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Depth dependent strain rate sensitivity and inverse indentation size effect of hardness in body-centered cubic nanocrystalline metals

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ABSTRACT

Size effects on hardness (H) and strain rate sensitivity (m) of nanocrystalline (NC) body-centered cubic Mo thin film were examined under nanoindentation testing. Contrast to existing reports that there was no indentation size effect on hardness in NC metals, inverse *indentation size effect* (ISE) in NC Mo was observed for the first time at penetration depths ranging from 15 to 200 nm, at all the loading strain rates applied. In addition, the strain rate sensitivity of NC Mo exhibited strong dependence on penetration depth, increasing dramatically with decreasing penetration depth. Surface effects related to two deformation mechanisms were proposed to be responsible for the observed inverse ISE on H and depth dependent m. Specifically, the mobility of screw dislocation/component and the diffusion length of interfacial diffusion were altered as the deformed region underneath the indenter was approaching the free surface, resulting in the unusual size effects in NC Mo.

1. Introduction

Indentation size effect (ISE), i.e., indentation hardness increasing with decreasing penetration depth, has been observed and well documented in the past two decades for a wide range of materials. Despite that ISE was considered in the earlier years as an artifact caused by either sample surface preparation or measurement error, both the Taylor dislocation model [1] and the model related to geometrically necessary dislocations within the deformed region [2] have been widely applied and accepted in interpreting specific size effect in indentation testing. However, ISE was only observed in single crystal and coarse grained metals and only few studies reported that nanocrystalline (NC) metals exhibit ISE during indentation [3]. The absence of ISE in fine-grained polycrystals (NC metals in particular) if the indentation size is comparable to or exceeds grain size is attributed to the fact that grain boundaries are strong barriers for dislocations [4], restricting the aforementioned two dislocation related models to operate in NC metals.

While significant efforts were devoted to characterizing ISE in face-centered cubic (fcc) metals, few concerned body-centered cubic (bcc) metals. Due to different dislocation mechanisms, the plastic deformation behavior of bcc metals is quite different from fcc metals. Unlike fcc metals, as a result of non-planar core structure and the associated high Peierls barrier, dislocation movement in bcc metals occurs on various slip systems and screw dislocations are slower than edge dislocations below critical temperature [5]. Moreover, it has been indicated that, opposite to trend observed in fcc metals, the strain rate sensitivity of bcc metals decreases with decreasing grain size [6]. The high strain rate sensitivity in coarse grained bcc metals was attributed to kink-pair assisted motions of screw dislocations. In contrast, the density of screw dislocations is quite low in NC bcc metals, results in their low strain rate sensitivity. In addition, Kaufmann et al. [7] indicated recently that surface effect could significantly enhance screw dislocation mobility in bcc metals, and surface effect may attribute to ISE significantly in a certain way [4]. Therefore, upon indentation testing, the penetration depth dependent hardness may be quite different from the ISE reported previously. This may therefore provide further insights to the physical mechanisms underlying the plastic deformation behavior of near-surface layers in crystalline metals.

In the present study, the penetration depth dependent hardness and strain rate sensitivity of NC Mo thin films were investigated by nanoindentation testing. Intriguingly, at all the loading strain rates applied in indentation hardness testing, inverse-ISE was observed and the strain rate sensitivity of hardness increased dramatically with decreasing penetration depth in the NC Mo. It was proposed that surface effects affecting the mobility of screw

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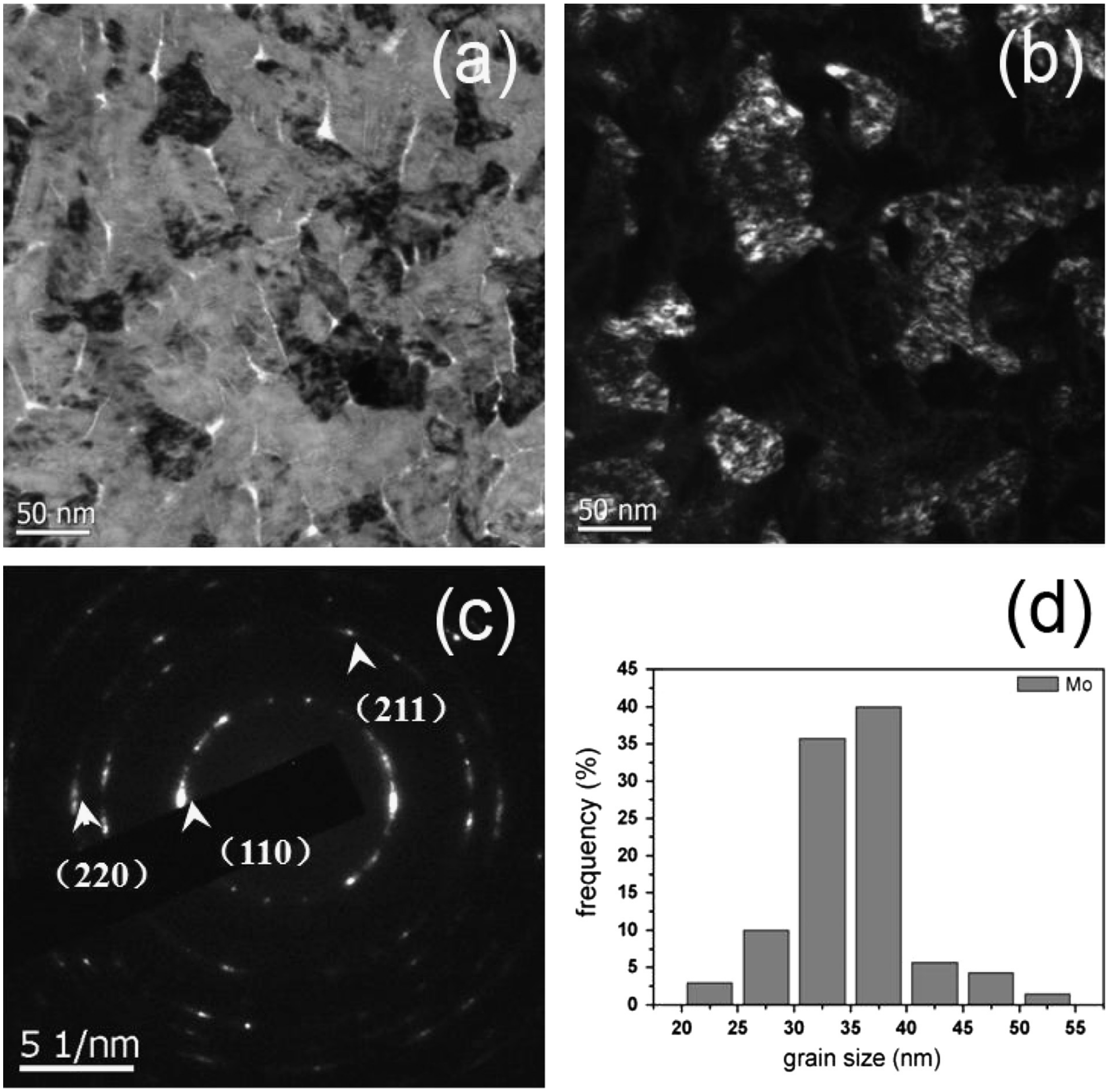

Fig. 1. . (a) Typical plan-view TEM bright-field image of NC Mo thin film; (b) corresponding dark-field image of (a); (c) electron diffraction (SAED) pattern of (a) and (b); (d) grain size distribution in NC Mo.

dislocation and the diffusion path along the indenter-specimen interface were the dominant mechanisms underlying the unusual size effects observed.

2. Experiments

2.1. Sample preparation and microstructure characterization

NC Mo thin film with thickness of 1350 nm was deposited via *d.c.* magnetron sputtering on a single silicon wafer (100) using a high purity target Mo (purity of 99.999%) under argon pressure of 0.3 Pa at room temperature. Transmission electron microscopy (TEM) and high resolution TEM (HRTEM) observations were carried out by using a JEOL 2100F microscope (Tokyo, Japan) under accelerating voltage of 200 kV.

Fig. 1(a) and (b) presented the plan-view bright-field and dark-field TEM micrographs of the Mo film, while Fig. 1(c) displayed the corresponding electron diffraction patterns. The average grain size of the Mo film was estimated to be about 34.8 nm, with relatively narrow grain size distribution.

Fig. 2 displayed the HRTEM images of the lattice structure within a typical grain in the Mo film, in which the corresponding zone axis is [001]. As shown in the magnified figure of Fig. 2(b), four dislocations could be identified, i.e., full dislocations of $1/2[\bar{1}\,\bar{1}1]$(h, n) and $1/2[11\bar{1}]$(k, m). Clearly, the Burgers vectors were neither parallel nor perpendicular to the dislocations. Therefore, the observed four dislocations in Fig. 2 were all mixed dislocations having both edge and screw components. In addition, the density of dislocations in the grain shown in Fig. 2(b) was estimated to be about $\sim 10^{16}\ m^{-2}$.

2.2. Nanoindentation testing

Nanoindentation tests were performed on MTS Nanoindenter XP system (MTS, Inc) under Continuous Stiffness Measurement (CSM) mode at ambient temperature, with the applied loading strain rate (LSR) ranging from $0.005\ s^{-1}$ to $0.2\ s^{-1}$. After the