[Article ID] 1003 - 6326(2000)06 - 0757 - 04

Fabrication and characteristics of magnesium alloys produced via powder metallurgy ①

CAO Peng(曹 鹏)[1,2], LU Li[1], Man On LAI[1]
(1. Department of Mechanical and Production Engineering,
National University of Singapore, Singapore 119260;
2. State Key Laboratory for Powder Metallurgy,
Central South University, Changsha 410083, P.R.China)

[Abstract] Magnesium aluminum alloy materials were fabricated via powder metallurgy. Microstructural evolution, thermal stability, tensile properties and fracture mechanism of the hot extruded magnesium alloys were investigated. Microstructural observation revealed that all alloys have fine equiaxed grains due to dynamic recrystallization during hot extrusion with $Mg_{17}Al_{12}$ precipitating along grain boundaries. The DSC analyses exhibited that because diffusion rate of Al into Mg is slow in solid state sintering process, Al concentration in localized region after sintering is still high enough to cause eutectic reaction. The tensile test showed that 0.2% yield strength, ultimate tensile strength and elastic modulus increase and elongation decrease with increasing Al content. Mg-9%Al obtains the best combination of mechanical properties among the investigated alloys. Fracture surface observation showed ductile fracture to be a dominant failure mode. Abundant dimples and tear ridges are found in the fracture surfaces. Grain boundary weakening results from the precipitate $Mg_{17}Al_{12}$ caused intergranular cracks during tensile test.

[Key words] magnesium alloys; mechanical properties; powder metallurgy; hot extrusion
[CLC number] TG146 **[Document code]** A

1 INTRODUCTION

Magnesium alloys, especially Mg matrix composites, are excellent candidates for engineering light structural materials because of their high specific stiffness and specific strength, good dimensional stability and high damping capacity. Thus, Mg alloys are expected in advanced structural applications such as automotive and aerospace[1,2]. Most Mg alloys have been fabricated by cast technology due to cost-effective[3]. Powder Metallurgy (PM) is an advanced technology in materials manufacture process, especially for metal matrix composites (MMCs)[4~7]. Because of the difficulty in wetting ceramic particles with molten metal, the powder metallurgy route was the first method developed[8~12]. A near-net-shape product can be fabricated via powder metallurgy. In this route, the material is prepared from powder without passing through the fully melting state. Hence, the PM technique can attain more homogeneous microstructures in the alloys. More importantly, a unique advantage of powder metallurgy is the ability to tailor microstructures and properties of different materials.

With the wide range of powder variables and process variables available, it is possible to design microstructures in such a matter as to obtain specific properties. The present work is to investigate the influence of Al on the properties of Mg alloy.

2 EXPERIMENTAL

The alloys used in the present work were fabricated by PM route. Pure Mg (> 98.5%) powders with the size of 0.60 ~ 0.03 mm and Al powder of about 5.00 μm, supplied by Merck, were mixed using a V blender for 4 h. The content of Al was 3%, 5%, 9% and 12%, respectively. The mixtures were cold-pressed into green compacts with gauge of 35 mm in diameter and about 40 mm in length followed by vacuum sintering at 400 ℃ for 1 h. The as-sintered round billets were hot extruded at 400 ℃. The extrusion ratio was 25:1. The extruded rods were cut along extrusion direction. The samples were polished followed by etching with acetic glycol solution for 15 s prior to microstructure analysis using an optical microscope.

A Rehometric 1500 simultaneous thermal analyzer (STA) was used for the thermal analysis of the extruded specimens. The heating rate was controlled within 10 ℃/min. Commercial pure argon gas was used in all cases to minimize contamination. A Shimadzu X-ray 6000 diffractometer with $CuK_{\alpha 1}$ radiation operated at 30.0 kV and 20.0 mA was employed to determine phase characteristics in the materials. The standard tensile specimens were machined with a gauge of 5 mm in diameter and 25 mm in length, according to ASTM E8M standard. The tensile properties, such as tensile strength, yield strength, elastic

① [Received date] 1999 - 11 - 05; [Accepted date] 2000 - 04 - 18

modulus and strain of as-extruded materials were measured at ambient temperature with a constant crosshead speed of 0.5 mm/min using an Instron 8500 series universal testing machine. The fracture surfaces of specimens after tensile testing were examined using a JEOL JSM-5800LV scanning electron microscope at an accelerating voltage of 15 kV, to study the fracture mechanism.

3 RESULTS AND DISCUSSION

3.1 Microstructure evolution

Fig.1 shows the microstructures of the samples with different Al contents. It was revealed that powders were well bonded each other although few voids were found. The samples contained fine equiaxed recrystallized grains because of dynamic recrystallization during hot extrusion, and some lamellar interdendritic eutectic were decorated the grain boundaries. The amount of interdendritic was found to increase with increasing the Al content. However, due to relative low and short sintering temperature and holding time, Al was not fully diffused into Mg to form Mg-Al alloy.

The results of XRD shown in Fig.2 proved the presence of $Mg_{17}Al_{12}$ in all the magnesium alloys. With increasing Al contents, precipitate $Mg_{17}Al_{12}$ increased. No other phases were detected in X-ray diffraction patterns.

3.2 DSC determination

The thermal behaviour of the samples was analyzed using a DSC at a heating rate of 10 ℃/min. As shown in Fig.3, all samples exhibit a remarkable endothermic peak at about 437 ℃, which is the melting temperature of eutectic with less than 37.7% Al (mole fraction). This indicated that because aluminum diffusing into magnesium is slow in solid state sintering process, Al concentration in localized region was high enough to cause eutectic reaction. To confirm the eutectic reaction in materials, the as-extruded billet was heated at 400 ℃ under argon atmosphere for additional 2, 3 and 5 h for Mg-5%Al alloy and 2, 4, 5, 7 h for Mg-9%Al alloy, respectively. These samples were then analyzed by DSC. In case of Mg-5%Al alloy, the results show that the endothermic peak became weaker with extending sintering time. As for the sample sintered for additional 3 h the endothermic peak could hardly be seen. Other Mg-Al materials with different Al contents exhibit the same behaviour, showing the disappearance of the endothermic peak of Mg-9%Al after additional 7 h of sintering. From DSC tests, it can be concluded that the sintering time at 400 ℃ should be 8 h for Mg-9% Al to enable the full reaction of Al with Mg. Increasing temperature may have similar effects as extending sintering time.

3.3 Tensile properties

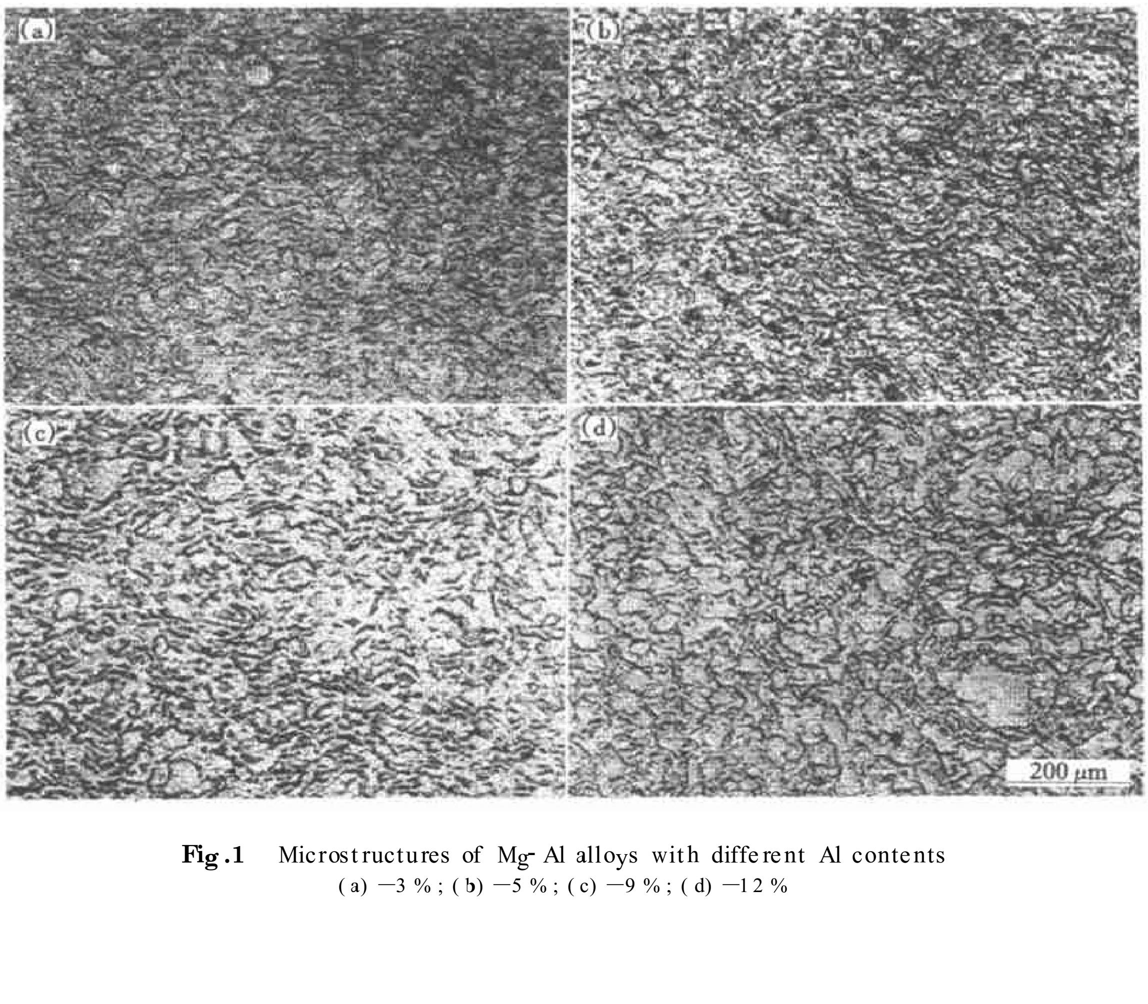

Fig.1 Microstructures of Mg-Al alloys with different Al contents
(a) —3%; (b) —5%; (c) —9%; (d) —12%

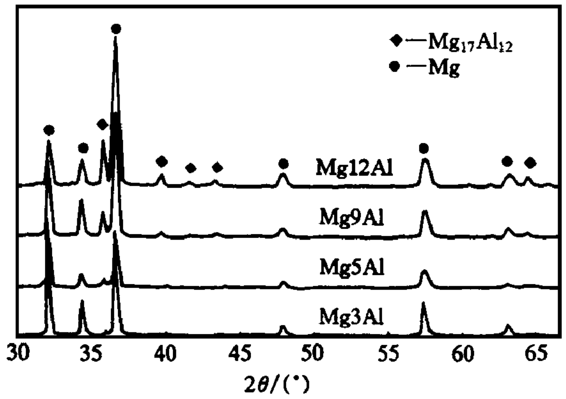

Fig.2 XRD spectra of as-extruded Mg-Al alloys

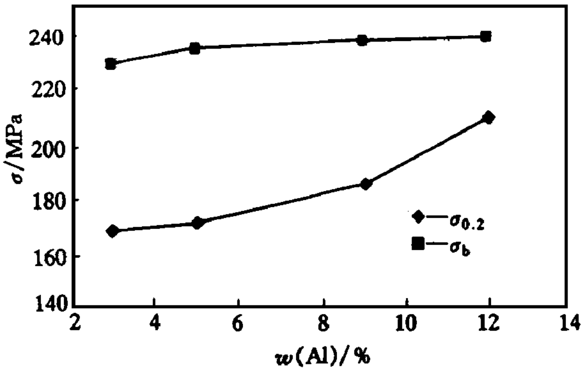

Fig.4 Tensile strength (σ) of Mg-Al alloys

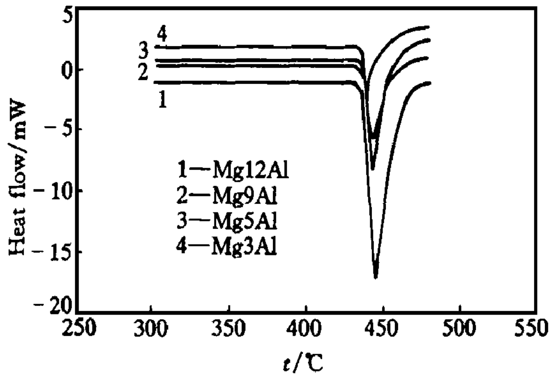

Fig.3 DSC scans of as-extruded Mg-Al alloys

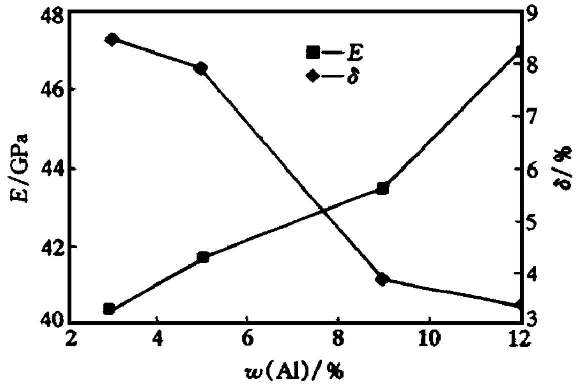

Fig.5 Elastic modulus and elongation of Mg-Al alloys

Mechanical properties including 0.2% yield strength ($\sigma_{0.2}$) , elastic modulus (E) , ultimate tensile strength (σ_b) and ductility behavior for as-extruded specimens, in terms of elongation, are summarized in Figs.4 and 5. Every value is the average of three specimens. As seen from Figs.4 and 5, there are only small differences in $\sigma_{0.2}$, σ_b for the specimens containing 3% ~ 5% Al. For the 0.2% proof strength, specimens containing 9% Al possesses a much higher value than those containing 3% and 5% Al, however, just little lower than Mg-12%Al. This may be mainly attributed to aluminium solid solution strengthening, and magnesium could obtain best solid solution strengthening with 9% aluminium content. From the values depicted in Figs.4 and 5, it could be found that powder metallurgy Mg-Al alloys are superior to casting magnesium aluminum alloy and equal to wrought Mg-Al alloys in tensile strength[3]. As we know, alloying has only small effect on elastic modulus. Nevertheless, the elastic modulus of the as-extruded Mg-12% Al alloy increased markedly due to excess single Al, whose elastic modulus is much greater (about 70 GPa) than that of Mg. Elongation decreased with increasing the aluminium content. However elongation of Mg-5% Al decreased much greater than Mg-9%Al. Magnesium alloy with 9%Al had the best combination of mechanical properties.

The fracture surfaces show that all the samples have a similar fracture mechanism. Ductile fracture is a dominant failure mode, which was revealed by the abundant dimples and tear ridges in the fracture surfaces. From Fig.6, crack propagation along the grain boundaries was eminent and intergranular fracture could be clearly observed. The alloys with more Al content showed more intergranular cracks, hinting the precipitate $Mg_{17}Al_{12}$ might be prone to cause grain boundary weakening. Though the materials attained nearly fully dense microstructure after hot extrusion, there still existed microvoids. In addition, during the tensile testing, precipitate may break into micro-void. These microvoids grew up after nucleation to coalesce finally into a micro-crack, and micro-cracks merged into a large crack to cause failure of the alloys. The dimples in the fracture surfaces result from the nucleation, growth and coalescence of micro-voids.

4 CONCLUSIONS

1) The magnesium alloys show fine equiaxed recrystallized grains because of dynamic recrystallization during hot extrusion. No other precipitates than $Mg_{17}Al_{12}$ phase are detected using XRD technique.

2) The DSC analyses exhibit that because al-

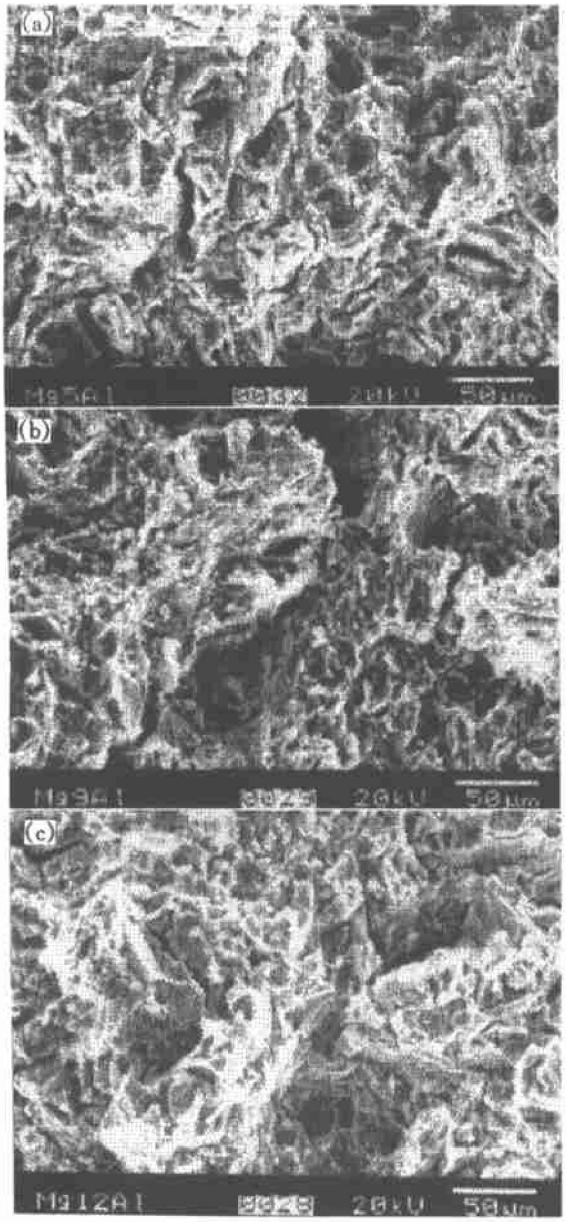

Fig.6 Fractographs of as-extruded Mg-Al alloys
(a) —Mg5Al; (b) —Mg9Al; (c) —Mg12Al

uminum diffusing into magnesium is slow in solid state sintering process, Al concentration in localized region is high enough to cause eutectic reaction. For Mg-9% Al powder compaction, the sintering time should be extended or sintering temperature should increased.

3) In the cases of Mg-3% Al and Mg-5% Al, there are only small differences in mechanical properties. As for Mg-9%Al, it has much better combination mechanical properties than other three alloys.

4) Ductile fracture is the dominant failure mode, which is revealed by the dimples and tear ridges in the fracture surfaces. The alloys with more Al content show more intergranular cracks, the precipitate $Mg_{17}Al_{12}$ is prone to cause grain boundary weakening.

[REFERENCES]

[1] Wilks T W. Cost-effective magnesium MMCs [J]. Adv Mater Processes, 1992, 8: 27 - 29.

[2] Lee D M, Suh B K, Kim B G, et al. Fabrication, microstructures and tensile properties of magnesium alloy AZ91/SiC_p composites produced by powder metallurgy [J]. Mater Sci Tech, 1997, 13: 590 - 595.

[3] Greenfield P. Magnesium [M]. London: Mills & Boon Ltd, 1972. 18 - 22.

[4] Liu Y B, Kwok J K M, Lim S C, et al. Fabrication of Al-4.5Cu-15SiC_p composite: I. Pressing using mechanical alloying [J]. J Mater Proc Tech, 1993, 37: 441 - 452.

[5] Wilks T E. Lightweight magnesium composites for automotive applications [A]. Society of Automotive Engineers, Technical Paper 940846 [C]. Warrendale, PA, USA, 1994. 49 - 54.

[6] Lu L, Kwok J K M, Lai M O, et al. Fabrication of Al-4.5Cu-15SiC_p composite: II. Effects of pressing parameters on fracture properties [J]. J Mater Proc Tech, 1993, 37: 453 - 462.

[7] FAN Jian-zhong, YAO Zhang-kai and DU Shou-yi. Interfacial structure of SiC_p/Al composites made by powder metallurgy [J]. The Chinese Journal of Nonferrous Metals, (in Chinese), 1998, 8(1): 20 - 23.

[8] Seidel M, Eckert J, Zueco-Rodrigo E, et al. Mg-based amorphous alloys with extended supercooled liquid region produced by mechanical alloying [J]. J Non-Cryst Solids, 1996, 205/207: 514 - 517.

[9] Purazrang K, Abachi P and Kainer K U. Mechanical behavior of magnesium alloy MMCs produced by squeeze casting and powder metallurgical techniques [J]. Composites Eng, 1993, 3(6): 489 - 505.

[10] Lloyd D J. Particle reinforced aluminium and magnesium matrix composites [J]. International Materials Reviews, 1994, 39(1): 1 - 23.

[11] Gross K J, Spatz P, Zuttel A, et al. Mechanically milled Mg composites for hydrogen storage: the transition to a steady state composition [J]. J Alloys and Compounds, 1996, 240: 206 - 213.

[12] Yang J, Ciuyeanu M and Roberge R. Preparation and hydrogen storage properties of $Mg_{1-x}Ni_x$ (x = 0 ~ 45%) composites [J]. J Alloys and Compounds, 1999, 287: 251 - 255.

(Edited by HUANG Jin-song)