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EVALUATION OF OXIDE DISPERSION STRENGTHENED (ODS) MOLYBEDNUM AND MOLYBDENUM-RHENIUM ALLOYS

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Abstract

Oxide dispersion strengthened (ODS) molybdenum alloys being developed for high temperature applications possess excellent high temperature strength and creep resistance. In addition they exhibit a ductile-to-brittle transition temperature (DBTT) in the worked and stress-relieved condition under longitudinal tensile load well below room temperature. However, in the recrystallized condition, the DBTT may be near or above room temperature, depending on the volume fraction of oxide dispersion and the amount of prior work. Dilute rhenium additions (7 and 14 wt.%) to ODS molybdenum were evaluated to determine their effect on low temperature ductility. The addition of 7 wt.% rhenium to the ODS molybdenum did not significantly enhance the mechanical properties. However, the addition of 14 wt.% rhenium to the ODS molybdenum resulted in a DBTT well below room temperature in both the stress-relieved and recrystallized condition. Additionally, the tensile strength of ODS Mo-14Re is greater than the base ODS molybdenum at 1000 to 1250°C.

Introduction

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 improvement in the low temperature fracture behavior is realized, the improvement in high-temperature strength is not as dramatic as illustrated by the plot of Figure 1. The creep-rupture life of unalloyed molybdenum at 0.65 of the absolute melting temperature (T_m) (1600°C or 2912°F) is only increased approximately an order of magnitude with up to 50 weight percent addition of rhenium. However, an oxide dispersion strengthened molybdenum (ODS Mo) alloy at 1800°C (3272°F) exhibits a creep-rupture strength many orders of magnitude greater than unalloyed molybdenum exhibits at 1600°C (2912°F).[2] In fact, the ODS Mo alloy is stronger in creep-rupture at 1800°C (3272°F) than either unalloyed tungsten or rhenium are at 1600°C (2912°F). In conjunction with this extraordinary strength, good low temperature ductility is displayed by the ODS Mo alloy in the cold-worked and stress-relieved condition, and the tensile ductile-to-brittle transition temperature (DBTT) is approximately -100°C (-148°F). The DBTT for unalloyed molybdenum (PM-Mo) can be at or below room temperature and depends on its metallurgical condition; i.e., stress relieved or recrystallized. In addition, a number of other factors such as strain rate, grain size, stress state, and impurity content also influence the DBTT. The dissolved interstitial elements (e.g., oxygen, carbon, and nitrogen) have the most profound effect on the DBTT of molybdenum. Of the interstitials, oxygen is the most severe in raising the DBTT, nitrogen is intermediate, and carbon has the least effect.[4] By reducing the oxygen and nitrogen content to less than one (1) weight ppm, the tensile DBTT has been observed to be less than -196°C (-321°F).[4-6] In addition to low oxygen, the atom ratio of carbon to oxygen is also reported to be a factor.[7] Room temperature elongation measured for unalloyed molybdenum, consolidated by either powder metallurgy or arc melting, in the recrystallized condition is sensitive to the carbon/oxygen atom ratio. At a carbon/oxygen atom ratio of 2/1 or greater, recrystallized molybdenum was ductile at room temperature, but when the carbon/oxygen atom ratio was less than 2/1, recrystallized molybdenum exhibited brittle behavior at room temperature.[7] However, no information on either the grain size for the test material or the character of the fracture surfaces was reported by the investigator, nor was the DBTT of the test material determined.

The addition of large amounts of rhenium to the Group VIA elements molybdenum and tungsten has been well characterized, and the dramatic improvement in the low temperature ductility of molybdenum and tungsten has been referred to as the "rhenium ductilizing" effect.[8-10] There has also been a significant amount of investigation on adding dilute amounts of rhenium, 20 weight percent or less to molybdenum; and, even at this level, improvements in low temperature ductility have been observed. The improvement in the low temperature ductility of molybdenum at dilute rhenium additions has been termed "Alloy Softening".[11-14] Interestingly, one investigation reported a lowering of the DBTT for molybdenum to below room temperature by the addition of 14% rhenium. This was observed even though the carbon/oxygen atom ratio for the material was less than 1.0.[11] Apparently, the influence of the carbon/oxygen atom ratio on room temperature ductility is not the only parameter that influences DBTT.

As discussed in reference [2], ODS Mo 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 volume fraction of oxide dispersion which influences the recrystallized grain size. Grain size is the parameter which is most often used for presenting DBTT data for the refractory metals. The effect of grain size on the DBTT for molybdenum alloys is presented in Figure 2, which displays data for unalloyed powder metallurgy molybdenum (PM-Mo), ODS Mo with 2 and 4 volume percent oxide, and binary molybdenum-rhenium (Mo-Re) alloys in both the worked/stress-relieved condition as well as in the recrystallized condition. For a given grain size, a dilute amount of rhenium further lowers the DBTT for recrystallized molybdenum. Based on this observation, a program was initiated to determine whether dilute rhenium additions to ODS Mo would yield comparable benefits to the DBTT. Both the low and high temperature mechanical behavior of oxide dispersion strengthened molybdenum (ODS Mo) will be discussed in this paper. The focus will be on the additional effect that dilute rhenium additions, 7 and 14 weight percent, have on the mechanical properties of the ODS Mo, particularly the low temperature ductility.

Experimental Procedures

Material Processing: Batches of molybdenum - 14 wt.% rhenium - 1 wt.% La_2O_3 (ODS Mo-14Re) and a molybdenum - 7 wt.% rhenium - 1 wt.% La_2O_3 (ODS Mo-7Re) composition were prepared using a proprietary powder metallurgy procedure. Each new composition was prepared by adding the desired amount of rhenium to ODS Mo powder, compacting the powder mixture, densifying the compacted billet, and then thermomechanically processing the billet into the final product. The procedures are summarized in Table 1 and discussed below.

The desired amount of rhenium powder, with an average particle size of 5 μm, was added to molybdenum alloy powder containing one weight percent lanthana particles and mixed (ball milled) thoroughly for at least eighteen hours. The mixed alloy powder was cold isostatically pressed (CIP) into a 38 mm (1.5 inch) diameter cylindrical compact using 207 - 276 MPa (30 - 40 ksi) pressure for at least fifteen minutes, cleaned in flowing dry (dew point < -100°C or -148°F) hydrogen at 1100°C (2012°F) for at least four hours, and sintered in dynamic vacuum (< 10^{-5} Torr) at 1950°C (3542°F) for at least four hours. Each sintered billet was placed into an unalloyed molybdenum extrusion canister and sealed by tack welding. Each clad billet was preheated in flowing dry hydrogen at 1350°C (2462°F) for at least thirty minutes and then dynamically compacted at a 6:1 reduction ratio using a high-rate extrusion press. Finally, each extruded billet was preheated in flowing dry hydrogen and hand swaged to a 5 mm (0.200 inch) diameter with the molybdenum cladding in place. The cladding was removed by centerless grinding, producing an alloy rod with a 4.6 mm (0.180 inch) final diameter. The total reduction in area as a result of the extrusion and swaging steps was greater than 98%.

Tensile and creep rupture specimens were machined from the 4.6 mm (0.180 inch) diameter rod. Each specimen was electropolished in a room temperature solution, containing four parts concentrated reagent grade sulfuric acid and one part distilled and/or deionized water, using a 0.25 mm (0.010 inch) thick Type 304 stainless steel cathode and a direct current accelerating potential of 6 - 7 volts to remove 5 - 8 μm (0.002-0.003 inches) from the gauge section diameter.

Material Characterization: Samples were removed from each end of the extruded and swaged rod for light optical metallographic examination and Vickers hardness measurements. A longitudinal and transverse orientation 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 µm alumina powder. The mounts were swab etched with Murakami's etchant (10 g potassium ferricyanide, 10 g potassium hydroxide, and 100 ml distilled water). The average grain size of the final rod product was measured 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. The average of at least five Vickers hardness (VHN) indents along the longitudinal section of each metallographically prepared specimen was determined using an automated hardness tester, Wilson Tukon Series 200. A diamond indenter with a five kilogram load and a ten second dwell time was used.

Mechanical Properties: The following mechanical properties were measured for the ODS molybdenum - rhenium alloys: (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 swaged and recrystallized (one hour vacuum anneal at 1600°C or 2912°F) alloys were measured by uniaxially loading a round bar tensile specimen to failure at temperatures ranging from +200 to -196°C (+392 to -321°F) per ASTM Standards E8 and E21 and a strain rate of 0.05 min^{-1} or 0.00083 sec^{-1}. The surface of each fractured tensile specimen was analyzed with a scanning electron microscope (SEM), to identify the mode of fracture; i.e., ductile or dimpled, cleavage, intergranular, or mixed-mode. The DBTT was identified as the lowest test temperature at which no evidence of brittle cleavage fracture was observed. The elevated temperature tensile properties of the swaged and recrystallized (one hour vacuum anneal at 1600°C or 2912°F) alloys were measured by uniaxially loading a round bar tensile specimen to failure in dynamic vacuum ($< 5 \times 10^{-5}$ Torr) at temperatures ranging from 1000 to 1600°C (1832 to 2912°F) and a strain rate of 0.05 min^{-1} or 0.00083 sec^{-1}.

The creep behavior of this new alloy was also evaluated. Constant load uniaxial creep tests were performed at 1000°C (1832°F) and at 1600°C (2912°F) in dynamic vacuum ($< 10^{-7}$ Torr). A round bar creep rupture specimen in the swaged condition was pin loaded in the creep test vacuum chamber. The entire system including the test specimen was sealed, baked-out for fourteen hours under dynamic vacuum at 225°C (437°F) to remove all adsorbed moisture and gases from all internal surfaces, and subsequently leak rate checked. The system including 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 between each shoulder was made optically off the shoulder 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 specimen. The fracture surface of the specimen was again analyzed with the SEM to identify the type of fracture.

Results and Discussion

Material Characterization: The chemical composition of each alloy powder mixture was measured using both inert gas fusion and plasma spectrometry techniques. The results for the starting ODS Mo and each ODS Mo-Re alloy are presented in Table 2. Representative light optical photomicrographs of the swaged ODS Mo-Re alloys containing one weight percent of lanthana particles are presented in Figure 3. The microstructural properties of the ODS Mo-Re alloys are listed in Table 3 and compared with typical characteristics of PM-Mo, a Mo-14Re (solid-solution) alloy, and ODS Mo.[2,11] The microstructure of the new alloys in the as-swaged condition are characterized by elongated grains with average widths of 8 µm (0.3 mils) and 5 µm (0.2 mils) for the ODS Mo-7Re and ODS Mo-14Re alloys, respectively. The average aspect ratio (average grain length/width) for the as-swaged alloys was 10 and 5, respectively. Both agglomerates and discrete oxide particles were observed in the microstructures of the two new alloys. The oxide particle sizes were 1-2 µm (0.04-0.8 mils) and 3-5 µm (0.12-0.2 mils) for the ODS Mo-7Re and the ODS Mo-14Re alloys, respectively. The occurrence of agglomerates and coarser oxide particles may have contributed to the larger grain size in the ODS Mo-Re alloys relative to the base ODS Mo.

Low Temperature Mechanical Behavior: Results for room temperature yield and ultimate tensile stress, percent elongation to failure, and reduction in area are listed in Table 4. The effect of temperature on the tensile reduction in area for molybdenum, Mo-14Re, and ODS Mo-14Re are presented in Figure 4 for material tested in the worked and stress-relieved condition and in Figure 5 for material in the recrystallized condition. The ODS Mo-14Re alloy exhibits significantly better ductility than the Mo-14Re binary alloy both in the worked and stress relieved condition as well as in the recrystallized condition. The effect of grain size on the DBTT of PM-Mo and binary Mo-Re alloys is shown in Figure 6. For a given grain size, the addition of a modest amount of rhenium has resulted in a significant decrease in the DBTT of the ODS Mo.

The effect of temperature on yield strength of the ODS Mo-Re alloy is also very interesting. As shown in Figure 7, the most significant impact of the dilute rhenium additions is on the temperature dependence of the flow stress. For body centered cubic metals at temperatures below $0.2T_m$ the flow stress is strongly temperature dependent and is a characteristic that has been well studied.[16] For molybdenum, $0.2T_m$ is 305°C (581°F). As test temperatures are lowered, the yield strength of molybdenum increases as the DBTT is approached, the rate of change increases until the yield strength exceeds the fracture strength and failure is characterized by brittle cleavage type fractures. From Figure 7, the slope of the yield strength curve for unalloyed molybdenum and ODS Mo are similar even though the ODS Mo has a higher yield strength. The addition of 14 wt.% rhenium to molybdenum decreases the temperature at which the yield strength increases to approximately -20°C (0.09 T_m) below which temperature the rate of yield strength increase is similar to unalloyed molybdenum. The change in slope below -80°C (-112°F) is a result of the fracture going from predominantly ductile to predominantly brittle in nature. The combination of the 14 wt.% rhenium with the oxide particle dispersion lowers the transition temperature, where the flow stress becomes temperature dependent, to -150°C (-238°F or 0.04 T_m). The reason for this behavior has not been determined but may be related to a change in the Peierls' Stress.

Elevated Temperature Mechanical Behavior: The yield and ultimate tensile stress, percent elongation to failure, and the reduction in area were determined over the range from 1000 to 1600°C (1832 to 2912°F). A graphic comparison of the ultimate tensile stress of the ODS Mo-14Re alloy with unalloyed molybdenum, a Mo-14 Re alloy, and ODS Mo is presented in Figure 8. The ODS Mo-14Re alloy exhibits the highest UTS of the four between 1000 and 1250°C (1832 - 2282°F). On the other hand, ODS Mo, containing a more homogeneous oxide distribution and finer oxide particle size, exhibits the highest UTS above 1250°C (2282°F).

An ODS Mo-14Re specimen tested in creep rupture at a test temperature of 1000°C (1832°F), and an applied stress of 207 MPa (30 ksi) ruptured in 140 hours. When compared with the creep rupture behavior of unalloyed arc-cast molybdenum and the Mo-14Re binary alloy, Figure 9, the ODS Mo-14Re is significantly stronger. For example, at 1000°C (1832°F) and 83 MPa (12 ksi), unalloyed molybdenum ruptures in 100 hours. Also on this plot is data for ODS Mo tested at 1800°C (3272°F) which again illustrates the benefit of the rare-earth inert oxide particle strengthening.

Summary

The addition of small amounts of rhenium, at the 14 wt.% level, results in further improvement in the low temperature ductility of ODS Mo. This effect has been referred to as "Alloy Softening". However, this designation does not appear correct and a more accurate description of the changes in ductility at dilute additions of rhenium is an affect on the temperature dependent component of the flow stress. Dilute additions of rhenium in combination with oxide dispersion strengthening would appear to enhance this behavior.

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Table 1. ODS Molybdenum - Rhenium Alloy Processing Schedule

Process Step	**Starting and Finishing Diameter, mm (inch)**	**Reduction in Area (%)**	**Process Temperature, °C (°F)**
Cold Isostatic Press	50 to 43 (2 to 1.7)	28	20 (68)
Hydrogen Clean	43 to 42 (1.7 to 1.65)	6	1100 (2012)
Vacuum Sinter	42 to 38 (1.65 to 1.5)	17	1950 (3542)
Extrude	38 to 16 (1.5 to 0.612)	83	1350 (2462)
Hand Swage	16 to 10 (0.612 to 0.403)	57*	1200 (2192)
	10 to 8 (0.403 to 0.302)	44*	1000 (1832)
	8 to 5 (0.302 to 0.188)	61*	800 (1472)
Declad and Trim	4.5 (0.180)	n/a	21 (70)

* A result of several passes with no one pass > 15% reduction in area.

Table 2. Results of Chemical Analysis

Lot ID	**Average Alloy Composition (weight percent)**					
	Mo	Re	La‡	O‡	C	Other
2LW-901551*	Balance	-	0.73	0.36	0.009	0.08 W
TEX-337	Balance	14.7	0.76	0.35	0.015	0.05 W
TEX-360	Balance	14.9	0.70	0.33	0.018	0.07 W
TEX-362	Balance	7.4	0.67	0.36	0.016	0.05 W

* Results for ODS molybdenum alloy powder lot used as starting material.
‡ Equivalent to one weight percent (2 volume percent) La_2O_3 in alloy.

Table 3. Microstructural properties of the as-swaged ODS Molybdenum - Rhenium Alloys, Unalloyed Molybdenum, Binary Molybdenum - 14 wt.% Rhenium, and ODS Molybdenum.

Alloy	Average Grain Width, µm (mils)	Average Oxide Particle Size, µm (mils)	Average Vickers Hardness, kg/mm^2
ODS Mo-7 Re	7.6 (0.3)	1.5 (0.06)	260
ODS Mo-14 Re	5.1 (0.2)	4.1 (0.16)	305
PM-Mo [2]	15.2 (0.6)	n/a	225
Mo-14 Re [11]	1.0 (0.04)	n/a	300
ODS Mo [2]	0.5 (0.02)	0.25 (0.01)	262

PM = Powder metallurgy unalloyed molybdenum

Table 4. Room Temperature Tensile Properties of the ODS Molybdenum - Rhenium Alloys, Unalloyed Molybdenum, Binary Molybdenum - 14 wt.% Rhenium, and ODS Molybdenum.

Alloy	0.2% Offset Yield Stress MPa (ksi)	Ultimate Tensile Stress MPa (ksi)	Elongation to Failure (%)	Reduction in Area (%)	DBTT °C (°F)
Swaged Alloys (> 90% reduction in area)					
ODS Mo-7 Re	593 (86)	889 (129)	13	42	-151 (-240)
ODS Mo-14 Re	827 (120)	1075 (156)	12	40	-195 (-320)
PM-Mo [2]	538 (78)	620 (90)	15	72	-23 (-10)
Mo-14 Re [11]	848 (123)	896 (130)	13	50	-73 (-100)
ODS Mo [2]	745 (108)	827 (120)	13	59	-60 (-76)
Recrystallized Alloys					
ODS Mo-7 Re	255 (37)	586 (85)	30	30	24 (75)
ODS Mo-14 Re	345 (50)	724 (105)	27	38	-50 (-58)
PM-Mo [2]	296 (43)	483 (70)	5	14	49 (120)
Mo-14 Re [11]	317 (46)	531 (77)	38	49	-18 (0)
ODS Mo [2]	365 (53)	524 (76)	29	65	24 (75)

PM = Powder metallurgy unalloyed molybdenum

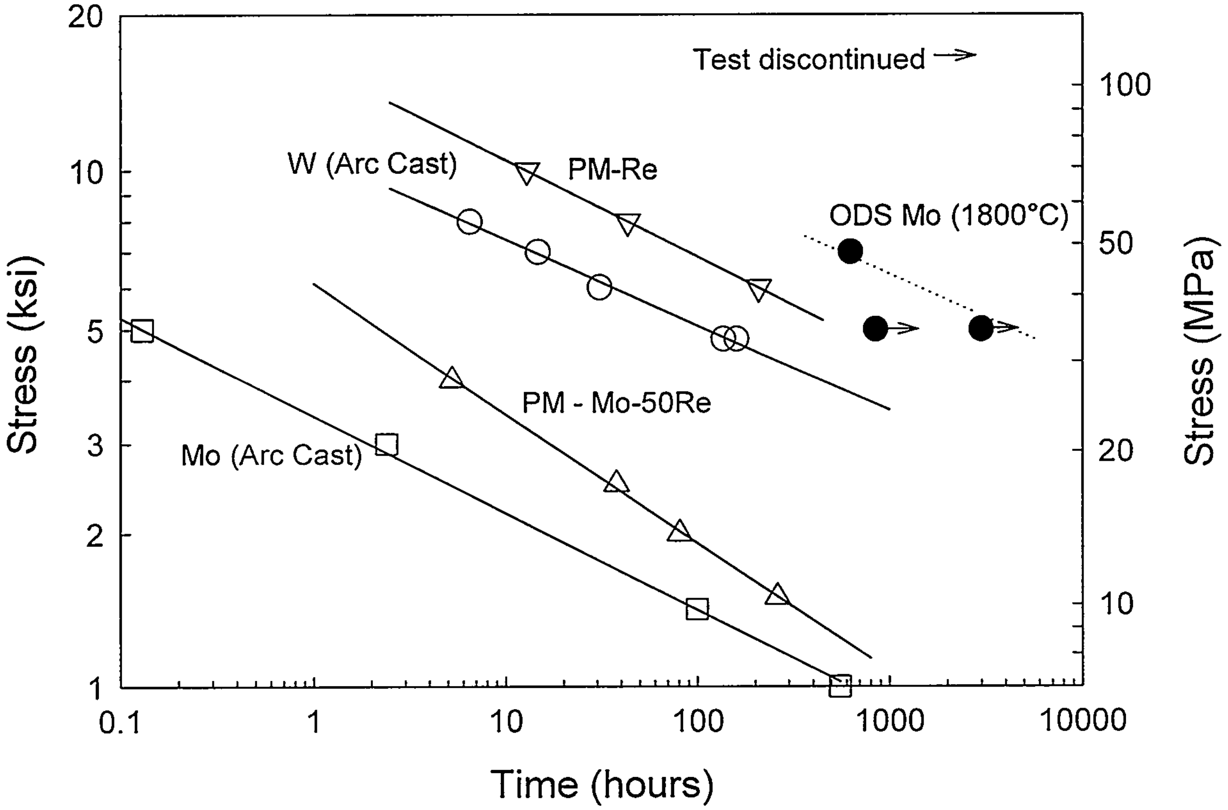


Figure 1. Creep-rupture of unalloyed molybdenum and molybdenum alloys at 1600°C (2912°F). [2,3]

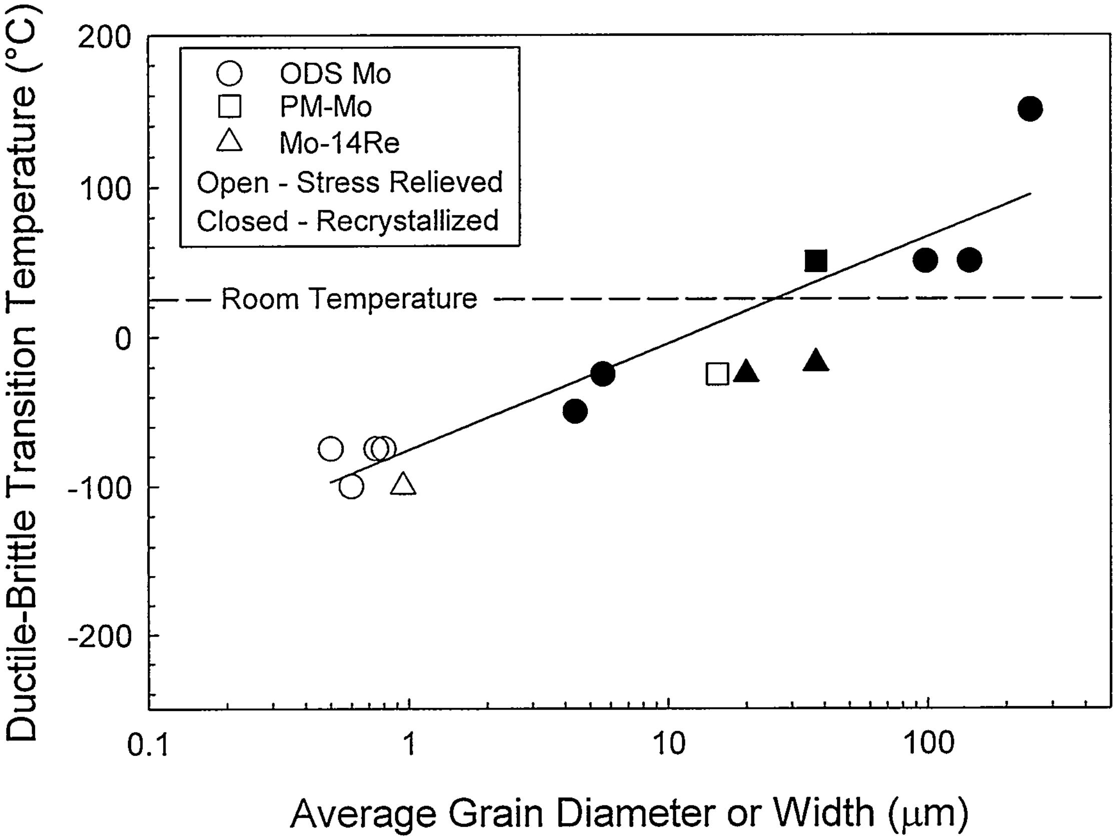
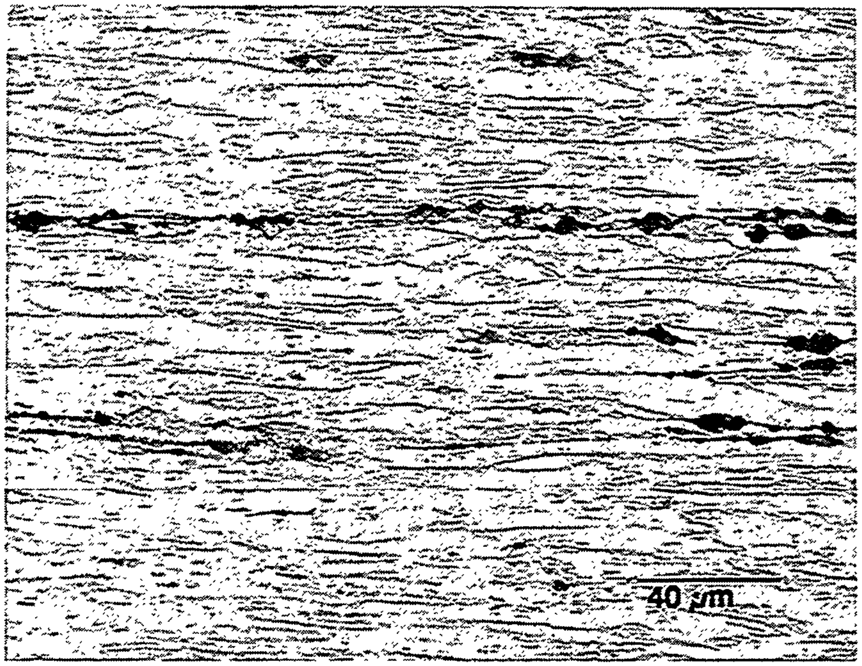
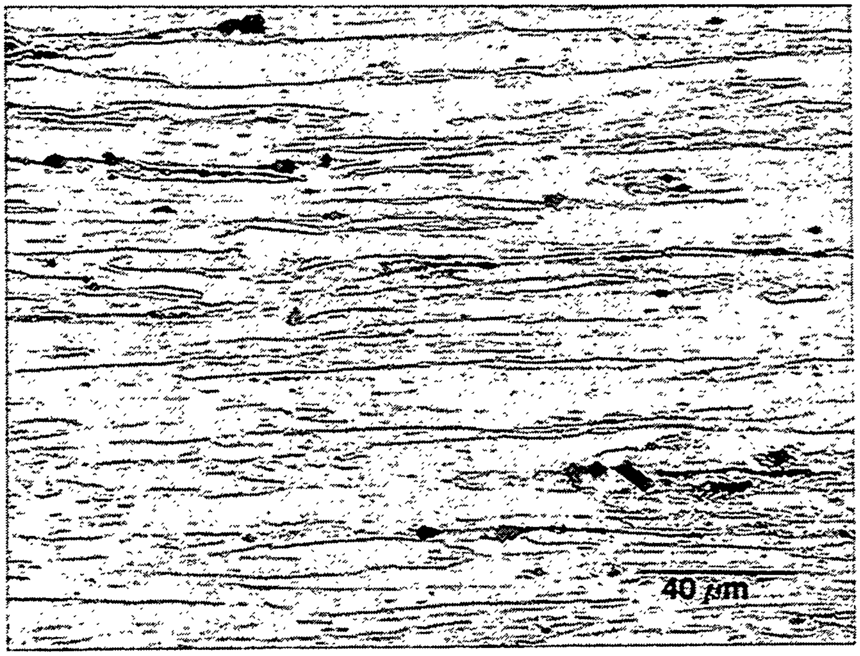


Figure 2. Effect of grain size on the ductile-to-brittle transition temperature (DBTT) of molybdenum alloys. [2,11,15]

Longitudinal Orientation (0.180 inch diameter rod)



A. ODS Mo - 14 wt.% Re Alloy



B. ODS Mo - 7 wt.% Re Alloy

Figure 3. Light optical photomicrographs of the swaged oxide dispersion strengthened (ODS) molybdenum - rhenium alloys. (500x magnification)

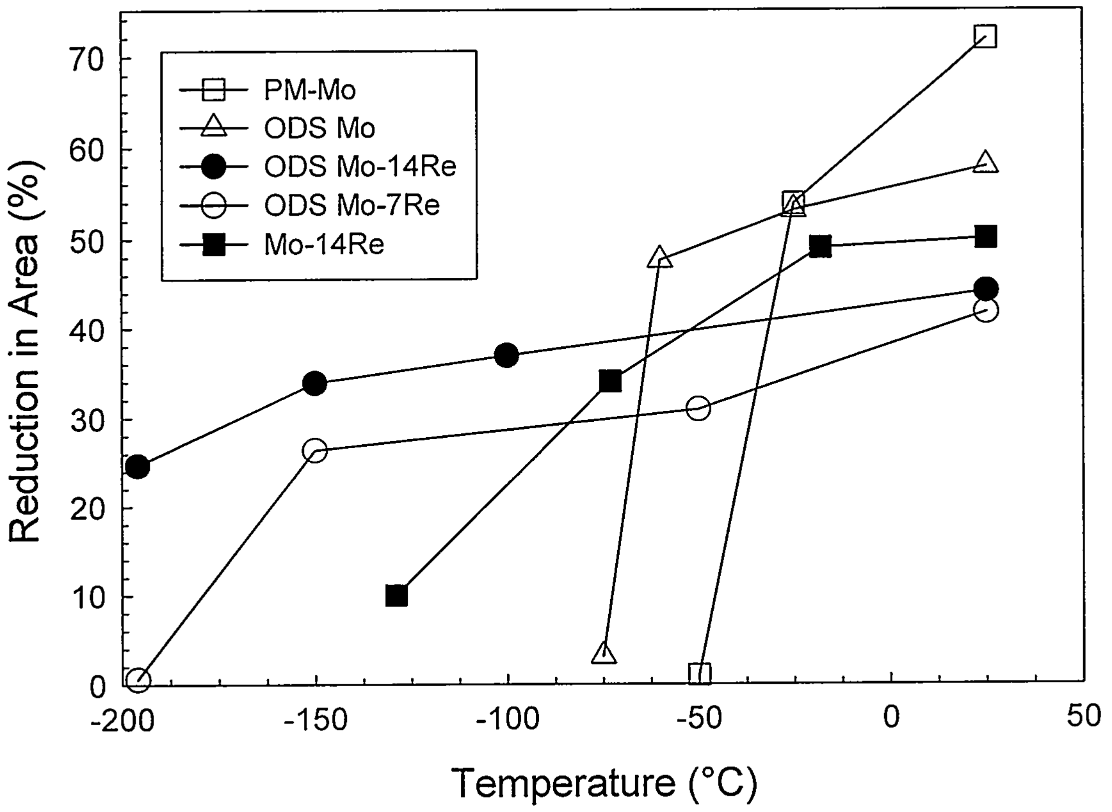


Figure 4. Effect of temperature on the ductility of molybdenum and molybdenum - rhenium alloys tested in the swaged and stress-relieved condition. [2,11]

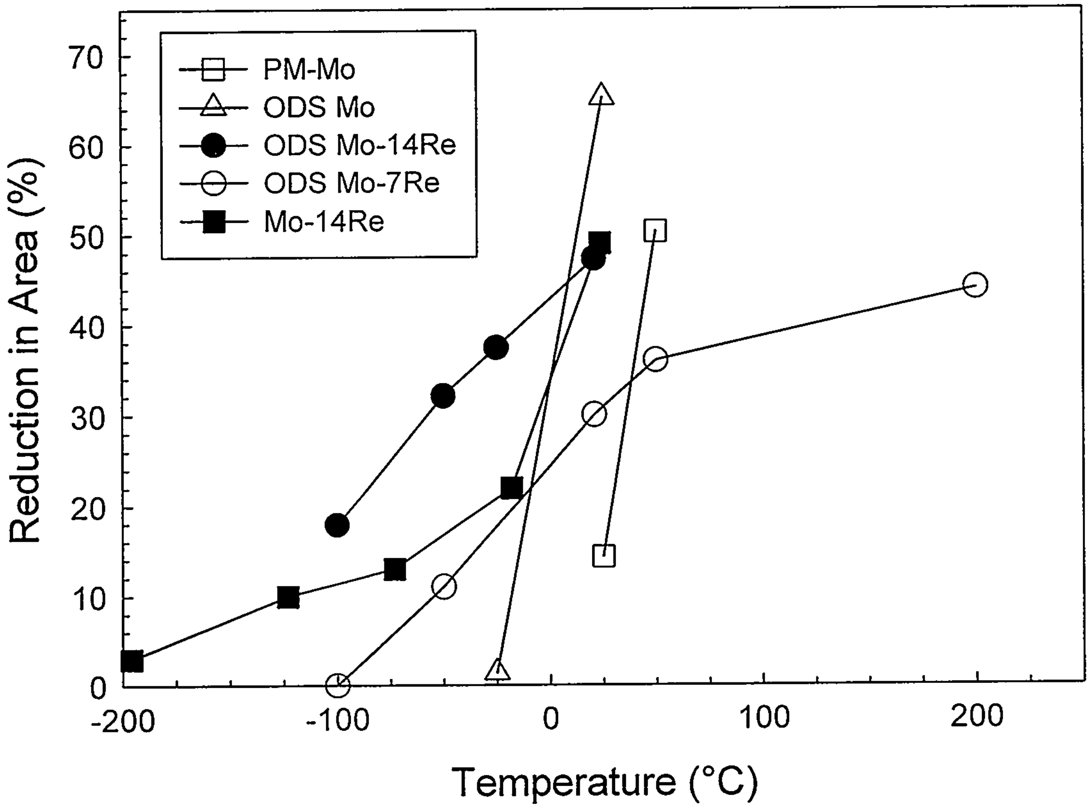


Figure 5. Effect of temperature on the ductility of molybdenum and molybdenum - rhenium alloys tested in the recrystallized condition. [2,11]

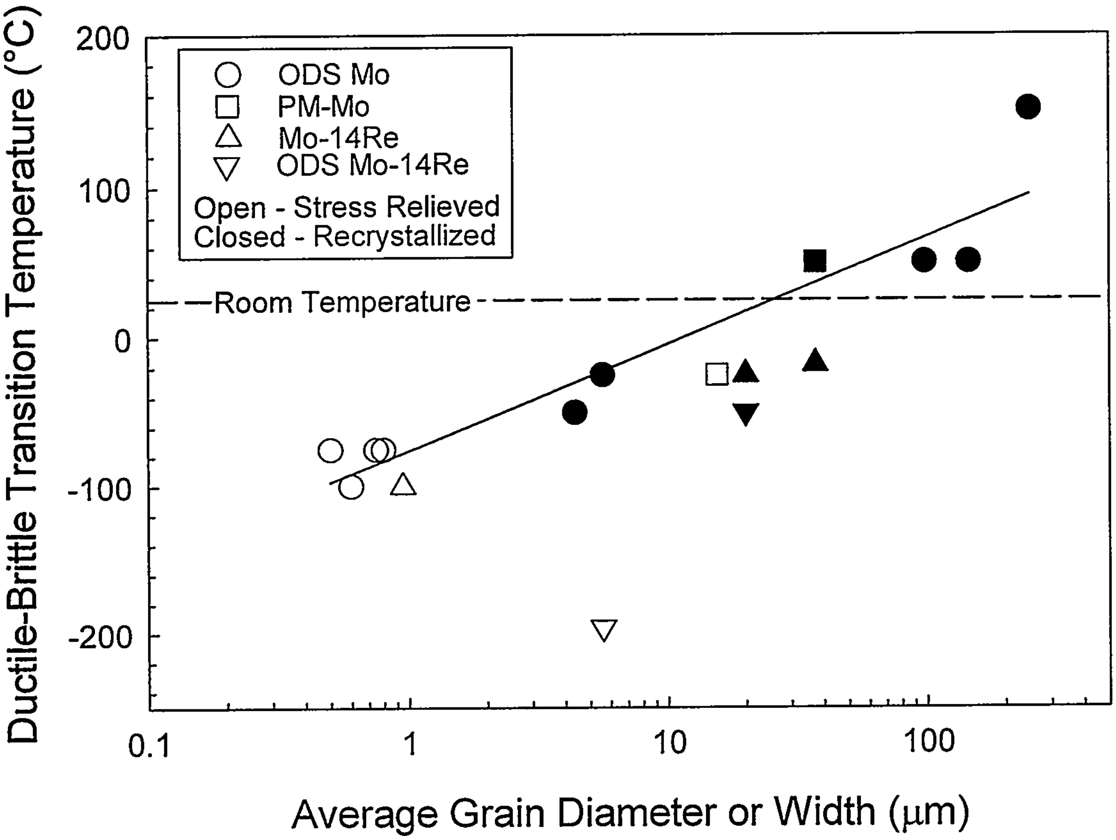


Figure 6. Effect of grain size on the ductile-to-brittle transition temperature (DBTT) of molybdenum alloys including the ODS Mo-14Re Alloys. [2,11,15]

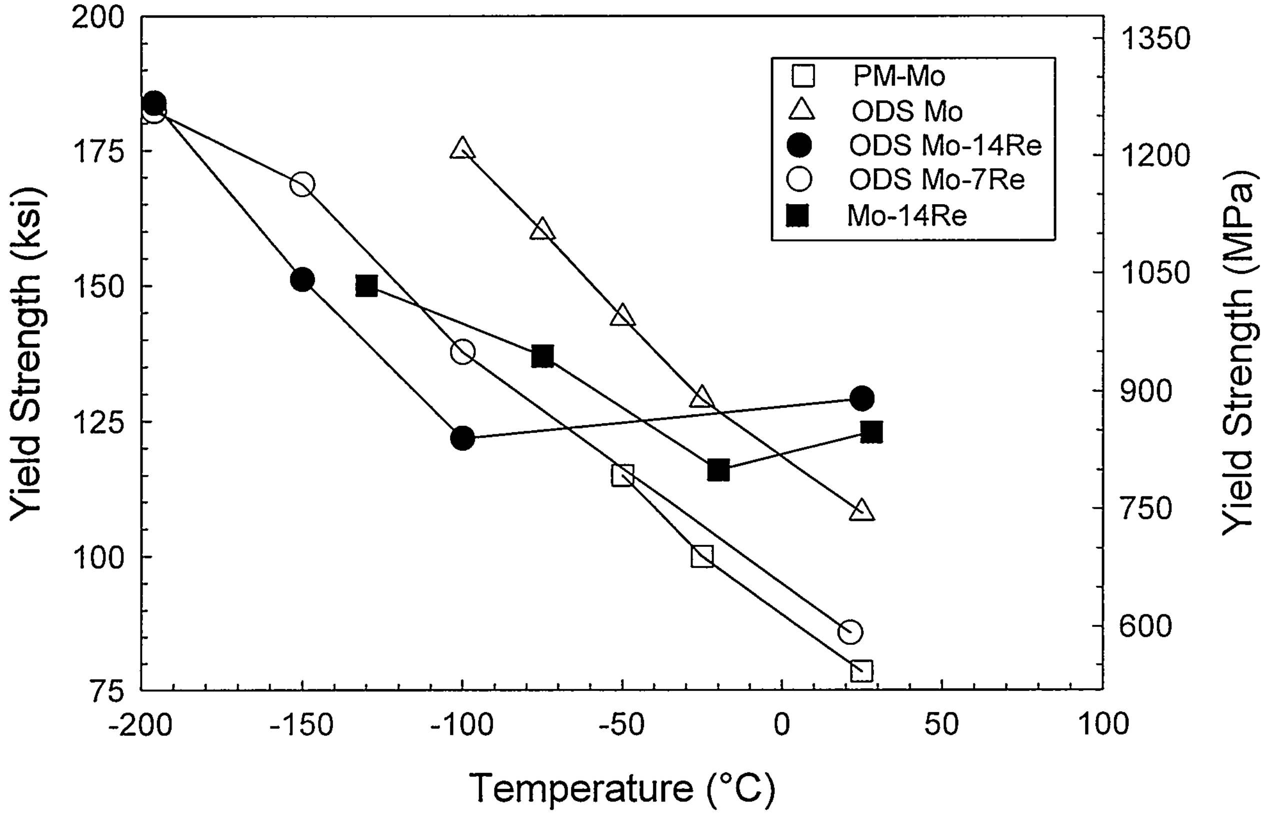


Figure 7. Effect of rhenium on the temperature dependence of yield strength for molybdenum and molybdenum alloys tested in the swaged and stress-relieved condition. [2,11]

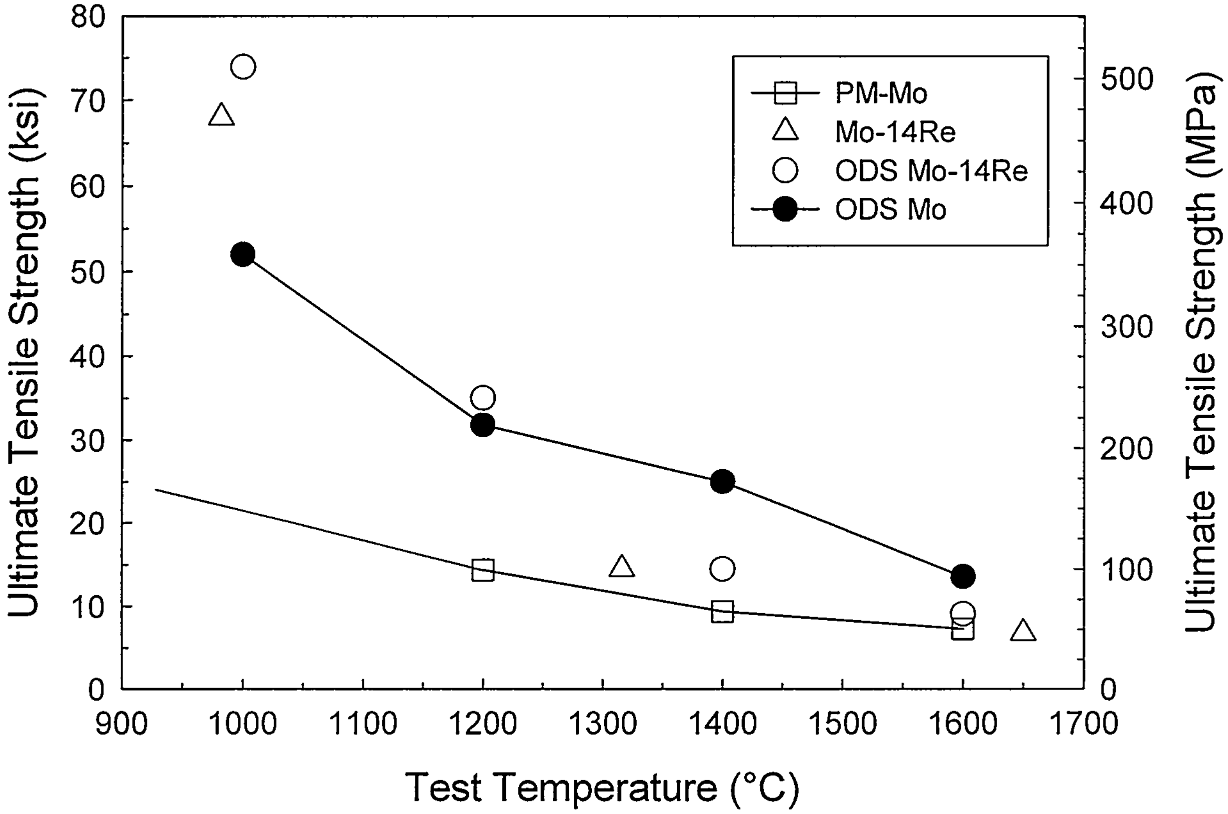


Figure 8. Effect of temperature on the tensile strength of molybdenum and molybdenum - rhenium alloys tested in the swaged and stress-relieved condition. [2,11]

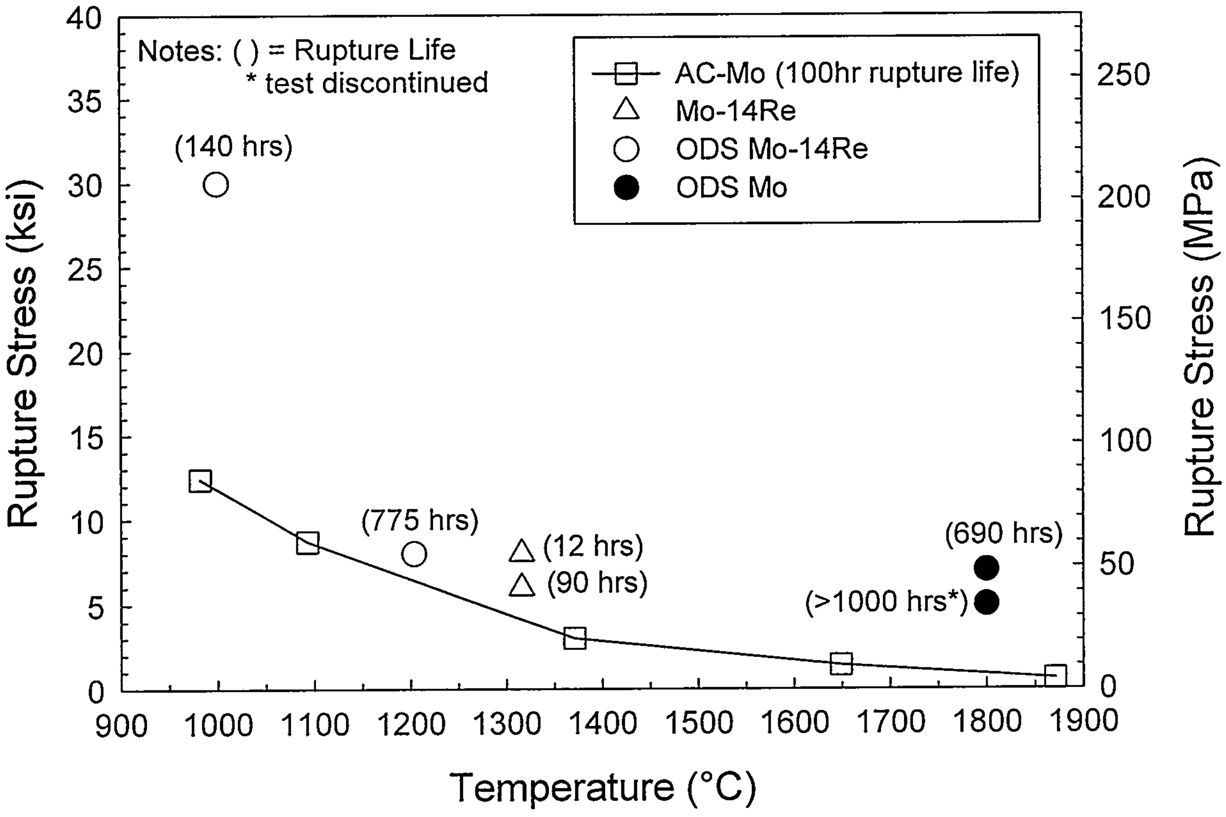


Figure 9. Comparative creep-rupture properties of molybdenum and molybdenum alloys. [3,11]