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Grain size dependent strain rate sensitivity in nanocrystalline body-centered cubic metal thin films

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ABSTRACT

The strain rate sensitivity (m) and activation volume (v^*) of three nanocrystalline (NC) body-centered cubic (bcc) metals, i.e., W, Mo and Ta, with various grain sizes were evaluated by nanoindentation testing. Opposite to the conventional trend that NC bcc metals exhibit reduced m as the grain size was decreased, elevated m was observed as the grain size was reduced from ~90 nm to ~30 nm for all the samples concerned. It was proposed that the unusual variation trends of m for NC bcc metals were dominated by GB-related mechanisms when the grain size drops below a critical value.

1. Introduction

Because of the confined nanoscale intragranular structure and enhanced grain boundary (GB) activities, the mechanical properties of nanocrystalline (NC) metals have been extensively studied in recent years [1–3]. For NC metals, GB-mediated mechanisms such as GB sliding, Coble creep and GB rotation played a key role during plastic deformation. The strain rate sensitivity m and apparent activation volume v^* of a NC metal were two crucial parameters that could shed light on its rate controlling deformation mechanisms during plastic deformation. Decreasing grain size led to increasing volume fraction of GBs, which would change dramatically m and apparent v^* relative to their bulk counterparts. Whilst existing studies concerning the variation trends of m and v^* focused mainly on face-centered cubic (fcc) metals, few concerned NC metals having body-centered cubic (bcc) lattice structures.

For NC fcc metals, e.g., Cu [4–6], Al [7,8] and Ni [9,10], m increased with decreasing grain size as shown in Fig. 1a. The enhancement was attributed to highly localized dislocation activities, such as dislocation nucleation from and/or dislocation de-pinning at GBs [9,11,12]. In contrast to fcc metals, as shown in Fig. 1b, bcc metals (e.g., Fe [13,14], Ta [4,15],W [16] and V [17]) exhibited a considerably reduced m as the grain size was reduced from coarse to ultra-fined or even nanoscale grains [4]. It was proposed that, as the grain size was reduced, the low mobility of screw dislocations played a crucial role for the reduced m [4]. However, a few exceptions opposing the reducing trend of m in bcc metals were also reported: for instance, an unusual enhanced m was observed in ball-milled Fe powder particles when the grain size was reduced to nanoscale [18]. Apparently, the variation trend of m and related deformation mechanisms in bcc NC metals needed to be explored further.

In the present study, the strain rate sensitivity and apparent activation volume of NC W, Ta and Mo thin films were examined under nanoindentation testing. Special focus was placed upon the contribution of elevated volume fraction of GBs and confined nanoscale granular structure to the plastic deformation of the selected NC bcc metals.

2. Experimental

A series of W, Ta and Mo thin films (total thickness 1 μm) were deposited on Si (100) substrate by *d.c.* magnetron sputtering. The base pressure prior to sputtering was 6.3×10^{-5} Pa and Ar pressure during sputtering was 5.4×10^{-1} Pa. The deposition rates were 5 nm/min for W, 9 nm/min for Ta, and 7 nm/min for Mo films. In addition, another Mo film with much smaller grain size was deposited by hundreds short depositions separated by dwell intervals of 100 s as described in Ref. [19]. All the specimens were annealed at temperature ranging from 573 K to 1073 K for 30 min. The microstructures of the samples were investigated by means of X-ray diffraction (XRD), transmission electron microscopy (TEM, JEM200CX) and high resolution TEM (JEOL 2100F) operating at 200 kV. All the TEM foils were prepared via Gatan Precision Ion Polishing System 691 using Ar ion.

Indentation experiments were performed using a dynamic contact module device equipped with the Nanoindenter XP system

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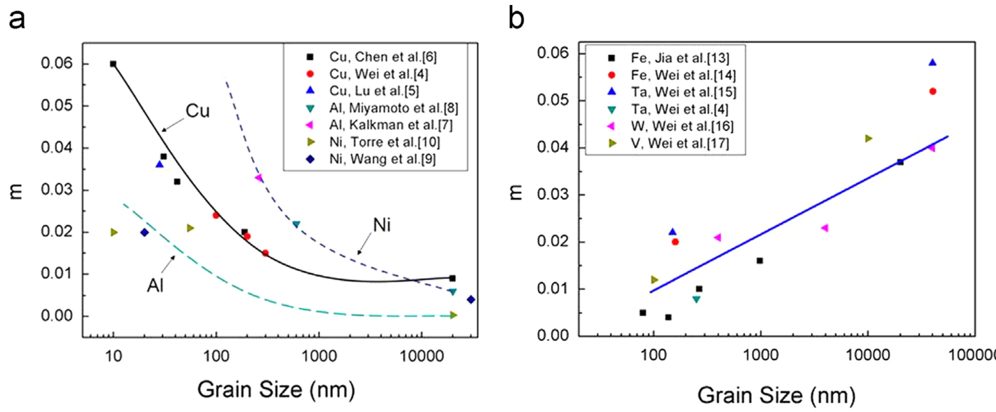

Fig. 1. Strain-rate sensitivity of (a) fcc metals and (b) bcc metals summarized using data from existing literatures.

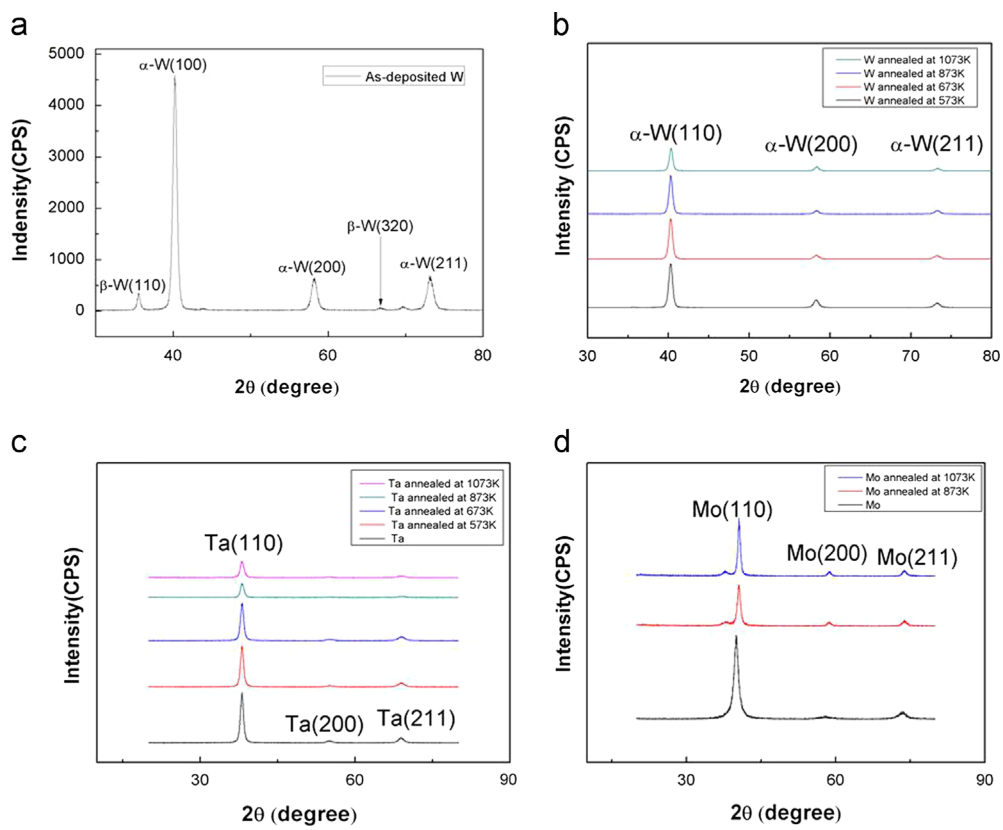

Fig. 2. X-ray diffraction scan for (a) as-deposited W films, (b) annealed W films, (c) Ta films and (d) Mo thin films.

(MTS, Inc.), under continuous stiffness measurement (CSM) mode at room temperature. Upon calibration on standard fused silicon, the tip of the Berkovich diamond indenter was estimated to have a radius of ~50 nm. The hardness of the prepared thin films was examined using depth control mode at varying loading strain rate (LSR) from 0.001 to 0.2 s^{-1}, and the LSR $\dot{\varepsilon}$ was given by [20]:

$$\dot{\varepsilon} = \frac{1}{h}\frac{\partial h}{\partial t} \tag{1}$$

where h was the indentation penetrating depth and t was time. The maximum penetration depth for all the indentation tests was set at 200 nm; and the hardness measured by CSM mode was calculated by averaging the hardness values obtained by varying the penetration depth from 100 to 150 nm to avoid the substrate effect. After the indenter reached the prescribed depth limit, the indenter was unloaded to 10% of the maximum load and held for thermal drift correction before the indenter was withdrawn from the sample surface to terminate the indentation test. At each LSR for every sample, at least 15 effective indentation tests were conducted and used for subsequent analysis.

3. Results and discussion

XRD analysis shown in Fig. 2a indicated that whilst peaks of both α-W (bcc structure) and β-W (A15 cubic lattice) existed in as-deposited W films, only α-W appeared in annealed W films as shown in Fig. 2b. Therefore, in subsequent analysis, we only examined the annealed W films. In addition, the XRD patterns shown in Figs. 2c and d suggested that both Ta and Mo films had bcc structure.