In-situ quasicrystal-reinforced magnesium matrix composite processed by equal channel angular extrusion (ECAE)

M. Y. ZHENG*, X. G. QIAO, S. W. XU, K. WU
School of Materials Science and Engineering, Harbin Institute of Technology, Harbin 150001, People's Republic of China
E-mail: zhenghe@hit.edu.cn

S. KAMADO, Y. KOJIMA
Department of Mechanical Engineering, Nagaoka University of Technology, Nagaoka 940-2188, Japan

Magnesium alloys have a strong potential for applications in structural components because of their low density. Considerable effort is currently being devoted to the development of high performance magnesium-based materials for aerospace and outer space applications. Apart from conventional alloying practice, there has been a rapid development of particle or fiber reinforced magnesium matrix composites [1, 2]. The use of ceramics as reinforcement in magnesium alloys resulted in significant improvement of strength and stiffness. However, such strength and stiffness benefit is at the expense of a rather poor ductility [1, 2].

Quasicrystals are isotropic and possess quasiperiodic lattice structure [3]. Due to the difficulty of the movement of dislocations in the quasicrystals at room and elevated temperatures, quasicrystals exhibit high hardness and high strength [4]. Therefore, quasicrystals have been successfully used as reinforcements in metal-matrix composites [5–7]. For example, quasicrystal-reinforced aluminum matrix composites with high strength combined with good ductility have been developed by powder-metallurgical method [5, 6] and casting process [7].

Recently, it was reported that a thermal stable icosahedral quaiscrystalline phase (I-phase) with chemical composition of Mg_3YZn_6 was formed as a coarse eutectic structure in the α-Mg matrix during conventional solidification in Mg-Zn-Y alloy system [8]. The existence of the two-phase (I-phase + α-Mg) region in the Mg-Zn-Y alloy system provides an opportunity to develop quasicrystal-reinforced magnesium matrix composites by thermal processing. Conventional thermomechanical processes, such as hot rolling [9, 10] and extrusion [11], have been employed for the alloy system to separate and distribute the I-phase in the α-Mg matrix, thus *in-situ* I-phase/magnesium matrix composites with high strength and ductility have been achieved [9–11].

Equal channel angular extrusion (ECAE) is a novel metal forming process to produce severe plastic strains in bulk material without changing the cross-section of the material [12]. This procedure has been recognized as one of the most effective methods in producing bulk ultra-fine grained (UFG) materials with sub-microcrystalline structure for a wide range of materials, including magnesium alloys [13]. Therefore, in the present study, ECAE was performed on a Mg-Zn-Y alloy containing quasicrystal in order to obtain a high performance *in-situ* quasicrystal reinforced magnesium matrix composite, and the microstructures and mechanical properties of the composite were examined.

Mg-Zn-Y alloy having a composition of Mg-11 wt.% Zn-0.9 wt.% Y was melted in an electrical furnace under a dynamic SF_6 and CO_2 mixed gas atmosphere, and cast into an air-cooled metal mould. After casting, $10 \times 10 \times 60$ mm^3 rectangular billets were machined from the ingot for ECAE processing. The ECAE was carried out on the billets through a die with a channel angle of 90° and a curvature angle of 30°. All pressings were conducted by rotating each billet about the longitudinal axis by 90° in the same direction between consecutive passes (designated as route Bc). The specimen was extruded at 325 °C for the first pass, 275 °C for the second up to the eighth pass at a pressing speed of 40 mm/min. MoS_2 paste was used as lubricant during ECAE. Samples for microstructure observation and mechanical testing were cut from the ECAE processed billets in the longitudinal direction. Phase analyses were performed with a Philips X'Pert X-ray diffractometer (XRD) with Cu Kα radiation. The microstructures of the samples were examined by optical microscope and Philip CM 12 transmission electron microscope (TEM). Tensile tests were conducted on dog-bone shaped samples with 12 mm gauge length, 6 mm width and 2 mm thickness at a crosshead speed of 1 mm/min.

XRD pattern of the as-cast Mg-Zn-Y alloy is shown in Fig. 1. The peak analysis revealed that the alloy consisted of two phases of α-Mg and icosahedral quasicrystal phase (I-phase).

Fig. 2 shows the typical microstructure of the as-cast Mg-Zn-Y alloy. The grain sizes of the α-Mg were in the range of 60–100 μm, as shown in Fig. 2a. Eutectic phases located in the interdendritic region were clearly observed in Fig. 2b. In Fig. 2c, the selected area diffraction pattern (SADP) taken from the eutectic lamellar shows 5-fold symmetry, which is a distinct

*Author to whom all correspondence should be addressed.

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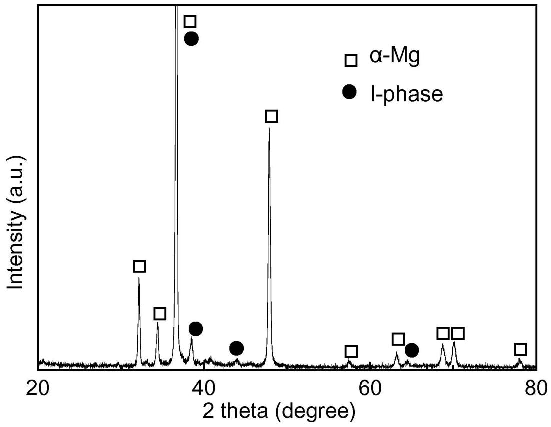

Figure 1 XRD patterns of the as-cast Mg-Zn-Y alloy.

characteristic of the I-phase. The volume fraction of the I-phase in the alloy was about 9%.

Fig. 3 shows the optical micrographs of Mg-Zn-Y alloy after ECAE for different passes. With increasing passes of ECAE, the α-Mg matrix grains were significantly refined, while the eutectic I-phases were gradually broken, and dispersed in the matrix. After 4-pass ECAE, as shown in Fig. 3a, the Mg-Zn-Y alloy consisted of fine α-Mg matrix grains with grain size about 4–8 μm, suggesting that dynamic recrystallization took place during the ECAP processing. However, deformation structures were generally inhomogeneous and coarse grains with grain size above 15 μm were still present. On the other hand, the broken I-phase tended to distribute along the direction of about 45° inclined to the longitudinal axis. After 8-pass ECAE, the α-Mg matrix grains were refined further, as shown in Fig. 3b, and a homogeneous equiaxed grain structure with an average grain size of about 3–4 μm was obtained, and the I-phase did not show obvious distribution orientation. The microstructure of the alloy after ECAE processing indicated that *in-situ* quasicrystal/magnesium matrix composites can be fabricated by ECAE.

Fig. 4 shows the TEM micrographs of the Mg-Zn-Y alloy subjected to 8-pass ECAE. As shown in Fig. 4a, the eutectic I-phases were broken into fine irregular shaped particles of 0.5–1 μm in size, and distributed in the α-Mg matrix close to the original I-phase pocket during ECAE. It is noted that no cavity was observed at the I-phase/α-Mg interface in the composite subjected to severe plastic straining, in spite of the different strength and plastic deformation behavior of I-phase and α-Mg. In addition, some massive I-phases were remained unbroken even after 8-pass ECAE, as shown in Fig. 4b. Small amount of deformation twins were observed in the 8-pass ECAE processed material. Furthermore, TEM observations revealed that fine precipitates were formed in the ECAE processed material, as shown in Fig. 4b. The fine I-phase precipitates have

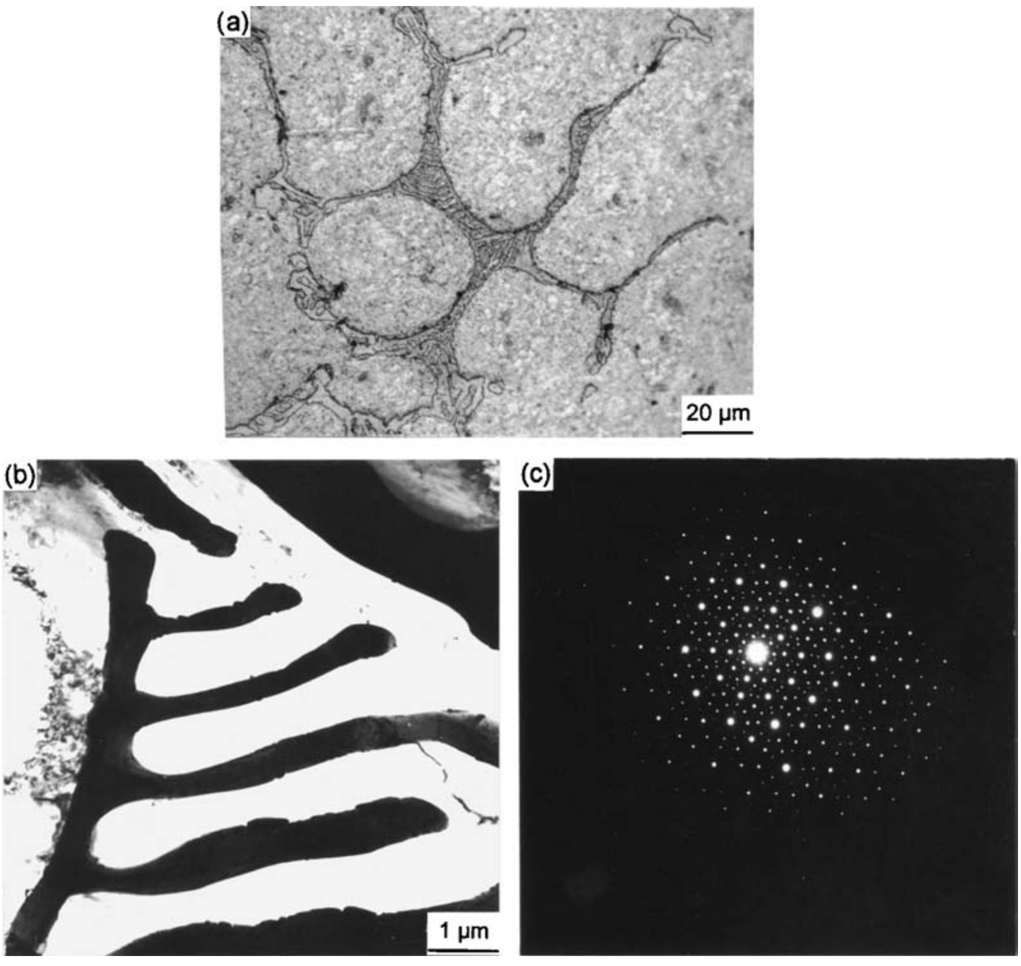

Figure 2 Typical microstructures of the as-cast Mg-Zn-Y alloy: (a) optical microstructure showing the eutectic pockets, (b) TEM micrograph of the eutectic I-phase and (c) SADP from the I-phase exhibiting 5-fold symmetry.

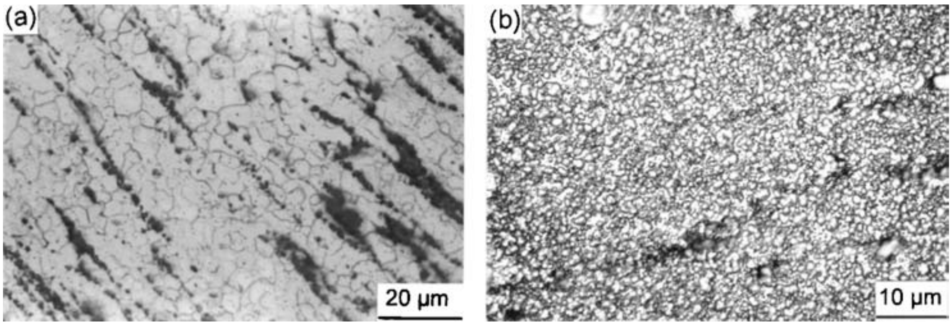

Figure 3 Optical microstructures of ECAE processed magnesium matrix composites: (a) after 4-pass ECAE and (b) after 8-pass ECAE. The extrusion direction is horizontal.

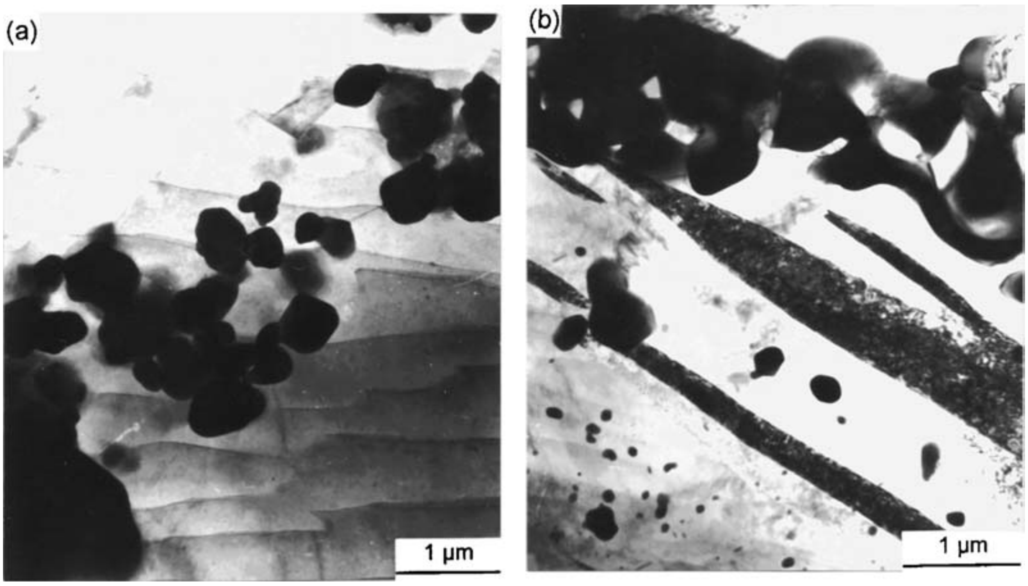

Figure 4 TEM micrographs of the 8-pass ECAE processed magnesium matrix composite: (a) dispersed I-phase near the original eutectic pocket and (b) undestroyed eutectic I-phase, twins and fine I-phase precipitates in the matrix.

been observed to be formed during hot extrusion and hot rolling of this kind of Mg-Zn-Y alloys [9, 11]. This suggests that the fine precipitates formed during the ECAE processing may also be I-phase.

Table I shows the tensile properties of the *in-situ* I-phase/magnesium matrix composites processed with different thermomechanical processes. The tensile strength and ductility of the ECAE processed composites were increased significantly compared with those of the as-cast material, indicating that ECAE is very effective in the improvement of tensile properties of material. After 8-pass ECAE, the ultimate tensile stress was about 287 MPa, the tensile elongation reached 26%. This suggests that a good combination of high strength and high ductility for this composite can be achieved by ECAE. The high strength and ductility is considered to be resulted from the fine grain sizes of the matrix alloy and the dispersion of I-phase in the matrix.

TABLE I Tensile properties of *in-situ* quasicrystal reinforced magnesium matrix composites

Materials	0.2% proof stress ($\sigma_{0.2}$, MPa)	UTS (σ_b, MPa)	Elongation to failure (ε, %)
As-cast	80	105	0.8
4-pass ECAE	151	276	10
8-pass ECAE	160	287	26
Normal extrusion [15] (350 °C, extrusion ratio 9:1)	273	376	19

During ECAE processing, dynamic recrystallization led to an equiaxed and homogeneous fine grains of α-Mg matrix alloy. Furthermore, a small amount of I-phase particles effectively acted as dynamic recrystallization sources during ECAE, helping to refine the grains of the α-Mg matrix.

The I-phase particle had a good strengthening effect on the Mg matrix alloy. The TEM observations indicate that the I-phase was bonded well with the magnesium matrix alloy. The strong I-phase/Mg matrix interface may allow effective load transfer from the matrix to I-phase. In addition, the broken I-phase particles and the fine I-phase precipitates can provide effective obstacles to dislocation movement during deformation [9, 11] and have a dispersed strengthening effect on the Mg matrix. Furthermore, the stable quasicrystalline -matrix interface with a low interfacial energy [14] can provide the improved elongation in the composite.

Compared with the normal extruded composite with the same composition [15], the ECAE processed composite exhibited a higher elongation to failure and lower tensile strength, in spite of the finer matrix grain size

(3–4 μm) in the ECAE processed composite than that in normal extruded composite (6–7 μm) [15]. Chino *et al.* [16] also reported that the flow stress of the ECAE processed AZ91 magnesium alloy was lower than that of the normal extruded AZ91 alloy in spite of almost the same grain size. The lower tensile strength in the ECAE processed composite is considered to be attributed to the different textures of α-Mg matrix alloy and the different distribution of I-phase obtained in the two different extrusion processes. Further work is needed to understand the effect of both α-Mg texture and the distribution of I-phase on the mechanical properties of the composite.

As a conclusion, ECAE can effectively refine the α-Mg matrix grain size, break and disperse the I-phase in Mg-Zn-Y alloy, providing an *in-situ* I-phase/magnesium matrix composite. The composite exhibited a good combination of high strength and high ductility, which is ascribed to the fine grain size of α-Mg matrix and fine dispersed I-phase particles and I-phase precipitates in the matrix.

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