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Hot deformation of U-9 wt% Mo

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HIGHLIGHTS

- U-9Mo was deformed from 850 to 1000 °C and 3×10^{-3} to 1 s^{-1}.
- Strain rate sensitivity of 0.33 was observed at 1000 °C–3×10^{-3} s^{-1}.
- At 1000 °C the dominant texture was <111> along the compression axis.

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ABSTRACT

Uranium – 9 wt% molybdenum in the as-extruded condition was deformed in compression in vacuum at temperatures from 850 to 1000 °C and strain rates from 3×10^{-3} to 1 s^{-1}. The strain rate sensitivity (m) was computed and plotted as iso-strain rate sensitivity contour plots. m was around 0.33 at 950–1000 °C at strain rate of 3×10^{-3} s^{-1}. Electron backscatter diffraction showed that at 1000 °C–3×10^{-3} s^{-1} grains refined, fraction of high angle boundaries increased and the average local misorientation reduced, all indicative of the occurrence of dynamic recrystallization. In comparison, at 950 and 900 °C both the fraction of low angle boundaries and local misorientation was higher. At 1000 °C–3×10^{-3} s^{-1} the [111] direction was aligned along the compression axis, whereas at lower temperature of 900 °C and 3×10^{-3} s^{-1} it was the orientations close to [001].

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1. Introduction

The global threat reduction initiative (GTRI) and the Reduced Enrichment for Research and Test Reactor (RERTR) programs require that low enriched uranium (LEU, $^{235}U < 20\%$) replace high enriched uranium (HEU, $^{235}U > 85\%$) as fuel in all research reactors [1–3]. Given the neutronics of research reactors this change-over requires fuel with a high density of uranium (>15 g U cm^{-3}) which can only be achieved if metallic monolithic LEU is used in the fuel. Unalloyed metallic U is orthorhombic and has poor irradiation growth and swelling resistance and hence is not used as fuel [4]. γ miscible alloy systems of U (the bcc phase) are desirable as they have increased high temperature strength, good thermal and irradiation stability and improved corrosion resistance [5]. U-Mo is one such system where the γ phase undergoes a eutectoid reaction to α + δ at 10.5 wt% Mo at 565 °C, where α is orthorhombic U and δ is tetragonal U_2Mo [6]. However, this eutectoid reaction is sluggish and for Mo content between 7 and 12 wt% moderate cooling rates retains γ in a metastable state at room temperature [7]. Specifically alloying U with 9–10 wt% Mo, in addition to stabilizing the bcc γ phase at room temperature, also improves the swelling resistance [4]. U-Mo fuels are fabricated either by vacuum induction melting and subsequent working [2,8,9] or by the powder metallurgy route [10]. Subsequently the cast microstructure is broken by hot working. This is an essential step in fabrication and can be effectively used to produce a component with a desired microstructure and free of macroscopic defects such as cracks, voids, macroscopic shearbands etc. In some early work the extrusion of U-12 wt% Mo was reported to be carried out at 850–880 °C, whereas that for U-9 wt% Mo the forging and rolling temperatures were reported to be 980 and 900 °C, respectively [5,11]. Recently hot working has also been reported to be carried out at 550–650 °C [1,7,9,12]. Studies on U-Mo alloys have been carried out in the area of fuel development [1,2,10,13], phase transformation and stability of γ phase [7,14–17], irradiation performance [8,18], physical properties [19,20], room temperature mechanical properties [12,19,21], and some

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intermediate temperature deformation behavior at 400–800 °C and strain rates from 10^{-5} to 10^{-2} s^{-1} [22,23]. Although hot working conditions have been reported, the flow behavior of U-Mo alloys at temperatures greater than 800 °C and in the strain rate range of 10^{-3}–1 s^{-1} have not been reported. Further, the relation between flow stress, strain rate and microstructure are largely unexplored. The present work characterizes the deformation behavior of U-9 wt% Mo in a higher temperature range of 850–1000 °C and higher strain rate range of 3×10^{-3}–1 s^{-1} and correlates the mechanical response with the developed microstructure.

2. Experimental

Uranium-9 wt% molybdenum alloy was obtained in the form of a rod extruded at 600 °C. The optical micrograph of this alloy after extrusion (Fig. 1) showed dark bands parallel to the extrusion direction. Similar bands in U-Mo alloys have been seen by other researchers and have been attributed to the segregation of Mo due to insufficient homogenization [9,19]. In order to identify the reason for these bands elemental mapping of Mo was done using energy dispersive spectroscopy (EDS) as described in Appendix. EBSD analysis also showed unindexed regions in the form of bands (Fig. 2a). The indexed regions showed equiaxed grains of 19 μm average grain size (Fig. 2a) and a high fraction of low angle boundaries as revealed through the misorientation angle distribution plot (Fig. 2b). The inverse pole figure (IPF) plot showed that most grains had the [001] direction oriented along the compression axis (Fig. 2c). Uniaxial compression tests were carried out on solid cylindrical samples of length 10 mm and diameter 5 mm at temperatures of 850, 900, 950 and 1000 °C and strain rates of 3×10^{-3}, 10^{-2}, 10^{-1}, 3×10^{-1}, and 1 s^{-1}. To prevent oxidation during high temperature tests, all the compression tests were carried out in a vacuum of 10^{-4} mbar using a deformation dilatometer. The temperature of the sample was controlled using an S-type thermocouple which was spot welded at the middle of the sample. The temperature of the sample was controlled to within an accuracy of ±1 °C. The samples were induction heated to the desired test temperature, held for 10 min and deformed to a true strain of 0.6. Immediately after deformation the samples were quenched with argon gas to retain the microstructure prevailing during hot deformation. The load (F) – displacement (d) data obtained in the compression tests were converted into true stress (σ) versus true strain (ε) curves using standard equations as $\sigma = S(1-e)$ and $\varepsilon = -\ln(1-e)$ where $S = F/A_o$ is the engineering stress and $e = d/h_o$ is the engineering strain, both S and e taken as positive values, and A_o and h_o are the initial cross-sectional area and initial height of the sample, respectively. The deformed samples were sectioned longitudinally along the compression axis, mounted, polished to 1 μm finish and electropolished using 50% phosphoric acid 50% water at 23 V, 8 °C for time of 10 s. The samples were observed using electron backscatter diffraction (EBSD) technique at 25 kV accelerating voltage (orientation image mapping system provided by Oxford Instruments) with in a Field Emission Scanning Electron Microscope instrument from Ziess.

Fig. 1. Optical micrograph of as-extruded sample showing dark bands along extrusion axis (horizontal).

3. Results

3.1. Flow stress

Fig. 3 shows the flow stress behavior of U-9 wt% Mo at various temperatures and strain rates. At temperatures of 850–1000 °C and at the higher strain rates of 10^{-1}, 3×10^{-1} and 1 s^{-1} U-9Mo showed work softening. At 950 and 1000 °C and at the lower strain rates of 3×10^{-3} and 10^{-2} s^{-1} the flow stress showed a near steady state behavior. For a fixed ε of 0.5 the flow stress increased with increasing strain rate and decreasing temperature (Fig. 4), as is expected for a thermally activated deformation process.

3.2. Strain rate sensitivity

The strain rate sensitivity $m = \partial \ln \sigma / \partial \ln \dot{\varepsilon}$ (slope of the log σ vs. log$\dot{\varepsilon}$ plot) was calculated and mapped out in the temperature and strain rate space as follows. To compute the strain rate sensitivity, σ was first interpolated at finely spaced T values using a spline fit for all strain rates tested. In the next step ln σ was interpolated at finely spaced ln$\dot{\varepsilon}$ intervals using cubic spline fit for all temperatures (tested as well as fitted). The coefficients of the spline function were then used to determine the strain rate sensitivity m at each value of strain rate and temperature. This was then mapped out as an iso-m contour plot on a T – log $\dot{\varepsilon}$ space as shown in Fig. 5. High strain rate sensitivity was observed in the temperature range of 900–1000 °C both at low strain rates of 3×10^{-3}–10^{-2} s^{-1} and at high strain rate of 3×10^{-1}–1 s^{-1}. At the intermediate strain rates the strain rate sensitivity was found to be lower. This change in strain rate sensitivity can also be seen from the difference in slopes of curves shown in Fig. 4a.

3.3. Macrostructure after deformation

The compression tested samples were sectioned longitudinally, polished, etched and observed using an optical microscope. It was seen that the sample tested at 850 °C–1 s^{-1} showed non-uniform deformation in form of a macroscopic shear band (Fig. 6). In comparison samples tested at higher temperatures showed no such shear band, an example of which is shown for sample tested at 950 °C–1 s^{-1} (Fig. 6). The bands as were seen in the as-extruded sample (Fig. 1), appear broken after deformation. As samples tested at 850 °C showed inhomogeneous deformation, EBSD was carried out only for samples which underwent relatively uniform deformation, i.e. samples tested from 900 to 1000 °C.

3.4. EBSD analysis

3.4.1. EBSD maps

At 1000 °C and all strain rate conditions the resultant microstructure showed equiaxed grains (Fig. 7). In each test condition unindexed regions were observed corresponding to the bands representing chemical heterogeneity (as observed in Figs. 1 and 2).

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