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Orientation dependence of stored energy release and microstructure evolution in cold rolled tantalum

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ABSTRACT

The bulk stored energy of cold rolled tantalum (Ta, 87% thickness reduction) was measured by differential scanning calorimetry, and related microstructure evolution during the stored energy release was studied by electron backscatter diffraction method. In addition, transmission electron microscopy was employed to reveal the substructure of the deformed Ta. Results showed that the deformed Ta consisted of {1 1 1} texture (<1 1 1> // ND) and {1 0 0} texture (<1 0 0> // ND), and its substructure was characterized by dislocation cells or dense dislocation walls. The total stored energy of as-rolled Ta was about 30.6 J/mol, most of which was released by recovery. The release of the stored energy suffered a complicated process and was orientation dependent. Correlative microstructure evolution path during heating referred to (i) recovery of all grains, (ii) primary recrystallization of {111} grains and recovery of {100} grains, and (iii) recrystallization of {100} grains.

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Introduction

When a metal is deformed, a fraction of energy is stored in the form of lattice defects, such as dislocations. Such stored energy will release during subsequent annealing process, and facilitate recovery and recrystallization to occur. Generally, the release of stored energy during recovery refers to annealing out of point defects, dislocation annihilation and rearrangement, and subgrain growth, while during recrystallization involves the formation of new strain-free grains and the subsequent growth of these to consume the deformed or recovered microstructure. Although the microstructure feature during the energy release process is identifiable, the borderlines between the various stages are often unclear [1]. However, understanding microstructure evolution is necessary and will be very helpful for optimizing heat treatments during material processing.

In this study, the stored energy of cold rolled tantalum (Ta) is measured and related microstructure evolution during stored energy release is investigated. Ta is selected as a research material for the following two reasons: (i) The stored energy of most of the metals has been measured in the early years, seeing the review by Bever et al. [2]. These materials have low melting points and relatively low recrystallization temperatures. In contrast, the data of the stored energy for high melting points metals, especially refractory metals (W, Mo, Ta), can be seldom found. The latest research on the stored energy measurement is carried out on Fe by Scholz et al. [3]. (ii) Ta is a refractory metal with a bcc structure. Due to unique properties, Ta has been widely used in many fields, such as electronics industry, cutting-tool industry, and chemical industry, medical and military fields [4–6]. Unfortunately, its fundamental studies drop behind. Specially, refining the grain size from a cast microstructure is notoriously problematic and thermomechanical processing is commonly employed in the manufacture of Ta products [7–10]. Producing a uniform fine grain structure largely relies upon multiple annealing steps between the mechanical deformation steps. Multiple annealing steps are considered to be costly and inefficient and it is necessary to shorten such process flow. Giving an insight into microstructure evolution during annealing is essential for us to find out optimal heat treatment conditions.

Experimental

The starting material in this study was high purity Ta ingot in the diameter of 97 mm with 99.99% minimum purity level. The chemical composition was determined by glow discharge mass spectrum and shown in Table 1. Such ingot was up-forged (with 50% deformation) followed by side-forging (with 50% deformation) to break down initial coarse grain structure. The forged Ta had 20 mm in thickness and then was annealed at 1250 °C for 2 h in a vacuum environment to get a fully recrystallized microstructure [10]. Finally, such ingot was processed to 2.6 mm thickness (about 87% deformation) at room temperature by multiple clock rolling, which referred to a continuous 135° rotation of rolling direction between the rolling pass [10].

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Table 1
Chemical analysis of Ta ingot (in wt. ppm).

Element	Content
C	8
H	2
N	18
O	30
S	<0.015
Si	<0.06
Total non-metallic impurities <60	
W	3.0
Nb	0.67
Mo	0.12
Al	<0.005
Ca	<0.005
Co	<0.003
Cr	<0.005
Cu	<0.005
Fe	<0.005
K	<0.005
Li	<0.003
Mg	<0.005
Mn	<0.003
Na	<0.005
Ni	<0.005
Sn	<0.005
Ti	<0.005
V	<0.003
Zn	<0.005
Zr	<0.005
U	<0.001
Th	<0.001
Total metallic impurities <4	
Ta	Balance

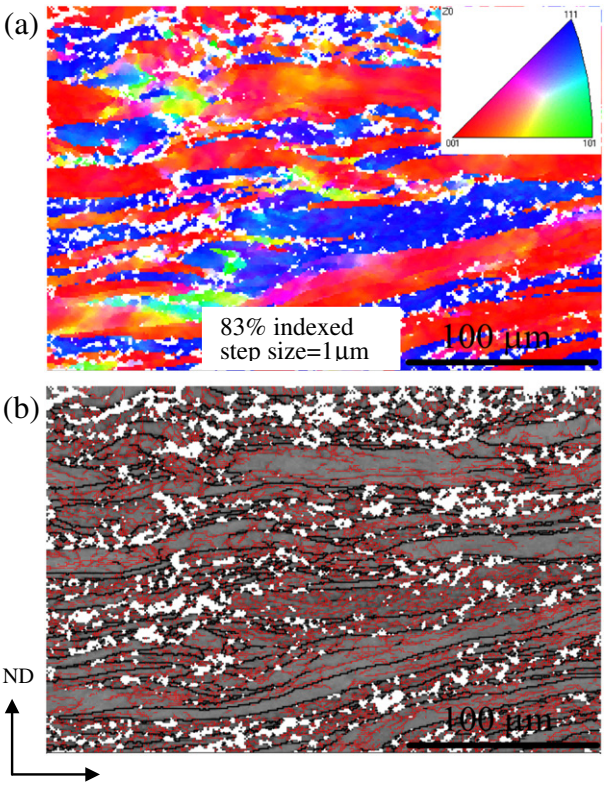

Fig. 1. Orientation image map (a) and grain boundary map (b) of cold rolled tantalum. White color is zero solutions, red lines represent boundaries 2°–10°, and black lines ≥10°. Legend of the figure is inserted at the top of the right corner.

Differential scanning calorimetry (DSC) was employed to measure the bulk stored energy of deformed Ta. A sample was cut from the rolled tantalum plate. The size of the sample was about 2 mm × 2 mm × 3 mm, which was smaller than the volume of a ceramic crucible. Prior to DSC measurement, the sample surface was ground and chemically polished in order to remove any deformation layer and surface contamination. DSC measurement was carried out in a NETZSCH STA instrument with a heating rate of 20 K/min going up to 1300 °C. The sample was kept at 1300 °C for 30 min to obtain complete recrystallization. Subsequently, the fully recrystallized sample was reheated to 1300 °C to acquire a baseline for quantitative measurement of the stored energy. During the heating process, N_2 was adopted to protect the sample from oxidation.

In order to acquire an overview picture of microstructure evolution during the stored energy release, other four samples sectioned from the same position of the rolled plate were heated to 950 °C, 1100 °C, 1200 °C, and 1300 °C respectively with the same condition as that used in the first DSC measurement. All the samples were prepared for microstructure examination by the electron backscatter diffraction (EBSD) technique. The microstructure was detected in longitudinal sections (rolling direction (RD)-normal direction (ND) plane) on a FEI Nova 400 microscope equipped with a fully automated electron backscatter pattern analysis system (Oxford Instruments-HKL Channel 5). The samples for EBSD analysis were mechanically ground, and then chemically polished in a solution of 50 ml H_2SO_4, 20 ml HNO_3 and 20 ml HF for 20 s at ambient temperature.

The release of the stored energy was involved with dislocation movement. It was essential to give an insight upon dislocation morphology. Thus, transmission electron microscopy (TEM) was applied to the exposure dislocation structure of the deformed sample. Samples for TEM observation were prepared by a chemical immersion method suggested by Wei [11]. TEM was conducted using a Zeiss Libra 200 operated at 200 kV.

Results

Deformation microstructure

Fig. 1 shows the deformation microtexture and microstructure of cold rolled Ta. After 87% thickness reduction, the deformed sample consists of a typical texture mixed with {1 1 1} texture (<1 1 1> // ND) and {1 0 0} texture (<1 0 0> // ND). The microtexture from EBSD results is consistent with the macrotexture measured by X-ray diffraction method [10]. The deformed sample contains large numbers of low angle boundaries (LAB, 2°–10°), as revealed by red lines in the Fig. 1b. Further qualitative analysis displays that the density of LAB in {1 1 1} grains is larger than that in {1 0 0} grains. Zero solutions (white color in the maps) represent severe lattice distortion regions associated with local dislocations. As shown in Fig. 1a, zero solutions nearly cluster in {1 1 1} grains or grain boundaries. These results indicate that the stored energy in cold rolled Ta is grain orientation dependence.

The orientation dependence of the stored energy in Ta has been reported by Sandim et al. with very coarse grains [9,12]. In {1 0 0} oriented grains, deformation occurred in a stable mode resulting in homogeneous deformation and low misorientation of neighboring regions, whereas in other oriented grains, such as {1 1 1} grains, a banded structure with large orientation spread within a single coarse grain often developed. In our study, the initial grains have a fine grain size and the orientation spread in {1 1 1} grains, most of which is within 10°, is much narrow than that in very coarse grains reported by Sandim et al.

DSC results

Fig. 2 is the DSC curves of cold rolled Ta heated to 1300 °C. The lower curve is obtained during the first heating ramp, while the upper curve is obtained during reheating of the recrystallized sample. Two exothermic

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