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Growth of preexisting abnormal grains in molybdenum under static and dynamic conditions

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ABSTRACT

This investigation compares the growth rates of preexisting abnormal grains under both static and dynamic conditions. Abnormal grains several millimeters in length were produced in two commercial-purity molybdenum (Mo) materials by tensile straining at temperatures from 1923 to 2073 K (1650–1800 °C). This process is termed dynamic abnormal grain growth (DAGG) because it produces abnormal grains during concurrent plastic straining. DAGG creates abnormal grains at much lower temperatures than does static abnormal grain growth (SAGG). Abnormal grains created through DAGG were characterized with their surrounding microstructures and were then subjected to annealing treatments. Only one-third of the preexisting abnormal grains subsequently grew by SAGG. Among these, SAGG occurred only in those specimens that required the largest strains to initiate DAGG when creating the abnormal grain(s). The rates of boundary migration observed for SAGG were approximately two orders of magnitude slower than those for DAGG. When DAGG in one specimen was interrupted by extended static annealing, it did not recur when straining resumed. The dislocation substructure developed during hot deformation, which includes subgrains typical of five-power creep, is critically important to both DAGG and SAGG of preexisting abnormal grains under the conditions examined.

1. Introduction

Abnormal grain growth (AGG) is the process of one or more grains growing to be significantly larger than grains of the surrounding microstructure [1,2]. These large grains are called abnormal grains. AGG often produces a bimodal microstructure consisting of several abnormally large grains surrounded by many smaller grains. In extreme cases, abnormal grains can consume the entire microstructure to produce a uniformly coarse grain size or even a single crystal [3–7,2]. AGG is thus of interest for producing single crystals in the solid state, a capability demonstrated in the laboratory for molybdenum (Mo) [8–14] and tantalum (Ta) [15]. For grain-oriented silicon steels, AGG is desired and necessary to produce the coarse, highly-textured microstructures required for optimal performance in electrical transformers [16]. In other cases, such as the Ni-based superalloys used in jet engines, the large grains produced by AGG can be quite detrimental [17–22]. The accidental occurrence of AGG during thermomechanical processing must be avoided for such materials. These are some of the many reasons to seek a predictive understanding of AGG phenomena, which are to date only understood in quite basic terms [23].

AGG phenomena may be divided into two broad categories [10,11]: AGG that occurs during static annealing at elevated temperature, termed static abnormal grain growth (SAGG), and AGG that occurs during concurrent plastic deformation at elevated temperature, termed dynamic abnormal grain growth (DAGG). SAGG is synonymous with the behavior called secondary recrystallization [1,2]. DAGG is a recently discovered phenomenon that produces AGG at lower temperatures than does SAGG. For the case of commercial-purity Mo sheet materials, DAGG was observed at approximately 1773 K (1500 °C) and has been repeatedly produced at 1923 K (1650 °C) [10–12]. SAGG, however, has only been observed in Mo at temperatures of 2073 K (1800 °C) and higher [8,24]. Important features of DAGG are illustrated in Fig. 1. This figure presents data from a tensile test at 1923 K (1650 °C) and a true-strain rate of 10^{-4} s^{-1} for the commercial-purity Mo sheet material examined in the present study. During initial tensile straining, the specimen displays high-temperature plasticity typical of creep deformation. Upon reaching the critical strain for DAGG initiation, ϵ_c, which is 0.13 for the data in Fig. 1, DAGG initiates and causes a concurrent sharp drop in flow stress. Plastic straining immediately beyond ϵ_c continues the growth by DAGG of the abnormal grain(s)

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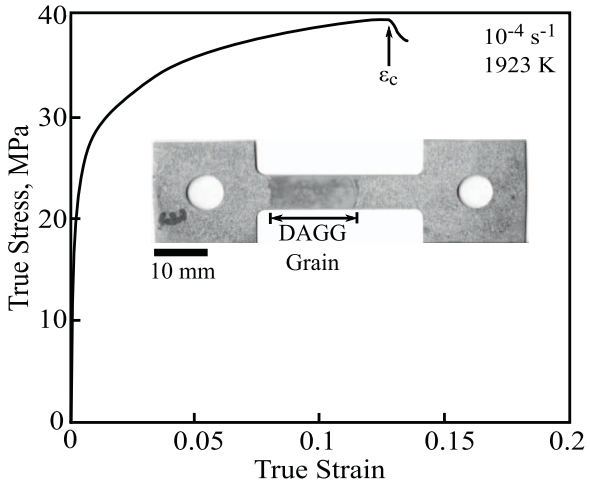

Fig. 1. True-stress *versus* true strain data from a tensile test of the commercial-purity Mo sheet material are presented. This test was performed at a temperature of 1923 K (1650 °C) and a true-strain rate of 10^{-4} s^{-1}. Tensile elongation of this specimen was halted shortly after DAGG initiation. An image of the tensile specimen shows the one large abnormal grain produced by DAGG, the DAGG grain.

initiated at ϵ_c, typically until the entire microstructure of the specimen gauge region is consumed. Once the entire gauge region is consumed by one or more abnormal grains, DAGG is complete and the flow stress stabilizes at a value significantly lower than before ϵ_c. Abnormal grains do not grow into the undeformed grip region of the tensile coupon. Continued straining simply deforms the abnormal grain(s) spanning the specimen gauge region until specimen rupture. When tensile deformation is halted prior to DAGG completion, DAGG also ceases. This produces one or more abnormal grains, such as the one shown in Fig. 1, that span only part of the specimen gauge region.

The reasons why SAGG is only observed at temperatures significantly above those for which DAGG occurs are not well understood. An important first step toward better understanding both DAGG and SAGG is to differentiate between the initiation, sometimes termed nucleation, of abnormal grains and their subsequent growth. The failure of SAGG to produce abnormal grains in Mo below 2073 K (1800 °C) could be because of either an inability to initiate abnormal grains at these relatively low temperatures ($T_H < 0.72$) or an extremely slow growth rate following initiation. The question of relative growth rates between SAGG and DAGG in the same material at identical temperatures has not been previously investigated. This study addresses this question by using DAGG to initiate abnormal grains, such as that shown in Fig. 1, and then studying the subsequent growth of these preexisting abnormal grains under both static and dynamic conditions.

Prior investigations demonstrated the importance of grain-boundary curvature and concurrent plastic deformation in the growth of abnormal grains by DAGG [10,11,15,14,12,13]. Grain-boundary curvature was demonstrated to supply the most important driving force for DAGG in Mo, and concurrent deformation is thought to primarily increase boundary mobility during DAGG [11–13]. However, the critical strains required for DAGG initiation indicate that significant dislocation substructure likely forms within the interiors of deformed grains prior to DAGG [25]. This may affect boundary migration in other ways. To address this possibility, specimens for this study were statically annealed following the initial creation of abnormal grains in order to eliminate as much of the dislocation substructure from the unconsumed, deformed polycrystalline microstructure as is practical through recovery. It will be shown that this met only limited success. The annealed specimens were then tested to investigate the potential for further growth of the preexisting abnormal grains created through DAGG. The following two specific questions are probed: 1. will a preexisting abnormal grain grow by SAGG during static annealing, and 2. will a preexisting abnormal grain in an annealed specimen grow again by DAGG when hot deformation is resumed? Where growth of preexisting abnormal grains was observed, measurements of boundary migration rates were made.

To avoid the complications produced by normal grain growth, this study uses a commercial-purity Mo sheet material that resists normal grain growth at temperatures up to 2073 K (1800 °C) [11,12]. This facilitates meaningful comparisons between tests at different temperatures and after different annealing times. To observe the effect of crystallographic texture on the growth of abnormal grains, an additional test used a separate commercial-purity Mo material in rod form with a deformation texture different from that of the sheet material.

2. Experimental

To study the growth of abnormal grains in Mo, DAGG was used to produce abnormal grains in two commercial-purity Mo materials, one in sheet form and the other in rod form. Individual specimens of the Mo sheet and rod materials are designated as Mo S and Mo R, respectively, with a different number appended for each specimen. Powder-metallurgy techniques were used to fabricate the Mo sheet material. This material meets the ASTM B 386, Grade 361 specification for commercially-pure Mo [26] and is the same sheet material designated as Mo PMB in a previous study by Noell et al. [12]. This sheet material was rolled to the as-received thickness of 0.76 mm, and the final rolling direction was used as a reference for the orientations of tensile test specimens. The chemical composition of the Mo sheet was evaluated by Noell et al. [12] and is provided in Table 1. The Mo rod material was produced using arc-melting techniques. The composition of the Mo rod prior to machining, provided by the manufacturer, is also shown in Table 1.

Creating abnormal grains by DAGG requires plastic deformation at high temperatures. For this first step in the study, tensile specimens of each Mo material were produced. Pin-loaded tensile coupons of the Mo sheet material were machined from the as-received Mo sheet. The gauge length and gauge width of the Mo sheet specimens were 25.4 mm and 6.4 mm, respectively. The radius between the grip and gauge regions was 1.6 mm. Specimens of the Mo sheet material

Table 1
The maximum limits reported by the manufacturer for chemical impurities in the powder used to produce the Mo sheet (Mo S) material are listed by weight percent. The concentrations of C, N, O and S in the Mo sheet material, measured independently using an inert-gas-fusion technique [27], are shown in parts per million (ppm) by weight [12]. The manufacturer's reported chemical composition of the Mo rod (Mo R) material before machining is also shown by weight percent.

Mo S	Mg	Mn	Ni	Al	Cu	Pb	Ti	Ca	
	≤0.001	≤0.001	≤0.002	≤0.002	≤0.002	≤0.002	≤0.002	≤0.003	
	Si	Sn	Cr	Fe	C	N	O	S	Mo
	≤0.003	≤0.003	≤0.005	≤0.005	10 ppm	10 ppm	40 ppm	10 ppm	bal.
Mo R	Mg	Mn	Ni	Al	Cu	Pb	Ti	Ca	
	<0.0010	<0.0010	<0.0010	<0.0010	<0.0010	<0.0010	<0.0010	<0.0010	
	Si	Sn	Cr	Fe	C	Mo			
	<0.0010	<0.0010	<0.0010	0.0011	<0.0050	bal.			