Materials and Design 97 (2016) 464–472

Contents lists available at ScienceDirect

Materials and Design

journal homepage: www.elsevier.com/locate/matdes

Largely alleviating the orientation dependence by sequentially changing strain paths

Haiyang Fan, Shifeng Liu *, Lijuan Li, Chao Deng, Qing Liu *

College of Materials Science and Engineering, Chongqing University, Chongqing 400044, China

ARTICLE INFO

Article history:
Received 9 November 2015
Received in revised form 19 February 2016
Accepted 20 February 2016
Available online 23 February 2016

Keywords:
135° clock rolling
Strain path change
Orientation dependence
Stored energy
Shear bands
Tantalum

ABSTRACT

Grains with γ-fiber texture (⟨111⟩ direction // normal direction (ND)) and θ-fiber texture (⟨100⟩ direction // ND) show different subdivision behaviors during unidirectional rolling, which leads to orientation-dependent stored energy. This orientation dependence in tantalum can be largely alleviated by a novel approach named 135° clock rolling, which is attributed to two reasons. One is that the clock rolling can weaken the micro-shear bands and destroy the parallel dislocation boundaries in γ-fiber grains, thus reducing their stored energies; and the other is that the clock rolling changes the stability of θ-fiber orientations and introduces plenty of "veins" into θ-fiber grains, thereby increasing corresponding stored energies. Therefore, 135° clock rolling narrows the stored energy difference between these two types of grains, which is beneficial for homogenizing the annealing microstructure of tantalum.

1. Introduction

Since the activity of slip systems depends on orientations, the deformation substructure and stored energy also differ from grain to grain [1], which is called the orientation dependence. This kind of dependence has adverse effects on the microstructural and textural uniformity of annealed samples [2,3]. For years, systematic studies about this topic have been done on some face-centered cubic (fcc) metals like copper and aluminum [4–6], while the body-centered cubic (bcc) transition metals with high melting points, e.g. niobium, molybdenum and tantalum, have received less attention than the above fcc metals, even though these refractory metals also show some degree of orientation dependence [7–9].

In these transition metals, representative components with orientation dependence are grains with {100} and {111} orientation, respectively [10–12], which own entirely different deformation substructures, stored energies and even recrystallization tendencies [8,13,14]. In this study, tantalum (Ta) is chosen as a model material for its application of sputtering target [15]. It is commonly accepted that orientation dependence of Ta is detrimental to the recrystallization homogeneity since it often leads to residual deformation bands and texture clusters during annealing [14]. As a result, the sputtering performance of Ta targets is largely damaged by this uneven recrystallization structure [16]. Unfortunately, relevant work about the orientation dependence of Ta is so lacking that only few studies based on single crystals and coarse columnar grains could be referred to [8,13,14,17]. Nevertheless, most of the available literatures including those above have just focused on characterizing the orientation dependence rather than alleviating it.

Strain path change should be an approach to weaken the orientation dependence as it activates slip systems from different directions and then destroys the substructure developed in preceding deformation courses [6,18]. Although some severe deformation modes like equal channel angular pressing (ECAP) can introduce a fine and uniform microstructure [19–21], these modes are not particularly applicable in industry for the limitation to billet's dimensions [19,20,22,23], especially for materials with a high hardness, e.g. Ta. Considering that rolling has long been popular in many industrial applications to produce sheet materials, this study focuses on rolling instead of some severe deformation modes.

The previous studies have reported that the 45° clock rolling cannot only weaken the basal texture and generate a uniform annealed microstructure of magnesium alloy [24], but also enhance the isotropy of zirconium [25]. The clock rolling is also a desirable industrial technique to produce sputtering targets [15]. Therefore, this study focuses on a clock rolling schedule with a rotation angle of 135°. Subdivision behaviors of θ-fiber and γ-fiber grains under unidirectional rolling (UR) and 135° clock rolling (CKR) were analyzed by techniques of electron back-scattered diffraction (EBSD), X-ray line profile analysis (XLPA), transmission electron microscope (TEM) and electron channeling contrast imaging (ECCI).

---

* Corresponding authors.
E-mail addresses: liusf06@cqu.edu.cn (S. Liu), qingliu@cqu.edu.cn (Q. Liu).

http://dx.doi.org/10.1016/j.matdes.2016.02.084

2. Material and experimental details

Initial tantalum plates (high purity, 99.95 wt%) were rolled by CKR (i.e. sequentially changing the rolling direction (RD) by 135° about the normal direction (ND)) and UR (i.e. rolling along a same direction), respectively. A schematic diagram of CKR is described in Ref. [26] and Table 1 shows more details of these two rolling schemes. The Von Mises equivalent strain εvm can be calculated by the equation: $\varepsilon vm = -\frac{2}{\sqrt{3}}\ln(1-\delta)$, where δ is the value of thickness reduction. Plates adopted were of 60% and 87% rolling reductions and thus the corresponding equivalent strains of these two rolling reductions were 1.06 and 2.36, respectively.

The following EBSD, ECCI and TEM measurements were performed at the center of RD-ND plane of samples rolled by different rolling schemes. After electrochemical polishing [27], grains with {001}⟨110⟩, {001}⟨100⟩, {111}⟨112⟩ and {111}⟨110⟩ orientations were selected and analyzed by EBSD attached to an extreme-resolution analytical field emission SEM (JEOL JSM-7800F) operated at 20 kV. In order to reveal the severely deformed microstructure accurately, these grains were scanned with a small electron beam spot size (12 nm) and a small step size (50 nm). An AZtec EBSD system (Oxford Instruments) and a HKL Channel 5 software were used for data acquisition and analysis, respectively. The TEM and ECCI measurements were conducted to observe the dislocation boundary arrangements in some of these grains. Foil preparation was in accordance with the method suggested by Wei et al. [28] and a Zeiss Libra 200 with an operating voltage 200 kV was adopted for TEM observation. ECC images were taken using JEOL JSM 7800F (working voltage 15 kV and working distance 8 mm).

To quantify the orientation-dependent stored energy, the RD-transverse direction (TD) plane with an area of 10 × 12 mm^2 was measured by XLPA for every sample. Measurements were conducted on a high-power diffractometer (Rigaku D/max 2500PC, rated power 18 kW) with a Cu Kα radiation (40 kV/150 mA). The θ-fiber (200) and γ-fiber (222) line profiles were recorded by step-scan mode with a step size of 0.01° and timing 1 s per step. After background subtraction and instrumental broadening correction, the (200) and (222) peaks were fitted by an XRD analysis software, JADE 6.0 (http://www.materialsdata.com/).

3. Results and discussion

3.1. 135° clock rolling

The rotation angle plays a critical role in changing rolling paths. Some typical rolling schedules including CKR are showed in Table 2. Schmitt et al. [29] introduced a scalar parameter α to indicate the effects of changing strain path. When α = 1, the slip systems are triggered from one direction. If α = −1, the same systems are activated from the opposite direction. For α = 0, some other latent systems can be awoken. Based on this theory, the α value of 90CR, 45CR and CKR are showed in Table 2, respectively. Perhaps 45CR and CKR are two confusing schemes, which can be differentiated below. According to the principle of force decomposition, the strain of one rolling pass is always partly strengthened by the following pass during 45CR but greatly neutralized during CKR, which should be an essential difference between these two modes. Taking the R2 (the second rolling pass) as an example, the strains introduced by 45CR in R2 are monotonic and orthogonal strains (α is a value from 0 to 1), while these strains are replaced by reverse and orthogonal ones during CKR (α is a value from −1 to 0). Thus, the influence of R1 (i.e. the monotonic strain) is partly enhanced by the R2 during 45CR but largely weakened by the R2 of CKR. It follows that the microstructure is always damaged by the next rolling pass of CKR.

Table 1
The rolling scheme of UR and CKR.

Number of rolling pass	Entrance thickness d_0/mm	Exit thickness d/mm	Rolling reduction of per pass %	Total rolling reduction %
1	20.0	17.2	14.0	14.0
2	17.2	14.7	14.5	26.5
3	14.7	12.6	14.3	37.0
4	12.6	10.8	14.3	46.0
5	10.8	9.3	13.9	53.5
6	9.3	8.0	14.0	60.0
7	8.0	6.9	13.8	65.5
8	6.9	6.0	13.0	70.0
9	6.0	5.3	11.7	73.5
10	5.3	4.7	11.3	76.5
11	4.7	4.2	10.7	79.0
12	4.2	3.8	9.5	81.0
13	3.8	3.5	7.9	82.5
14	3.5	3.2	8.6	84.0
15	3.2	3.0	6.3	85.0
16	3.0	2.6	13.3	87.0

3.2. Changing the substructure of γ-fiber grains

Different rolling histories result in different microstructural features. M.Y. Huh et al. [30] concluded that rotating RD of 30°, 45°, 60° or 90° respectively after an initial 30% reduction of straight rolling can promote the formation of micro-shear bands (MSBs). M. Lewandowska [6] found that changing the orthogonal strain path can cause a partial destruction of previous dislocation walls, contributing to a more homogeneous distribution of the dislocations. In this study, CKR leads to a γ-fiber grain substructure with very few MSBs, as shown in Figs. 1 and 2.

The UR and CKR (εvm = 1.06) introduce very different microstructural features into γ-fiber grains, as revealed in Fig. 1. Both {111}⟨112⟩ and {111}⟨110⟩ grains in the UR-Ta include numbers of MSBs (Fig. 1(a)), while these bands develop in a very moderate way for the CKR-Ta (Figs. 1(c and e)). Case seemingly still goes like this when εvm reaches 2.36, as shown in Fig. 2. It should be clarified that the local severe deformation circled in Fig. 2(c) is most likely attributed to the effect of grain boundary rather than rolling path, since the strain near the boundary is normally larger than that in the grain interior after deformation [31]. To prevent this misleading information, the regions involving grain boundaries were neglected when doing statistical analysis.

The misorientation distribution in Fig. 2(e) shows that the high angle boundaries (i.e. misorientation angle >15°) occur almost only in UR-γ grains, which is in agreement with the results obtained by Gurao et al. [32]. They concluded that the cross-rolled microstructure has lower intragranular misorientation when compared with samples rolled by UR. Apart from the MSBs, a set of parallel lamellar boundaries named geometrically necessary boundaries (GNBs) are also typical microstructures in deformed grains [5]. In the interstitial-free steel (IF steel) processed by UR, the average misorientation angle of GNBs is <10° [33]. Hence, misorientation angles ranging from 40° to 60° in Fig. 2(e) could be regarded as the effects of MSBs.

The UR often leads to a sharp and well-defined substructure, while changing rolling path introduces a substructure in a diffused state [32]. Similar results are observed as well in this study. Regularly arranged GNBs in the UR-Ta result in an obvious intragranular anisotropy, as evident from Fig. 3(a) and (b). As mentioned above, changing rolling paths can cause interaction, annihilation and rearrangement of previous dislocation structures, and at the same time create new ones. After 16 rolling passes of CKR, this annihilation and creation finally result in a substructure consisting of a huge number of blocks. Substructural features like irregular shapes, short-range regularity, distortion and chaos can be revealed from