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Deformation twinning and localized amorphization in nanocrystalline tantalum induced by sliding friction

Y.S. Zhang^{a,*}, L.C. Zhang^b, H.Z. Niu^a, X.F. Bai^a, S. Yu^a, X.Q. Ma^a, Z.T. Yu^a

^a Northwest Institute for Nonferrous Metal Research, Xi'an 710016, China

^b School of Engineering, Edith Cowan University, 270 Joondalup Drive, Joondalup, Perth, WA 6027, Australia

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1. Introduction

Severe plastic deformation (SPD) has been developed as an effective technique to prepare nanocrystalline (nc) materials. While the vast majority of investigations involving nc materials fabricated by SPD have concentrated on those metals with a facecentered cubic (fcc) crystalline structure, such as Cu [1,2], and Al [3]. A few systematical studies on microstructure evolution of Ti [4] and Mg [5], typical metals with a hexagonal close-packed (hcp) crystalline structure, have been conducted. Much less literature has reported on the processing and properties of nc metals with a body-centered cubic (bcc) crystalline structure probably owing to their high plastic resistance and the limitation of the technologies adopted. For example, as a typical bcc metal, usually ultrafine or coarse grained Ta was obtained by cold forging [6] or equal channel angular pressing (ECAP) [7]. Recently, nc Ta with an average grain size of about 40 nm [8] and 70 nm [9] have been processed by high-pressure torsion (HPT), which are to our knowledge the only two exclusive reports up to now on the formation of nanocrystalline Ta fabricated by SPD.

In this work, a nc surface layer of tantalum with extremely small grains of less than 10 nm was achieved by means of a technique called sliding friction treatment (SFT) [10]. Deformation twinning and localized amorphization were observed in nanocrystalline tantalum induced by sliding friction. Our results shed light on the understanding of the microstructure evolution of the

* Corresponding author. E-mail address: y.sh.zhang@163.com (Y.S. Zhang).

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2. Experimental

We reported the formation of nanocrystalline structure in the surface layer of a pure tantalum plate

under sliding friction treatment. Nanocrystalline grains of 7 nm with amorphous boundaries were

formed in the topmost surface layer of Ta plate. Deformation twins were also observed in the

nanocrystalline grains with size below 20 nm. Deformation twinning and localized amorphization are

the possible deformation mechanisms in the top surface layer of tantalum induced by sliding friction.

The SFT was performed with a ball-on-disc contact configuration, in which a Ta plate $(200 \times 200 \times 3 \text{ mm}^3 \text{ in dimension})$ with a purity of 99.95 wt% and a surface roughness (R_a) of 0.4 μ m was pressed with a 10-mm-diameter WC-Co ball under high pressure and SPD is imparted by sliding the Ta plate with respect to the ball. Compared with the traditional friction and wear testers, this unique SFT equipment employed in this work possesses a higher sliding velocity and a displacement offset (e.g. 100 µm in this work) along the perpendicular direction after each sliding path. As such, a large treated surface with refined grains can be obtained in our SPT treatments. The treatments were carried out under chlorcosane without any additives and the given processing parameters were selected as follows: 500 N in load, 0.2 m/s in sliding velocity, 50 mm in amplitude, and 100 in cycle.

The microstructure of the surface layer of the treated samples was characterized by using a JEOL JEM-2100 transmission electron microscope (TEM) operated at a voltage of 200 kV. Thin foil samples for TEM observations were cut from the treated surface layer and thinned by ion thinning at low temperatures.

3. Results and discussion

Fig. 1 shows a bright-field and the corresponding dark-field TEM image for the topmost surface layer of the SFT Ta sample. It is evident that the microstructure of the top surface layer is











Fig. 1. (a) A typical low magnification TEM bright-field and (b) the corresponding dark-field TEM image for the microstructure at the topmost surface layer in the SFT Ta sample. The inset shows the statically distribution of grain size in the layer.

characterized by extremely ultrafine equiaxed grains with random crystallographic orientations. In terms of a number of TEM images, a statistical grain size distribution was derived as shown in the Fig. 1(b) inset. The average grain size of the top layer is about 7 nm.

Ultrafine-grained (UFG) structures are typical steady-state characteristics of those bulk metals processed by means of SPD. The current extremely small grain obtained in tantalum can be attributed to the unique processing technology and characteristics of the adopted metal. In the process of SFT a very-high-rate shear deformation with high strain gradients was applied to the top surface layer of a pure bulk Ta sample. The calculated maximum strain gradient was ~ 1 according to the measurements of the displacements of the flow lines [11], which is one order of magnitude greater than that for the high-pressure torsion process [12]. High strain gradients during SPD may be a key factor relating to the grain refinement limitation of the metals [12]. Furthermore, room temperature dynamic recovery of dislocations is difficult to occur for tantalum, which is known at a very high melting temperature (3290 K). Mohamed et. al [13] has proposed that the minimum grain size increases exponentially with the stacking fault energy (SFE) of pure metals through ball milling. Apparently, our findings are somewhat in contradiction with this model. This may be attributed to the difference in the grain refinement mechanism between these two processing techniques.

It is evident that a diffused halo, which is a typical feature of an amorphous phase, superimposes on rather sharp continuous diffraction rings from nanocrystalline tantalum grains with random orientations (Fig. 2 inset). Therefore, the as-treated microstructure for the topmost surface layer of the SFT Ta plate consists of a mixture of amorphous phase and fine grains. Such unique characteristics in the topmost layer of SFT Ta are typically depicted by high-resolution TEM (HRTEM) image, as shown in Fig. 2, in which the amorphous structures (indicated by solid arrows) are clearly located at the grain boundaries of equiaxed nanocrystalline Ta. A localized amorphization occurring at grain boundaries has been often reported in multicomponent complex alloy systems under various mechanical deformation and extreme conditions of severe strains and/or high strain rate, e.g. HPT [14] and surface mechanical attrition treatment (SMAT) [15]. However, in this study, amorphous structures as well as the diffused halo cannot be found at the depth just adjacent to the topmost surface, as shown in Fig. 3. So, it is suggested that the initiation and development of amorphization are responsible for the extreme localized strain or strain rate greater than a certain level, as a result of an elastic instability loss of shear rigidity [16]. At very fine grain sizes of several nanometers in the topmost layer of SFT Ta,



Fig. 2. A high-resolution TEM bright-field image and the corresponding selected area electron diffraction (SAED) pattern for the microstructure at the topmost surface layer in the SFT Ta sample. Amorphous structures are clearly located at the grain boundaries of equiaxed nanocrystalline Ta.

where dislocation activities cease to operate, amorphization also acts as a new deformation mechanism relative to the grain boundary migration [17] and grain boundary sliding [18] based on computer simulations.

It is noted that deformation twinning is also observed in this sample within a grain with short directional size of ~ 20 nm, where the twins are distinct with two characteristic flat interfaces parallel to each other (Fig. 3a and b). In rather small grains less than 5 nm in size, deformation twins can also be observed (Fig. 3c). For the grain size of less than tens of nanometers, partial dislocation activity becomes the preferred deformation mode, resulting in the formation of deformation twins [19,20]. This is consistent with our TEM results that no full dislocation was observed in the grains in Fig. 3d.

The observation of deformation twins in nc grains has generated considerable interest and debate due to the very different behavior in the formation of deformation twins between nc and coarse-grained materials. For example, the deformation twins, which have never been observed in coarse-grained Al due to its high stacking fault energy, were confirmed in nc Al film produced by physical vapor deposition [21]. It should be noted that the Download English Version:

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