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MECHANICAL PROPERTIES OF OXIDE DISPERSION STRENGTHENED (ODS)
MOLYBDENUM ALLOYS

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Abstract

Oxide dispersion strengthened molybdenum, Mo-ODS, developed by a proprietary powder metallurgy process, exhibits a creep-rupture life at $0.65T_m$ (1600°C) of three to five orders of magnitude greater than unalloyed molybdenum, while maintaining ductile fracture behavior at temperatures significantly below room temperature. In comparison, the creep-rupture life of the Mo-50Re solid solution strengthened alloy at 1600°C is only an order of magnitude greater than unalloyed molybdenum. The results of microstructural characterization and thermal stability and mechanical property testing are discussed.

Introduction and Background

The improvement in the low temperature ductility of molybdenum by additions of up to 50 weight percent rhenium is a phenomenon that has received considerable attention since its discovery by Geach and Hughes over forty years ago.[1] Although an improvement in the low temperature fracture behavior is realized, improvement in high-temperature strength is not as dramatic as illustrated by Figure 1. The creep-rupture life of unalloyed molybdenum at $0.65T_m$ (1600°C or 2910°F) is increased approximately an order of magnitude with 50 weight percent addition of rhenium. While the solid solution strengthening of molybdenum by rhenium is effective, another strengthening mechanism that has been demonstrated to improve the elevated temperature creep strength of metals is oxide dispersion strengthening. (ODS).[2]

Increasing the elevated temperature creep rupture strength of molybdenum by oxide dispersion strengthening was investigated by Jaffee and benefits in elevated temperature creep strength were observed.[3] Jaffee reported the effects of a number of oxide additions on the 982°C creep rupture life at 25 ksi. The oxides reported to have the greatest effect were alumina, titania, and zirconia, displaying improvements under these conditions of two orders of magnitude. However, the oxides were added on a constant weight percent basis, and because of oxide density differences, the volume fraction at a given weight percent addition varied by a factor of four. For the compositions being studied, this represented an oxide volume content of ten to forty percent. However, this early work clearly displayed the promise of oxide dispersion strengthening as a mechanism for improving the elevated temperature strength of molybdenum. One significant observation of this work was that none of the compositions could be hot worked with 10 wt.% oxide additions.

A commercially available ODS molybdenum containing 1 wt. % lanthanum oxide is produced by mechanical mixing of the oxide with molybdenum powder. Wire has been successfully drawn from this material and creep data reported indicate three orders of magnitude reduction in creep rate compared with unalloyed molybdenum or TZM at 3270°F and 4 ksi.[4] However, no creep rupture data were reported, and a lower creep rate does not always result in an increase in rupture life.

Low room temperature ductility is one of the properties that has limited broader application of molybdenum alloys. The ductile to brittle transition temperature (DBTT) for unalloyed powder metallurgical molybdenum (PM-Mo) can be at or below room temperature depending on the metallurgical condition, i.e., stress relieved or recrystallized, as well as other factors including strain rate, grain size, stress state, and impurity content. The content of dissolved interstitial elements (e.g., oxygen, carbon, and nitrogen) has the most profound effect on the DBTT of molybdenum. Of the interstitials, the effect of oxygen is the most extreme, nitrogen being

intermediate, and carbon the least.[6,7] By reducing the oxygen and nitrogen contents to less than one weight ppm, the tensile DBTT has been observed to be below -196°C (-320°F).[8] In addition to low oxygen, the atom ratio of carbon to oxygen has also been reported to effect the DBTT.[9] At a carbon/oxygen atom ratio of 2/1 or greater, recrystallized molybdenum was reported to be ductile at room temperature, but when the C/O atom ratio was less than 2/1, recrystallized molybdenum exhibited brittle behavior at room temperature. However, the authors did not report the grain size of the test material, the character of the fracture surfaces, nor the DBTT.

The successful development of a new class of oxide dispersion strengthened (ODS) molybdenum materials is reported here, and the mechanical behavior of these materials is discussed. In particular, the results of high-temperature creep-rupture testing and the low temperature fracture behavior are presented.

Experimental Procedures

Material Processing: A general process flow outline for preparation of the experimental material is shown in Figure 2. Individual material batch sizes ranging from 1 to 60 kg containing 2 and 4 volume percent of La_2O_3 , CeO_2 , Y_2O_3 , and ThO_2 additions, respectively, were prepared using a proprietary powder metallurgy procedure.[10] Starting billets ranging in size from 1.3 inch diameter to 2.75 inch diameter were consolidated by cold isostatic pressing (CIP) with green densities on the order of 65% of theoretical. Billets were fully densified by a combination of high temperature sintering followed by a) - swaging and drawing, b) - rod rolling and swaging, or c) - dynamic compaction using a high energy extrusion press (DYNAPAK) and swaging to a typical final rod diameter of 0.18 inch. The total reduction in area from the starting billet to the final rod diameter by the various working operations was always greater than 90%. Prior to mechanical working operations, the billets were preheated in hydrogen to temperatures in the range of 800 to 1500°C .

Tensile and creep rupture specimens of the configurations illustrated in Figures 3a and 3b, respectively, were machined from the 0.180 inch diameter rod. Prior to testing, each specimen was electropolished at room temperature in a solution of four parts concentrated reagent grade sulfuric acid and one part distilled and deionized water using a Type 304 stainless steel cathode and a dc potential of six to seven volts to remove 0.002-0.003 inches from the gauge section diameter.

Material Characterization: Samples removed at various stages of working were examined using light optical metallographic examination and Vickers hardness measurements were obtained. A longitudinal and a transverse section of each specimen was prepared by sectioning, mounting in Bakelite, and polishing through a series of declining grit sizes. The final relief polish used 0.05 micron alumina powder. The mounts were swab etched with Murakami's etch (10 grams potassium ferricyanide and 10 grams potassium hydroxide in 100 milliliters distilled water). The average grain size of the final rod product was determined using the linear intercept method, per ASTM Standard E89. The average size and shape of the oxide particles was determined from unetched optical micrographs using a similar method. A minimum of five impressions were made along the longitudinal section of each metallographically prepared specimen using a Wilson Tukon Series 200 automated hardness tester or a Vickers Hardness Tester. A pyramidal diamond indenter with a 0.5 or 1 kilogram load and a ten second dwell time was used on the Wilson machine, and a 5 kilogram load was used with the Vickers Hardness Tester.

Transmission electron microscopy (TEM) analysis was also performed on selected ODS compositions at the Major Analytical Instrumentation Center (MAIC) at the University of Florida, Gainesville, FL, under the direction of Professor Michael Kaufman. Longitudinal and transverse thin foil specimens were prepared by sectioning with a low-speed diamond saw, mechanically thinning by grinding on silicon carbide paper, and punching into 3 mm disks. An electrolytic etch in a room temperature solution of 1 part concentrated sulfuric acid and 7 parts methanol with an applied potential of 15 Vdc and a current density of 150 mA/cm² was used to thin each specimen until perforation. Ion milling using argon ions at an accelerating potential of 4 kV and incident angles of 15° and 11° was used to complete the final stages of thinning and polishing, respectively. The thinned areas around the perforation were approximately 500 - 1500 Å thick. Specimens were characterized using a JEOL 200CX transmission electron microscope (TEM) with an accelerating potential of 120 kV. Brightfield (BF) and darkfield (DF) micrographs were taken to document the shape and size of the strain-hardened microstructural features and oxide particles, respectively. Selected area electron diffraction (SAED) techniques were used to identify the oxide phase/crystal structure and the preferred orientation of the as-worked Mo-ODS rods.

Mechanical Properties: The following mechanical properties were measured for the Mo-ODS material: (a) 0.2% offset yield stress, (b) ultimate tensile stress, (c) percent reduction in area, and (d) total elongation to failure. The ductile-to-brittle transition temperature (DBTT) of the worked and recrystallized swaged rod was measured by uniaxially loading a round bar tensile specimen to failure at temperatures ranging from +200°C to -196°C (+390°F to -320°F) per ASTM Standards E8 and E21 at a strain rate of 0.05 min⁻¹ or 0.00083 sec⁻¹. The elevated temperature tensile properties of the swaged and recrystallized rod were measured by uniaxially loading a round bar tensile specimen to failure in dynamic vacuum (< 5x10⁻⁵ torr) at temperatures ranging from 1000°C to 2200°C (1830°F to 3990°F) and a strain rate of 0.025 min⁻¹ or 0.00042 sec⁻¹. The surface of each fractured tensile specimen was examined with a scanning electron microscope (SEM), Amray Model 1810, to identify the mode of fracture, i.e., ductile or dimpled, cleavage, intergranular, or mixed-mode.

The uniaxial creep-rupture behavior of the Mo-ODS material was evaluated at temperatures in the range of 1200°C (2190°F) to 1800°C (3270°F) under ultra high vacuum (< 10⁻⁸ torr) conditions using internally dead weight loaded machines as previously described.[11] Round bar creep rupture specimens were tested in the as-swaged condition and after a high temperature anneal. The test specimen was heated to the desired test temperature and held for thirty minutes with no load on the specimen. An initial length measurement was obtained by an optical technique using a Gaertner optical extensometer with a resolution of 50 microinches. Measurements were made using the shoulders of the gauge section for reference. The initial load was then applied, and the first strain measurement was made. Strain measurements were taken at frequent intervals to define the creep deformation behavior of the specimens. The fracture surface of each specimen was examined by SEM as described above to identify the mode of fracture.

Results and Discussion

Material Characterization: The chemical composition of each powder mixture was determined using both inert gas fusion and plasma spectrometry techniques. The oxygen content for the starting powder mixtures ranged from 0.2 to 0.5 wt.% oxygen depending on the volume fraction added; and the carbon content ranged from 25 to 200 ppmw (parts per million by weight).

Representative light optical photomicrographs and transmission electron micrographs of an as-worked Mo-ODS rod containing two volume percent of lanthana particles are presented in Figures 4 and 5. The microstructure of the Mo-ODS material in the as-worked condition is characterized by refined, elongated grains with average widths ranging from 0.02 mils (0.5 μm) to 0.04 mils (1 μm). The average aspect ratio (average grain length/width) for the as-worked material ranged from 5 to 50. The average oxide particle sizes were 0.05-0.5 μm for the Mo-ODS with lanthana, ceria and thoria, but the average oxide particle sizes were 0.5-1.5 μm for Mo-ODS with yttria. The oxide particles were mainly observed as polycrystalline films or stringers along grain boundaries with smaller discrete particles located within grains (Figure 5). The size and shape characteristics of the oxide particles can be classified into two groups: non-deformable, spherical particles (Mo + yttria) and deformable stringers (Mo + thoria, lanthana, or ceria). During subsequent thermomechanical processing of material containing the deformable variety, the as-centered oxide particles deform and are reduced in size. The size and shape of the harder yttria particles, on the other hand, remained unchanged. Selected area electron diffraction (SAED) analysis of the metal grains in transverse orientation specimens indicated a preferred orientation of $\langle 110 \rangle$ parallel to the rod axis. SAED analysis also verified that the dispersed particles were indeed the desired oxide dispersoids.

The recovery and recrystallization response of the as-worked material was directly related to the oxide particle size-distribution. The recrystallization temperature was measured from both microstructure and microhardness changes for each composition. The Mo-ODS material containing the finest oxide particle distribution—that is, thoria, ceria, and lanthana—exhibited the highest one-hour recrystallization temperature (up to 2000°C); whereas, the Mo-ODS rod containing a coarser oxide particle distribution, i.e., yttria, exhibited only a moderate improvement in the one-hour recrystallization temperature (up to 1500°C), compared to about 1100°C for unalloyed powder metallurgy molybdenum rod as illustrated in Figure 6. Also shown on Figure 6 are the resulting heat treated microstructures for the Mo-ODS containing yttria (a) which after one hour at 1500°C exhibited fully recrystallized equiaxed grains. The lanthana containing Mo-ODS (b) however, exhibited a high aspect grain structure, indicative of the effectiveness of the lanthana dispersion in promoting anisotropic recrystallization after heating for one hour at 1800°C.

Low Temperature Mechanical Behavior: The temperature at which the transition from ductile-to-brittle behavior occurred (DBTT) was determined by analyzing the tensile test results and examining the fracture surfaces. The effect of temperature on the tensile reduction in area for molybdenum and the various Mo-ODS compositions is presented in Figure 7a for material tested in the worked and stress-relieved condition and in Figure 7b for material in the recrystallized condition. The Mo-ODS compositions exhibit significantly better ductility than unalloyed molybdenum (powder metallurgy) in both the worked and stress relieved condition and especially in the recrystallized condition. The DBTT for the Mo-ODS materials in the worked and stress relieved condition ranged from below -150°C (-235°F) to approximately -100°C (-148°F), and the DBTT for the Mo-ODS material in the recrystallized condition was as low as -75°C (-100°F). A representative scanning electron photomicrograph of a fracture surface from a recrystallized test specimen uniaxially loaded to failure at -75°C (-100°F) is presented in Figure 8. The dimpled fracture surface is characteristic of a ductile overload failure.

The Mo-ODS material in the worked and stress-relieved condition exhibits a DBTT well below room temperature. However, in the recrystallized condition, the DBTT may be near or above

room temperature, depending on the recrystallized grain size, which in turn is influenced by the volume fraction and particle size of the dispersed oxide and the final amount of cold work. The effect of grain size on the DBTT is presented in Figure 9, displaying data for unalloyed molybdenum (PM-Mo) and Mo-ODS rod in the worked/stress-relieved condition and in the recrystallized condition. Both the Mo-ODS material and the unalloyed molybdenum exhibit a very fine as-worked grain size. However, the fine oxide particles in the Mo-ODS material inhibit grain growth promoting a smaller recrystallized grain size in comparison to the unalloyed molybdenum, providing a DBTT below room temperature.

In body centered cubic metals at temperatures below $0.2T_m$, the flow stress is strongly temperature dependent and has been thoroughly studied.[13,14] For molybdenum, $0.2T_m$ is 305°C (613°F). As the test temperature is lowered, yield strength increases until the DBTT is reached. The yield strength continues to increase until it exceeds the fracture strength of the material, where the failure is characterized by brittle cleavage type fractures. From Figure 10a, it is noted that the slopes of the yield strength curves for unalloyed molybdenum and Mo-ODS are similar even though the Mo-ODS has a higher yield strength. The presence of the fine oxide particles stabilizes the as-worked structure, maintains a fine grain size, and results in the transition temperature for recrystallized Mo-ODS being below room temperature. Another interesting feature displayed in Figure 10a is the temperature independence of the flow stress exhibited by the Mo-ODS lanthana over the temperature range of 300°C down to 150°C ($0.2-0.15T_m$). For bcc metals, this characteristic is not generally observed. How the oxide dispersion/microstructure influences the flow stress behavior is a subject worthy of further study.

Elevated Temperature Mechanical Behavior: Tensile yield strengths determined at elevated temperatures for the various Mo-ODS compositions are shown in Figure 10b and compared with similar data for unalloyed molybdenum. The tensile yield strength of the Mo-ODS material is two to three times greater than unalloyed molybdenum between 1000 and 2200°C (1830 and 4000°F). Specimens tested at 1600°C and above failed by intergranular separation. A representative scanning electron micrograph of the fracture surface is presented in Figure 11. Decohesion of grains was observed throughout the region of plastic instability, i.e., the necked region. This mode of failure is typical for metals at this fraction (0.65) of the melting temperature.

The creep-rupture behavior of the Mo-ODS material shows a significant improvement over unalloyed arc cast molybdenum at all test temperatures. Arc cast molybdenum properties were used as the basis for comparison. Powder metallurgy molybdenum properties are sensitive to source and processing, while the properties for the arc cast molybdenum were consistent, and independent of source.[5] The creep-rupture properties of the Mo-ODS rod and unalloyed arc cast molybdenum at 1200°C (2190°F), 1600°C (2910°F), and 1800°C (3270°F) are compared in Figures 12 - 14, respectively. At all three temperatures, the creep-rupture lifetimes of the Mo-ODS were greater by as much as 4 orders of magnitude compared with unalloyed arc cast molybdenum. The one exception was the Mo-ODS yttria material which exhibited only about a six-fold increase in creep-rupture lifetime over unalloyed molybdenum at 1600°C (2910°F) (Figure 13). This modest improvement in creep rupture lifetime was attributed to one of two reasons: the coarser oxide particle size distribution and/or the presence of a non-deformable oxide dispersed. In fact, the commercially available Z-6 Mo-ODS material, containing about 0.5 wt.% zirconia particles, exhibits a similar moderate improvement in creep-rupture life. The zirconia particle size distribution in this material is also much coarser than that of the Mo-ODS containing ceria, thoria, or lanthana particles. Also, the zirconia particles—similar to the yttria particles—are non-deformable.

With the exception of the Mo-ODS compositions containing yttria which failed intergranularly, all creep-rupture specimens containing deformable oxides failed transgranularly at test temperatures of 1200°C (2190°F)(0.5 T_m) to 1600°C (2910°F)(0.7 T_m). Over this range of fractions of T_m , solid solution strengthened alloys normally fail by intergranular separation.

The extraordinary improvement in creep strength resulting from the addition of a deformable particle is illustrated by the results of a temperature change test on a Mo-ODS composition containing two volume percent thoria. At an applied stress of 7 ksi (48.2 MPa), testing was initiated at 1400°C (2550°F). Maintaining the 7 ksi stress, the test temperature was increased in 100C° (180F°) increments up to a maximum temperature of 1800°C (3270°F). The measured creep elongation versus time is presented in Figure 15. The stress-rupture lifetime (in minutes) for unalloyed arc cast molybdenum at 7 ksi is shown in parentheses at each test temperature for comparison.[5] For example at 1700°C (3090°F), unalloyed Mo would have ruptured in 0.3 minutes, whereas the Mo-ODS material was tested for more than 800 hours without rupture and less than 0.1% elongation. Currently, Mo-ODS material has been tested at 1800°C (3270°F) and 5 ksi for up to 3000 hours without failure. At 1800°C (3270°F), unalloyed molybdenum would have ruptured in less than one tenth of a minute. The Mo-ODS material is stronger in creep-rupture at 1800°C (3270°F) than either unalloyed tungsten or rhenium at 1600°C (2910°F)- (Figure 16).

Summary

The creep-rupture life of unalloyed molybdenum at 0.65 T_m (1600°C or 2910°F) was exceeded by 3-5 orders of magnitude (also at 0.65 T_m) by oxide dispersion strengthened (ODS) molybdenum, fabricated using a proprietary process. The DBTT for material in the recrystallized condition was determined to be significantly below room temperature, attributed to the stable, fine grained microstructure developed through appropriate thermomechanical processing. The combination of excellent workability, high temperature strength, and low temperature ductility make Mo-ODS material a candidate for numerous applications. The Mo-ODS material at 1800°C (3270°F) exhibits a creep-rupture life up to 5 orders of magnitude greater than that of unalloyed molybdenum and at least ten times greater than unalloyed arc cast tungsten and two orders of magnitude greater than the Mo-50Re alloy at 1600°C (2910°F). This outstanding creep rupture behavior, coupled with a DBTT below room temperature provides a material that could be substituted for tungsten in certain applications.

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Figure 1. Creep-rupture of unalloyed molybdenum, Mo-50 wt.% Re alloy, unalloyed tungsten, and unalloyed rhenium at 2910°F (1600°C).[5]

Figure 2. Summary of the fabrication and evaluation schedule for the Mo-ODS compositions.

(a) - Room and Cryogenic Test Temperatures

(b) - Elevated temperature creep - rupture

Figure 3. Test specimen configurations.

(a) Longitudinal orientation

(b) Transverse orientation

Figure 4. Light optical photomicrograph of the as-worked Mo-ODS containing two volume percent of lanthana.

(a) Longitudinal orientation

(b) Transverse orientation

Figure 5. Brightfield transmission electron micrograph of the as-worked Mo-ODS containing two volume percent of lanthana.

a) Mo-Yttria

b) Mo-Lanthana

Figure 6. Recovery and recrystallization behavior of unalloyed powder metallurgy molybdenum Rod (85% prior RA) and Mo-ODS rod (98.1% prior RA) with representative microstructure after heating for one hour at 1500°C for Mo-Yttria (a) and after heating for one hour at 1800°C for Mo-Lanthana (b).

(a) Worked and Stress-Relieved Condition

(b) Recrystallized Condition

Figure 7. Effect of temperature on the tensile ductility of Mo-ODS and unalloyed molybdenum.

(a)

(b)

Figure 8. Secondary electron micrograph of the fracture surface of a recrystallized Mo-ODS specimen containing two volume percent of lanthana particles uniaxially loaded to failure at -75°C (-100°F): (a) low magnification(35X), and (b) high magnification(1,000X).

Figure 9. Effect of grain size on the ductile-to-brittle transition temperature (DBTT) of Mo-ODS and unalloyed (P/M) molybdenum.[12]

Figure 10a. Effect of temperature on the yield strength of Mo-ODS compositions and unalloyed molybdenum tested in the worked and stress relieved condition, ambient temperature.

Figure 10b. Effect of temperature on the yield strength of Mo-ODS compositions and unalloyed molybdenum tested in the worked and stress relieved condition, elevated temperature.

a - Low magnification

b - High magnification

Figure 11. Scanning electron micrograph of the fracture surface of an as-worked Mo-ODS specimen containing two volume percent lanthana, uniaxially loaded to failure at 1600°C (2910°F) in dynamic vacuum.

Figure 12. Creep-rupture properties of Mo-ODS and unalloyed molybdenum(arc cast) at 1200°C (2192°F).

Figure 13. Creep-rupture properties of the Mo-ODS compositions and unalloyed molybdenum(arc cast) at 1600°C (2910°F).

Figure 14. Creep-rupture properties of Mo-ODS compositions and unalloyed molybdenum (arc cast) at 1800°C (3270°F).

Creep Elongation, percent

Figure 15. Constant-load, uniaxial creep curve of Mo-ODS (2 vol% thoria) tested with an applied stress of 7 ksi (48.2 MPa).

Figure 16. Creep-rupture Properties of unalloyed molybdenum (arc cast), Mo-50Re, rhenium and tungsten at 1600°C (2910°F) and Mo-ODS at 1800°C (3270°F).

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