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Texture evolution of high purity tantalum under different rolling paths

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ABSTRACT

Deformation behavior and texture evolution of the material can be significantly affected by strain path change. For this reason, two rolling methods, unidirectional rolling (UR) and clock rolling (CR), were employed to manufacture tantalum plates. Texture evolution during unidirectional rolling and clock rolling was studied respectively by orientation distribution function (ODF). Related annealed microstructures were investigated by orientation image map (OIM). Usually, unidirectional rolling led to a strengthening of the main texture component with increasing strain, but for tantalum dominant texture component {0 0 1} θ-fiber was stable after 70% deformation, while minor texture component {1 1 1} γ-fiber was enhanced with increasing strain. In clock rolling, both of the two fibers were not stable any more for their intensity varied with rolling pass. After the final deformation, a similar texture was produced by the two rolling methods. However, recrystallization texture revealed a big difference. Such different texture development was contributed to microstructural change resulted from rolling path change.

1. Introduction

Tantalum (Ta) is a body-centered-cubic (BCC) metal with very high melting point (2996 °C) and high density (16.7 g/cm^3). Due to unique properties, Ta and its alloys have been applied in many fields, such as electronics industry, cutting-tool industry, chemical industry, aerospace and military fields. In order to meet various applications, different microstructures and textures are desired to be obtained, which largely depend on the manufacture and fabrication processing. For example, in deep drawing, Clark et al. (1992) pointed out a structure with γ-fiber (⟨1 1 1⟩//ND) texture and fine grain size was favorable, while in sputtering, Michaluk (2002) suggested a random texture or texture distributed uniformly with fine grain structure was superior. Unfortunately, the microstructure of high purity Ta ingots consists of a few coarse columnar grains with the grain size in the cm-range. As revealed by Sandim et al. (2005), refining the grain size from a cast microstructure in Ta was notoriously problematic. According to the patent invented by Turner (2006), typical process techniques for forming the desired structure in Ta usually refer to forging, rolling, extrusion and combinations thereof. Producing a uniform fine grain structure and texture largely relies upon multiple annealing steps between the mechanical deformation steps. The heat treatment of Ta needs to be carried out at high temperature in vacuum environment, which is highly energy-consuming. Multiple annealing steps are considered to be costly and inefficient and it is necessary to shorten such process flow.

The strain path is different when materials deformed under different deformation modes, such as compression, extrusion, torsion, forging, and rolling. According to the review by Davenport and Higginson (2000), changing the strain path has significant influence on grain size, texture, dislocation arrangement, recrystallization kinetics, and thus the mechanical properties. In the manufacture of plates or sheet products, where rolling technique is often used, the conventional unidirectional rolling usually leads to anisotropy or through thickness gradient in texture and microstructure. Hence, many variant rolling techniques have been developed to enhance texture and microstructure uniformity. Among them, the most widely studied techniques are cross rolling, asymmetry rolling, and cross-roll rolling. Recently, the cross rolling was applied by Oertel et al. (2010) to manufacture molybdenum sheets with improved forming properties by increasing γ-fiber texture. The asymmetry rolling was used to obtain grain refinement and homogeneous γ-fiber texture in IF steels by Tóth et al. (2012), and the cross-roll rolling introduced by Chino et al. (2006) has been conducted on magnesium alloy sheet to enhance its formability. The above rolling techniques have also been applied on Ta. In the early years, the cross rolling was used to manufacture Ta plates with improved drawability by strengthening γ-fiber texture by Clark et al. (1992). The asymmetric rolling was employed by Field et al. (2005) to refine the grain structure and randomize the crystallographic texture in Ta. Nowadays, uniform fine grained bulk Ta has been successfully

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Table 1
Chemical composition of high purity Ta ingot (wt ppm).

C	N	H	O	Nb	Mo	W	Ti	Si	Fe	Ni	Ta
9	20	2	30	6.4	0.14	0.61	<0.001	<0.005	<0.005	<0.005	Balance

obtained through equal channel angular extrusion (ECAE) by Mathaudhu et al. (2005). However, it is restricted to laboratory scale and is not commercially viable. Therefore, changing the rolling path is still a preferred method in microstructure and texture control of Ta. As reported by Aditya et al. (2012), changing rolling path had a significant effect on the microstructure and mechanical properties in as-rolled and annealed Ta. Although the mentioned methods have been applied to process Ta with fine grain structures, related texture character is little investigated, which will have an impact on certain properties of the metal.

In this paper, two rolling paths, unidirectional rolling and 135-degree clock rolling, were employed to manufacture tantalum plates. The present work concentrated on the influence of different rolling paths on texture evolution of the deformed samples. In addition, microstructure and texture of annealed samples processed by the two rolling methods were also investigated.

2. Experimental procedures

2.1. Material and rolling experiments

The material used was high purity tantalum, with the chemical composition given in Table 1, supplied by Ningxia Orient Tantalum Industry Co. Ltd., China, provided as ingot with cylindrical shape with the diameter of 97 mm. Several discs were cut from this ingot and forged into rolling plates of 20 mm in thickness with round shape. The plates for rolling underwent a heat treatment (1250 °C for 2 h in vacuum environment) to get fully recrystallized microstructure.

These round plates were divided into two groups for next rolling experiments. One with round shape was processed directly by clock rolling, and another was cut into rectangle shape first and then processed by unidirectional rolling. Schematic diagram of metal working process is shown in Fig. 1a. During clock rolling, the rolling direction was counterclockwise rotated by 135° with respect to the previous rolling direction. After a set of 8 rolling passes, the rolling plate retained nearly round shape. Four thickness reductions of 70%, 76%, 82% and 87% were obtained by 8, 10, 12 and 16 rolling passes during clock rolling. Simultaneously, the other four rectangle plates were unidirectional rolled to the same thickness reductions as that in clock rolling. The rolling schedules are specified in Table 2. All the rolling experiments were carried out on an industrial rolling mill with a roll diameter of 1000 mm. The value of l/h (l/h, where l is the contact length between the specimen and the roll, and h is the

Table 2
Rolling schedule for each rolling pass.

Number of rolling pass	Entrance thickness, d_0 (mm)	Exit thickness, d (mm)	Geometry of the roll gap, l/h
1	20.0	17.2	2.01
2	17.2	14.7	2.22
3	14.7	12.6	2.37
4	12.6	10.8	2.56
5	10.8	9.3	2.72
6	9.3	8.0	2.95
7	8.0	6.9	3.15
8	6.9	6.0	3.29
9	6.0	5.3	3.31
10	5.3	4.7	3.46
11	4.7	4.2	3.55
12	4.2	3.8	3.54
13	3.8	3.5	3.36
14	3.5	3.2	3.66
15	3.2	3.0	3.23
16	3.0	2.8	3.45

Note: $l/h = 2\sqrt{r(d_0 - d)}/(d_0 + d)$, r is the radius of rolling mill.

average thickness of the sample for each rolling pass) was kept between 2 and 3.7 for each rolling pass, which was considered to produce homogeneous texture and microstructure. The rolling speed was 0.2 m/s, and no lubrication was adopted during experiments.

Samples for texture measurements and microstructure characterization were sectioned along the final rolling direction (Fig. 1b). Selectively, two specimens with 87% thickness reduction were sampled from clock rolled and unidirectional rolled plates respectively for heat treatment. Both samples were capsulized in quartz tubes with a pressure close to 10^{-5} Pa, then heated to 1300 °C at 10 °C/min and annealed for 1 h to obtain fully recrystallized microstructure.

2.2. Texture measurements

Textures of the rolled plates processed by two different rolling methods were measured by XRD technique. The textures were examined by measuring the four incomplete pole figures {1 1 0}, {2 0 0}, {2 1 1}, and {2 2 2} in the center layer (RD-TD plane) of the samples. The measurements were carried out in the range of the pole distance angle from 20° to 90° in the back reflection mode using Cu Kα1 radiation. During measurements, the specimens were oscillated along the TD to cover a larger specimen area. The

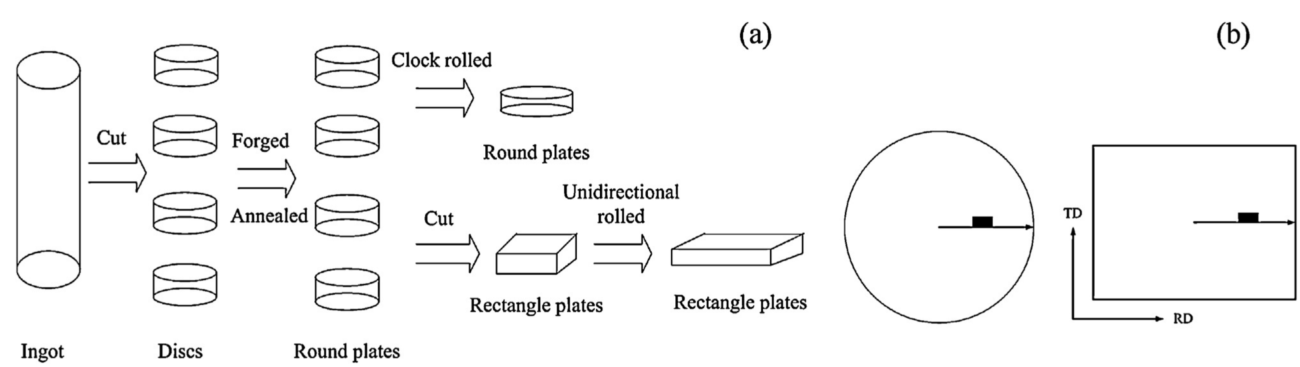

Fig. 1. Schematic diagrams of metal working process (a) and sample sections (b).