HALL-PETCH RELATIONSHIP AND STRAIN RATE SENSITIVITY OF NANOCRYSTALLINE Mg - 5WT% Al ALLOY

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Abstract: This study investigated the grain size dependence of mechanical properties and deformation mechanisms of microcrystalline (*mc*) and nanocrystalline (*nc*: grain size below 100 nm) Mg-5wt% Al alloys. The Hall-Petch relationship was investigated by both instrumented indentation tests and compression tests. The test results from the indentation tests and compression tests match well with each other. The breakdown of Hall-Petch relationship and the elevated strain rate sensitivity (SRS) of present Mg-5wt% Al alloys when the grain size was reduced below 58nm indicated the more significant role of GB mediated mechanisms in plastic deformation process. However, the relatively smaller SRS values compared to GB sliding and coble creep process suggested the plastic deformation in the current study is still dislocation mediated mechanism dominant.

Key words: nanocrystalline Mg alloy; Hall-Petch relationship; strain rate sensitivity, deformation mechanism.

1 INTRODUCTION

Generally, the grain size dependence of the activation of deformation mechanisms in polycrystals is described according to the Hall–Petch law (Bohlen et al., 2007). It describes the dependence of the yield strength σ on the average grain size d as $\sigma = \sigma_0 + k_{HP}d^{-1/2}$ where σ_0 is a friction stress for dislocation movement. The slope k_{HP}, called the ―Hall–Petch strength coefficient", depends on the orientation relation between the interacting grains as well as the critical shear stresses of the activated deformation modes in both grains. With decreasing grain size, the strength is increased due to the grain refinement strengthening mechanism. At some certain point, the positive Hall-Petch (H-P) relationship break down when the grain size reduces below a critical value. Up to now, most work on the deformation behaviour of *nc* metals and alloys have been focused on those with face-centered cubic (FCC) and body-centered cubic (BCC) crystal structures. Less attention has been paid to the *nc* alloys with a HCP crystal structure. How far this relation holds with decreasing grain size in *nc* HCP metals is less attended.

In the present study, a rate-change instrumented indentation method was used to examine the H-P relationship of *mc* and *nc* Mg-5 wt%Al alloys by precisely determining strength of materials at room temperature The advantages of instrumented indentation over other techniques lie in the precise load/displacement measurements with a nanoscale resolution and nearly defect-free deformation volume (Gouldstone et al., 2007; Pan & Chen, 2009). Also, the strength measurement is also carried out by conventional compression tests. The final results from the indentation tests and compression tests match well with each other. The breakdown of H-P relationship and elevated SRS suggests the change of deformation mechanism when the grain size is reduced below a critical value.

2 EXPERIMENTAL

Elemental powders of Mg (purity >98.5%) and Al (purity 99.5%) were used as starting materials for mechanical milling. The nominal composition of the composite is Mg-5 wt%Al. To prevent agglomeration and excessive cold welding of powders, 2–3 wt. % stearic acid was added to the powder mixture. To change the grain size, the powder mixture was mechanical milled under argon atmosphere for different durations (0, 10, 20, 30 and 40 h). After milling, the powders were cold-compacted and then sintered at 500 °C in an argon furnace for 2 h and hot-extruded to 7mm diameter rods. For simplicity, these rods were labeled using the milling durations, i.e., MA0 (as-blended), MA10, MA20, MA30 and MA40.

Nanoindentation tests were carried out on a Hysitron Triboscope with a Berkovich indenter. The load and displacement were recorded with a force resolution of about 1 nN and displacement resolution of about 0.2 nm (Bao, Xu, Li, & Li, 2010; Xiaodong, Xinnan, Qihua, & Eklund, 2005). Indentation tests were carried out at different loading rates (1 mN/s to 400 mN/s) to a maximum peak load at 200 mN. The uniaxial compression test was conducted using an Instron universal testing machine at room temperature over two magnitudes of strain rates, from 0.0001/s to 0.01/s. Cylinder specimens for compression test with 5 mm diameter and 10 mm in length with an aspect ratio of 2, according to ASTM Standard E9-09, (American Society for Testing and Materials International, 2009) were machined from the extruded bar along the extrusion direction. To check the repeatability of the results, 3~5 samples were tested for each condition. The variations in stress and hardness were within 5% in most cases.

The microstructures were observed using optical microscopy (OM) and transmission electron microscopy (TEM, Philips CM200) with an operating voltage of 200 kV for the coarse-grained and nc alloys, respectively. The average grain size in the samples was determined using the intercept method from the microstructural images.

3 RESULTS AND DISCUSSIONS

The OM and TEM images are shown in Fig. 01a-c. Fig. 01a shows that the average grain size of the unmilled Mg alloy is about 13µm. The TEM images of MA20 to MA40 reveal that finer grain structures after milling and extrusion, as shown in Fig. 01b-c. Grain size distribution of *nc* samples was estimated from the TEM images taken at different locations of the milled samples. The grain size dependence on milling hours are shown in Fig. 02.

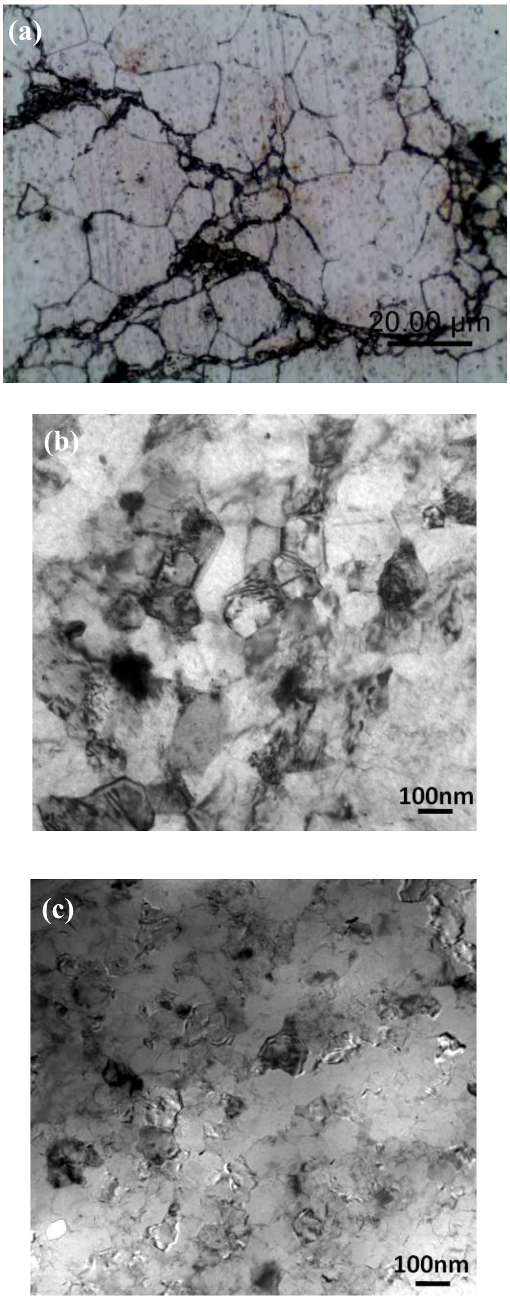

FIGURE 01: (a) OM image showing microstructures of MA0, and TEM bright field image showing fine grain structures of MA20 (b) and MA40 (c), respectively.

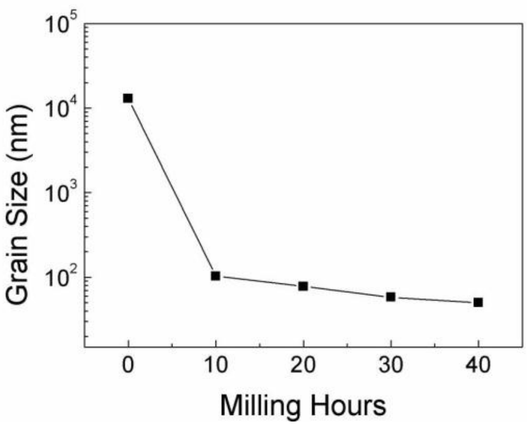

FIGURE 02: Grain size dependence on milling hours

The P-h curves of Mg-5wt%Al alloys under different loading rates are plotted in Fig. 03, in which three different loading rates were applied. At a given loading rate, the curves shift upwards with the increasing of the milling hours from MA0 to MA30 on loading, indicating that the resistance of the materials to indentation gradually increases with the grain refinement. The shift of P-h curves suggests that the enhanced strength with reduction of grain size from 13 µm to 58nm, as shown by the fact that a shallower indentation depth is produced at the same maximum force. However, after another 10 h mechanical millings, the P-h curves shift downwards with further reduce of grain size to 50 nm. This change suggests that the grain refinement strengthening is not quite effective when the grain size is below 50 nm, and the strength drops after milled for 40 h.

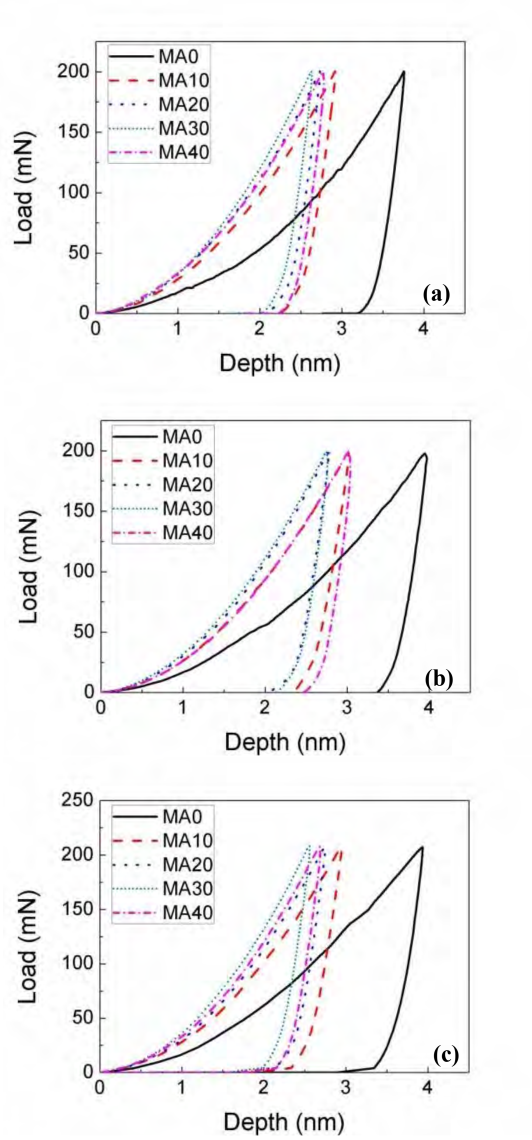

FIGURE 03: Force-Penetration Depth curves of *mc* and *nc* Mg-5%Al alloys at loading rates of (a) 1mN/s, (b) 20 mN/s and (c) 400 mN/s.

The SRS of a material is defined as the variation of flow stress with strain rate at a given level of strain for a fixed temperature and it can be expressed as:

$$m = \frac{\sqrt{3}kT}{\sigma V^*} \quad (1)$$

where k is Boltzman constant, T the absolute temperature, ζ the flow stress and v^* the activation volume, which can be considered as the derivative of the activation energy with respect to the effective shear stress.

By employing nanoindentation measurements, the flow stress can be related to the measured hardness ($H = 3\zeta$) and consequently the SRS is measured as:

$$m = \frac{3\sqrt{3}kT}{HV^*} \quad (2)$$

The strain rate sensitivity and activation volume calculated from compression and indentation tests are shown in Tab. 01. For *mc* MA0, the dominant intragrain dislocations activities during the plastic deformation process leads to a high activation volume value (~260 b^3), thus a negligible SRS value. When the grain size decreases down to nanometer region, the dislocation sources would switch from intragrain sources to grain boundary (GB) emission/escaping mechanism. The highly localized dislocation activities (dislocations leaving/escaping from boundaries) result in the small activation volume, which would be much smaller than those associated with the conventional mechanisms of forest dislocation intersection in the lattice, which in turn would be associated with a correspondingly elevated SRS (Dao, Lu, Asaro, De Hosson, & Ma, 2007).

TABLE 01: Strain rate sensitivity and activation volume values obtained from Indentation and Compression tests

	Indentation		Compression	
	m	$v^*(b^3)$	m	$v^*(b^3)$
MA0	0.003	273	0.006	259
MA10	0.013	33	0.014	41
MA20	0.017	22	0.020	26
MA30	0.046	8	0.048	10
MA40	0.068	6	0.107	6

The H-P relationship curves derived from present indentation and compression tests on extruded Mg-5wt%Al alloys are shown in Fig. 04. As noted, the H-P relationship curves could be divided into three regions: I) the *mc* region, where the yield strength follows the H-P relation; II) the *nc* region, where the yield strength departs from the conventional H-P relationship for coarse-grained material with a smaller k_{HP} value; III) grain size smaller than a critical value (d_{cri}), where the k_{HP} value is essentially negative. The hardness values increase monotonically with decreasing grain size when the grain size decreases from 13μm to 58 nm. However, the slope decreases and becomes negative as the grain size further decreases below 50 nm of MA40.

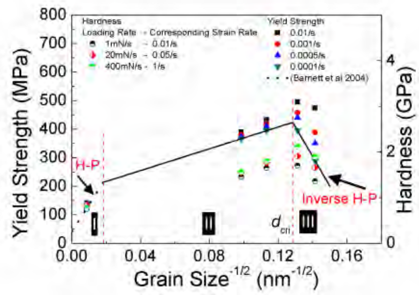

FIGURE 04: Hardness and strength of *mc* (Barnett, Keshavarz, Beer, & Atwell, 2004) and *nc* Mg-5% Al as a function of $\sqrt{d}$ under compression and indentation tests

The break-down of the H-P relationship at small grain size indicates the change of deformation mechanism. For *mc* materials, the positive H-P slope suggests that the dominant role of dislocation mediated plasticity during indentation process. At sufficiently small grain size, the H-P model which is based upon the lattice dislocations or deformation twins may not be operative. On the other hand, volume fraction of grain boundaries increases significantly with decreasing grain size, GB mediated mechanisms become more significant.

Conrad and Narayan (2000) proposed a thermally-activated grain-boundary shearing model assuming that GB shear was associated with many independent atomic shear events. Employing the concept of thermally-activated shear, the macroscopic shear rate produced by independent, atomic shear events at the GBs is given by:

$$\dot{\gamma} = \frac{6A\nu_D}{d}\sinh\left(\frac{v^*\tau_e}{kT}\right)\exp\left(-\frac{\Delta F}{kT}\right) \quad (3)$$

where A is the atomic diameter, ν_D (≈10^{13} Hz) is the Debye Frequency, $\eta_e=\eta-\eta_0$, where η is the applied stress and η_0 a back stress or threshold stress, $v^* = b^3$ the activation volume (b is Burgers vector), and ΔF is the activation energy for GB diffusion; for Mg it's 92kJ/mol (Frost, 1982).

The increasingly significant contribution from GB mediated mechanisms leads to the breakdown of the H-P relationship. The increasingly important role of GB sliding process in deformation also result in the enhanced ductility in MA40 at low strain rate (0.0001/s), exhibiting a true strain over 60%.

However, SRS of MA40 (m = 0.107) is still smaller than that expected for plastic deformation process controlled by GB sliding (m = 0.5) or coble creep (m = 1.0) (Dao, et al., 2007). These results indicate that grain boundary diffusion-mediated mechanisms are not yet dominant over dislocation-based processes.

4 CONCLUSIONS

In the present study, *mc* and *nc* Mg-5wt% Al alloys are fabricated by mechanical milling, with the grain size spectrum ranging from micro- to nanometer. The grain refinement has a significant effect on the mechanical response of extruded Mg-5wt% Al alloys. The grain refinement strengthening mechanism operates when the grain size is reduced from 13μm to 58nm, indicating a positive H-P relationship. However, further reduction of grain size would lead to the breakdown of H-P Relationship, where the k_{HP} value is essentially negative. The breakdown of H-P relationship and the significantly elevated SRS values of *nc* Mg-5wt% Al alloys suggests that GB mediated mechanisms play a more important role in plastic deformation when the grain size is reduced down to 50nm. However, the relatively smaller SRS in the current study compared to those processes dominated by GB sliding and Coble Creep indicated that grain boundary diffusion-mediated mechanisms are not yet dominant over dislocation-based processes. The comparable results of SRS from independent nanoindentaion and compression testing also suggests that nanoindentation is a reliable technique for probing mechanical behaviour of *mc* and *nc* Mg-5wt% Al alloys (at least the current Mg-5wt% Al materials).

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