Mechanical Properties and Size Effects of Microscale Molybdenum Wires: Postprint

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Date: 2023-03-18T00:00:00+00:00

Abstract

Tensile experiments and stress-controlled tension-tension fatigue experiments were performed on molybdenum wires (model material) with diameters of 125 mm, 140 mm, and 160 mm to investigate the mechanical properties and size effects of micron-scale body-centered cubic structured molybdenum wires. The tensile and fatigue fracture surfaces of the molybdenum wires were observed using scanning electron microscopy, and the tensile and fatigue fracture behaviors of metallic molybdenum wires were analyzed. The results indicate that both the tensile fracture strength and the fatigue strength under stress control of micron-scale body-centered cubic drawn molybdenum wires decrease with decreasing wire diameter. The size effect on the fatigue performance of micron-scale molybdenum wires originates from the different numbers of elongated grains in the radial direction of the wires. Consequently, the smaller the wire diameter, the more sensitive the molybdenum wire is to fatigue cracks or defects initiated on the surface, and the shorter the fatigue propagation life.

Full Text

Preamble

Vol. 29 No. 7
CHINESE JOURNAL OF MATERIALS RESEARCH
July 2015

Size Effect on Mechanical Property for Cold-Drawn Micron-Sized Molybdenum Wires

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Supported by the National Natural Science Foundation of China (Nos. 51171045 & 51371047) and the Fundamental Research Funds for the Central Universities (No. N130810003).

Manuscript received February 26, 2015; in revised form April 1, 2015.

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Abstract

Cold-drawn molybdenum wires with diameters of 125, 140, and 160 µm were selected as model materials to investigate the mechanical performance and size effects of micron-scale body-centered cubic (BCC) Mo wires through tensile tests and tension-tension fatigue tests under stress control. The fracture surfaces were examined by scanning electron microscopy to analyze the tensile and fatigue fracture behavior. The results show that both the tensile fracture strength and fatigue strength under stress control decrease with decreasing wire diameter. The size effect on fatigue performance originates from the different number of elongated grains along the radial direction, making thinner wires more sensitive to fatigue cracks or defects nucleated on the surface, thereby resulting in shorter fatigue crack propagation life.

KEY WORDS metallic materials, molybdenum wires, tension properties, fatigue fracture, size effect

Introduction

Molybdenum is a refractory metal with a melting point of 2610°C and maintains a body-centered cubic (BCC) crystal structure from room temperature to its melting point. Due to its high thermal stability, creep resistance, and thermal conductivity, Mo and its alloys are used in missile components, engines, and nuclear reactors. Molybdenum also possesses a high elastic modulus and a coefficient of linear expansion approximately one-third to one-half that of conventional steel. It exhibits excellent corrosion resistance as it does not react with acidic or alkaline solutions at room temperature. Additionally, with its relatively low density, Mo offers a high specific strength (strength-to-density ratio). Mo wires remain stable in molten glass and have a thermal expansion coefficient nearly identical to that of glass, making them suitable as reinforcement fibers for bulk metallic glass composites [1]. As a reinforcement fiber material, the mechanical behavior of Mo wires under dynamic and static loading is of great interest, yet few reports have documented the mechanical properties of pure Mo wires. Therefore, investigating the mechanical properties of micron-scale Mo wires holds significant theoretical and practical value.

Extensive research has been conducted on the tensile and fatigue properties of micron-scale face-centered cubic (FCC) metals [2-10]. When the geometric scale (wire diameter or foil thickness) ranges from several tens to hundreds of micrometers, tensile strength decreases with decreasing scale. The size effect on fatigue performance depends on the loading mode. Dai et al. [9] investigated size effects on tensile and fatigue properties of rolled and annealed micron-scale copper foils, finding that tensile strength decreased with foil thickness, and fatigue strength obtained under constant total strain control via dynamic bending also decreased with thickness. More recently, Xu et al. [11] studied the fatigue behavior of micron-scale FCC copper single crystals and observed size effects on fatigue performance. Despite these comprehensive studies on FCC metals, research on the mechanical properties of micron-scale BCC metals, particularly fatigue behavior, remains scarce, and the underlying mechanisms of size effects are not well understood. This paper selects cold-drawn micron-scale BCC Mo wires as the research object to investigate the tensile and fatigue properties of wires with different diameters and to explore the size effects on mechanical performance.

Experimental Methods

The cold-drawn pure Mo wires used in this study had diameters of 125, 140, and 160 μm, with a chemical composition (mass fraction, %) of C 0.025, N 0.005, Ti 0.16, Ni 0.02, Cr 0.014, and Mo 99.77%. Prior to testing, the Mo wire samples were ultrasonically cleaned with ether and absolute ethanol, then dried. Both tensile and fatigue samples were cut to a total length of 30 mm with a gauge length of 10 mm.

Mechanical testing was performed on an MTS 858 micro-force testing machine. Tensile tests were conducted at a strain rate of 1\$×10^{-3}\$ s^{-1}. Fatigue tests employed tension-tension loading at a frequency of 20 Hz with a sinusoidal waveform and a stress ratio of 0.1. Electron backscatter diffraction (EBSD) was used to characterize grain size, orientation, and boundary features. Scanning electron microscopy (SEM) was employed to observe tensile and fatigue fracture surfaces. For EBSD characterization, Mo wires were electropolished to achieve a smooth surface using an electrolyte consisting of sulfuric acid and absolute ethanol at a volume ratio of 1:3, with polishing conducted at 6 V and -30°C.

2.1 Microstructural Observation of Mo Wires

[Figure 1: see original paper] presents EBSD characterization of the microstructure of cold-drawn Mo wire with a diameter of 160 μm. Figure 1a shows grain orientation maps along the drawing direction (RD), transverse direction (TD), and normal direction (ND), with corresponding inverse pole figures (IPFs) shown in Figure 1b. Grain boundaries with misorientation angles above 5° were statistically analyzed. The results reveal that grains are significantly elongated along the drawing direction, forming a fibrous grain structure. The RD grain map in Figure 1a shows similar colors along the elongated direction, indicating consistent grain orientation and a pronounced texture. The RD IPF in Figure 1b confirms a strong <110> texture along the wire drawing direction. In contrast, the ND and TD grain maps show relatively random orientations, with corresponding IPFs indicating distributions near <111>. Statistical analysis of these elongated grains yields an average grain width of 289 nm, characteristic of submicron grains (Figure 1c). Figure 1d shows the grain boundary misorientation distribution, revealing that most boundaries between elongated grains have random misorientation angles, though some exhibit high-angle boundaries.

2.2 Tensile Properties of Micron-Scale Mo Wires

Figure 2a [Figure 2: see original paper] shows the engineering stress-strain curves for Mo wires of different diameters, while Figure 2b illustrates the relationship between tensile fracture strength and wire diameter. Although tensile strength decreases slightly with decreasing diameter, the overall size effect on tensile strength is not particularly pronounced. This trend resembles the size effect observed in tensile properties of micron-scale FCC copper foils by Dai et al. [9,10]. Furthermore, all wire diameters exhibit fracture soon after plastic yielding, indicating poor ductility.

Figure 3 [Figure 3: see original paper] shows SEM images of the tensile fracture surface of a 125 µm diameter Mo wire; other diameters exhibit similar fracture features. Low- and high-magnification images are presented in Figures 3a and 3b, respectively. The micrographs reveal slight necking after fracture, consistent with the stress-strain curves. When tensile stress is applied to the drawn Mo wire, the longitudinally aligned fibrous grains undergo limited plastic deformation. However, since these grains have already been deformed during drawing, their capacity for further plastic deformation is limited. The combined effects of incompatible deformation among elongated grains and stress concentration lead to grain fracture, with secondary cracks appearing on the fracture surface. The fibrous bundle morphology visible on crack surfaces likely results from intergranular cracking along elongated grain boundaries, as indicated by arrows in Figure 3b.

The Mo wires used in this study have high purity (99.77% Mo), so impurities have minimal effect on brittleness. Molybdenum has a BCC crystal structure, and the influence of lattice type on ductility primarily depends on the number of available slip systems. Although BCC metals have fewer slip systems, they exhibit relatively good isotropy. The BCC lattice is more open than the FCC lattice, and for transition metals like Mo, the asymmetric distribution of d-electrons creates electron vacancies, imparting directional atomic bonding and greater resistance to dislocation motion. Consequently, BCC metals are more brittle than FCC metals. Additionally, C and N atoms present in the Mo wire readily occupy interstitial sites in the BCC lattice, causing asymmetric lattice distortion and increasing brittleness. Moreover, the polycrystalline nature of these Mo wires results in poorer ductility compared to single-crystal Mo, as grain boundary strength is insufficient, leading to brittle intergranular fracture.

2.3 Fatigue Properties of Mo Wires

Tension-tension fatigue tests were conducted on cold-drawn Mo wires with diameters of 125, 140, and 160 μm at a loading frequency of 20 Hz and stress ratio of 0.1. The resulting stress amplitude-fatigue life (S-N) curves are shown in Figure 4 [Figure 4: see original paper]. The fatigue performance exhibits a clear size effect: as wire diameter decreases, the fatigue limit gradually decreases, and at the same fatigue life, thinner wires show lower fatigue strength.

Figure 5 [Figure 5: see original paper] presents SEM images of fatigue fracture surfaces for a 125 μm diameter Mo wire after 2.88\$×10$^{\{3\}, 3.39\times 10}$\{4\}, *and*9.44×10^\{5\}\$ cycles (Figures 5a, 5c, and 5e, respectively), with corresponding high-magnification views in Figures 5b, 5d, and 5f. Low-magnification images show that macroscopically, fatigue fracture surfaces are similar to tensile fracture surfaces (Figure 3). Fatigue microcracks nucleate at the sample surface (as indicated by left arrow in Figure 5b) and grow under cyclic loading, propagating radially inward until final fracture occurs. Additionally, prominent secondary cracks appear on fatigue fracture surfaces, causing axial splitting of the wire. High-magnification observations (Figures 5b, 5d, and 5f) reveal no fatigue striations; instead, local steps are visible (arrow in Figure 5d), indicating that crack propagation occurs locally along elongated grain boundaries. Therefore, after nucleating at the surface, fatigue cracks propagate radially inward; when encountering resistance from vertical grain boundaries, they redirect and propagate locally along elongated grain boundaries parallel to the wire axis.

For bulk FCC metals (e.g., pure copper) and BCC metals (e.g., pure iron), fatigue damage originates from cyclic strain localization at the surface, forming persistent slip bands (PSBs) with associated extrusions/intrusions [12]. Microscopically, edge dislocation dipoles form wall structures within PSBs, while screw dislocation motion in channels between walls accommodates most plastic strain. Accumulation of cyclic plastic strain eventually leads to microcrack formation at PSB/matrix interfaces [12]. For metal films a few micrometers thick constrained by substrates, when film thickness approaches or falls below the characteristic fatigue microstructure size in bulk materials (~1 μm), the tendency for surface extrusion/intrusion strain localization decreases with thickness, and typical dislocation structures gradually disappear [13], leading to increased fatigue strength with decreasing film thickness [14]. Luo et al. [15] found that 20 nm thick Au films exhibited significantly higher fatigue strength than 900 nm thick submicron-scale Au films; typical fatigue extrusion/intrusion damage was absent in nanoscale Au films, while grain boundary-related cyclic plasticity behaviors such as grain growth and twinning were observed. Zhang et al. [16] studied fatigue behavior of 100 nm thick Cu films in as-deposited and annealed states, finding that annealed films possessed better fatigue strength.

For unconstrained FCC metals with geometric scales of tens or hundreds of micrometers, Hofbeck et al. attributed the absence of fatigue extrusions in fine Cu wires to image forces on dislocations near surfaces and mutual annihilation [2]. Dai et al. [9] investigated fatigue strength of micron-scale FCC copper foils using dynamic bending under constant total strain control, finding that decreasing foil thickness reduced ductility, which caused the observed decrease in fatigue strength. Xu et al. [11,17] studied fatigue properties of micron-scale copper single crystal foils, applying symmetric reverse bending to micron-thick cantilever specimens and simulating dislocation structures using a modified reaction-diffusion model. They found that nominal fatigue life decreased with decreasing beam thickness (from 100 μm to 1 mm) at the same strain amplitude [11,18].

For the cold-drawn BCC Mo wires in this study, both tensile and fatigue strengths decrease with decreasing wire diameter. This size effect may be related to the grain structure. EBSD characterization in Figure 1 shows that Mo wire grains exhibit strong texture along the drawing direction with random boundary misorientations. Since these elongated grains have a width of only 288 nm, dislocation motion is hindered during fatigue loading, making it difficult to form typical dislocation cell structures (usually ~500 nm [16]). The fatigue life is thus governed by crack propagation resistance after surface nucleation. According to the model relating fatigue life to grain size in microscale materials proposed by Dai et al. [9], the higher fatigue strength of 160 μm diameter wire results from a greater number of elongated grains in the radial direction. When fatigue cracks propagate radially inward, they encounter resistance and may redirect to propagate locally along elongated grain boundaries parallel to the wire axis. Therefore, more grains in the radial direction more significantly affect crack propagation, making thinner wires more sensitive to surface cracks or defects.

Conclusions

1. Both the tensile fracture strength and stress-controlled fatigue strength of cold-drawn micron-scale BCC Mo wires decrease with decreasing wire diameter. This size effect originates from the different number of elongated grains in the radial direction, making thinner wires more sensitive to fatigue cracks or defects nucleated at the surface and resulting in shorter fatigue crack propagation life.

2. Fatigue cracks in Mo wires nucleate at the sample surface and form secondary cracks that split the wire axially while the main crack propagates. This behavior is related to the shape of the elongated submicron grains in the wire.

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Note: Figure translations are in progress. See original paper for figures.