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Nanocrystalline Mg-3%Al Alloy: its Synthesis and Investigation of its Tensile Behavior

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***Abstract*—**The tensile properties of Mg-3%Al nanocrystalline alloys were investigated at different test environment. Bulk nanocrystalline samples of these alloy was successfully prepared by mechanical alloying (MA) followed by cold compaction, sintering, and hot extrusion process. The crystal size of the consolidated milled sample was calculated by X-Ray line profile analysis. The deformation mechanism and microstructural characteristic at different test condition was discussed extensively. At room temperature, relatively lower value of activation volume (AV) and higher value of strain rate sensitivity (SRS) suggests that new rate controlling mechanism accommodating plastic flow in the present nanocrystalline sample. The deformation behavior and the microstructural character of the present samples were discussed in details.

***Keywords*—**Nanocrystalline, tensile properties, temperature effect.

I. Introduction

THE present demand of light weight materials such as magnesium and their alloys are growing very fast. Magnesium is one of the most abundant and lightest structural metallic materials (density ~ 1.74 gm/cm^3). High specific strength at room temperature and good damping characteristic of Mg alloys led to a potentially exploited in aerospace industry, automobile industry and for sports goods. However, low ductility, insufficient toughness and poor corrosion resistance due to the few slip system available for the dislocation movement in a hexagonal closed packed (HCP) structured is the major draw back of these alloy. Further more, Mg alloys exhibit poor mechanical properties at high temperatures. Hence, increasing interest has focused on the grain size refinement of these alloys [1-2]. It has been observed that most of the nanocrystalline materials attribute limited ductility because of the difficulties of dislocation slip and deformation twining with smaller grain size [3]. However, improved ductility and even low temperature superplasticity was observed in Mg and its alloys by the refinement of grain size [4-5].

The main objective of the present study is the synthesis of nanocrystalline Mg-3%Al alloy via mechanical alloying and the characterization of its microstructure and deformation behavior at different test conditions. The strain rate sensitivity and activation volume at room temperature was calculated from repeated stress relaxation tests [6]. The effect of temperature was examined by the quantitative analysis of temperature sensitivity [7].

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II. Experimental Procedures

A. Preparation of Bulk Nanocrystalline Sample

Nanocrystalline (NC) powders of Mg3wt%Al were prepared by mechanical milling from the elemental powders of Mg and Al carried out in a high-energy ball-milling machine operated for 30 hrs. NC powders were then consolidated into a cylindrical billet of 35 mm diameter and 35 mm length. The compacts were sintered in an argon furnace at 450^{0}C for 2hrs and followed by hot extrusion to get 7 mm diameter cylindrical rod. More details of the procedures are discussed in Ref. [1].

B. Tensile Test

Tensile specimens of 25 mm gauge length and 5 mm diameter were prepared by machining (in a CNC m/c) and followed by mechanical polishing of the extruded rod. Tests were carried out at different test temperatures (from room temperature to 250^{0}C) in a fully computer controlled INSTRON-8874 machine with a load cell of 25kN. Before the test start, the specimens were heated at a test temperature for 20 minutes inside the environment chamber. Repeated stress relaxation tests were conducted at room temperature in order to evaluate the physical activation volume and strain rate sensitivity.

C. Characterization of Microstructure and Fracture Surface

The crystal size and the structural evolution of the extruded samples were evaluated by X-ray diffraction analysis using Shimadzu Lab XRD-6000 X-ray diffractometer with CuK_α radiation, operated at 40 kV voltage with a scanning speed of 0.02 degrees/sec. The fracture surfaces of the broken tensile specimens (tested at room temperature and at 250^{0}C) were comprehensively examined to provide insight into the fracture mechanism during loading. The grain size of the microcystalline sample was calculated optically.

III. Results and Discussion

A. Structure and Grain Size

The XRD spectra of the powder samples were examined in Fig. 1 (a). The Al diffraction peak (1 1 1) was clearly

observed in unmilled powder sample. However, this peak was disappeared in the milled sample. It implies that the solid solution of Mg and Al were formed in the milled sample. The peak broadening and the decrease of its intensity is the result of reduction in crystal size and increase in internal strain. The new phase of $Mg_{17}Al_{12}$ and $MgAl_2O_4$ was detected in the milled sample. The crystal sizes of milled powders were calculated using Hall-Williamson method and to be about 35 nm.

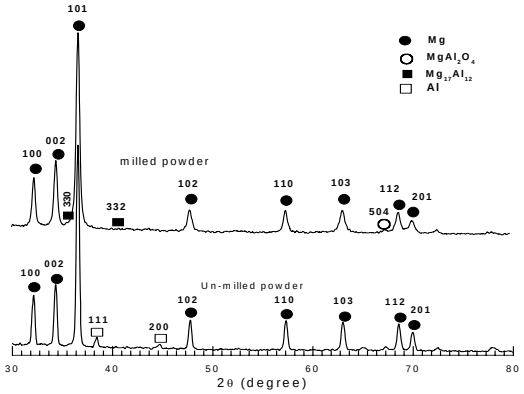

Fig. 1 (a) XRD spectra of milled and unmilled powders

Fig. 1 (b) represents the XRD spectra of the extruded samples. The variation of diffraction peak from that of powdered sample was observed in the extruded samples. The lower intensity of Mg (0 0 2) peak is the result of high extrusion ratio where the basal plane of Mg rotates in the direction of [1 0 1] to coincide the extrusion direction [2]. Significant phase of $MgAl_2O_4$ was detected in the extruded samples, especially when the sample was milled one. A dramatic increase in crystal size was observed in the extruded samples and to be about 91 nm. The mean grain size of the unmilled extruded sample was measured optically to be about 12 μm.

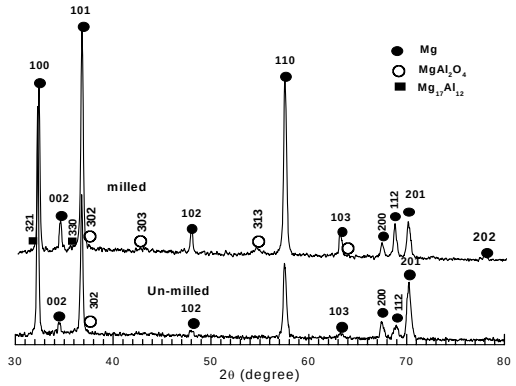

Fig. 1 (b) XRD spectra of milled and unmilled extruded sample

B. Tensile Behavior

Tensile tests with different test temperatures were conducted at constant strain rate ($\dot{\varepsilon} = 4\times10^{-4}$ s^{-1}). Fig. 2 represents the true stress-strain curve of NC and MC samples at different test environments. It has been clearly observed that higher ductility was attributed in NC sample at room temperature. The mechanism for plastic deformation in NC samples may be due to the grain boundary sliding and rotation which compensate the continuity of grain sliding [10].

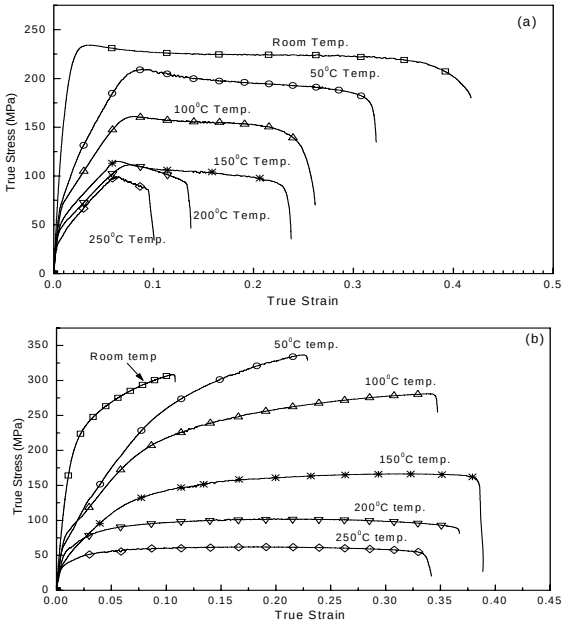

Fig. 2 True tensile stress-strain curve of (a) NC (30 hrs milled) sample (b) MC sample, at various testing temperatures

Rapid decrease of strength with increase of test temperatures was observed in both the samples. However, monotonic decrease of failure strain was attributed in NC sample only. The loss of ductility at higher temperatures may mainly be attributed by the formation of localized shear deformation.

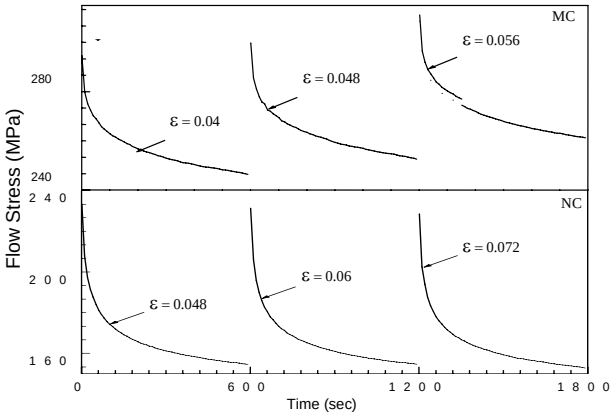

Fig. 3 Repeated stress relaxation curves with time interval of 600 s for MC and NC sample

Fig. 3 shows the repeated stress relaxation test for MC and NC sample at room temperature. No remarkable increase of stress level at the end of each consecutive cycles indicating

negligible work hardening was associated in the NC sample.

During the relaxation of stress, the rate of stress drop is proportional to the plastic strain that can be expressed as [8],

$$-\dot{\sigma} = C\dot{\varepsilon} \tag{1}$$

The strain rate sensitivity and the apparent activation volume can be expressed as [9],

$$m = \frac{\partial \log(\sigma)}{\partial \log(\dot{\varepsilon})}; \quad \text{and} \quad \Delta V = \frac{\sqrt{3}kT\partial \ln(\dot{\varepsilon})}{\partial \sigma} \tag{2}$$

where k is the Boltzmann constant and T is the absolute temperature.

Furthermore, The plastic strain rate is proportional to the slope of the relaxation curve ($-d\sigma/dt$). We calculate the strain rate sensitivity (m) from the slope of the linear fitting of $\ln(\sigma)$ vs. ($-d\sigma/dt$) plot. While, the apparent activation volume (ΔV) was estimated from the slope of the linear fitting of ($-d\sigma/dt$) vs. σ plot. It has been observed that relatively lower value of ΔV (order of ~ $14b^3$, where b is the burger vector) and higher value of m (~ 0.078) was attributed to the NC sample.

C. Strain Hardening Behavior and Ductility

The most general form of stress-strain curve can be described as,

$$\sigma = K\varepsilon^n \tag{3}$$

where σ and ε is the true flow stress and true plastic strain respectively. Term K is the strength coefficient and n is known as work hardening exponent. The changes in the work hardening exponent (measured from the true stress-strain curve) and the ductility measured by the % of reduction of area (%RA) at different test temperatures are plotted in Fig. 4.

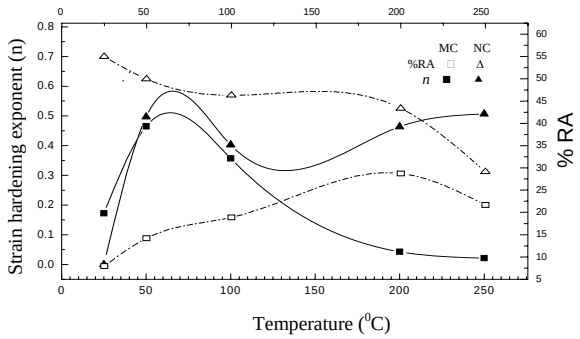

Fig. 4 Variation of strain hardening exponent and the %RA at various test temperatures for MC and NC sample

It is clear that NC sample exhibited almost zero work hardening and higher ductility at room temperature in comparison with that of MC sample. However, just opposite nature was observed both in work hardening and ductility at higher temperature tensile test.

D. Temperature Sensitivity

Temperature sensitivities (TS) were examined to understand the effect of temperatures. The parameter of TS (n_a) was calculated from the flow stress-strain data using the following empirical relationship [7]:

$$n_a = -\{\ln(\sigma_2/\sigma_1)\}/\{\ln(T_2/T_1)\} \tag{3}$$

where $T_2/T_1>1$ and $T_1 = 25^0$C. The value of flow stresses, σ_2 and σ_1 are calculated at temperature T_2 and T_1 respectively in a definite value of plastic strain.

Fig. 5 represent the variation of TS parameter for NC and MC samples at true strain of $\varepsilon = 0.05$ and $\varepsilon = 0.1$. Increasing trend of TS parameter was observed for the MC and NC sample at strain, $\varepsilon = 0.1$ with the incremental temperature range. However, almost a flat character of TS parameter was attributed in NC sample at lower value of true plastic strain. Hence, it is clear that the TS parameter is depending on the test temperature as well as the true strain points.

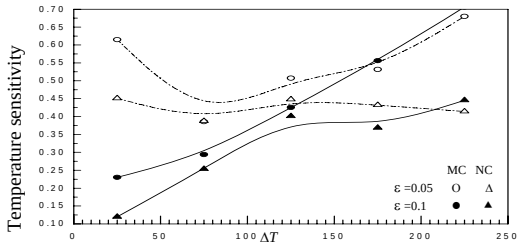

Fig. 5 Variation of temperature sensitivity parameter (n_a) for MC and NC sample

E. Behavior of Fracture Surface

Fig. 6 and Fig. 7 represent the fractograph of the deformed tensile specimen of MC and NC samples tested at room temperature and 250^0C temperature respectively. Cleavage marks indicative fracture surface with micro crack was observed in Fig.6 (a), which clearly indicates that the failure mode of MC sample is primarily brittle in nature. Dimple features along with some micro voids (considerably smaller than that of MC sample) are observed in Fig.6 (b) indicating some plasticity expected in NC sample at room temperature.

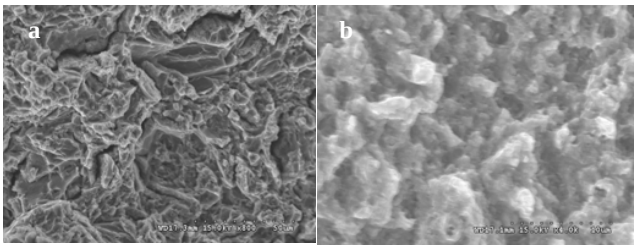

Fig. 6 Fractographs of fracture surface after tensile test at room temperature (a) MC sample (b) NC sample

The intergranular fracture behavior with some shallow dimples was observed in MC sample tested at 250^0C as shown in Fig. 7 (a). The tensile fracture surface of NC sample tested

at 250^0C temperature as shown Fig. 7 (b) consisting of a dimple structure which is larger than the mean grain size. The reduction in the width of the tensile samples close to the fracture point represents some plastic deformation produced by shear stresses. Probably this is the result of the formation and coalescence of submicron voids along in the direction of fracture path which provoked localized plastic deformation, instability as indicated by the sudden necking very near to the fracture surface.

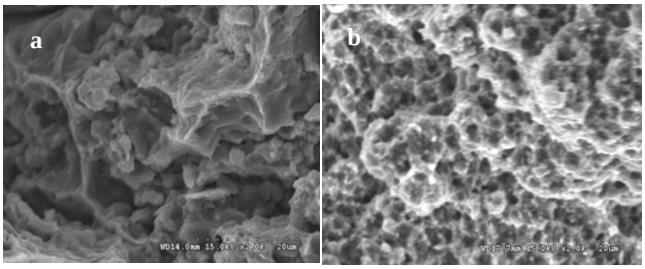

Fig. 7 Fractographs of fracture surface after tensile test at 250^0C temperature (a) MC sample (b) NC sample

IV. Conclusion

The aim of this research was to elucidate the deformation behavior of nanocrystaline Mg-3%Al alloy at various test conditions. Nanocrystalline samples were prepared successfully by mechanical milling. Present experimental results of NC sample give a very good comparison in different aspect of materials characterization with that of MC sample.

Structural characterization demonstrates the formation of new phases of Mg and Al during the milling process and after extrusion of the consolidated samples.

Enhanced ductility at room temperature observed in NC sample. The rapid degradation of in mechanical properties at elevated temperature may be due to the presence of large amount $Mg_{17}Al_{12}$ at the grain boundaries [11] and the development of localized shear deformation. Higher value of strain rate sensitivity and lower value of activation volume in NC sample suggest the possible emission of dislocation from the grain boundaries.

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