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Indentation size effect on hardness in the body-centered cubic coarse-grained and nanocrystalline tantalum

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ABSTRACT

Indentation size effect (ISE) on the hardness in the nanocrystalline (NC) (~24 nm) Ta and coarse-grained (CG) counterpart has been investigated by the nanoindentation tests at room temperature. The experimental results show that at shallow indentation depth ranging from 50 nm to 200 nm, the measured hardness is obviously smaller than the values predicted by the Nix-Gao model in the CG Ta, which is in contrast to the tendency in the NC Ta. At indentation depth less than 600 nm, obvious ISE can be observed in the both of CG and NC Ta. When the indentation depth is larger than 600 nm, the observed ISE in the CG Ta would gradually disappear in the NC Ta. Based on the deformation mechanism at different indentation depth, the density of geometrically necessary dislocations (GNDs) (ρ_{G-NC}) and statistically stored dislocations (SSDs) (ρ_{S-NC}) constructed by the Nix-Gao model are used to explain this phenomena.

1. Introduction

Over the past decades, nanoindentation techniques have been developed to a commercial method to make it possible to explore the micromechanical properties and behavior of very small dimensional materials, such as thin films or near-surface layers of solids, single phases in multiphase materials and etc. Extensive experiments have repeatedly exhibited strong indentation size effect (ISE), i.e., indentation hardness decreases with increasing indentation depth. Based on the theory of strain gradient plasticity, Nix and Gao [1] have established a model as description of the ISE for crystalline materials using the concept of geometrically necessary dislocations (GNDs) and statistically stored dislocations (SSDs), which have successfully predicted the experimental indentation hardness data for many crystalline materials. But Manika have reported when the indentation size is comparable to or exceeds grain size (d), ISE will not be observed because the fact that grain boundaries (GBs) are strong barriers for dislocations [2], that could restrict the aforementioned two dislocation related models to operate in nanocrystalline (NC) metals. Up to now, ISE have been widely observed in single crystalline and coarse grained (CG) metals, but the studies involving NC metals are rare, especially in the body-centered cubic (BCC) NC metals.

Tantalum serves as a prime candidate material for studying plasticity in BCC metals owing to its high phase stability because of high melting temperature/pressure [3]. Due to the difficulty in fabrication of bulk BCC NC metals, film materials (with thickness less than ~2 μm) of NC Ta deposited by direct current magnetron sputtering have always been used to study their mechanical properties via nanoindentation. The measured hardness (H) of these materials is likely to be too high (in the range of 10–18 GPa) [4–9] compared to the values estimated by the equation of $H = C\sigma_y$ (where C is the constant of 2.5–3.0 and σ_y is the yield strength), which is probably attributed to the small indentation depth adopted in these limited-scale materials. To obtain more information about mechanical properties of the NC BCC metals, the problem remaining should be explored in detail is how the hardness responses at different indentation depths over a wide range of loading strain rates ($\dot{\varepsilon}_L$).

The objective of this paper is to investigate the indentation size effect on the hardness in the CG and NC Ta. To shed light on this issue, nanocrystalline surface layer with a thickness of ~20 μm and an average d of ~24 nm was successfully synthesized by means of sliding friction treatment (SFT). Nanoindentation test in the CG and NC Ta were loaded systematically at different strain rate of 1×10^{-3}/s–4×10^{-1}/s to the indentation depth of 50–1000 nm at room temperature. Combination with the deformation mechanism in the CG and NC metals, the terms of the density of GNDs (ρ_G) and SSDs (ρ_s) in the Nix-Gao model are used to explain the hardness at shallow indentation depth, ISE in the CG Ta and no ISE in the NC Ta at larger

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indentation depth.

2. Experiments

A commercial Ta plate with a size of 200 mm×200 mm×3 mm and a purity of 99.95 wt% was subjected to the SFT process. More detailed descriptions of the SFT setup and procedures used in this study were reported previously [10,11]. The microstructure of the surface layer of the treated samples was examined by transmission electron microscopy (TEM) (JEM-2100F). Thin film samples for the TEM examination were cut, polished and dimpled by argon-ion milling (EMRES101) at 5 kV. In order to acquire reliable nanoindentation data, the surface of the test specimens were mechanically polished to mirror finishing. Quasi-static nanoindentation tests were performed at room temperature on a nanoindenter (Agilent-G200) with a Berkovich diamond indenter. The CG and NC Ta were loaded at the strain rate of 1×10^{-3}/s–4×10^{-1}/s to the indentation depth of 50–1000 nm without holding time at maximum load. The thermal drift calibration was performed prior to testing (limiting thermal drift rate valueless than 0.03 nm/s) and each indentation was repeated at least ten times.

3. Results

3.1. Microstructure

Fig. 1a shows the representative TEM bright field image of the microstructure and the corresponding selected area diffraction (SAD) pattern (the upper inset) of the section at 0–20 μm below the treated surface where severe plastic deformation occurred, respectively. The TEM image displays that a quite uniform and roughly equiaxed grain structure is mostly separated by high-angle GBs and twins can be observed inside some nano-sized grains. Fig. 1b shows the grain size distribution based on the statistical analysis of ~500 grains taken from the TEM images and the average d is ~24 nm. Fig. 1c gives residual impression in the different regions below the treat surface. Nanoindentation tests have shown the obtained data in the regions of the CG Ta and NC Ta can be well repeated at given loading condition because of uniform microstructure, which are beneficial for studying the mechanical properties of these two materials.

3.2. Load-displacement curves

Fig. 2a and b show the load-displacement ($P - h$) curves obtained at the strain rate of 1×10^{-3}/s–4×10^{-1}/s to the indentation depth of 50–1000 nm without holding time at maximum load on the CG and NC Ta, respectively. As shown in Fig. 2a, at $\dot{\varepsilon}_L$ of 4×10^{-1}/s, the loading curves at different depths are completely overlapped for these two samples indicating the accuracy of the tests. As shown in Fig. 2b, with decreasing $\dot{\varepsilon}_L$, the required load decreases more sharply in the NC Ta compared with that in the CG Ta. At lower $\dot{\varepsilon}_L < 1 \times 10^{-2}$/s, the displacement at maximum load would deviate from the setting depth of 1000 nm more seriously, due to the displacement drift induced by the thermal drift [12].

3.3. Calibration of the measured hardness

To obtain extremely accurate hardness, Liu et al. have proposed a new method recently that renders measured hardness insensitive to thermal drift at lower $\dot{\varepsilon}_L$, which has been successfully demonstrated its validation through the nanoindentation results in the film materials

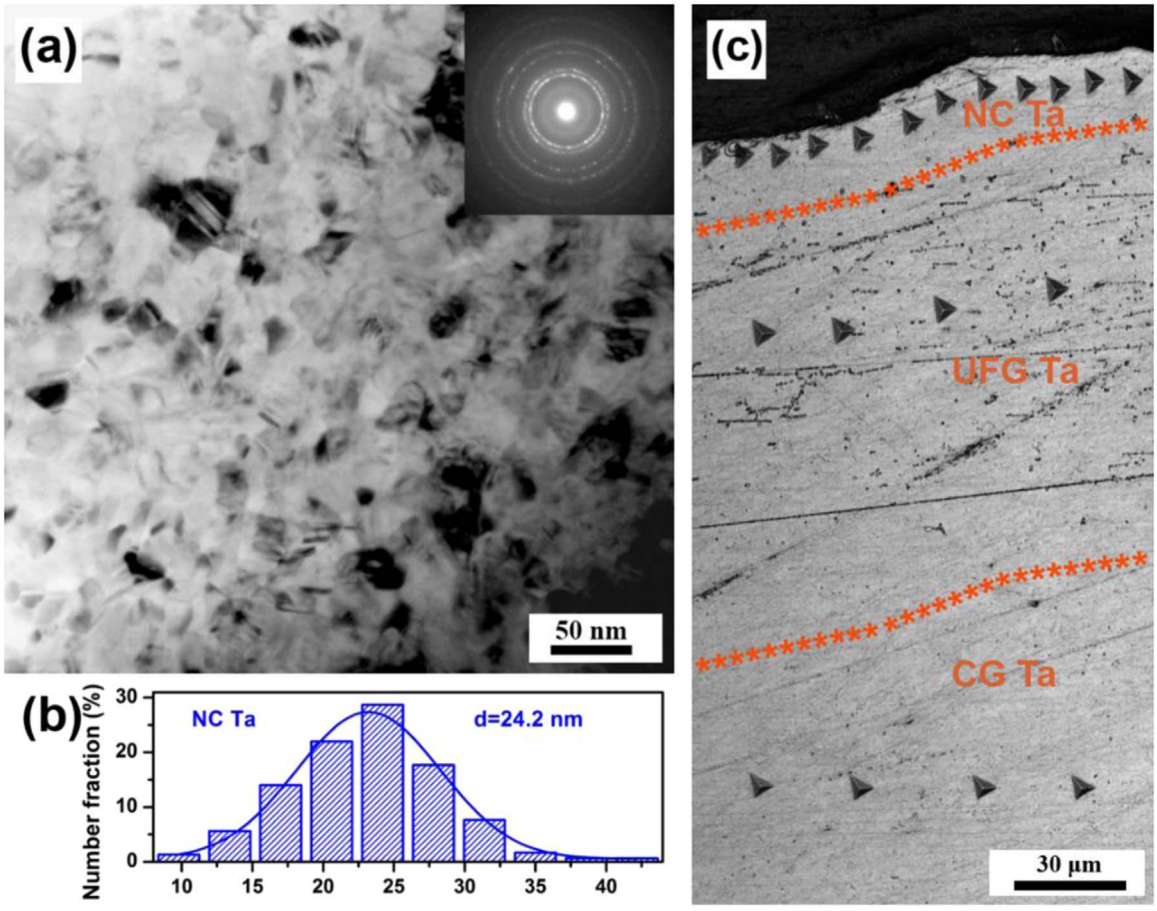

Fig. 1. (a) TEM bright-field images and the corresponding electron diffraction patterns (the inset) of the SFT NC Ta. (b) The grain size distribution of the NC Ta. (c) Residual impression in the NC, UFG (ultrafine-grained) and CG Ta.