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Dynamic abnormal grain growth in tantalum

Nicholas A. Pedrazas [a], Thomas E. Buchheit [b], Elizabeth A. Holm [c], Eric M. Taleff [a,*]

[a] The University of Texas at Austin, 204 East Dean Keeton St., Stop C2200, Austin, TX 78712-1591, USA
[b] Sandia National Laboratories, P.O. Box 5800, MS1411, Albuquerque, NM 87185-1411, USA
[c] Carnegie Mellon University, 5000 Forbes Avenue, Pittsburgh, PA 15213, USA

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ABSTRACT

Dynamic abnormal grain growth (DAGG) is a phenomenon that produces one or more very large, abnormal grains during plastic deformation of polycrystalline material at high temperatures. DAGG was previously observed in commercial-purity molybdenum (Mo) and was used to produce large Mo single crystals of centimeters in length. The present investigation is the first to demonstrate DAGG in commercial-purity tantalum (Ta) sheet, another body-centered-cubic refractory metal. DAGG occurs in Ta at temperatures from 1450–1850 °C across strain rates from 3×10^{-5} to 5×10^{-4} s^{-1}. Grain boundary migration rates during DAGG in Ta are on the order of 10 mm/min. DAGG produces large abnormal grains preferentially oriented with the ⟨101⟩ direction approximately parallel to the tensile axis. A unique observation of this investigation is a preponderance of Σ3 special boundary character along the boundaries of large abnormal grains produced in Ta through DAGG. The propensity toward this special boundary character is a result of a relatively large grain size and strong texture in the polycrystalline material prior to DAGG and the typically low energy of Σ3 boundaries, which suppress boundary migration.

1. Introduction

Abnormal grain growth (AGG) involves the rapid growth of one or a few "abnormal" grains to at least several times the size of other grains in a recrystallized microstructure [1–4]. Many prior investigations of abnormal grain growth only considered growth during static annealing at elevated temperatures. Abnormal grain growth under such conditions may be specifically termed static abnormal grain growth (SAGG) [5]. This terminology is helpful to distinguish SAGG from abnormal grain growth that occurs during plastic deformation (*i.e.*, dynamic conditions) at elevated temperature, which was termed dynamic abnormal grain growth (DAGG) [5]. This distinction is important because DAGG exhibits features uniquely different from SAGG [5–8]. DAGG occurs at lower temperatures and produces significantly more rapid boundary migration rates than SAGG, for example [5,8]. Previously, DAGG induced by tensile straining at elevated temperatures was used to produce large single crystals, several centimeters long, in commercial-purity molybdenum (Mo) sheet and wire [5–8]. DAGG induced during tensile deformation at constant strain rate produces a rapid drop in load as one or more large grains grow across and along the gage region of the specimen. Flow stress drops as previous grain boundaries are swept away until a single grain, or a small number of large grains, span the specimen gage region, at which time the flow stress assumes that of the single or few crystals remaining. The large DAGG grains have a reduced flow stress because they do not contain the subgrains and other barriers to dislocation creep that existed in the polycrystalline microstructure they consumed. Because DAGG requires concurrent plastic deformation, DAGG grains do not penetrate significantly into the grip regions of tensile coupons. An example of a very large grain produced in tantalum (Ta) through DAGG is presented in Fig. 1(a). This DAGG grain consumed the entire gage length of the specimen. Fig. 1(b) shows the corresponding true stress *versus* true strain data and indicates where DAGG initiated and terminated during the tension test.

The present study investigates DAGG in a commercial-purity Ta sheet material. Specimens were tested in tension at elevated temperatures to induce DAGG. The recrystallized microstructures and progression of normal grain growth during exposure to high temperature were examined prior to straining. The mechanical behavior associated with DAGG during tensile elongation is presented. The microstructures produced by DAGG are characterized, including residual island grains, DAGG grain boundary types, individual grain crystallographic orientations and crystallographic textures. Attention is given to the role of special boundary types and the crystallographic orientation variations within individual DAGG grains.

* Corresponding author.
E-mail address: taleff@utexas.edu (E.M. Taleff).

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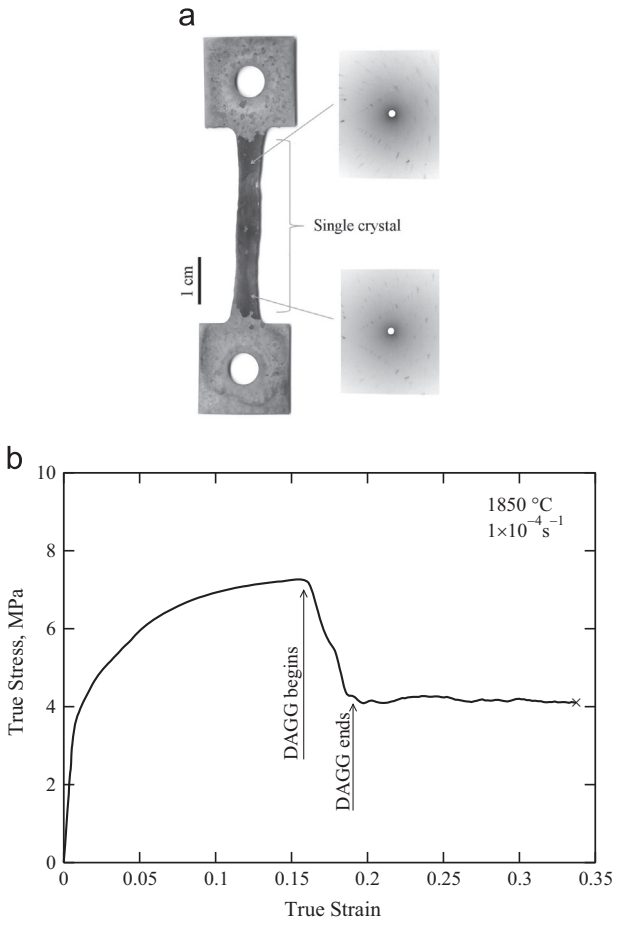

Fig. 1. This Ta specimen (a) evidences a DAGG grain produced at 1850 °C. Laue X-ray back-scatter diffraction images are shown from the extremes of the single-crystal region to demonstrate that the DAGG grain has a single orientation. Corresponding true stress *versus* true strain tensile test data (b) show a rapid drop in stress at approximately 0.16 true strain, indicating initiation and growth of the DAGG grain.

Table 1
Composition of Ta sheet material, in ppm by weight.

Ta	O	Si	H	C	Cr	Cu	Fe	Mg	Mn	Mo	Ni	Nb	N	Ti	W	Zr
Bal.	130	30	15	30	8	8	25	5	9	8	5	250	14	10	30	5

2. Experimental procedure

The material studied in this investigation is a commercial-purity, arc-melted, Ta sheet material that meets the ASTM R05200 grade specification [9]. Chemical composition of the sheet was provided by the supplier, Eagle Alloys Corporation of Talbott, TN, and is reproduced in Table 1. Tensile coupons were machined from the sheet with their tensile axes aligned perpendicular to the sheet rolling direction, *i.e.*, the rolling direction is along the long-transverse direction of the tensile coupons. Coupons were machined into a dog-bone geometry with a gage length of 25.4 mm, a gage width of 6.4 mm, a gage-grip transition radius of 1.6 mm, and the as-received thickness of 0.76 mm. Holes of 6.4-mm diameter were machined into the grip ends to accommodate loading pins used for testing of the coupons.

Tensile tests were conducted at constant temperature and constant true-strain rate in a high-temperature vacuum furnace attached to a computer-controlled, electromechanical test frame. Specimens were tested in tension using pin-loaded grips on tungsten pull rods within the hot zone of the furnace. Additional details of the test apparatus are provided by Worthington [7]. The range of true-strain rates investigated is approximately 3×10^{-5} to $5 \times 10^{-4}\ s^{-1}$, and test temperatures range from 1450 to 1850 °C, homologous temperatures (T_H) of 0.52–0.65. These strain rates and homologous temperatures were chosen for study based upon previous observations of DAGG in Mo. The vacuum level in the furnace during testing was approximately 10^{-4} Pa (10^{-6} Torr). Tungsten resistance elements provided the desired temperature to within ± 10 °C, as determined through separate furnace temperature profiling experiments. For each test, the furnace was brought to the desired test temperature and held at temperature until thermal expansion of the tungsten pull rods and specimen effectively stopped, which required 1–2 h. This assured accurate and consistent strain rate application throughout the test. Constant true-strain rates were applied in displacement control by continuously updating extension rate based on current elongation. Calculations to determine appropriate extension rate histories assumed conservation of specimen volume and no necking. Observations of tested specimens support these assumptions as quite reasonable. Tensile tests were stopped at either a predetermined elongation following the load drop associated with DAGG or after specimen rupture.

Specimens were prepared for optical and electron microscopy analysis using standard metallographic procedures and final-polished with a 0.05 μm colloidal silica solution. Specimens were etched using a solution of 10 ml nitric acid, 10 ml hydrofluoric acid, and 25 ml sulfuric acid prior to optical microscopy. Specimens were examined as polished in electron microscopy. Optical and back-scatter scanning electron microscopy observations were used to measure grain sizes by the lineal intercept method [10]. Large single-crystal regions, the DAGG grains, were characterized using Laue X-ray back-diffraction and electron back-scatter diffraction (EBSD). EBSD was also used to characterize the recrystallized, annealed microstructures in undeformed specimens. All EBSD data were collected on a Zeiss Supra 55VP SEM using Oxford HKL Channel5 [11] software.

3. Results

3.1. Recrystallized microstructures

During the heating and thermal soaking stages of tensile tests, each specimen had ample time to fully recrystallize and experience normal grain growth. Specimens were observed to recrystallize at all test temperatures, which indicates that the recrystallization temperature for this Ta material is below the lowest test temperature of 1450 °C. However, no SAGG was observed in any of the specimens. The recrystallized microstructures in the undeformed grip regions of tested specimens were examined and assumed to be representative of the microstructures in the specimen gage regions immediately before tensile straining. The effect of temperature on grain size is demonstrated in the pair of photomicrographs and the plot shown in Fig. 2. The initial, as-received microstructure in Fig. 2(a) demonstrates a lineal-intercept grain size of 50 μm. Normal grain growth during annealing at 1800 °C produced the microstructure shown in Fig. 2 (b). The lineal-intercept grain sizes after static annealing range from 247 to 550 μm, depending on temperature as shown in Fig. 2 (c). Variations in annealing time from 1–2 h did not significantly alter grain size because most grain growth occurred early in annealing. Thus, Fig. 2(c) provides a reasonable indication of normal grain growth as a function of temperature.

EBSD measurements were used to characterize the crystallographic texture of the recrystallized material. Results are referenced