniques, the W-base WHfC wires were produced by vacuum arc-melting process.

Hafnium carbide in the Mo alloys was formed from alloying of elemental Hf and C during sintering at high temperature. This Mo alloy wire (MoHfC+45W) is believed to have about 0.1% HfC and about 0.7% HfC for WHfC. The MoHfC wires also contained a small unintended amount (4%) of W, which can provide some solid-solution strengthening. The WHfC wires were strengthened by the 0.4% HfC dispersoids without additional alloying elements.

All wires were heavily drawn to their final nominal diameter of about 380 μm . Figure 1 shows the microstructures of the as-drawn wires in this study. The grain structures are noted as infinitely long and of a very fine width, so-called fibrous grain structures. The grain width of the MoHfC wires is about 0.5 μm , and that of the WHfC wires about 0.3 μm . The size distribution

TABLE I.—CHEMICAL COMPOSITION OF Mo AND W BASE ALLOY WIRES

| Material | Chemical composition, wt% (at.%) | | | | | |
|-----------|----------------------------------|--------|--------|---------------|----------------|---------|
| | C | N | O | Hf | W | Mo |
| MoHfC | 0.044 (0.36) | 0.0026 | 0.016 | 1.2 (0.66) | 4.4 (2.50) | Balance |
| MoHfC+25W | 0.020 (0.19) | 0.0023 | 0.0036 | 0.9 (0.56) | 30.4 (19.0) | Balance |
| MoHfC+45W | 0.0096 (0.10) | 0.0019 | 0.0038 | 0.8 (0.56) | 49.5 (34.8) | Balance |
| WHfC | 0.03 (0.45) | 0.0009 | 0.0039 | 0.4 (0.41) | Balance | ----- |

of the dispersoids in the MoHfC wires was inhomogeneous, ranging from less than 0.1 to 1.0 μm , whereas dispersoids in the WHfC wires were finely distributed with an average size of less than about 0.1 μm . MoHfC+25W and MoHfC+45W wires appear to result in a finer grain width and a more inhomogeneous spacing of the dispersoids than the MoHfC wires.

Test Procedure

Tensile and stress-rupture tests were conducted in a vacuum of 10^{-5} Pa at temperatures ranging from 1200 to 1600 K. Furnace test temperature was monitored with a platinum/platinum-13% rhodium thermocouple, and controlled within ± 3 K during the test. The experimental details of the tensile and stress-rupture test procedures have been given previously [3,7]. The wire was cut to about 40 cm lengths and then suspended through a vertically mounted resistance furnace. Tensile testing was at a constant cross-head speed and the load-time curves were recorded automatically. The proportional limit (PL), ultimate tensile strength (UTS) were determined from the load-time curves. For the stress-rupture test, the wire was loaded with an appropriate dead-weight, which was supported by a retractable support during specimen heating. The stress-rupture strength (σ_r) was determined from the stress versus rupture time plots. The reduction of area (RA) of failed specimens was measured using an optical split image microscope at 150 magnification.

Stress-rupture and tensile tests were performed on wires in the as-drawn condition. In addition tensile tests were performed on specimens electropolished [3] to produce a definite gauge section about 25.4 mm long and 280 ± 10 μm in diameter.

RESULTS

Tensile Stress-Strain Rate Behavior

The effect of the strain rates at 1400 and 1600 K on the PL of the as-drawn and electropolished wires is shown in Fig. 2. The electropolishing provided a tensile specimen with a well defined 25.4 mm long gauge section. The PL of the electropolished wires appeared to be higher than that of the as-drawn wires, about 650 and 480 MPa at 1400 K. The original 380 μm wire diameter was reduced to 280 μm . The strain rates were calculated based on the assumption that all deformation took place in the electropolished gauge section.

The decrease of the PL with decreasing strain rate was small or negligible at the high strain rate range of 3.3×10^{-4} to $3.3 \times 10^{-2} \text{ sec}^{-1}$. However, it is noted that the drop of the PLs is quite large at the slower strain rates, 3.3×10^{-4} to $3.3 \times 10^{-5} \text{ sec}^{-1}$. The drop of the PLs at the low strain rates is due to the onset of primary recrystallization at 1400 to 1600 K. The difference in the dependency of the strain rate between the MoHfC, MoHfC+25W and MoHfC+45W wires was negligible at 1400 K. At 1600 K the PL of MoHfC+45W wires is higher than that of the MoHfC wires over the strain rate range studied.

Stress-Rupture Properties

The stress-rupture properties of the as-drawn wires are shown in Fig. 3. Test temperature ranged from 1200 to 1477 K and the stress-rupture time from 0.1 to about 1000 hr. The error bar

indicates a representative range of rupture time data at one stress. The 10- and 100-hr stress-rupture strengths, $\sigma_{t=10}$ and $\sigma_{t=100}$, determined from the stress-rupture curves, are summarized in Table II. The difference in the σ_t between the Mo-base alloy and the W-base alloy wires increased with increasing rupture time and testing temperatures. At 1200 K, the $\sigma_{t=10}$ (10-hr stress-rupture strength) and the $\sigma_{t=100}$ were, respectively, 1237 and 1038 MPa for MoHfC, and 1275 and 1221 MPa for WHfC. At 1366 K, the $\sigma_{t=100}$ of MoHfC was much lower than that of WHfC, i.e., 480 MPa versus 950 MPa.

It is noted that the difference in the σ_t between the simple MoHfC and the MoHfC+45W also increased with increasing temperature and time. At 1200 K, the $\sigma_{t=10}$ of MoHfC was comparable with that of MoHfC+45W (about 1237 MPa). From 1366 to 1477 K, the 100-hr stress-rupture strength of the MoHfC+45W wires was substantially higher than that of the MoHfC wires, whereas the 10-hr short-term strength of the MoHfC+45 wires was slightly higher than that of MoHfC wires. This suggests that the W containing Mo alloy wires may be a candidate composite fiber reinforcement wires for the long-term and high temperature applications, and the simple MoHfC wires may be suitable for short-term and low temperature application below 1400 K.

The relationship between the initial stress and the rupture time, t_R , is shown in Fig. 4 as a function of testing temperature for the MoHfC wires. At 1200 and 1600 K a unique slope existed for each temperature, and the stress-rupture strength value at 1200 K was clearly higher than that at 1600 K for all the rupture times. The slope at 1200 K was steeper than that at 1600 K. From 1366 to 1477 K, however, two slopes existed, one for rupture times above about 10 hr, and another one for the relative short-term rupture time of less than 10 hr. The two different slopes at 1366 and 1477 K resulted in that the long-term $\sigma_{t=100}$ of MoHfC wires at 1366 K was considerably higher than that at 1477 K, 500 and 300 MPa, respectively. The sharp drop of the stress rupture strength for the long-term rupture time is associated with the change in the fibrous grain structures, such as grain broadening.

The relation between the initial stress and the rupture time of the MoHfC was correlated by using the conventional power-law expression [8,9]:

$$t_R = A\sigma^{-p} \exp (Q/RT) \quad (1)$$

where t_R = rupture time (hr), A = constant, σ = applied stress (MPa), p = stress exponent for stress-rupture, T = testing temperature (K), R = gas constant (8.314 J/mol.K) and Q = apparent activation energy for stress-rupture (kJ/mol). The stress exponent, p , the slope of the curves in

TABLE II.—10- AND 100-HR STRESS-RUPTURE STRENGTH OF Mo AND W BASE ALLOY WIRES

| Wire | 10-Hr stress-rupture strength at K, MPa | | | | | 100-Hr stress-rupture strength at K, MPa | | | | |
|-----------|--|------|------|------|------------------|---|------|------|------------------|-----------------|
| | 1200 | 1366 | 1400 | 1477 | 1600 | 1200 | 1366 | 1400 | 1477 | 1600 |
| MoHfC | 1237 | 728 | 564 | 464 | ^a 160 | 1038 | 480 | 350 | 310 | ^a 91 |
| MoHfC+25W | ----- | 675 | ---- | 494 | ---- | ---- | 574 | ---- | 405 | ---- |
| MoHfC+45W | 1237 | 808 | ---- | 615 | ---- | 1038 | 679 | ---- | 589 | ---- |
| WHfC | 1275 | 1176 | ---- | ---- | ---- | 1221 | 950 | ---- | ^b 780 | ---- |

^aExtrapolated from the measured short-term data.

^bFrom Petrasek et al.'s report.

Fig. 4, is high, about 14, at 1200 K over the entire test time range, and at 1366 and 1477 K for the short-term tests. A high p value at low temperatures is indicative of a high sensitivity of the rupture time with the applied stress, the observed stress value for $\sigma_{t=10}$ is almost equivalent to $\sigma_{t=100}$ at 1200 K. The long-term stress-rupture data at 1366 K displayed considerable scatter, however, the data appeared to correlate best with a stress exponent of about 5. The apparent activation energy, Q for the stress versus rupture-time was determined to be about 480 kJ/mol over the stress range of 400 to 500 MPa, for temperatures of 1366 to 1477 K, and for the long-term rupture time data of about 10 hr or larger. This value compares well with the steady-state creep value of 470 kJ/mol determined for recrystallized TZM Mo alloy at 1573 to 1673 K [10].

DISCUSSION

Strengthening by the Dispersoid Particles

The tensile and stress-rupture properties of the HfC dispersed Mo alloy wires appear to have comparable strengths to the WHfC wires at about 1200 K. However, for temperatures above 1200 K, the Mo-base alloy wires are weaker than the W-base alloy wires. The addition of W as an alloying element enhances the stability of the microstructure at high temperature and yields a higher strength MoHfC base alloy.

The short-term and/or low temperature tensile and stress-rupture strength results from the fibrous grain structures and Hall-Petch type grain boundary strengthening. The fine fiber grain contains more elastically stored energy [3,11], which results from the large amount of cold working employed in the wire drawing process, and contributes considerably to the wires strength. For example, the high tensile strength of the heavily drawn Mo-33Re alloy was reported to be due to the highly developed, fine scale, cell structures with a high background dislocation density [12]. For the HfC dispersoids strengthened Mo alloy, the long-term and high temperature stress-rupture and tensile strengths, however, may be the combined effects of strengthening from the fine dispersoids, Orowan stress from the dispersoids, and/or the solid solution hardening from the strong and hard alloying element. The fine and closely distributed HfC dispersoids have been shown to effectively block dislocation motion and to affect the formation of the cell and wall structures [11].

Table III shows the increase in the tensile and stress-rupture strength of the hafnium carbide dispersed wires compared to the literature values of the unalloyed wires and recrystallized W or Mo sheets at 1366 K. The increase in the tensile strength of the hafnium carbide dispersed wires was about fivefold and sevenfold for the stress-rupture strength in comparison to W and Mo sheets, respectively. The increase in tensile strength due to the addition of HfC particles to Mo or W and the cold work was about 650 MPa, which is somewhat higher than earlier estimates: Previous work indicated about a 120 MPa increase for the 200 μm WHfC wires with a 1.55 vol% fraction of HfC [3].

Property Comparisons

The stress-rupture data for the wires indicated that in the higher temperature range the WHfC wires have a higher strength than the MoHfC wires and that the W addition to the MoHfC is an effective strengthener. The density compensated specific strength values, stress/density, i.e.,

**TABLE III.—THE INCREASE IN THE TENSILE AND STRESS-
RUPTURE STRENGTH OF THE HfC PARTICLE DISPERSED
MoHfC AND WHfC WIRES IN COMPARISON TO THE
UNALLOYED MOLY AND TUNGSTEN
WIRES AT 1366 K**

| Material | Condition | Particle size, nm | Units, MPa | 100-Hr stress-rupture strength, MPa | Young's modulus, GPa |
|----------|------------------------|-------------------|------------------|-------------------------------------|----------------------|
| MoHfC | Wire (380 μm) | 150 | 970 | 480 | ---- |
| Mo | Wire (380 μm) | ---- | ^a 320 | ^b 80 | ---- |
| Mo | Sheet (recrystallized) | ---- | ^b 172 | ^b 57 | ^b 220 |
| WHfC | Wire (380 μm) | 35 | 1340 | 950 | ---- |
| W | Wire (380 μm) | ---- | ^a 650 | ^a 460 | ---- |
| W | Sheet (recrystallized) | ---- | ^b 241 | ^b 145 | ^b 365 |

^aUnpublished work by H.M. Yun.

^bFrom reference [5].

MPa/(g/cm³) or m, are an important criteria in choosing a candidate wire for fiber composite reinforcement. Figure 5 shows the comparison of the density compensated stress-rupture strength of the MoHfC, MoHfC+25W, MoHfC+45W and WHfC wires, including the 218, ST300, and W4ReHfC [13]. It is noted that the specific rupture strength of Mo-base wires is almost equivalent to that of the WHfC wires. The 100-hr specific rupture strength of the Mo-base alloy wires appeared to be lower at 1366 K than that of the W4ReHfC wires.

The present HfC dispersed Mo-base wires (MoHfC, MoHfC+25W and MoHfC+45W) also have a higher 100-hr specific rupture strength than the lamp grade 218 W or the thoria dispersed ST300 wire at 1366 or 1477 K. These results indicate that the MoHfC wire reinforced composites, such as Nb alloy matrix composites, may have a greater stress-rupture strength than similar composites reinforced with the 218 or ST300 W wire [1].

SUMMARY

Tensile and stress-rupture behavior of molybdenum (Mo) and tungsten (W) alloy wires, 380 μm diameter, have been studied in the temperature range of 1200 to 1600 K, and the results are summarized below:

- (1) Long-term stress-rupture strength of the MoHfC wires was improved by W addition.
- (2) The tensile strength of the MoHfC wires increased with increasing strain rates, and the strain rate dependency increased with increasing temperatures.

CONCLUSION

The hafnium carbide dispersed Mo-base alloy wires have a higher stress-rupture strength than the commercially available W-base alloy wires. The density compensated specific strengths of MoHfC wires is comparable to those of the strongest experimental W-base alloy wires. These

Mo-base alloy wires, therefore, appear to be an attractive alternative candidate for metal matrix composite fiber reinforcements.

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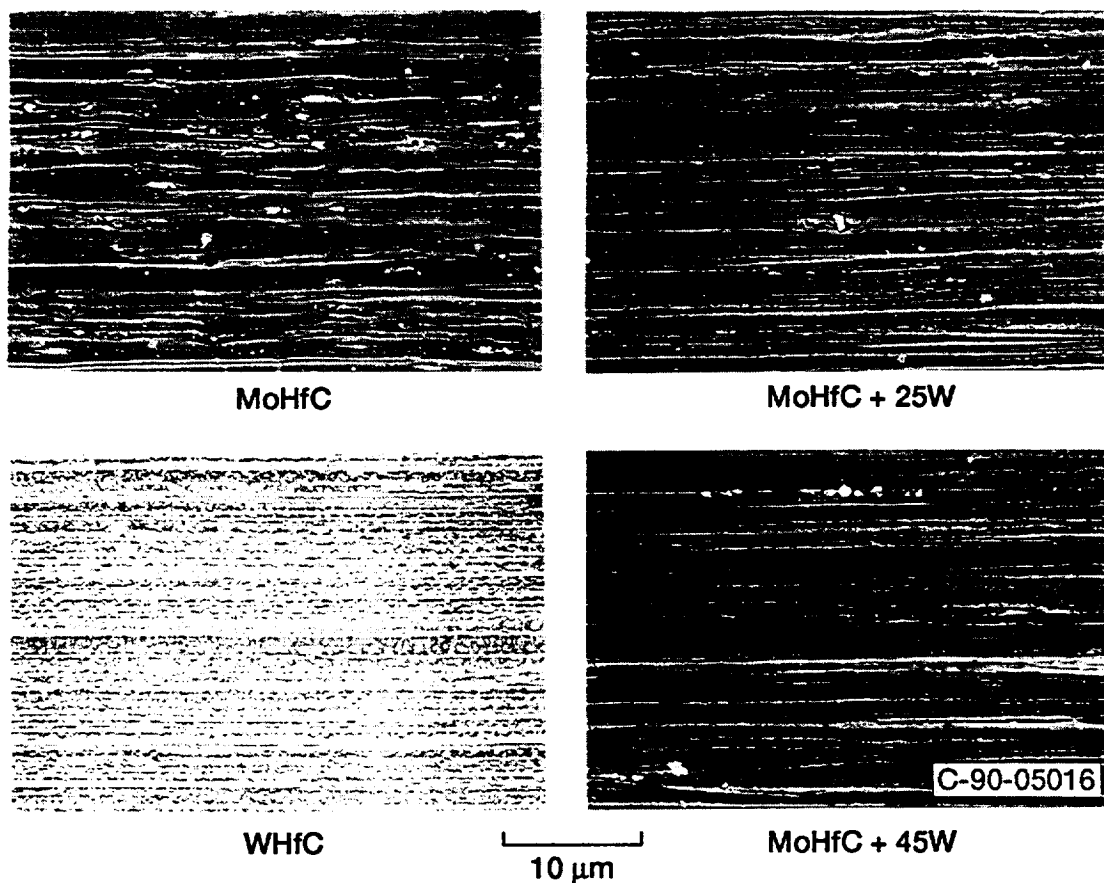


Figure 1.—As-drawn microstructures (SEM secondary electron images) of Mo and W base alloy wires.

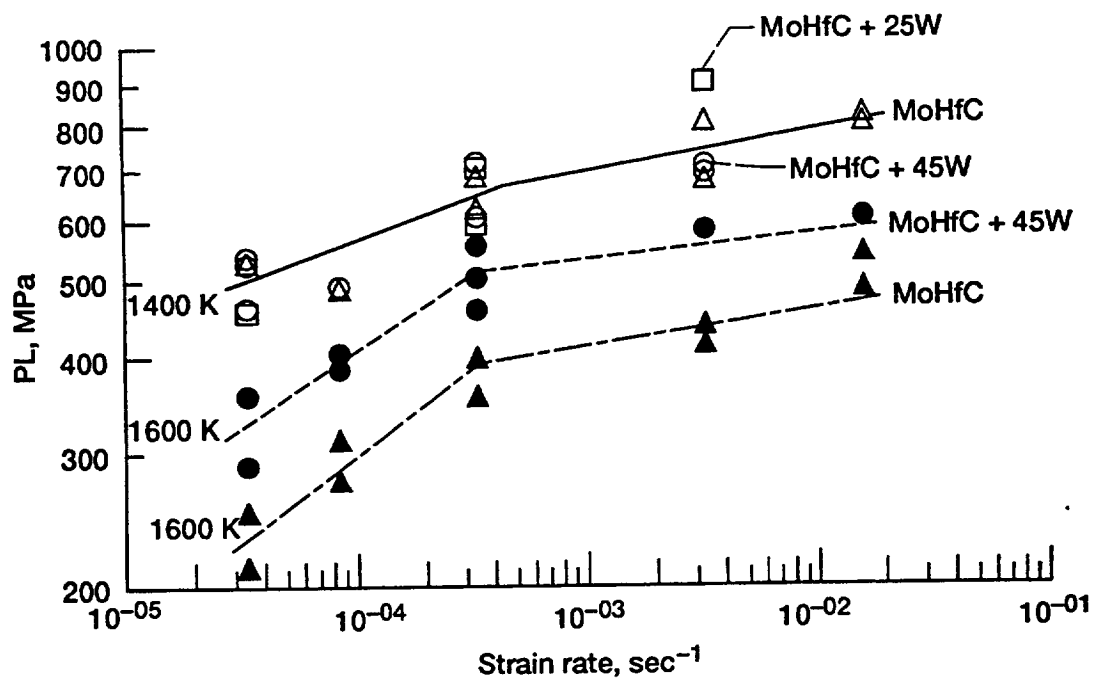


Figure 2.—Effect of the strain rate on the tensile strength of Mo-base alloy wire at 1400 and 1600 K.

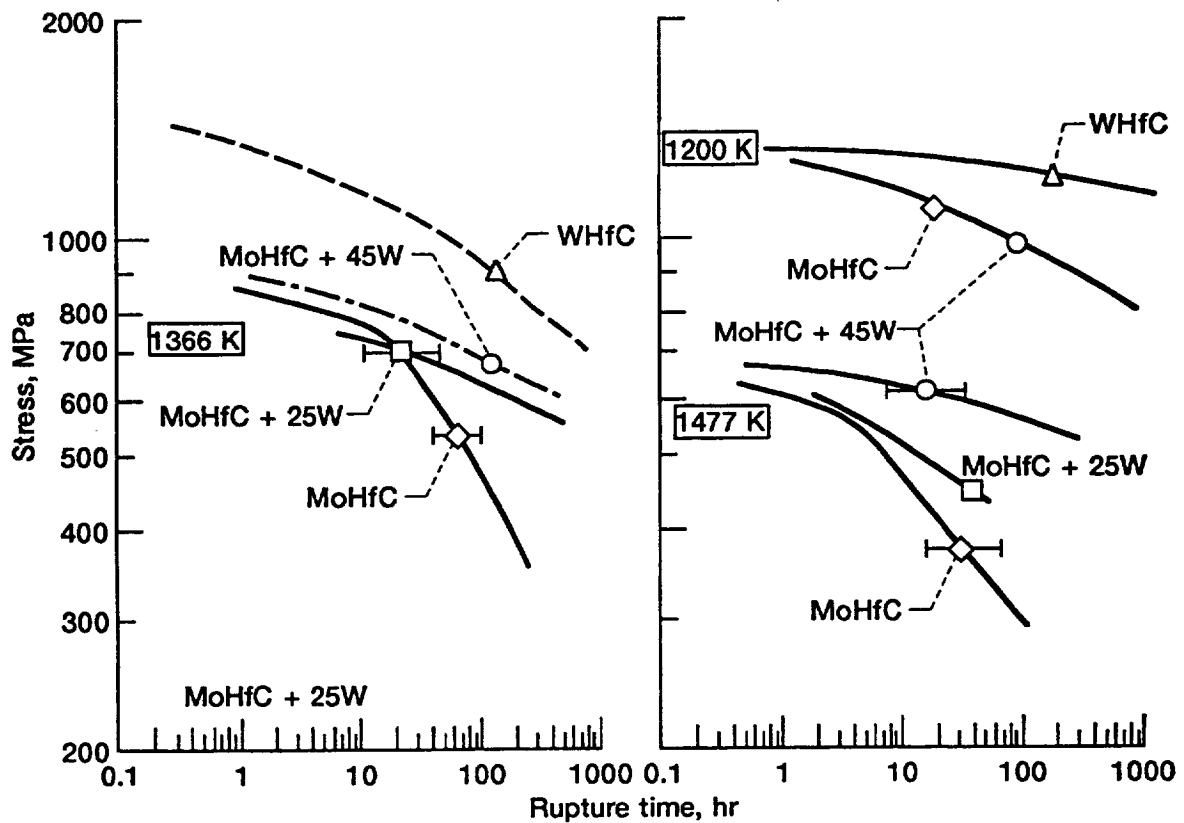


Figure 3.—Stress-rupture behavior of Mo- and W-base alloy wire at 1200, 1366 and 1477 K.

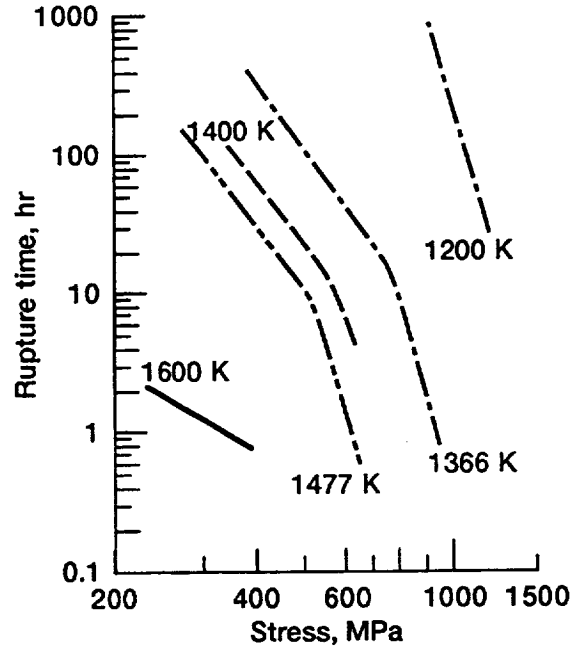


Figure 4.—Stress-rupture behavior of as-drawn MoHfC wires in the temperature range of 1200 to 1600 K.

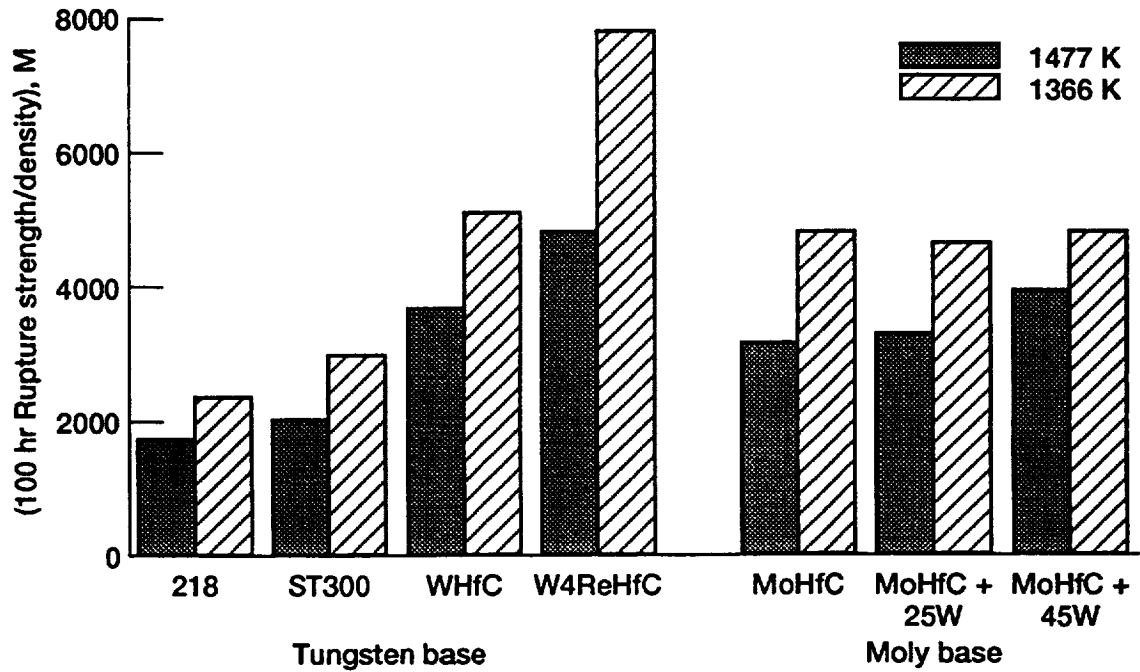


Figure 5.—Comparison of density compensated 100 hr-stress-rupture strength of candidate Mo- and W-base alloy wires for fiber reinforcements. The value of 218, ST300 and W4ReHfC wires came from the reference [4].

| REPORT DOCUMENTATION PAGE | | | Form Approved OMB No. 0704-0188 | |
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| 1. AGENCY USE ONLY (Leave blank) | 2. REPORT DATE December 1993 | 3. REPORT TYPE AND DATES COVERED Technical Memorandum | | |
| 4. TITLE AND SUBTITLE Tensile and Stress-Rupture Behavior of Hafnium Carbide Dispersed Molybdenum and Tungsten Base Alloy Wires | | 5. FUNDING NUMBERS WU-590-13-11 | | |
| 6. AUTHOR(S) Hee Mann Yun and Robert H. Titran | | | | |
| 7. PERFORMING ORGANIZATION NAME(S) AND ADDRESS(ES) National Aeronautics and Space Administration Lewis Research Center Cleveland, Ohio 44135-3191 | | 8. PERFORMING ORGANIZATION REPORT NUMBER E-8128 | | |
| 9. SPONSORING/MONITORING AGENCY NAME(S) AND ADDRESS(ES) National Aeronautics and Space Administration Washington, D.C. 20546-0001 | | 10. SPONSORING/MONITORING AGENCY REPORT NUMBER NASA TM-106350 | | |
| 11. SUPPLEMENTARY NOTES Final Report. Prepared under Interagency Agreement DE-AI03-86F16310. Hee Mann Yun, Cleveland State University, Cleveland, Ohio 44115; and Robert H. Titran, NASA Lewis Research Center. Prepared for The Materials Research Society Fall Meeting, Boston, Massachusetts, December 1-5, 1993. Responsible person, Hee Mann Yun, (216) 433-6089. | | | | |
| 12a. DISTRIBUTION/AVAILABILITY STATEMENT Unclassified - Unlimited Subject Category 26 | | 12b. DISTRIBUTION CODE DOE Category UC-504 | | |
| 13. ABSTRACT (Maximum 200 words) The tensile strain rate sensitivity and the stress-rupture strength of Mo-base and W-base alloy wires, 380 μ m in diameter, were determined over the temperature range from 1200 K to 1600 K. Three molybdenum alloy wires; Mo + 1.1w/o hafnium carbide (MoHfC), Mo + 25w/o W + 1.1w/o hafnium carbide (MoHfC+25W) and Mo + 45w/o W + 1.1w/o hafnium carbide (MoHfC+45W), and a W + 0.4w/o hafnium carbide (WHfC) tungsten alloy wire were evaluated. The tensile strength of all wires studied was found to have a positive strain rate sensitivity. The strain rate dependency increased with increasing temperature and is associated with grain broadening of the initial fibrous structures. The hafnium carbide dispersed W-base and Mo-base alloys have superior tensile and stress-rupture properties than those without HfC. On a density compensated basis the MoHfC wires exhibit superior tensile and stress-rupture strengths to the WHfC wires up to approximately 1400 K. Addition of tungsten in the Mo-alloy wires was found to increase the long-term stress-rupture strength at temperatures above 1400 K. Theoretical calculations indicate that the strength and ductility advantage of the HfC dispersed alloy wires is due to the resistance to recrystallization imparted by the dispersoid. | | | | |
| 14. SUBJECT TERMS Tensile test; Strain-rate; Stress-rupture; Recrystallization; Tungsten; Molybdenum; Hafnium carbide | | | 15. NUMBER OF PAGES 12 | |
| | | | 16. PRICE CODE A03 | |
| 17. SECURITY CLASSIFICATION OF REPORT Unclassified | 18. SECURITY CLASSIFICATION OF THIS PAGE Unclassified | 19. SECURITY CLASSIFICATION OF ABSTRACT Unclassified | 20. LIMITATION OF ABSTRACT | |

