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Uniaxial, load-controlled cyclic deformation of recrystallized molybdenum sheet

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ABSTRACT

Monotonic tensile tests and uniaxial load-controlled tension–tension cyclic tests were performed on commercial purity (99.99%) molybdenum in the recrystallized state in the temperature interval spanning RT (25 °C) to 500 °C range. Cyclic deformation behavior was characterized by analyzing the ratcheting strain, cyclic hardening response, and fatigue life. The response of unrecrystallized and recrystallized Mo was compared at room temperature; the influence of test variables including mean stress, stress amplitude and *R* ratio on ratcheting strain in recrystallized Mo at room temperature was also examined. The magnitude and rate of ratcheting strain evolution with cycles appear to depend on both the mean stress and the stress amplitude. Microstructural evolution during cyclic loading was characterized by conducting interrupted tests at room temperature as well as at 400 °C. Observations confirm dislocations cell structure formation during cyclic plastic deformation at room temperature and at 400 °C, the process occurring more rapidly at the higher temperature; in addition, at the higher temperature some of the cell walls appeared to have transformed into subgrain boundaries, suggesting partial recovery.

1. Introduction

Refractory alloys including Mo, Mo solid solution alloys and Mo–Si–B multiphase alloys are of research interest to the aerospace and aircraft engine communities as they are considered potential candidates for high-temperature applications beyond the capabilities of Ni-based superalloys [1–6]. Such alloys have been characterized for their monotonic tensile properties in tension and compression as well for their creep resistance [7–11]. Likewise multiphase Mo–Si–B alloys have been studied in terms of monotonic and cyclic crack growth behavior and creep–fatigue interactions have been examined as well [12–15]. Considerably less is known about the cyclic deformation response of Mo and Mo-base alloys and yet, for the applications that these alloys are being examined and developed, this mode of loading is pertinent.

There have been a few studies on the cyclic deformation response of single and polycrystalline Mo; the single crystal was examined in tension–compression till about 130 °C [16,17] and the results were rationalized in terms of dislocation structure evolution and dislocation mobility. The polycrystalline material was also studied in tension–compression at room temperature using stress- and strain-controlled tests [18] to understand the role of discontinuous yielding on cyclic response. More recently, Chen et al. [19] have studied cyclic plasticity in recrystallized Mo at low temperatures where screw dislocation mobility is limited and used stress-controlled tests to differentiate between the microplasticity and macroplasticity regimes and attributed the former to unpinning and reversibility of edge dislocation segments and the latter to contributions from screw dislocations. To the best of our knowledge, there are not studies performed on Mo in the tension–tension mode which is considered a relevant mode for many aerospace components and Mo-based alloys are receiving research attention for hot-component applications. Accumulation of inelastic deformation during cyclic loading (ratcheting strain) can occur under asymmetric stress-loading conditions such as tension–tension loading and can affect fatigue life adversely and this aspect has not been systematically studied for the case of molybdenum. Furthermore, the fatigue response at higher temperatures where dynamic recovery is a possibility has not been studied either. It is the purpose of the work presented here to fill some of these gaps in the literature on Mo and to then extend the research to Mo-based solid solution alloys.

In this paper, we have examined the cyclic deformation response of both, unrecrystallized and recrystallized commercial Mo sheet in tension–tension fatigue in the temperature regime of 27–500 °C. Tests were performed in the stress-controlled mode and ratcheting strain, cyclic hardening response, and fatigue life were documented. Microstructural observations were made using optical microscope, scanning electron microscopy (SEM) and transmission electron microscopy (TEM) and the microstructural findings have

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Table 1
Interstitial content (wt.%) in the as-received Mo sheets.

Mo sheet	1	2	3	4	5	6
Carbon	.014	.012	.015	.012	.023	.010
Nitrogen	<.001	.001	.001	<.001	<.001	.001
Oxygen	<.001	<.001	.003	.001	.002	<.001

been used to interpret the cyclic behavior of Mo as a function of temperature.

2. Experimental procedure

The material used in this study was 99.99% purity commercial molybdenum sheet that was obtained in the unrecrystallized condition from Goodfellow Corporation in Oakdale, PA (USA). The interstitial levels in the sheets were measured and are reported in Table 1. Due to sheet size limitations, multiple sheets were procured and these are labeled 1–6 in Table 1. The C level in these sheets varied from 0.01 wt.% to 0.015 wt.% except in sheet 5 where it was 0.023 wt.%. The N and O levels were uniformly low. Dog bone-shaped flat specimens were electrodischarge machined from these sheets with a gauge length of 18 mm, a gauge width of 1.8 mm, and thickness of 1.5 mm. All specimens were annealed in vacuum for 2 h at 1300 °C resulting in a recrystallized grain structure with an average grain size around 30 μm. Specimens were ground and then electropolished with a solution of sulfuric acid in methanol (125 ml sulfuric acid + 875 ml methanol) to eliminate the surface scratches. Microstructures of the as-received and annealed samples were characterized using optical microscopy. The optical metallography samples were electrolytically etched to reveal grain boundaries using a solution of oxalic acid (10 g oxalic acid + 100 ml water) and by applying 5 V for 1–2 s.

Room temperature mechanical tests were conducted in air as were tests at 100 °C and 200 °C. Tests at higher temperatures (300 °C, 400 °C and 500 °C) were performed in vacuum as Mo is prone to oxidation at these temperatures. Displacement-controlled monotonic tensile tests were first performed at these different temperatures at a nominal strain rate of $10^{-4}\,s^{-1}$ to obtain the stress–strain response as well as to determine the value of the ultimate tensile strength of the material, a parameter used for the cyclic loading. Load-controlled cyclic tests were then performed using asymmetric tension–tension loading and sinusoidal waves with a frequency of 10 Hz. During the test, load and position values were continuously recorded.

Subsequently, the deformed specimens were analyzed by SEM and TEM. Fracture surfaces were examined in the SEM and substructure evolution was studied in the TEM. Thin foil TEM specimens were obtained from the gauge section of the deformed specimens by grinding the section down to 50 μm thickness and then extracting segments using a standard 3-mm diameter punch. These disks (or segments thereof) were then electro-polished to perforation using a Struers Tenupol-5 twin jet polisher and an electrolyte of 12.5% sulfuric acid and 87.5% methanol. Optimal thinning conditions were obtained at a temperature of −25 °C and 20 V.

Table 2
Monotonic tensile properties of the recrystallized Mo sheet.

Temperature (°C)	Yield stress (MPa)	Ultimate tensile stress (MPa)	Plastic strain at failure (%)
23	347	446	55
100	182	375	48
200	101	320	44
300	143	278	43
400	80	243	41
500	75	230	40

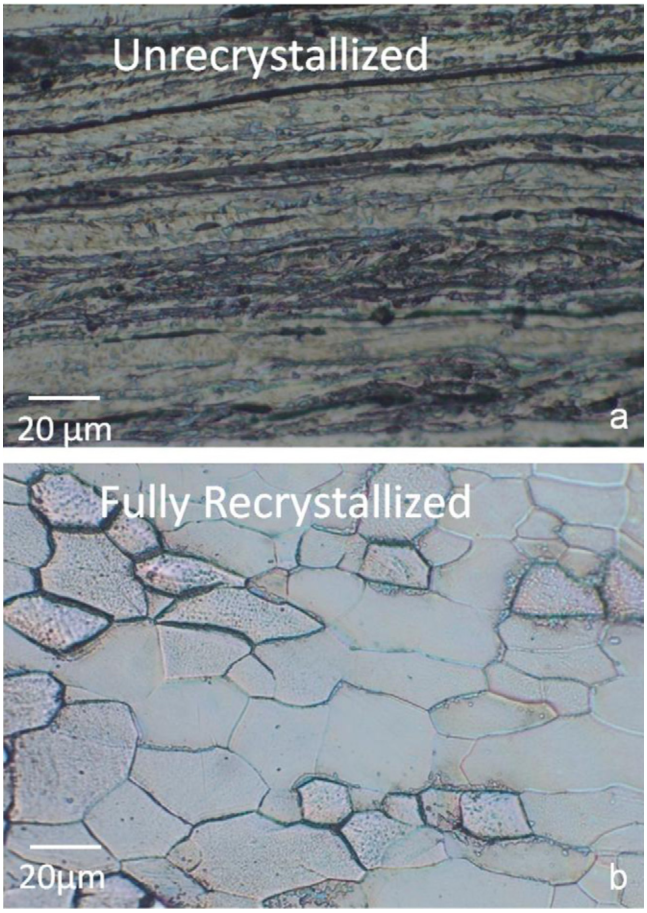

Fig. 1. Representative optical micrographs of the Mo sheet in (a) as-received and (b) annealed conditions.

3. Results

3.1. Mechanical response

A representative optical micrograph of the microstructure of the Mo sheets in the thickness plane in the as-received condition is shown in Fig. 1a and it is evident that the material is in an unrecrystallized condition with the grains being elongated in the rolling direction. After heat treatment at 1300 °C for 2 h, a fully recrystallized microstructure with an average grain size of ∼30 μm is obtained (Fig. 1b). Examination of the texture of the as-received sheet and after annealing using electron backscatter diffraction (EBSD) did not reveal a significant change in texture and is in agreement with previous reports on the evolution of recrystallization texture in Mo [20,21].

The monotonic tensile response of the material in these two conditions is compared in Fig. 2a. In the as-received condition, the material exhibits a high yield strength in the vicinity of 675 MPa, followed by almost immediate softening, and failure eventually occurs with tensile elongation of about 18–20%; this is characteristic of a highly-worked material. In contrast, the recrystallized material (annealed) shows a much lower yield strength of ∼350 MPa, a visible ultimate tensile strength (UTS) of about 450 MPa and an elongation of ∼55%. In both cases, serrated flow is noted, being more prominent in the annealed condition. These serrations are attributed to the presence of interstitials (notably C but also N and O) in the sheet and their interactions with dislocations.

The corresponding cyclic response at room temperature for the as-received (unrecrystallized) and annealed (recrystallized)