

# IMPROVED PROCESSING OF Mg-Zn-Y ALLOYS CONTAINING QUASICRYSTAL PHASE FOR ISOTROPIC HIGH STRENGTH AND DUCTILITY

Alok Singh<sup>1</sup>, Y. Osawa<sup>1</sup>, H. Somekawa<sup>1</sup>, T. Mukai<sup>1</sup>

<sup>1</sup>Lightweight Alloys Group, Structural Metals Center, National Institute for Materials Science, 1-2-1 Sengen, Tsukuba 305-0047, Japan

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## Abstract

The stable quasicrystal phase in Mg-Zn-Y alloys has been proved to be beneficial for strength and ductility and has also been shown to impart high fracture toughness. However, the strength of these alloys has been limited to 300MPa with an average grain size of about 1  $\mu\text{m}$ . We show here a simple procedure in which a  $\text{Mg}_{93}\text{Zn}_6\text{Y}$  alloy was chilled cast and then extruded. Yield strengths of up to 400 MPa in tension and compression, accompanied by ductility of  $\sim 14\%$  were obtained with grain of size of about 1  $\mu\text{m}$ . Compression to tension yield anisotropy ratios were in the narrow range of 0.95 to 1.03. These alloys also showed ageing response with two peaks. The effect of precipitation ageing on the mechanical properties has been studied.

## Introduction

Various approaches are being taken to improve strength and ductility of magnesium alloys. These include alloying to form new precipitates as well as mechanical deformation to produce finer microstructure to activate non-basal slip and inhibit twinning. An icosahedral quasicrystalline phase (i-phase) occurs in Mg-Zn-RE alloys in which the rare earth element RE is Y, Gd, Tb, Dy, Ho or Er, which is now known to be a thermodynamically stable  $\text{Mg}_3\text{Zn}_6\text{RE}$  phase [1, 2]. It also exists in direct equilibrium with  $\alpha$ -Mg phase [3, 4]. Since this phase has special properties such as high hardness and low surface energy [5], it becomes a natural choice as a strengthening phase in magnesium alloys. However, this phase forms interdendritically; therefore it must be finely dispersed in the alloy by means such as hot rolling or extrusion [6, 7, 8, 9, 10, 11, 12]. These alloys have been shown to have a good balance of strength and ductility, have very high fracture toughness [13], good fatigue strength [14], weak texture [15] and excellent formability [16].

By direct extrusion and hot rolling, the grain size has

been limited to 1  $\mu\text{m}$ , with tensile and compressive yield strength (YS) of about 300MPa [11, 12]. To raise this limit, powder metallurgy was performed, which produced a grain size of about half a micron and tensile YS at the level of 400MPa [17]. It has been shown that the strength levels follow Hall-Petch relationship indicating grain boundary as the main strengthening mechanism [12, 17]. In this study, we show that higher strengths can be obtained by improved processing for better distribution of the i-phase. Precipitation in the matrix and its effect on tensile and compressive YS is shown.

## Experimental Procedure

An alloy of composition Mg-6at%Zn-1at%Y was made by melting in an electric furnace under a cover gas, and then cast into a chill cast mold. The alloy was extruded at an extrusion ratio 25:1 at temperatures 390 $^\circ$ , 300 $^\circ$  and 250 $^\circ\text{C}$ . Ageing was performed in an oil bath at 150 $^\circ\text{C}$ . Microstructure was observed by transmission electron microscopy (TEM) using a JEOL 2000FX microscope. Samples for TEM were thinned by ion milling. Tensile and compression tests were performed on an Instron machine at a strain rate of  $10^{-3}$ . Tensile specimens had a diameter 3mm and gauge length 15 mm. Compression specimens were 4mm in diameter and 8mm in height. Mechanical test data were averaged from three tests. To determine the average grain size, the average linear intercept length measured was multiplied by a factor of 1.74 to account for stereographic effects [18]. Vickers microhardness tester was used for measuring hardness of the samples.

## Results and Discussion

### Extruded microstructure

The chill cast alloy had a dendritic structure with dendritic arms about 20  $\mu\text{m}$  thick. Fine grain size was obtained after extrusion. Figure 1 shows grain structures after extrusion at 300 $^\circ\text{C}$  and 250 $^\circ\text{C}$ . The grain size are

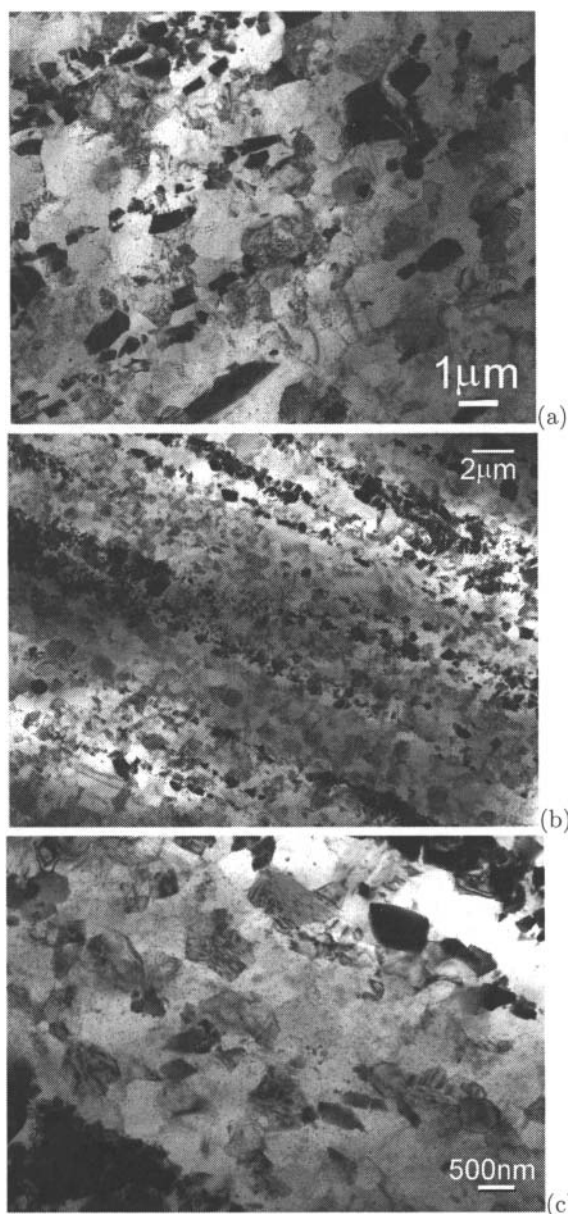


Figure 1: Bright field electron micrographs showing grain structures and i-phase distribution after extrusion at (a) 300°C and (b) 250°C. (c) Details from (b) show nano-sized precipitates in the matrix.

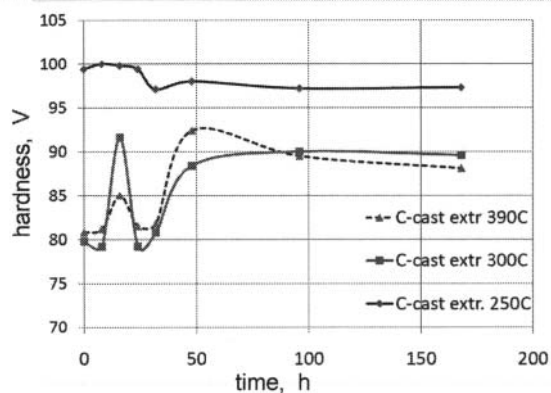


Figure 2: Vickers hardness curves on ageing for the alloys extruded at 390°C, 300°C and 250°C.

about a micron. Comparable size of i-phase particles (dark contrast) were distributed along the extrusion direction. The grain size were measured to be  $5.14 \pm 0.87$ ,  $1.13 \pm 0.15$  and  $0.98 \pm 0.16$  μm after extrusion at 390°, 300° and 250°C, respectively. Very fine nanometer-size precipitation was observed in the matrix.

#### Ageing response of the extruded alloys

Since extrusion temperatures of 300°C and higher were also used, some ageing effect was expected in the alloys. Therefore ageing response was measured, shown in Fig. 2. For extrusions at 390° and 300°C, a sharp peak is observed at about 17h. This was confirmed to be due to the precipitation of rod-like  $\beta_1'$  precipitates (Fig. 3). After this peak, the hardness rises again to peak at ~48h, beyond which the hardness remains nearly at the peak level, with very little decrease. The origin this second peak is yet to be ascertained.

The initial hardness of the sample extruded at 250°C was higher than the peak hardness of the other two alloys (Fig. 2). There were no sharp peaks, but instead a small plateau corresponding to the first peak in the other two alloys, and a small rise corresponding to the second peak. The final hardness was lower than the initial. Since the extrusion temperature was lower than 300°C, it is expected that precipitation has occurred during the extrusion. In such cases the precipitate morphology is round, instead of rod-shaped.

Fig. 3 shows precipitates in the alloy extruded at 390°C aged at 150°C for 17h and 48h. Rod-like  $\beta_1'$  precipitates

are clearly observed in (a), aligned along the  $c$  axis of the matrix. Dark rounded particles are of the  $i$ -phase. Attached to these are precipitates of monoclinic  $Mg_4Zn_7$  phase. It is observed in (b) that some of these rod-like precipitates persists after longer ageing for 48h. Fig. 3(c) shows both rounded and rod-like precipitates in the alloy extruded at  $250^\circ C$  and aged for 48h. The rounded precipita

### Mechanical Properties

The mechanical properties of the as-extruded alloys are shown as tensile and compression stress-strain (SS) curves in Fig. 4. After extrusion at  $390^\circ C$ , the tensile yield stress (TYS) was  $268.3 \pm 2.0$  MPa and compression yield stress (CYS) was  $252.9 \pm 0.7$  MPa. For extrusion at  $300^\circ C$ , the TYS was  $340.5 \pm 3$  and CYS was  $342.3 \pm 9.0$  MPa. Thus the CYS was comparable or even larger than the TYS. This is similar to the extruded alloy  $Mg_{93}Zn_6Ho$  of a comparable grain size [11]. In case of the alloy extruded at  $250^\circ C$ , TYS was  $386.3 \pm 2.8$  MPa and the CYS was  $354.6 \pm 8.0$  MPa. In this case, the TYS is larger than the CYS again. This may be due to the precipitation condition of the  $250^\circ C$  extruded alloys, in comparison to the  $300^\circ C$  extruded alloy, as mentioned above. It is noted that after yielding in compression, there is a plateau in the SS curve. This plateau becomes longer with finer grain size, as reported earlier [19]. Elongation to failure in compression are in the range 14-16% strain.

The tensile properties show a decrease in strain hardening as the grain size decreased. This may be understood as less number of dislocations are generated in a small grain. What is noteworthy is that the strain to failure (elongation or the ductility) increased with decreasing grain size. The elongation for the alloys extruded at  $390^\circ$ ,  $300^\circ$  and  $250^\circ C$  were  $12.88 \pm 1.03$ ,  $14.98 \pm 0.46$  and  $16.00 \pm 0.30\%$ , respectively.

### Effect of Ageing on Strength and Ductility

The extruded alloys were aged at  $150^\circ C$  for 48h and mechanically tested again. There was a slight increase in the TYS, but a decrease in the CYS in case of the finer grained alloys. The TYS for the aged  $390^\circ$ ,  $300^\circ$  and  $250^\circ C$  extruded alloys were  $288.7 \pm 1.6$ ,  $346.4 \pm 7.7$  and  $399.9 \pm 2.3$  MPa, respectively. The corresponding CYT were  $258.1 \pm 1.1$ ,  $326.2 \pm 0.8$  and  $377.6 \pm 9.5$  MPa. The yield asymmetry ratio YAS ( $=CYS/TYS$ ) can decrease after ageing because the  $\beta'_1$  precipitates are easily sheared [20]. Particularly in the case of extrusion at  $300^\circ C$ , the

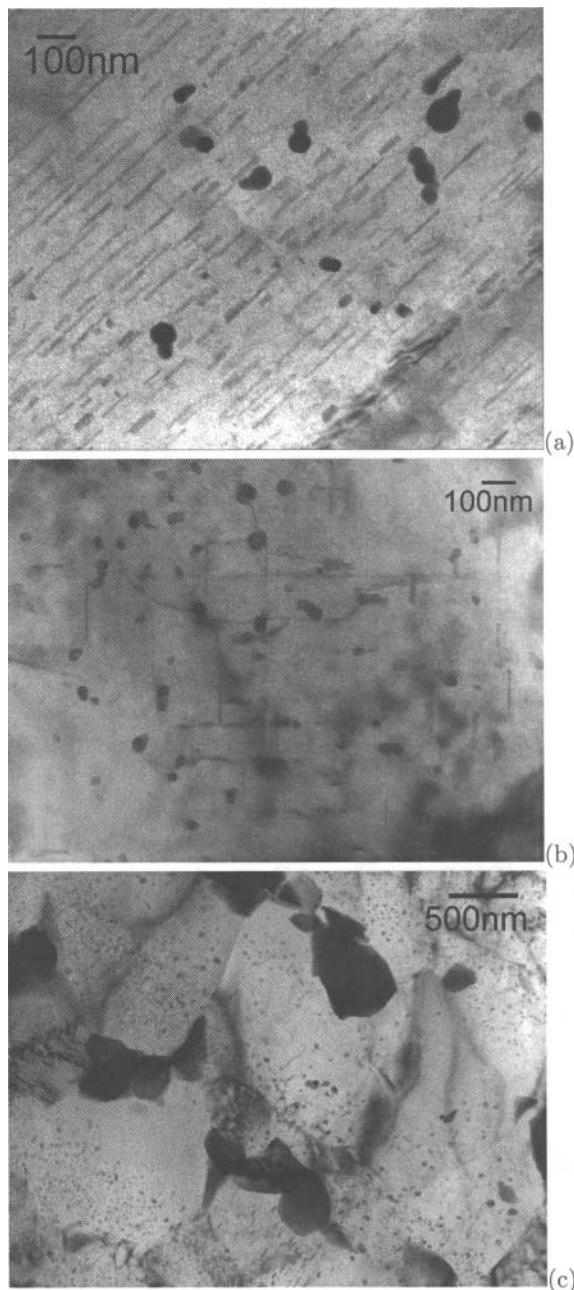


Figure 3: Precipitation in the matrix of alloy extruded at  $390^\circ C$  after ageing at  $150^\circ C$  for (a) 17h and (b) 48h, and (c) in alloy extruded at  $250^\circ C$  after ageing for 48h. Round dark particles in (a) and (b) are  $i$ -phase.

YAS ratio decreased from 1.1 before ageing to 0.94 after ageing.

Fig. 5 compares the tensile and compressive SS curves of as-extruded with the aged alloys for extrusions of 390° and 250°C. In case of the tensile curves of the alloy extruded at 390°C, the as-extruded sample has higher strain hardening and lower elongation, as compared to the aged sample. After ageing the TYS is higher, as well as the elongation is higher at nearly 18%. Precipitates would slow the slip, resulting in lower strain hardening and delaying pile-ups at grain boundaries, resulting in larger elongation. The compressive SS curves of the same samples (Fig. 5(b)) shows similar curves before and after ageing; however, after ageing the yield point is more pronounced, and the plateau after the yielding is longer (the ends of plateau of curves before and after ageing are marked with arrows). The plateau marks the region of predominant twinning. The plateau region ends when, following twinning, considerable slip occurs. The extended plateau region indicates that slip is impeded by precipitation. The rod-like precipitates which are bent or sheared by twins do not have significant effect on reduction of twinning [20]. Precipitation in Mg-Zn-RE alloys is reported to reduce the YAS ratio [21].

A slight increase in the TYS is observed after ageing in case of alloy extruded at 250°C too, Fig. 5(c), but there is no strain hardening after yielding in case of the aged sample, leading to an early failure (elongation 10.5±1.3%). It would appear that there were no barriers to dislocation motion after ageing. In case of compression (Fig. 5(d)), the SS curves before and after the ageing are similar, except that the CYS after the ageing is lower. The width of the plateau after yielding is (about 0.5% in strain) smaller than before ageing, indicating that slip is more active after ageing. Both in the case of extrusion of 300° and 250°C the CYS fell after ageing.

#### Effect of microstructure on ductility

Tensile elongations in as-extruded condition and after ageing are plotted against reciprocal of grain size in Fig. 6. In the as-extruded condition, the ductility rises with decreasing grain size. This would be due to activation of non-basal slip near grain boundaries to maintain compatibility stress across the grain boundaries [22]. Texture would also play a role. Finer grains after wrought processing are accompanied by a weaker texture, which is associated with better ductility [23]. In coarse grained alloys, in absence of precipitates to impeded dislocation

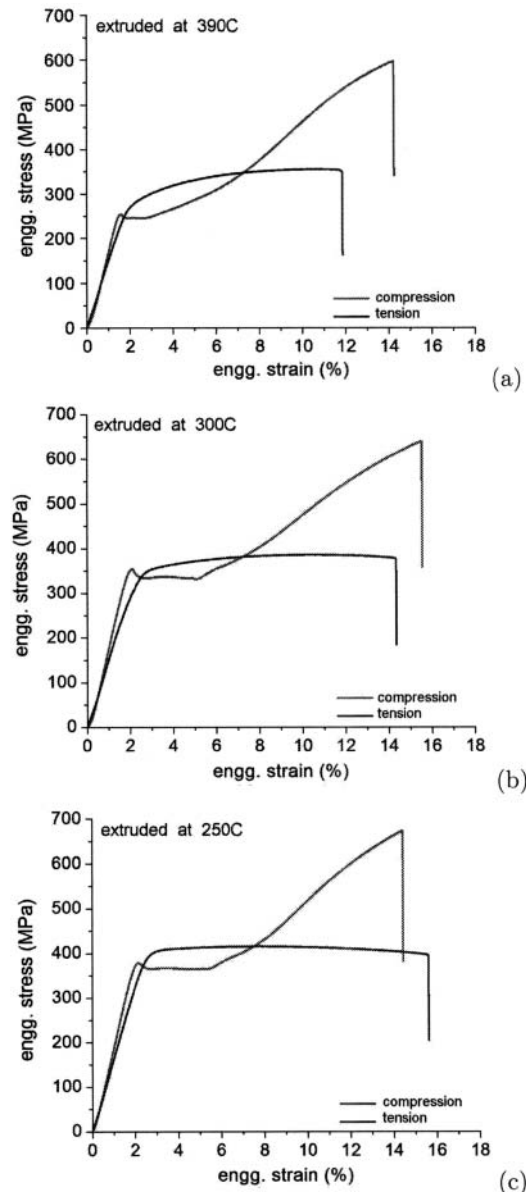


Figure 4: Stress-strain curves in tension and compression after extrusion at (a) 390°C, (b) 300°C and (c) 250°C.

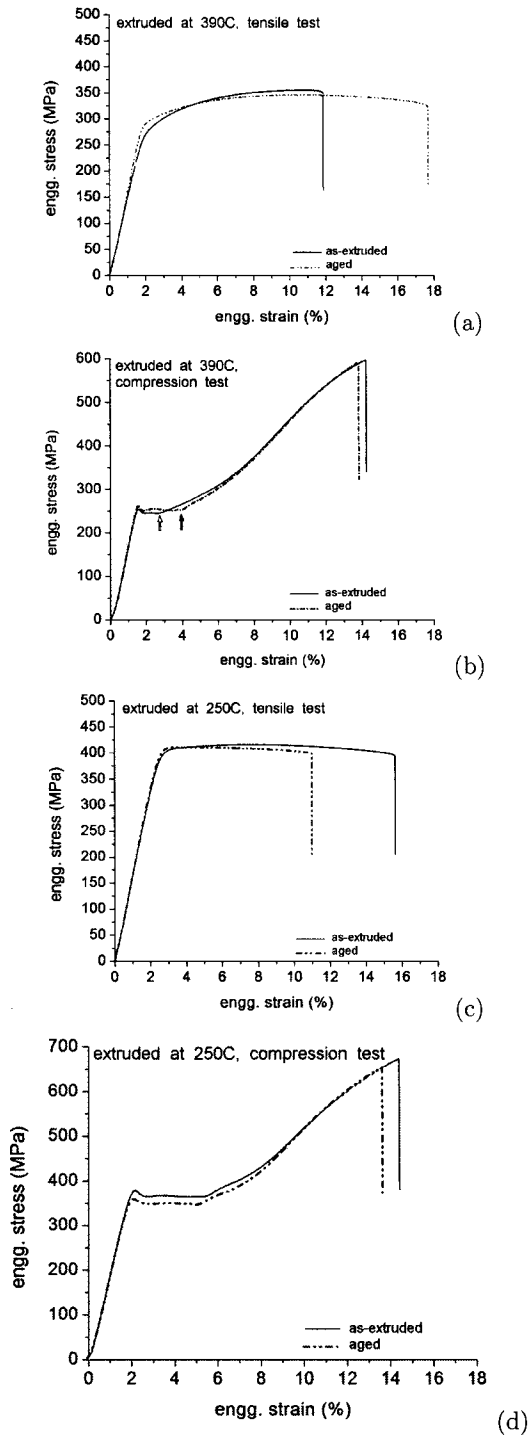


Figure 5: Comparison of stress-strain curves in as-extruded and aged (150°C for 48h) for extrusion at 390°C in tension (a) and compression (b), and for extrusion at 250°C in tension (c) and compression (d).

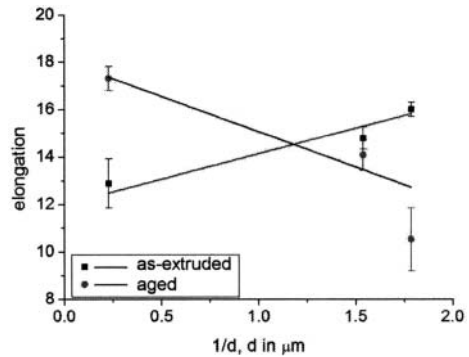


Figure 6: Tensile elongation to failure of alloys in as-extruded and aged condition plotted against reciprocal of grain size.

motion, pile-ups will occur easily, resulting in crack formation. Twinning also occurs easily in larger grained alloys which, coupled with dislocation pile ups, would lead to fracture. After ageing, the precipitates impede the motion of dislocations, preventing and early fracture. However, the lowering of ductility in fine grained alloys is not immediately clear. Long precipitates in fine grains may themselves become a source of brittleness.

## Conclusions

A simple modification of processing of a quasicrystalline i-phase containing Mg-Zn-Y alloy resulted in excellent mechanical properties. The alloy was chill cast, and then wrought processes by direct extrusion. Very high yield strengths of about 400 MPa, both in tension and compression, were obtained with average grain size of about a micron. This strength was accompanied by reasonable ductility between 12 to 18%. On ageing at 150°C, two peaks were observed at 17h and 48h. Beyond 48h, the hardness showed very little decrease. After ageing for 48h, there was a slight increase in the tensile yield strength, but a slight decrease in the compressive yield strength in case of the fine grain size. The ductility increased in case of larger average grain size (~5μm) but decreased in case of fine grained (~1μm) alloys.

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