Deformation Behaviour of Nanocrystalline Mg-Al Alloys during Nanoindentation

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Abstract - Deformation Behaviour of Nanocrystalline Mg-5%Al alloy was investigated using instrumented indentation tests. Nanocrystalline Mg-5%Al alloy were obtained via mechanical alloying method with the grain size of the alloy ranges from micrometer to nanometre regime. A pronounced effect of different loading rates on hardness behaviour was observed in the nanoindentation tests. This contributes to the ascending effect of mechanically alloying on materials’ grain refinement and strength increase. The nanocrystalline material is found to be more rate sensitive than their microcrystalline counterparts, which suggests the strong effect of grain refinement on the strain rate sensitivity. When the grain size is decreased down to UFC and nc regimes, the cutting forest dislocations as the dominant plastic deformation mechanism is not applicable due to the scale effect. The smaller activation volumes and the plastic deformation mechanisms involves grain boundary activities contribute to the increase of strain rate sensitivity in nc Mg-5% Al alloys.

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1 Introduction

Magnesium alloys has been a growing interest in various engineering applications, such as automobile, aerospace, communication and computer industry due to their low density, high specific strength and good machinability and availability as compared to other structural materials. However, most Mg alloys suffer from poor plasticity due to their hcp structure. Recent research has shown that ductility and strength of Mg alloy are affected by the grain size [1]. Mohri et al. [2] reported the ductility enhancement of a Mg–Y–RE alloy by hot extrusion. Mukai et al. [3] have also demonstrated that the ductility in WE43 magnesium alloy can be enhanced by the grain refinement even at a dynamic strain rate of $\sim 2 \times 10^3 \text{ s}^{-1}$. Several methods such as mechanical alloying and severe plastic deformation have shown the effectiveness of grain size refinement [4]-[7]. When the grain size is reduced down to the Ultra fine grained (UFG) and nanocrystalline (nc) regime, attractive properties of nc metals and alloys begin to surface, such as the high yield and fracture strengths, the improved wear resistance and the super-plastic behaviours at relatively low temperatures. Also, nc materials show high strain rate sensitivity (SRS) compared to their microcrystalline counterparts.

There are reports of both increased and decreased strain rate sensitivity with decreasing grain size in metallic materials. Three representative FCC metals, i.e., Cu, Ni and Al show enhanced strain rate sensitivity when the grain size is decreased into the UFG/NC regime [8]-[13]. However, UFG/NC metals with BCC structure exhibit an opposite tendency, which is the strain rate sensitivity of BCC metals decreases with reduced grain size [14]-[18]. Only a limited number of papers are available in the literature addressing the grain size effect on the SRS of HCP metals. For the time being, any hypothesis is questionable about the grain size effect on SRS for HCP metals due to the extreme paucity of experimental results [19].
Many of the results available in the literature can’t be easily compared due to the different techniques for the fabrication techniques of the materials, which lead to widely different microstructures, processing induced defects, contamination and residual stress. These differences render it difficult to identify the mechanisms responsible for differences in mechanical properties. These differences render it difficult to identify the mechanisms responsible for differences in mechanical properties. There is a critical need for examining the SRS of nc metals and alloys in a systemic way over a broad range of strain rates with well-characterized materials [20].

In this paper, the effects of grain size and strain rate on the mechanical behaviour of Mg-5%Al alloys fabricated via mechanical milling were studied using nanoindentation. The constituent metal powders were mechanical milled under argon atmosphere with different durations of 0 hours (MA0 as-blended), 20 hours (MA20) and 30 hours (MA30). The milled powders were cold-compacted using 35 mm diameter die and sintered at 500ºC for 2 h. The sintered billets were then hot-extruded with an extrusion ratio of 25:1 to 7 mm diameter. The microstructures were examined using transmission electron microscopy (TEM) with a Philips CM200 Analytical Scanning TEM with an operating voltage of 200 kV. The sample was prepared using twin-jet polishing technique.

2 Experiment

Nanoindentation experiments were carried out using A Hysitron TI-700 Ubi nanomechanical test instrument with a diamond Berkovich indenter of which the nominal tip radius is 150 nm. The load and displacement were monitored continuously by a three-plate capacitive force/displacement transducer. The indentation tests were carried out under constant loading rate. The samples were indented to a peak force using linear loading rates of 0.05mN/sec, 1mN/sec, 20mN/sec, and 400mN/sec in the range
50-200mN maximum load, respectively. Before starting the indentation test, efforts were made to minimize the effects of thermal drift by allowing several hours for thermal equilibrium to be reached.

The strain rate sensitivity 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 [21][21]:

$$m = \frac{\sqrt{3}kT}{\sigma v^*}$$  \hspace{1cm} \text{Equation 1}

Where $k$ is Boltzman constant, $T$ the absolute temperature, $\sigma$ 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 ($H=3\sigma$) and consequently the strain rate sensitivity is measured as:

$$m = \frac{3\sqrt{3}kT}{HV^*}$$  \hspace{1cm} \text{Equation 2}

3 Results and Discussions

The microstructures of the Mg-Al alloys with different grain sizes are shown in Fig.1. The average grain sizes are 13 µm, 80 nm and 60 nm for MA0, MA20 and MA30, respectively. Therefore, the grain size was reduced to below 100 nm after 20-30 hours mechanical milling. Previous results [21] revealed that the grain boundaries were atomically sharp and that no amorphous phase was present at the grain-boundary regions. Compression tests were performed on an Instron Machine with a strain rate of $10^{-3}$/s on cylinder samples with a dimension of 3mm diameter and 5 mm in length. The compressive yield strength of the samples of MA0, MA20 and MA30 were shown in Figure 2. Figure 3 shows the nanoindentation load-displacement curves of MA30 at different loading rates. A pronounced effect of loading rate on the load-penetration depth curves is observed. In contrast, the indentation response
from MA0 and MA20 is not very sensitive to the loading rate. As can be seen from the curves, for the milled samples, higher indentation forces are required to achieve the same depth with the increasing loading rates. This contributes to the ascending effect of mechanically alloying on materials’ grain refinement and strength enhancement. Figure 4 shows the variation of hardness with loading rate. A more pronounced effect of loading rate on hardness was observed with the alloy with finer sized grains, i.e., MA30. The indentation hardness of nc Mg-5% Al is almost twice as high as the hardness of mc Mg-5% Al.

![Microstructure of the (a) MA0 (b) MA20 and (c) MA30.](image)

![Yield Strength obtained from compression tests](image)

![Load vs displacement curves of (a) MA0, (b) MA20 and (c) MA30 at different loading rates](image)
By measuring the nanoindentation hardness and calculating the activation volume, it becomes possible to obtain the strain rate sensitivity of the material. The strain rate sensitivity of the ultra-fine grained nc Mg-5% Al alloy was calculated from Equation 2 based on the activation volumes for UFG and nc Mg alloy available from the literatures [23]-[26]. The calculated strain rate sensitivity of nc Mg-5%Al alloy and some other HCP metals for comparison are shown in Figure 5. From Fig 5 we can see that, the strain rate sensitivity of the Mg-5% Al Alloy decreased as the grain size increased. The values of the milled samples MA20 and MA30 were very close with each other, while the SRS of the mc Mg Alloy valued one third that of the nc Mg Alloys.

Depth-sensing indentation tests revealed that the strain-rate sensitivity is a strong function of grain size [27]-[28]. The increased strain rate sensitivity is directly related to a change in the rate controlling mechanism for plastic deformation [29].

Molecular dynamics simulations [30]-[31] found that grain-boundary atoms as well as atoms up to 7–10 lattice parameters away from the grain boundary are heavily involved in plastic deformation.
Deformation was mostly found to be taken up by atoms at and nearby grain boundaries. Based on this, Schwaiger et al. [32] explained the observed effect of grain size on the rate-dependent plastic response on the premise that a rate-sensitive grain-boundary affected zone (GBAZ) exists, which broadly refers to a region adjoining the grain boundaries in nc metals where the crystalline lattice is elastically strained despite the ostensible absence of any point defects. Atoms within this GBAZ are more likely to be involved in the deformation process.

In addition, the different behaviour observed for mc, ufc and nc metals can be explained in terms of activation volume. For coarse grained materials, the dislocation sources are assumed to be in the centre of the grain, leading to positive and negative dislocation pile-ups generated by the activation of a Franck–Read source. As the grain size is decreased, the number of dislocations piled up against a grain boundary decreases, at a fixed stress level, since this number is a function of the applied stress and of the distance to the source. Conversely, an increased stress level is needed to generate the same number of dislocations at the pile-up. At a critical grain size, we can no longer use the concept of a pile-up to explain the plastic flow. In nc metals and alloys, in fact, dislocations are generated at grain boundaries, and grain boundaries act as dislocation sinks due to image forces. Recent model analyses [33] indicate that, due to the small volumes involved in the process of dislocations leaving/escaping from boundaries, the activation volume would be much smaller than those associated with the conventional mechanisms of forest dislocation intersection in the lattice. In addition, mechanisms of generation and annihilation at grain boundaries, coupled with grain rotation and migration are involved in the plastic deformation in nc materials since cutting forest dislocations is a mechanism that can’t operate at UFG and nc regime due to the scale effect. Such mechanisms disappear when the grain size increases from nc to UFG or even coarse grain, leading to the decrease of the strain rate sensitivity.
4 Summary

To sum up, instrumented indentation tests were performed on both micro-crystalline and mechanical milled UFG and nc Mg-5% Al alloys. The results suggest that grain refinement to nc structures and ultra-fine regime leads to a strong effect on the SRS. The SRS parameter m increased from 0.016 to 0.068 when the grain size decreased from 13μm to 60nm. When the grain size is decreased down to UFC and nc regimes, the cutting forest dislocations as the dominant plastic deformation mechanism is not applicable due to the scale effect. The smaller activation volumes and the plastic deformation mechanisms involves grain boundary activities contribute to the increase of SRS in nc Mg-5% Al alloys.

Reference

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