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Size-Induced Strengthening in Nanostructured Mg Alloy Micropillars

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Size effects on the compressive strength of nanostructured Mg-micropillars were investigated. Mg–10Al alloy micropillars with diameters ranging from 1.5 to 8 μm were prepared by focused-ion-beam and tested under micro-compression. A significant improvement in strength was found by reducing the pillar diameter $<3.5 \mu\text{m}$. The deformation mechanisms of the compressed pillars were characterized using transmission electron microscopy. We attribute the size-induced strengthening to a less number of dislocation sources along with a higher activity of non-basal deformation mechanisms.

Keywords: Nanostructured Mg Micropillars, Micro-compression Tests, Size Effect, Transmission Electron Microscopy

Studies on size-scale effects i.e. grain size and specimen dimension have attracted considerable attention in recent years because of their influence on the mechanical behavior of materials.[1] In general, it is well accepted that the strength of metals can be improved through refinement of the grain size by the Hall–Petch mechanism. Interestingly, it has also been demonstrated that the strength of the material can be improved by reducing the specimen dimensions, as first reported by Uchic et al. [2] in Ni-based superalloys and by Greer et al. [3] in single crystal Au.

Nowadays, the optimization of the mechanical properties of materials at the micron and submicron scales is crucial for the design of high-performance micro-electro-mechanical devices. Consequently, researchers have focused on investigating both grain size and specimen size effects on the mechanical behavior of micron/submicron-pillars of polycrystalline materials such as Ni,[4,5] Ag,[6] TiAl,[7] Cu,[8] and Pt.[9]

In the case of Mg, many efforts have been dedicated to study the compressive behavior of single crystal Mg micron/submicron-pillars.[10–15] These studies have shown that the compressive strength of single crystal Mg-micropillars mainly depends on the initial dislocation density [11] and the orientation of the basal planes with respect to the loading direction (LD).[10–12] Byer and Ramesh [11] reported a size effect on the yield strength by decreasing the initial dislocation density. Lilleodden [12]

observed a remarkable increase in compressive strength along the *c*-axis for single-crystal Mg-micropillars with diameters $<10 \mu\text{m}$. It is well known that when the *c*-axis is nearly parallel or perpendicular to the LD, alternative deformation mechanisms such as non-basal slip and/or twinning can be activated since the Schmid factor for basal plane sliding is very small. For instance, a significant hardening was found on single-crystal Mg-micropillars loaded in compression along the *c*-axis by the activation of multiple slip systems on pyramidal planes.[10,12] However, it is interesting to note that no sign of twinning is found in those works. It appears that a size effect is also associated with the deformation by twinning. As reported by Yu et al.,[15] the deformation by twinning becomes unfavorable when the specimen size decreases. Ye et al. [13] demonstrated a strong size effect associated with basal plane sliding and extension twinning in single-crystal Mg nano- and micropillars. In fact, it has recently been demonstrated by Prasad et al. [14] that the required stress to nucleate twins in single crystal Mg-micropillars is higher than that in single crystal Mg bulk samples.

While many efforts have been devoted to study the size effect on single-crystal Mg micropillars, the effect of specimen size on polycrystalline Mg micropillars has not been reported yet. In this work, we report the influence of specimen size (diameter) on the compressive strength of nanostructured Mg–10Al micropillars with a

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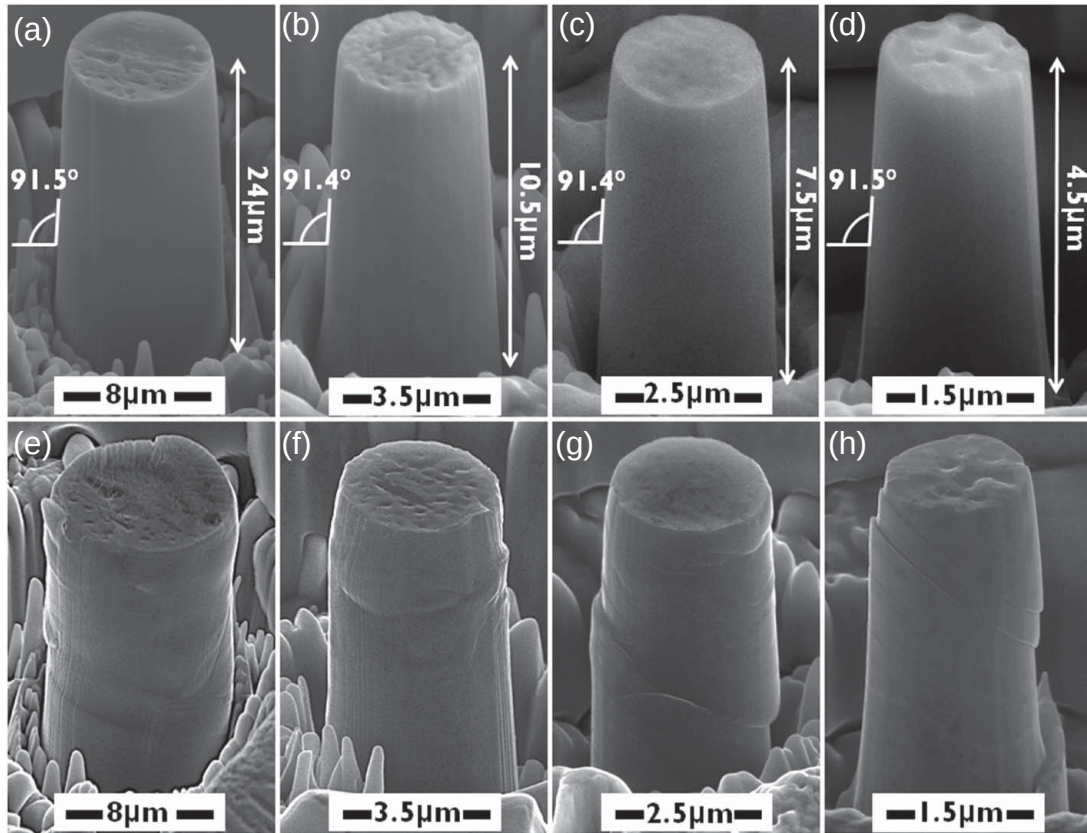


Figure 1. SEM images of nanostructured Mg–10Al micropillars with different diameters (a)–(d) before and (e)–(h) after compression (2% of compressive strain).

bimodal grain size distribution. Using transmission electron microscopy (TEM) we investigate the underlying deformation mechanisms.

Nanocrystalline Mg₉₀Al₁₀ (wt.%) powders were prepared by cryomilling for 8 h under liquid nitrogen atmosphere in a Union Process, Szegvari mill at California Nanotechnologies. Cryomilled powders were consolidated using a spark plasma sintering (SPS) system (Syntex Inc., Dr. Sinter Lab TM, model SPS-515S). The sintering process was carried out in vacuum for 5 min at 400°C and under uniaxial pressure of 100 MPa. More details about the material processing can be found in Refs. [16,17] Mg–10Al micropillars with diameters ranging from 1.5 to 8 μm and aspect ratio of 1:3 were fabricated by focused ion beam (FIB). A FEI Nova600 Nanolab Dual-Beam FIB-scanning electron microscope (SEM) operated at 30 kV was used for this purpose. A series of concentric annular milling patterns with different currents were applied. In order to tailor the pillars into the desired shape and minimize the tapering, [18,19] a low beam current (<1 nA) was used as final milling step. Uniaxial micro-compression tests were performed using a MTS Nanoindenter XP. A diamond indenter with a flat-end tip of 10 μm in diameter was used. The strain rate was controlled in the range of 3×10^{-3} and 5×10^{-4} (s⁻¹). TEM samples of the compressed pillars were prepared by

FIB and examined using a FEI-Titan TEM operating at 300 kV.

Four characteristic Mg–10Al micropillars before and after compression are shown in Figure 1. Figure 1(a)–(d) show 52° tilted-view SEM images of those micropillars with top diameters of 8, 3.5, 2.5 and 1.5 μm, respectively, before compression. It is evident to observe that all pillars are slightly tapered as a result of the annular cutting method by FIB. The sidewall taper angle and pillar length are indicated in all images. We measure taper angles $\leq 1.5^\circ$, which are smaller than the values reported in the literature (2–5°) for typical micropillars machined by FIB. [20,21] Figure 1(e)–(h) show 52° tilted-view SEM images of the compressed pillars up to 2% of strain. Shear bands are apparent on the pillars surface, especially for pillars with diameters ≤ 3.5 μm. In contrast, 8 μm pillars plastically deform showing a barreling shape similar to the bulk samples under compression. This observation suggests that a transition from homogeneous-like to shear-band deformation occurs by reducing the pillar diameter.

The results of the micro-compression tests are shown in Figure 2 and presented in Table 1. At least five samples were tested for each pillar diameter as shown in Table 1. The average yield strength and the standard deviation have also been included. In order to avoid any

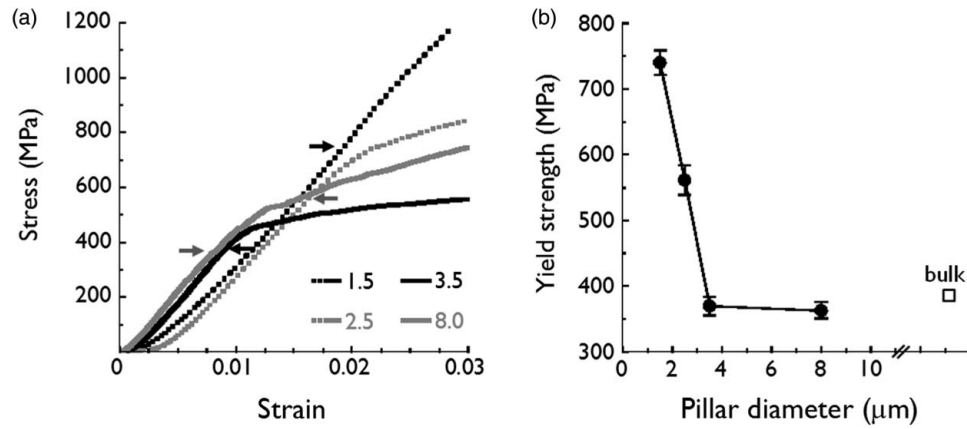


Figure 2. (a) Compressive stress–strain curves of nanostructured Mg–10Al micropillars. (b) Compressive yield strength as a function of pillar diameter.

Table 1. Compressive properties of nanostructured Mg–10Al micropillars with different diameters.

Pillar diameter (μm)	Yield strength (MPa)	Strain at failure (%)	Average yield strength (MPa)	Average strain at failure (%)
1.5	742	2.4	746 ± 19	2.6 ± 0.2
	729	2.7		
	778	2.7		
	746	2.8		
	734	2.5		
2.5	577	3.6	554 ± 21	3.9 ± 0.3
	536	3.7		
	549	4.4		
	575	3.9		
	533	3.7		
3.5	347	4.2	364 ± 15	4.5 ± 0.2
	374	4.6		
	373	4.8		
	379	4.3		
	349	4.7		
8.0	373	4.8	362 ± 12	4.7 ± 0.3
	362	5.5		
	350	4.1		
	373	4.3		
	350	4.7		

overestimation of the measured stress due to the tapering, the stress was obtained using the load divided by the middle cross-section area of the pillars. Figure 2(a) shows representative engineering stress vs. strain curves for nanostructured Mg–10Al pillars with different diameters. From the linear slope of the stress–strain curve, we estimate the elastic modulus, E , ~50 GPa, which is in good agreement with that measured by nanoindentation with a Berkovich tip (52 GPa). The yield strength, which is determined as the end of the proportional limit of each curve is highlighted by an arrow.

Pillars with 8 and 3.5 μm diameters present similar yield strength values i.e. 373 and 374 MPa, respectively. It is interesting to note that the yield strength increases

significantly when decreasing the pillar diameter. In particular, the yield strength values for 2.5 and 1.5 μm pillars are 549 and 746 MPa, respectively. Therefore, an increase of around 50% and 100% is obtained when decreasing the pillar size to 2.5 and 1.5 μm, respectively. Additionally, the stress–strain curves of Figure 2(a) indicate a less ductile behavior for the smallest pillars as confirmed by their values of strain at failure in Table 1. Thus, in addition to a significant increase in strength, a reduction in ductility is observed for pillars with smaller diameter.

The trend of the yield strength as a function of pillar diameter can be better seen in Figure 2(b). These results suggest a strong size effect on the strength of nanostructured Mg micropillars with diameters smaller than 3.5 μm. In contrast, no size effect was observed when the diameters are higher than 3.5 μm. In fact, the compressive strength of micropillars with diameters higher than 3.5 μm is similar to that of the bulk sample (397 MPa).[17]

TEM analysis was performed in order to study the underlying deformation mechanisms during micro-compression. Figure 3(a) is a bright-field TEM image of a compressed 2.5 μm micropillar showing the characteristic bimodal grain size distribution after SPS sintering. As described in our previous work,[17] the bimodal grain size distribution consists of fine-grains ~35 nm and coarse-grains ~400 nm. No apparent texture was found. See Ref. [17] for more microstructural details. We identify at least three shear bands forming an angle with respect to the LD of ~70° (labeled 1 and 2 at the top of the pillar) and ~60° (labeled 3 at the bottom of the pillar). High-resolution TEM (HRTEM) image from the shear band 1 (Figure 3(b)) reveals extension twins on the characteristic {10 $\bar{1}$ 2} planes (highlighted by yellow solid lines) in a grain with the c -axis almost perpendicular to the LD. In fact, nanotwins of <1 nm widths can be observed. This has been confirmed by indexing its fast Fourier transform (FFT) (inset). Interplanar distances measurements indicate the misorientation relation between the parent

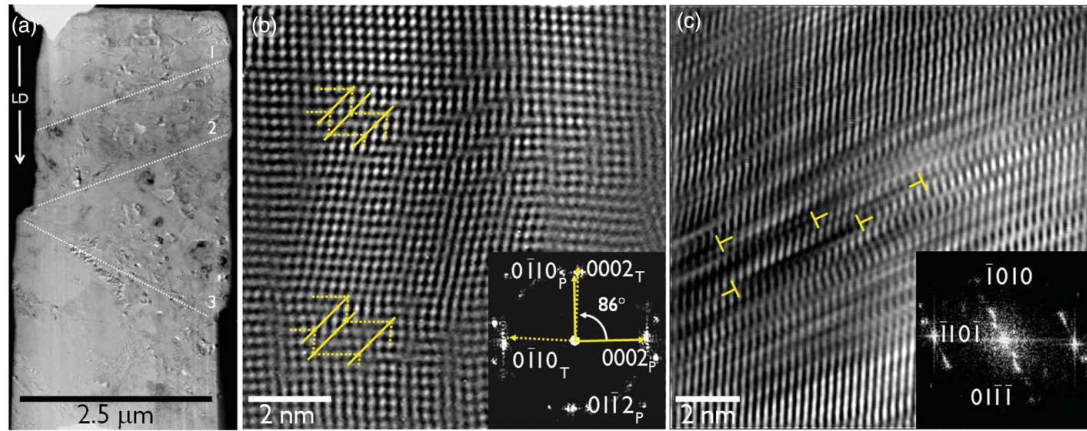


Figure 3. (a) Bright-field TEM image of a compressed 2.5 μm micropillar. (b) HRTEM image showing extension twins in a grain oriented to the $(2\bar{1}10)$ zone axis. Inset is its FFT. (c) HRTEM image showing prismatic dislocations terminated at SFs in a grain oriented to the $(1\bar{2}1\bar{3})$ zone axis. Inset is its FFT.

(solid lines) and the twin (dashed lines) close to the expected angle of 86° for a grain oriented to the $(2\bar{1}10)$ zone axis. Note that $\pm(0002)_T$ (basal planes from the twin) and $\pm(0002)_P$ (basal planes from the parent) are almost perpendicular to each other.

In addition to the twinning mechanism, we find prismatic dislocations (highlighted by $\perp$) terminated at stacking faults (SFs) in a grain close to the shear band 2 (Figure 3(c)). Inset is the indexed FFT of the image indicating the $\{1\bar{1}00\}$ planes where dislocations occur for a grain oriented to the $(1\bar{2}1\bar{3})$ one axis. The characteristic streaks parallel to the $\langle 1\bar{1}00 \rangle$ direction as a result of prismatic SFs are also apparent.

From the TEM analysis, we find that extension twinning and prismatic slip can be activated during compression of 2.5 μm nanostructured Mg-micropillars. While extension twinning is a common deformation mechanism seen in Mg bulk samples, higher stress is required to nucleate extension twins in micropillars.[14] In addition, it is well known that the critical resolved shear stress for prismatic slip and twinning is always higher than that for the basal slip.[22] Therefore, we believe that the enhanced strength observed in nanostructured Mg–10Al micropillars with 2.5 μm diameter is mainly due to the activation of the aforementioned deformation mechanisms.

From our previous work based on *in situ* SEM micro-compression studies of nanostructured Mg–10Al micropillars with 4 μm diameter,[23] we found in addition to basal plane sliding, contraction twins in a grain oriented with the c -axis almost parallel to the LD.

These results highlight the important role that the grain orientation plays in the strengthening of nanostructured Mg micropillars. As demonstrated by the TEM analysis of 2.5 μm micropillars, alternative deformation mechanisms such as non-basal slip and/or twinning can be activated in grains with the c -axis nearly parallel or perpendicular to the LD, since the Schmid factor for basal plane sliding is very small. These observations suggest

that the contribution from grains favorably oriented for basal slip (within a bimodal grain distribution randomly oriented without any preferential texture [17]) to the pillar strength decreases with decreasing pillar diameter. Additionally, by reducing pillar diameter the number of grain boundaries also decreases,[24] which significantly reduces the number of potential dislocation sources.[4] Hence, the applied stress required for nucleation or activation of dislocations, i.e. yield strength also increases. Based on this, we conclude that the size-induced strengthening is due to a smaller number of dislocation sources along with a higher activity of non-basal dislocation sliding and twinning.

In summary, we have demonstrated a strong specimen size effect on the compressive strength of nanostructured Mg–10Al alloy micropillars machined by FIB. We find that the yield strength increases significantly when the pillar diameter is $<3.5 \mu\text{m}$. An increase of around 50% and 100% is obtained when the pillar diameter decreases to 2.5 and 1.5 μm , respectively. In contrast, no size effect is observed for pillar diameter $\geq 3.5 \mu\text{m}$. We attribute the size-induced strengthening to a less number of dislocation sources along with a higher activity of non-basal dislocation sliding and twinning.

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