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Discerning size effect strengthening in ultrafine-grained Mg thin films

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Microtensile experiments have been performed to elucidate the mechanical response of ultrafine-grained Mg thin films. Strengths of 160 MPa and elongations up to 8% were measured. Post-deformation electron microscopy indicates a lack of intragranular dislocation confinement. While strength does increase with decreasing grain size, the size effect for hexagonal Mg is not as strong as that reported for face-centered cubic metals. Strength appears to be governed by a lack of dislocation pile-up as well as texture and Peierls effects.

Keywords: Size effect; Mechanical properties; Hall–Petch strengthening; Hexagonal; Thin films

Due to its low density, magnesium has garnered much attention as a potential metal for lightweight aerospace and automotive components [1]. Reductions in grain size should benefit the design of Mg-based alloys by providing Hall–Petch [2,3] strengthening and potentially activating grain-boundary (GB)-mediated mechanisms that could enhance deformability [4]. Size effects at the submicron level have been widely studied for face-centered cubic (fcc) metals but investigations of hexagonal close-packed (hcp) systems are still relatively nascent. Work on Ti pillars by Norfleet [5] measured a weak size effect in the micron regime, suggesting that the confinement of dislocations provided less stress enhancement than the Peierls stress [6,7] needed to move dislocations through the lattice. A similar trend was found in body-centered cubic (bcc) metals [8,9] with the strong Peierls friction metals exhibiting lower size effects. This work seeks to elucidate the size-specific mechanical properties of ultrafine-grained (ufg) Mg free-standing thin films.

Test specimens were prepared by depositing Mg onto a Si platform patterned with a tensile geometry following the process outlined in Refs. [10] and [11]. The films were synthesized using electron beam evaporation from a 99.999% pure source. The deposition was pulsed; at a base pressure of 4×10^{-7} Torr, six 33 nm deposition steps each separated by 1 min dwells were repeated to build a nominally 200 nm thick film. Measurements with a stylus tip profilometer revealed that the actual film thickness was 250 ± 10 nm. Prior to testing, the films were made free standing by removing the Si under the gauge region with a dry gas etch of XeF_2. Mechanical testing was conducted using a small-scale tensile apparatus described in Ref. [12]. The system was outfitted with a 10 g load cell and the experiments were displacement controlled using a linear actuator with 30 nm step resolution. Specimens were gripped using UV-curable adhesive, and alignment prior to permanent bonding was performed optically with a stereoscope, adjusting the sample position with a five-axis motorized stage. All tensile tests were conducted at an initial specified strain rate of $5 \times 10^{-4}\ s^{-1}$. Strain was measured using digital image correlation processed with a MATLAB®-based code [13].

Analysis of the as-deposited Mg by transmission electron microscopy (TEM, Phillips 420) found the grains to be essentially free of dislocations and growth twins. The area-weighted as-deposited grain size was 197 nm with an 89 nm standard deviation, putting the Mg in the ufg regime. An amorphous oxide has been reported to form on scratched Mg films [14] and the Mg of this

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study did have some small amorphous pockets, most likely oxide, randomly dispersed amongst the grains. As Mg is anisotropic [15], an out-of-plane orientation map to discern texture was measured using a Phillips CM20 field emission gun transmission electron microscope equipped with a NanoMEGAS orientation mapping unit. A 4 μm × 4 μm scan, given in Figure 1a, shows the orientation distribution of the grains. The 0001 pole figure (Fig. 1b), which reveals the basal texture, indicates that ~9.5% of grains are positioned with their basal plane parallel to or within 10° of the film surface.

The stress–strain responses from five tensile experiments are recorded in Figure 2. All specimens exhibited similar behavior; each yielded at around 131 MPa, reached a peak stress near 160 MPa, and then strained to failure without significant hardening. Elongation at fracture varied from 3% to 8%. Specimens A and B failed prematurely and this was attributed to an undetected edge flaw. The other specimens failed via necking with local strain levels of the order of 13–15% inside the neck.

Post-fracture TEM analysis was conducted and a representative image of the deformed Mg is provided in Figure 3. Only a very small fraction of the grains contained dislocations. Experiments and simulations suggest that small grain metals exhibit GB-mediated flow mechanisms such as GB dislocation emission [16,17], sliding [18] or coupled migration [19]. The area-weighted grain size of the deformed Mg was 188 nm with a 76 nm standard deviation. While smaller than the as-deposited grain size, in statistical terms no refinement occurred. As coarsening due to GB coupling is not detected, deformation is most likely occurring through dislocation or twin emission and absorption at GBs. Simulations on small-grain Mg also suggest this deformation mode with dislocation and twin emission reported to occur at GBs [20,21]. Deformation twins were found in a small fraction of the grains. The observed twinning system was (10–11) <10–12> (see Fig. 3 inset) which occurs in crystallites whose c-axis contracts. This compression twinning (CT) is recognized in hcp systems [22] and has been reported in single-crystal Mg experiments on bulk [23] and nanoscale [24,25] specimens but is absent in micron-size samples [26,27]. Tensile twinning (TT), which occurs when the c-axis of a grain extends, was not widely detected. GB sliding was not confirmed here but other studies [4,28] suggest that this mechanism may be active.

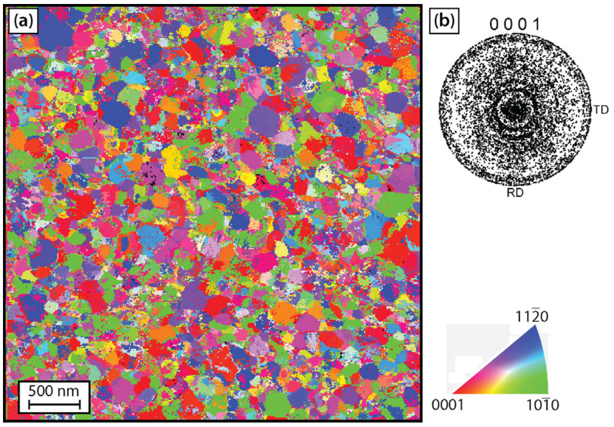

Figure 1. (a) Out-of-plane orientation map of as-deposited Mg with (b) corresponding 0001 pole figure.

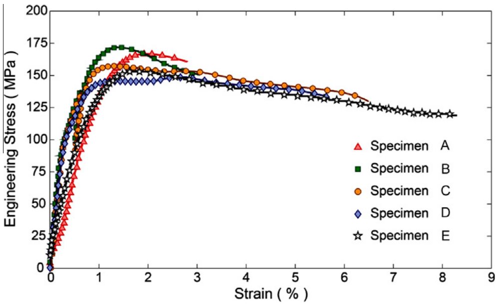

Figure 2. Tensile response data for 250 nm thick free-standing Mg thin films.

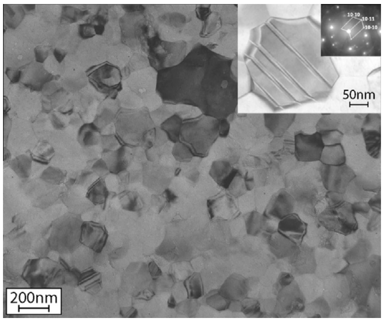

Figure 3. Representative bright-field TEM image of deformed Mg with image of a compression twin inset.

A Hall–Petch plot (Fig. 4a) was assembled using data extracted from published studies [28–33] to see how yield strength (σ_y) varied with the inverse square root of grain size (d). Mg appears to follow the Hall–Petch trend until d = 1 μm where, as noted by Choi et al. [28], a decrease in the strengthening slope occurs. Curiously, σ_y for the present study is exceptionally low as some of the reference data for grain sizes larger than 1 μm have higher yield strengths.

To better understand why such a low yield stress is measured in the ufg Mg, the overarching Hall–Petch constructs were first considered. The strengthening is chiefly based upon dislocation pile-up, and in ufg Mg there is a clear lack of intragranular dislocation storage (see Fig. 3). If no pile-up occurs, the strengthening trend is not likely to be followed. Furthermore, Hall–Petch assumes a $\sigma_y \propto d^{-0.5}$ relationship which may not be valid [34]. If d vs. σ_y is plotted as in Figure 4b, it is seen that smaller grains are associated with higher yield points, but the breakdown of the strengthening slope is not as evident. A power-law fit to the individual data sets reveals that the size effect ranges from $d^{-0.1}$ to $d^{-0.35}$ which is less than $d^{-0.5}$ and also less than the $d^{-0.6}$–d^{-1} size effect found in fcc single-crystal experiments [35]. Three fits are provided in Figure 4b and fits for all data sets are denoted in the legend. The values are in line with those measured for bcc metals [8,9] from single-crystal pillar experiments by Schneider et al., who