Microstructural Evolution of Friction Stir Processed Cast Mg-5.9 mass%Y-2.6 mass%Zn Alloy in High Temperature Deformation

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Cast Mg-Y2-Zn1 alloy was friction stir (FS) processed to obtain fine grain structure. Then the elevated temperature tensile tests were carried out to investigate the deformation behavior of the FS processed alloy. The starting structure for FS processing was coarse dendritic microstructure (secondary dendrite arm spacing ca. 75 µm) with a plate-like second phase, which developed from inter-dendritic eutectic pockets after homogenization heat treatment. The α-Mg matrix and second phases were broken mechanically and distributed homogeneously by FS processing. The refined grain size at the fully stirred zone had the average size of ca. 2.0 µm. Different deformation behavior occurred as a result of varying microstructural evolution according to temperatures (623 K and 723 K).

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1. Introduction

Magnesium alloys have been investigated in the transportation industry for lightweight structural applications. To date, die-casting has been used to produce most large structural Mg-based parts, however, cast Mg alloys are known to have poor mechanical properties because of coarse grain size and inhomogeneous microstructure. Therefore, high strength is required for wider applications of Mg alloys. Reportedly, yttrium suppressed basal slip and caused secondary slip systems of cross slipping. Second phase particles, which show close coherency between the α-Mg matrix and icosahedral quasicrystal (I-phase of Mg51YZn6), also increased in Mg-Y-Zn alloy strength. Rapidly solidified powder metallurgy (RS P/M) Mg-Y2-Zn1 alloy has high creep-resistance, high strength (up to ca. 600 MPa) at room temperature, and super-plasticity at 623 K. Various strengthening methods were applied to this material: solid solution strengthening (Y and Zn), second phases (Mg2Y5 particles or quasi-crystals), fine grains by hot extrusion (average grain size and particles of ca. 200 nm) and long period ordered structure (6H structure). Watanabe et al. reported a fine-grained Mg-Y2-Zn1 alloy produced using ECAE that showed higher hardness than AZ91 or even RS P/M Mg-Y2-Zn1 alloy. This strength resulted from the fine grain and distribution of the second-phase particles (Mg12-YZn). Recently the authors reported that FS processing is a viable alternative process to produce bulk fine grained Mg-Y2-Zn1 from a cast. Morishige et al. reported good performance of FS-processed Mg-Y2-Zn1.5 alloy at room temperature (σy of 322 MPa, UTS of 345 MPa, 6.5% elongation) and higher hardness when loaded to the Hall-Petch relationship with Mg-Y-Zn alloys produced using other metalworking processes. Therefore, strengthening and forming of processed bulky alloy by elevated temperature forging requires understanding of either second phases or grain size.

In this work, the microstructural evolution of FS processed cast Mg-Y2-Zn1 alloy by elevated temperature tensile tests is examined using microscopic analyses, X-ray diffractometry, and temperature measurement.

2. Experimental Procedures

The material used in this study was Mg-5.9 mass%Y-2.6 mass%Zn (Mg-Y2-Zn1) alloy. Casting was carried out by melting high-purity Mg, Y, and Zn in an Ar gas atmosphere (0.15 MPa). A 200 mm wide × 200 mm long × 10 mm thick block was cut from the center of the ingot. Homogenization treatment (95 h at 773 K) was carried out before FS processing. The microstructures of as-cast and as-homogenized Mg-Y2-Zn1 alloys are shown in Figs. 1(a) and 1(b). X-ray diffractometry (XRD) was carried out to check the second phase particles in as-cast and as-FS-processed material using a CuKα X-ray.

Using an appropriate tool (5.5 mm probe length and 20 mm shoulder diameter), FS processing was performed on the cut homogenized block. The conditions of FS processing were: rotation speed of 450 rpm and traveling speed of 0.833 mm/s at constant loading of 32 kN, which was reported as a good weld condition in the former report. The FS-processed area was ca. 80 × 100 mm after 30 passes. Temperature histories at more detail (see more detail in Fig. 2(a)) were detected using K-type thermocouples during FS processing. Tensile...

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Fig. 1 Optical microstructures of the as-cast Mg-Y2-Zn1 (a) and after homogenization (b).
After FSP colloidal Almond paste. Subsequently, they were fine-polished with
the second phase of large Mg3. The constant strain rate of
m3 varied with temperatures of 623, 673 and 723 K (at a
tensile tests).

Fig. 2 Schematic illustration of FS processing and position of thermocou-
pltes (a), and temperature–time diagrams during processing (b).

Fig. 3 Optical microstructure of SZ (a) and TMAZ (b).

Fig. 4 XRD analysis of as-cast (a) and as-FS-processed (b).

3. Results

As-cast Mg-Y2-Zn1 alloy showed dendrite structures surrounded by eutectic pockets in Fig. 1(a). The secondary dendrite arm spacing (SDAS) was estimated as about 75 µm and the measured cooling rate in casting was 0.1–1 K/s. Figure 1(b) showed that a lamellar structure developed from the eutectic pockets into the matrix, as reported.10,11

In Fig. 3(a), drastic dynamic recrystallization (DRX) is apparent in the stir zone (SZ) when compared to the as-homogenized one (Fig. 1(b)). In SZ, DRX occurred fully. The measured size of the lamellar colony that existed in the fully stirred zone was ca. 2.0 µm. In Fig. 3(b), TMAZ is shown in the process of DRX (mixture of elongated coarse grains and refined grains). The elongation of grains aligned on the tangential direction to the tool’s rotating axis. This indicates that the deformation in TMAZ was shear deformation around the rotating probe. However, insufficient thermo-

e-mechanical energy produced insufficient DRX compared to that of SZ.

The value of TMAX 0.3 mm distant from the probe tip surface was detected as 665 K at the position of P3 in Fig. 2(b). The maximum temperature in the SZ is somewhat higher than the measured temperature. It might activate recrystallization or grain growth for Mg-Y2-Zn1 alloys.10,11

The second phase of large Mg3YZn6 quasi-crystal (stable to ca. 723 K4) was not affected thermally. From the temperature histories shown in Fig. 2(b), the cooling rate after FS processing was roughly calculated.7 At the positions contacted by thermocouples, the cooling rate was ca. 4 K/s during cooling from 665 K to 473 K; it then decreased slightly to ca. 0.5 K/s (close to furnace cooling). The second phase of Mg12YZn is reported stable to ca. 673 K.7 Cast and heat treated fine plate-like second phase might be resolved and re-precipitate by the rapid heating and immediate cooling during FS processing. Anyhow, these second-phase particles were distributed homogeneously by FS processing (stirring the eutectic envelope by sever plastic deformation (SPD)) at TMAX of ca. 665 K and at a cooling rate of ca. 4 K/s.

Figure 4 shows XRD results of as-cast (a) and FS-

processed Mg-Y-Zn (b). As-cast and FS-processed Mg-Y2-

Zn1 displayed similar peaks, which were identified as a mixture of α-Mg matrix and second phases. Reported second-phase particles in Mg-Y-Zn systems are Mg3YZn6,10,11 Mg12YZn,7 an inter-metallic compound of Mg3Y2Zn3, and Mg624Y5.6 The peaks indicated that the second phases that existed in both as-cast and FS-processed materials were quasi-crystal (Mg3YZn6), Mg12YZn, and α-Mg matrix. Therefore, second-phase particles (Mg3YZn6 and Mg12YZn) that had developed during casting and homogenization treatment remained after FS processing.

Figure 5 shows the tensile elongation-to-failure test results for various temperatures of 623, 673 and 723 K at a constant strain rate of 10^{-3} s^{-1}. It is noteworthy that the flow stress at 623 K increases, then decreases gradually to a plateau, and finally to fracture. This σ-ε profile resembles that of DRX of
superplastic Al5083 alloy. On the other hand, when tensile tested at 723 K, flow stress increased gradually to UTS, indicating slight work hardening. Figure 5 displayed different deformation behavior in tensile tests at various temperatures.

The SEM microstructures after tensile tests at 623 K and 723 K are shown respectively in Figs. 6(a) and 6(b). The Mg$_3$YZn$_6$ particles (B in Fig. 6) were distributed homogeneously in both photos. However, grain growth occurred clearly (2.0 µm to ca. 7.5 µm) after tensile tests at high temperature (723 K) although little change of grain size occurred in the tensile sample at 623 K (ca. 2.3 µm). The grains that were tensile-tested at 723 K were elongated to the tensile direction when analyzed using an optical microscope (aspect ratio ca. 3.2, horizontal/vertical length of grains in Fig. 6(c)). Segregations of second phases were visible (C in Fig. 6).

Micro-Vickers hardness results of selected tensile tested samples were of a similar level ($H_v$ ca. 130) for the samples tensile tested at 623 K, but decreased ($H_v$ ca. 80) for the sample tested at 723 K.

4. Discussion

In the case of insufficient Y or Zn elements in Mg-Y-Zn alloy systems, second phases did not occur. However, Y or Zn atoms moved on the basal plane during creep deformation, and suppressed dislocation’s basal glide, subsequently activating cross slip or secondary slip systems. For sufficient Y and Zn to form a second phase, the possible second phases in Mg-Y-Zn alloys have been investigated: Mg$_3$YZn$_6$, Mg$_3$Y$_2$Zn$_3$, and Mg$_{12}$YZn. It has been suggested that the cooling rate might rule the formation of second phases in Mg-Y$_2$Zn$_1$ alloys. In fact, RS P/M Mg-Y$_2$Zn$_1$ is believed to have had the fastest cooling rate and formed Mg$_{24}$Y$_5$. Rapid quenching followed (estimated cooling rate was ca. 50 K/s) and formed Mg$_{12}$YZn. In conventional casting of Mg-Y-Zn alloys, usually Mg$_3$YZn$_6$ occurs as a quasicrystal. These second phases have been well observed in the microstructure of as-cast samples and are called ‘eutectic pockets’ or ‘eutectic envelopes’ (see Fig. 1(b)). Figure 1(b) shows that, on the other hand, Y or Zn elements segregated in the eutectic pockets moved into the matrix and generated a needle-like second phase. The second phases (quasicrystal of Mg$_3$YZn$_6$ and second phase of Mg$_{12}$YZn) are thermally stable to up to ca. 673 K. Therefore, second phases after FS processing were believed to be mechanically broken and stirred eutectic pockets at $T_{MAX}$ ca. 665 K (Fig. 2(b)).

These second phases were changed after tensile tests at various temperatures. Grain growth occurred at the high temperature of 723 K (Fig. 6(b) and 6(c)). Usually, second phases are stronger than the matrix and suppress grain growth because mobile dislocations move only slightly through the grains.

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**Fig. 5** Tensile elongation-to-failure test results shown for various temperatures (623–723 K) at a constant strain rate of $10^{-3}$ s$^{-1}$.  

**Fig. 6** SEM microstructures after tensile testing: at 623 K (a) and 723 K (b) and optical microstructure at 723 K (c).
second phase. However, the grains in Fig. 6(c) were elongated and large when compared to samples at the low temperature of 623 K (Fig. 6(a)). These results suggest that fine grains after FS processing were changed to a mixture of an $\alpha$-Mg matrix (A in Fig. 6(c)), quasicrystal (B: Mg$_3$YZn$_6$), and segregated second phases (C: Mg$_{12}$YZn) during grain growth and plastic deformation at the high temperature (723 K). However, understanding the segregation of second phases in Mg-Y-Zn alloys will require more detailed study using widely various temperatures and strain rates.

The micro-Vickers hardness results indicated strengthening of the material. The Hall-Petch relation can show the strengthening of a material considering the grain size.$^{7,9}$ In Fig. 7, the Hall-Petch relationship of Mg-Y-Zn alloys is shown with different processing histories. Morishige et al. reported an increase of room temperature (RT) hardness of FS-processed Mg-Y$_2$-Zn$_1$ and Mg-Y$_2$-Zn$_{1.5}$ alloys over the hardness displayed by$^9$ FS-processed Mg-Y$_1$-Zn$_{0.2}$ alloy,$^{15}$ ECAE-processed,$^7$ or RS P/M Mg-Y$_2$-Zn$_1$ alloys.$^{5,6}$ However, hardness decreased with the grains grown when tensile tested at 723 K, but did not decrease with similar grain size when tensile tested at 623 K (see Fig. 7). This high level of hardness might result not only from grain refinement, but also distribution of second phases. Bae et al. reported that the quasicrystal and $\alpha$-Mg matrix had strong coherency, which engendered high strength of Mg-Y-Zn alloy.$^4$ According to Watanabe et al.,$^7$ ECAE processed Mg-Y$_2$-Zn$_1$ containing Mg$_{12}$YZn particles, which increased in strength at RT.

In this study, we investigated microstructural evolution of Mg-Y$_2$-Zn$_1$ alloy after casting, FS processing and tensile tests. Second phases depended on thermo-mechanical processes. Very high hardness at RT and high ductility at high temperature underscored the reliability of bulk FS-processed Mg-Y$_2$-Zn$_1$ alloy.

5. Summary

(1) Microstructure of cast Mg-Y$_2$-Zn$_1$ alloy has evolved by thermal and/or mechanical treatments (homogenization, FS processing and tensile elongation-to-failure tests at high temperature). A lamellar structure developed from the eutectic pockets by homogenization treatment. Then the microstructure was changed considerably by FS processing to very fine lamellar colonies (ca. 2 $\mu$m). Grain growth and segregation of second phases occurred at the very high temperature of 723 K, but did not change at 623 K.

(2) Tensile tests at high temperature showed slight work hardening when tested at 723 K, but it showed DRX at 623 K.

(3) The FS-processed Mg-Y$_2$-Zn$_1$ alloy, which was tensile tested at 723 K, lost its strength to the extent. In contrast, another sample, which was tensile-tested at 623 K, showed considerably increased hardness compared to the FS-processed one.

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