



Understanding the mechanical behavior of nanocrystalline Al–O thin films with complex microstructures

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Abstract

The mechanical behavior of nanocrystalline (NC) metals has attracted widespread interest, though the majority of efforts have focused on (nominally) pure metals. By comparison, the mechanisms of deformation and strengthening in NC alloys, especially those with high segregation propensity and strong chemical interactions, are less well understood. Here we present a quantitative investigation on the mechanical behavior of such an alloy system. NC Al–O thin films are synthesized by means of confocal co-sputtering, which enables a wide-range and quasi-independent control over impurity content and grain size. Detailed characterization combining transmission electron microscopy with three-dimensional atom probe tomography identify the multiple morphologies of O impurities in a composite-like microstructure, including nanosized α -Al₂O₃ precipitates, O-rich clusters segregated along grain boundaries, and O solute atoms inside Al grains. Individual contributions of these strengthening features to the mechanical properties of NC Al–O thin films, as measured by instrumented nanoindentation, are then well delineated by a microstructure-informed analytical model. Dislocations emitted from grain boundaries are pinned by the stronger obstacles and cut through the weaker, and we show that the strong chemical interactions of this Al–O system play a dominant role in its pronounced strengthening capability. The influence of O impurities on the plasticity and deformation mechanisms in NC Al films is also discussed based on microtensile testing.

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1. Introduction

Nanocrystalline (NC) metals have attracted widespread interest due to their outstanding mechanical behavior including high strength [1–4], improved flaw tolerance [5,6], and high resistance to corrosion [7], fatigue [8] and wear [9]. However, the mechanisms governing plastic

deformation and strengthening in NC metals have not been fully explored on a quantitative level. While some experiments have shown that the conventional Hall–Petch (H–P) relationship can be extrapolated from the coarse- and ultrafine-grained regime to grain sizes as small as 10–20 nm [2,10–12], more recent studies by means of in situ testing [6,13–23] and atomistic simulations [10,24–29] have underlined the emergence of new deformation mechanisms at reduced size scale. These include twinning/detwinning [13,22], dislocation emission/absorption at grain boundaries (GBs) [14,21,24,26], GB sliding [10,27–29],

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grain rotation [15,25], GB migration [16,27] and stress-coupled grain growth [15–20], which provide new prospects for tailoring the mechanical properties of NC metals.

However, most studies to date have focused on (nominally) pure NC metals. As a classic strengthening strategy in bulk structural materials [30], alloying is also expected to play a prominent role in governing the microstructure and mechanical properties of NC metals. It has been recognized that GB migration can be retarded by solute atoms, either through the kinetic drag effect [31] or by the reduction of GB energy due to solute segregation along GBs [32,33]. A more refined model to predict the thermodynamic stability of NC alloys based on the free energy of mixing was recently proposed by Schuh et al. [34,35]. Consequently, NC alloys can be adequately stabilized against thermal grain growth, thus retaining their fine grain size and concomitant high strength [36,37]. The mechanical stability of NC alloys [38,39], in contrast, has received much less attention despite many reports of stress-driven grain growth in (nominally) pure NC metals [15–20]. In one study, Tang et al. confirmed the segregation of O atoms at GBs of NC Al thin films by using three-dimensional atom probe tomography (3D-APT) [40], and showed that the GB excess of O increased with the base pressure of sputtering deposition [41]. Additions of O also led to the suppression of stress-driven grain growth, accompanied by a transition of tensile behavior of the freestanding NC Al thin films [18] from “ductile and moderately strong” to “very strong and brittle”, which qualitatively corroborated atomistic simulations by Elsener et al. [42] showing that the critical stress for coupled GB migration increased with the GB excess of impurity atoms. Beyond these studies of nanostructural stability, the applicability of classic strengthening models (i.e. based on dislocation–obstacle interactions) in NC alloys remains an open question. To this end, Rupert et al. [43] recently proposed a new mechanism for solid–solution strengthening of NC alloys. By combining the classic Fleischer model [44] with the GB-pinning effect of dislocations, which was augmented by a modification of global lattice properties due to alloying, the strengthening behavior of their Ni–W alloys as well as several other NC alloys were well described.

Experimentally, alloy strengthening of NC thin films has mainly focused on metal–metal binary systems including Cu–Fe [45,46], Cu–Nb [46], Ni–Cu [45], Ni–W [11,44], Al–Fe [47] and Al–Mo [48]. Taken as a whole, the effectiveness of strengthening was found to increase with segregation energy and the size misfit between solute and solvent atoms [49]. However, the strengthening behavior of NC alloy systems with both high segregation propensity and strong chemical interactions, such as Al–O [42] and Ni–P [50], is still far from understood. Systematic investigations of this type of NC alloys have special significance due to their extraordinary effectiveness for grain size refinement [34] and strengthening, thus serving as promising candidates for applications such as wear-resistant coatings [51,52] and oxide-dispersion-strengthened alloys [53,54].

On the other hand, quantitative modeling of the impurity strengthening mechanisms in these complex microstructures also faces special difficulties, as the cooperation between the multiple strengthening features, including but not limited to solute atoms, precipitates and GBs, requires a thorough characterization of both spatial distribution and chemical states of the impurities. However, atomistic simulations are of limited assistance in this regard since accurate interatomic potentials with strong charge-transfer characteristics are still relatively immature [42]. In addition, convolution of the strengthening effects coming from impurity and grain size, a common and elusive issue in NC alloys [45,48], also needs to be addressed. Accurate control of impurity content through film deposition is another practical issue (at least for the Al–O system), since the previous route of changing base pressure [41] showed only limited control of impurity content and species.

In the present study, NC Al–O thin films were synthesized by means of co-sputtering, which enables wide-range and quasi-independent control over impurity content and grain size. By using transmission electron microscopy (TEM) combined with 3D-APT, the multiple morphologies of O are identified in a complex composite-like microstructure, and their strengthening contributions to the mechanical properties of NC Al–O thin films, as measured by instrumented nanoindentation, are distinguished based on an analytical model. The influence of O impurities on the deformation mechanisms of NC Al films is also discussed based on microtensile testing.

2. Materials and methods

NC Al thin films with varied O content were synthesized by magnetron co-sputtering in an Explorer 14 sputtering system (Denton Vacuum). A 99.999% pure Al target and a 99.995% pure α -Al₂O₃ target were configured in confocal geometry, and the chamber stage was rotated to maintain uniformity of film thickness and composition. The Al target was sputtered by DC powers of 200 and 300 W, whereas the RF power on the α -Al₂O₃ target was varied from 0 to 150 W, yielding 12 deposition batches with 8 different DC/RF power combinations: 200/0, 200/10, 200/50, 200/100, 300/0, 300/15, 300/75 and 300/150. Hereinafter, all batches are referred to by their DC (the first number) and RF (the second number) powers in Watts. For each batch, NC Al–O thin films were deposited on Si(100) with the native oxide layer intact [55], fused silica glass and Cu TEM grids with supporting C films. The chamber was evacuated to $\sim 4 \times 10^{-7}$ Torr and then filled with Ar gas flow at 25 sccm. Deposition was performed at a chamber pressure of $\sim 5 \times 10^{-3}$ Torr. Deposition rates (for each DC power) were calibrated beforehand so that similar film thicknesses (*t*) of 160–180 nm were achieved for most batches.

For each batch, global O content (C_O) was measured in the films deposited on Si(100) by using energy-dispersive spectroscopy (EDS) in an FEI Quanta 600 scanning electron microscope (SEM) operated at 10 kV. Plan-view

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