Rev.Adv.Mater.Sci. 6 (2004) 28-32

NANOSTRUCTURED HIGH STRENGTH Mg-5%Al-*x*%Nd ALLOYS PREPARED BY MECHANICAL ALLOYING*

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Received: July 24, 2003

Abstract. Nanostructured Mg-5%Al-*x*%Nd (*x* = 0.5, 1 and 5 wt.%) alloys were prepared by mechanical alloying. Microstructural characterization revealed average crystalline size to be about 30 nm after mechanical alloying while it increased to about 90 nm after sintering and extrusion. Mechanical properties showed increase in 0.2% yield stress, ultimate tensile strength and ductility after 20 h of mechanical alloying. The increase in yield stress and ultimate tensile strength was attributed to reduction in grain size as well as to the enhanced diffusion after mechanical activation. Although ultra high yield stress was observed from the specimen with 5%Nd, its ductility was reduced to about 1.6%.

* Presented at International Conference Nanomaterials and Nanotechnologies (Crete, Greece, August 30-September 6, 2003)

1. INTRODUCTION

Low ductility and strength and poor corrosion resistance of Mg and its alloys caused by the nature of its hexagonal close-packed structure limit applications of Mg alloys for decades. Improvement of ductility and strength is therefore necessary to enable Mg alloys to be used in structural applications. Recent research has shown that ductility and strength of Mg alloys are affected by the grain size. High ductility can be obtained when the grain size is typically less than about 10 μm [1]. Several methods such as mechanical alloying and equal channel angular deformation have demonstrated the effectiveness of refinement of grain size [2-5]. It was found that with grain size refinement ductility was observed to increase [6] and even at low temperature [7]. Due to difficulty in handling ultra-fine Mg powders, up to now, only few research papers are there on mechanical alloying of Mg alloys. The present paper focuses on the effect of mechanical alloying of Mg and development of new nanostructured Mg5Al-Nd alloy.

2. EXPERIMENTAL PROCEDURES

Mg alloys of nominal composition Mg-5%Al-*x*%Nd (*x* = 0.5%, 1% and 5%) were prepared via mechanical alloying method. Elemental powders of Mg (purity > 98.5%), Al (purity 99.5%) and Nd (purity > 99.0%) were used for milling. A Fritsch planetary ball mill operating at 250 rpm was used. Forty 15 mm diameter balls were employed with a ball-to-powder weight ratio of about 20:1. Process control agent of stearic acid was added to the powder mixture to prevent agglomeration and excessive cold welding of powders. Prior to mechanical alloying, powder mixtures were sealed in milling vials with 99.9% pure argon gas. After mixing and mechanical alloying, the powders were cold-compacted into rods of 35 mm in diameter and about 35 mm in length. The compacts were then sintered at 400 °C or 500 °C in a vacuum furnace for 2 h. Extrusion of the compacts with an extrusion ratio of 25:1 was carried out at 400 °C.

X-ray diffraction (XRD) analysis was carried out using a Shimadzu Lab XRD-6000 X-ray diffractometer

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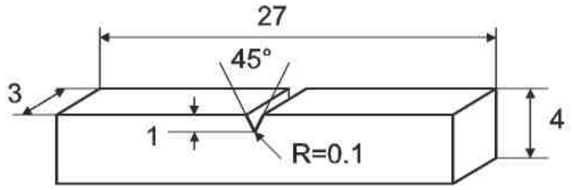

Fig. 1. Specimen for impact test.

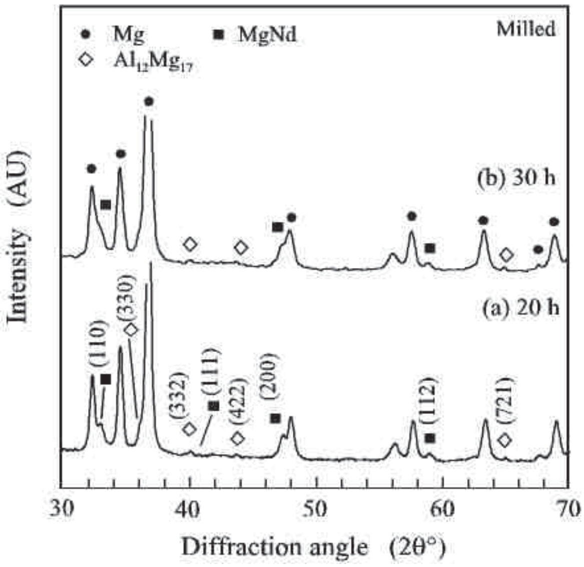

Fig. 2. X-ray spectra of mechanically alloyed powder.

with Cu K_α diffraction. Crystallite sizes of mechanically alloyed powders and extruded samples were determined from Scherrer's equation. A Philips CM20 (200 kV) analytical transmission electron microscope (TEM) was used for microstructural characterization.

The extruded rods were machined into tensile test specimens of 5 mm diameter and 25 mm gauge length in accordance with the ASTM E8M-96. Tensile test was performed using an Instron machine. Impact test was also carried out using the specially designed small notched specimens, as shown in Fig. 1.

3. RESULTS AND DISCUSSION

3.1. Structures

The XRD diffraction spectra of mechanically alloyed powders are shown in Figs. 2a and 2b. As shown in Fig. 2a, two new phases of $Al_{12}Mg_{17}$ and MgNd can be clearly identified after 20 hours of mechanical alloying. A peak at about 56.2° is close to $Mg_{12}Nd$ but is unable to confirm. Except for peak broadening, there is almost no change in diffraction. Fig. 3 shows the XRD diffractions of the sintered and extruded specimens. Very different diffraction patterns can be seen in this figure. For the unmilled specimen, only one $Al_{12}Mg_{17}$ (330) peak can been seen from Fig. 3a because of easy reaction between Mg and Al; all other peaks are from Mg. After mechanical alloying there is no trace of $Al_{12}Mg_{17}$ after sintering (Figs. 3b and 3c). This may be due to the fine size of $Al_{12}Mg_{17}$ in the milled samples. It appears that these finer $Al_{12}Mg_{17}$ particles are not stable and decompose. A new phase of $Mg_{12}Nd$ appears in the cost of MgNd. At the same time, a broad hump peaked at 43.06° can been seen in Fig. 3b. It is interesting to note that the strongest diffraction peak of $MgAl_2O_4$ (orthorhombic) appears at 42.97°. It is reasonable to assume the formation of $MgAl_2O_4$ at the expanse of $Al_{12}Mg_{17}$ phase.

The Mg grain sizes of mechanically alloyed powders were found to be about 23 nm and 18 nm after 20 and 30 hour of mechanical alloying, respectively. However, high temperature sintering has led to increase in grain size. The grain sizes of the specimens mechanically alloyed for 20 hours, and sintered at 400 or 500 °C are about the same, varying from 43 nm to 90 nm. The grain sizes of specimens mechanically alloyed for 30 hours, and sintered at 400 or 500 °C range from 37 to 88 nm.

3.2. Mechanical properties

Fig. 4 compares the mechanical properties of some of the specimens. Three distinguish features can be identified. Firstly, strengths of the mechanically alloyed specimen is higher than those of unmilled ones. Secondly, ductility of the mechanically alloyed specimens is larger than those of unmilled ones. Thirdly, strength of the specimens is strongly dependent on the amount of Nd. For the specimens with 0.5%Nd and sintered at 400 °C, the yield stress of A0 is 227 Mpa, while the samples mechanically alloyed for 20 and 30 hours show higher values of 276 and 296 MPa, respectively. For the un-milled specimens with 0.5%Nd, the value of ductility is 4.6%, while the mechanically alloyed specimens exhibit significantly higher values of 10.8% after 20 hours of milling even though Mg has only three basal slip systems. This observation suggests that in ad-

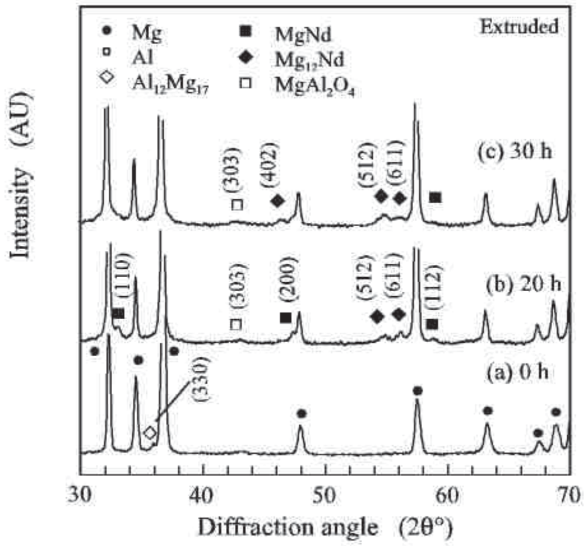

Fig. 3. XRD spectra of extruded specimens.

dition to slip and twinning of Mg, other mechanisms such as low temperature creep may play a very important role in ductization. Further milling led to decrease in the ductility. If the specimens are sintered at 500 °C, the yield stresses of the three specimens become about the same. The ductility of unmilled one does not show much difference but the values of ductility of the both mechanically alloyed ones increase.

For the specimens with 5%Nd, as shown in Figs. 4c and 4d, there is no big difference in the yields stresses between the unmilled specimens with 0.5%Nd and with 5%Nd. This implies that Nd atoms are unable to successfully diffuse into Mg lattice forming solid solution at the temperature of 500 °C and hence there is no solid solution strengthening. Compared with Fig. 4a, the specimens with 5%Nd sintered at 400 °C show large reduction in its ductility after milling. Specimens sintered at 500 °C exhibit higher ductility compared to those of the same composition sintered at 400 °C; which is attributed to the enhanced particle bonding at 500 °C. The high temperature sintering at 500 °C does not show the draw back in terms of strength and ductility. However, for unmilled specimens high temperature sintering neither increase in strength nor enhance ductility.

Mechanical properties in terms of 0.2% yield stress, ultimate tensile strength (UTS), elongation obtained from averaging three readings are given in Table 1. The 0.2% yield stresses of specimens A and B sintered at 500 °C were lower than those sintered at 400 °C. Specimens C with 5wt.%Nd always show higher yield stresses at the higher sintering temperature. Similar to hardness measurement, yield stresses and UTS of the specimens

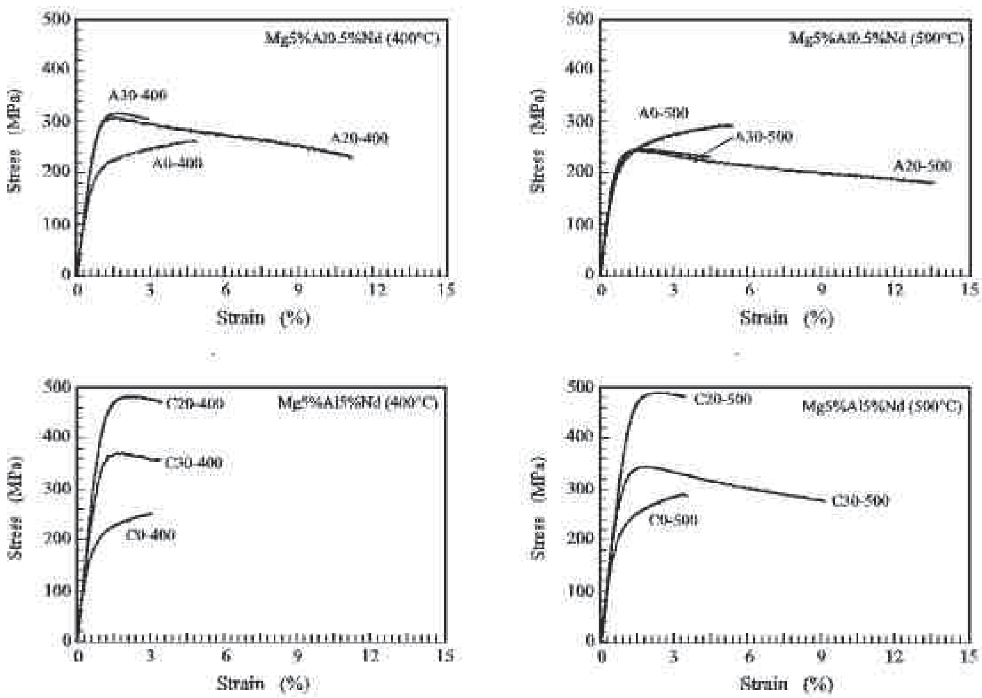

Fig. 4. Tensile load *vs* strain of specimens.

Table 1. Tensile properties of Mg5%Al-Nd alloys.

Specimen	0.2%YS(MPa) zzz = 400 °C	0.2%YS(MPa) zzz = 500 °C	Elongation (%) zzz = 400 °C	Elongation (%) zzz = 500 °C
A0-zzz	227	216	4.6	4.9
A20-zzz	276	223	10.8	17.4
A30-zzz	297	217	2.2	5.8
B0-zzz	212	208	3.9	5.1
B20-zzz	351	331	9.2	5.6
B30-zzz	346	225	3.6	12.3
C0-zzz	190	211	2.7	3.3
C20-zzz	422	436	1.7	2.6
C30-zzz	271	333	1.6	5.9

without mechanical alloying do not show much change when Nd content is increased. However, once the specimens were synthesized via mechanical alloying, not only yield stress and UTS but also ductility were dramatically improved. Both yield stress and elongation of specimen A increase from 227 MPa and 4.6% to 276 MPa and 10.8% after 20 h of milling. The yield stress peaks at 297 MPa after 30 h of milling but elongation is lowered to 2.2%. Though highest yield stress can be obtained after 20 h of mechanical alloying of specimen C, ductility is dropped to 1.7% and 2.6 for C20-400 and C20-500 specimens, respectively.

The results of the impact test are shown in Table 2. Except for A30-500, there is an apparent reduction of impact energy for the sample after ball milling. Several other investigations also demonstrated highly brittle behavior of nanocrystalline metals [8-10]. In Fig. 4, it is clear that there is no work hardening for the samples after 20 or 30 h milling although considerable strengthening has been achieved. Normally, with decreasing the grain size, dislocation slip and deformation twining are getting more difficult, which results in a high strength but a lower ductility. In Table 1, it can be seen that there is an apparent increase in both the strength and elongation for the samples after the ball milling. Hahn *et al*. [11] shown that for a material with very fine grain sizes, the deformation mechanism was dominated by grain boundary sliding. Grain boundary sliding is considered to take place along some preferential grain boundaries and the unsuitable boundaries can be accommodated via grain rotation. Grain boundary sliding is a thermally activated process in which the boundary volume elements need to be moved. Obviously, this process is time dependent. This can explain why the ductility could be maintained or even improved for the samples with nano-sized grains after ball milling during the quasi-static tensile test (Table 1) but the impact toughness was greatly deteriorated (Table 2) as the period of loading was too short to allow the movement of elements at grain boundaries to be accomplished.

4. CONCLUSIONS

Nano-grained Mg5%Al-Nd alloys have been successfully fabricated through mechanical alloying of elemental Mg, Al and Nd powders. Though grain grow took place during sintering, it is still in the nano-range. Higher temperature sintering at 500 °C did not lead to dramatic grain growth. Very high yield strength has been obtained from the specimens which were mechanically alloyed. Much large elongation of the mechanically alloyed specimens has

Table 2. Average impact energy of Mg5%Al-Nd alloys.

Specimen	Energy (J) zzz = 400°C	Energy (J) zzz = 500°C
A0-zzz	0.23	0.23
A20-zzz	0.05	0.0465
A30-zzz	0.054	0.55
B0-zzz	0.192	0.35
B20-zzz	0.0465	0.048
B30-zzz	0.042	0.056
C0-zzz	0.198	0.205
C20-zzz	0.056	0.05
C30-zzz	0.053	0.045

been achieved. The maximum elongation is about 13%. Impact results indicate ductility of the specimens to be deformation rate-dependent.

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