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Scripta Materialia 65 (2011) 335–338

Scripta MATERIALIA

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Achieving high strain rate superplasticity in Mg–Zn–Y–Zr alloy produced by friction stir processing

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Received 24 March 2011; revised 28 April 2011; accepted 29 April 2011
Available online 4 May 2011

Friction stir processing was applied to hot-rolled Mg–Zn–Y–Zr alloy to produce a fine-grained structure 4.5 μm in size with fine, uniformly distributed $Mg_3Zn_3Y_2$ particles and predominant high-angle grain boundaries (HAGBs) of 91%. A maximum superplasticity of 1110% was achieved at a high strain rate of $1 \times 10^{-2}\ s^{-1}$ and 450 °C. The superior superplasticity at this high strain rate is attributed to the excellent thermal stability of the fine-grained structure and the high percentage of HAGBs.

Keywords: Superplasticity; Magnesium alloys; Friction stir processing; Microstructure; Grain boundaries

Superplastic forming (SPF) is an effective method of fabricating hard-to-form materials into complex shapes [1]. For SPF to be used in industry, there is a need to develop high strain-rate superplasticity (HSRS, strain rate $\geqslant 1 \times 10^{-2}\ s^{-1}$), especially for magnesium alloys with poor workability. The constitutive equation for superplasticity of fine-grained materials predicts that HSRS could be achieved by reducing the grain size and/or increasing the deformation temperature. However, the current experimental data show that it is difficult for magnesium alloys to exhibit HSRS even when the grain size is reduced to less than 1 μm [2–4]. This is mainly attributed to the poor thermal stability of magnesium alloys at higher temperature due to the absence of thermally stable pinning particles. In this case, the superplasticity is usually developed at relatively low temperatures, and HSRS is hard to achieve due to significant grain growth at higher temperatures. It is suggested that, by increasing the thermal stability of fine-grained magnesium alloys, HSRS may be achieved at higher temperatures [5].

Mg–Zn–Y–Zr alloy has been developed as a new high-strength magnesium alloy for elevated temperature applications [6]. With the presence of two ternary thermally stable equilibrium phases, i.e. the icosahedral quasicrystal I phase (Mg_3Zn_6Y, with a eutectic point of ~450 °C) and the W phase ($Mg_3Zn_3Y_2$, with a eutectic point of ~510 °C) [7,8], these alloys are more thermally stable than conventional magnesium alloys [9–11]. Therefore, it is expected that HSRS could be achieved in Mg–Zn–Y–Zr. However, several previous studies, using different processing methods, were unable to achieve HSRS in such alloys. For example, Bae et al. [10] reported a maximum elongation of 780% in a hot-rolled Mg–3.0Zn–0.5Y–1.5Zr alloy at 450 °C and a strain rate of $5 \times 10^{-4}\ s^{-1}$. Zheng et al. [12] found that an equal-channel angular processed (ECAP) Mg–4.3Zn–0.7Y exhibited a maximum elongation of ~600% at 350 °C and $1.5 \times 10^{-4}\ s^{-1}$. Similarly, Tang et al. [11] reported that an ECAP Mg–5.8Zn–1.0Y–0.48Zr alloy exhibited an elongation of 800% at 350 °C and $1.7 \times 10^{-3}\ s^{-1}$.

Friction stir processing (FSP) has been developed as a solid-state processing technique, based on the concepts of friction stir welding [13]. FSP results in the generation of a fine-grained structure and the break-up and uniform distribution of second-phase particles, thereby enhancing the superplasticity of the stir zone (SZ) significantly [14]. A number of studies indicated that HSRS and/or low-temperature superplasticity could be easily achieved in fine-grained aluminum alloys prepared via FSP [15–17]. For magnesium alloys, it was reported that FSP could result in enhanced superplasticity [18].

Xie et al. [19] produced a fine-grained Mg–Zn–Y–Zr alloy containing only W phase via FSP at a tool rotation

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1359-6462/$ - see front matter
doi:10.1016/j.scriptamat.2011.04.041

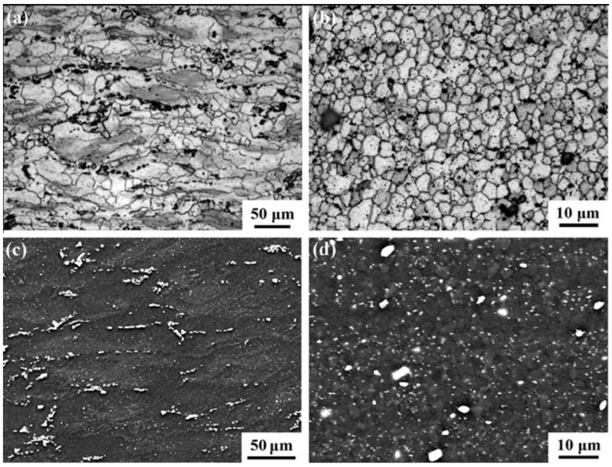

Figure 1. OM and SEM micrographs showing the microstructure of Mg–Zn–Y–Zr alloy: (a and c) rolled BM; (b and d) the FSP sample.

rate of 800 rpm and a traverse speed of 100 mm min^{-1}. A maximum elongation of 635% was obtained at 450 °C and a relatively high strain rate of 3×10^{-3} s^{-1}. However, the W particle size was relatively large in their study. It is expected that enhanced superplasticity would be achieved at higher strain rates if the size of the W phase particles was further reduced. In this study, Mg–Zn–Y–Zr alloy containing only W phase was subjected to FSP at a higher tool rotation rate in order to obtain finer W phase particles, and superplastic behavior was examined. The aim is to achieve HSRS in the magnesium alloys.

An 8 mm thick Mg–7.12Zn–1.2Y–0.84Zr (wt.%) hot-rolled plate was subjected to FSP at a rotation rate of 1500 rpm and a traverse speed of 100 mm min^{-1}. A steel tool with a concave shoulder 20 mm in diameter and a threaded conical pin 8 mm in root diameter and 4.9 mm in length was used. Microstructural characterization was performed on the cross-section of the SZ by optical and scanning electron microscopy (OM and SEM). Electron backscatter diffraction (EBSD) orientation maps were obtained using a ZEISS SUPRA 35. Samples for the EBSD observation were electropolished in a solution consisting of 60 ml nitric acid and 140 ml ethanol at 15 V, followed by ion milling.

Mini tensile specimens (2.5 mm gage length, 1.4 mm gage width and 1.0 mm gage thickness) were machined perpendicular to the FSP direction, with the gage length being centered in the SZ. The specimens were subsequently ground and polished to a final thickness of 0.8 mm. Each specimen was fastened in the tensile testing apparatus when the furnace was heated to the selected testing temperature, then held at this temperature for 20 min to establish thermal equilibrium prior to the tensile test. Tensile tests were conducted at a constant crosshead speed using an Instron 5848 microtester. For comparison, tensile tests of the rolled samples were also conducted with the tensile axis parallel to the rolling direction.

The microstructures of the Mg–Zn–Y–Zr alloy are shown in Figure 1. The as-rolled base metal (BM) exhibited a typical strip-like rolling structure containing coarse and nonuniform grains and large and heterogeneously distributed W phase particles (Fig. 1a and c). By comparison, in the FSP sample, a uniform fine-grained structure 4.5 μm in size was observed (Fig. 1b) and the W particles were remarkably refined and uniformly distributed (Fig. 1d). While most of the W particles had a size of less than 1 μm, a small number of large W particles about 2–4 μm in size were also observed.

Figure 2a shows the microstructure of the FSP Mg–Zn–Y–Zr obtained by EBSD mapping. High-angle grain boundaries (HAGBs, grain boundary misorientations ⩾15°) and low-angle grain boundaries (grain boundary misorientations <15°) are shown by black and white lines, respectively. The microstructure was characterized by equiaxed grains with predominant HAGBs. The fraction of the HAGBs was determined to be 91% (Fig. 2b).

The microstructural change during FSP suggested that FSP could effectively refine the grains and break up the W particles due to extensive plastic deformation and dynamic recrystallization. Compared to the FSP Mg–Zn–Y–Zr in Ref. [19], the FSP Mg–Zn–Y–Zr in this study exhibited finer grains and much finer, more uniform W particles. This is attributed to more intense plastic deformation resulting from the higher rotation rate in this study.

Figure 3a shows the variation in elongation with initial strain rate for the Mg–Zn–Y–Zr alloy at 400 and 450 °C. Elongation of the rolled Mg–Zn–Y–Zr was low and did not exhibit any appreciable dependence on the strain rate. In contrast, the FSP Mg–Zn–Y–Zr showed superior superplasticity. At 400 °C and strain rates of 1×10^{-3}–1×10^{-2} s^{-1}, elongations of above 600% were achieved. The optimum strain rate was found

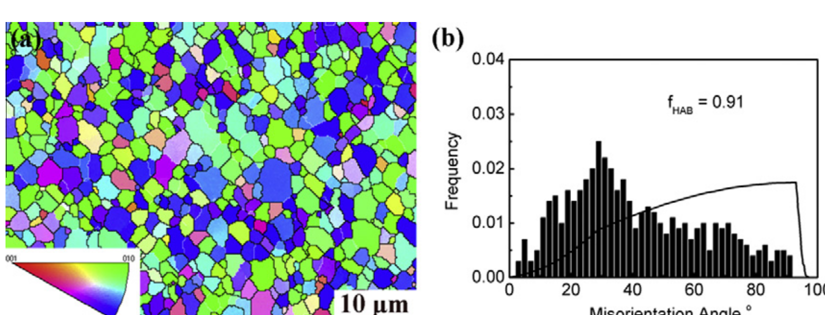

Figure 2. Microstructure of FSP Mg–Zn–Y–Zr: (a) EBSD map and (b) boundary misorientation angle distribution.

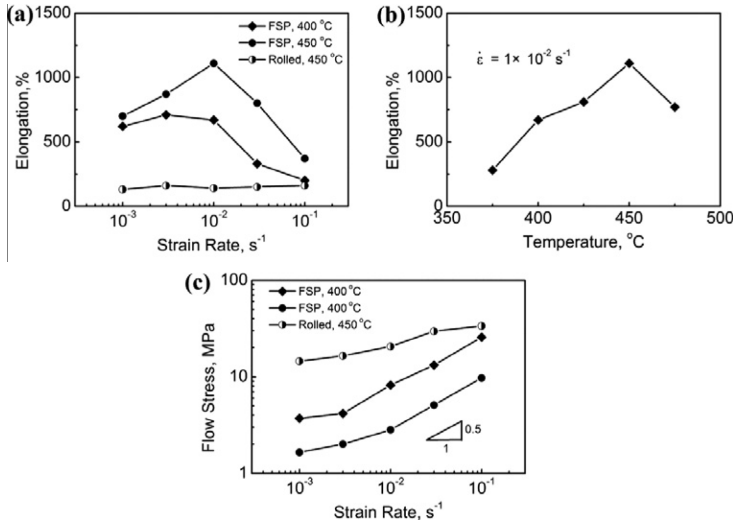

Figure 3. Variation in elongation with (a) initial strain rate and (b) temperature; (c) variation in flow stress with initial strain rate for Mg–Zn–Y–Zr alloy.

to be $3 \times 10^{-3}\ s^{-1}$. At 450 °C, the elongations at all strain rates increased significantly and the optimum strain rate increased to $1 \times 10^{-2}\ s^{-1}$, with a maximum elongation of 1110% being obtained. Even at $3 \times 10^{-2}\ s^{-1}$, an elongation of 800% was observed.

Figure 3b shows the effect of temperature on the elongation of the FSP Mg–Zn–Y–Zr at an initial strain rate of $1 \times 10^{-2}\ s^{-1}$. The FSP alloy exhibited superplasticity within a wide high-temperature range of 375–475 °C at $1 \times 10^{-2}\ s^{-1}$. The optimum temperature was determined to be 450 °C. Thus, a maximum superplasticity of 1110% was achieved at 450 °C and a high strain rate of $1 \times 10^{-2}\ s^{-1}$.

The variation of flow stress with the initial strain rate for both the FSP and rolled Mg–Zn–Y–Zr is shown in Figure 3c. The rolled Mg–Zn–Y–Zr exhibited a strain-rate sensitivity m of ~0.1; this accounts for the absence of superplasticity in the rolled alloy. For the FSP sample, the m value of ~0.5 was observed at strain rates of 3×10^{-3} to $1 \times 10^{-1}\ s^{-1}$ for 400 °C and at strain rates of 1×10^{-2} to $1 \times 10^{-1}\ s^{-1}$ for 450 °C. This is consistent with the optimum superplasticity in the FSP Mg–Zn–Y–Zr. The m value of ~0.5 is associated with a grain boundary sliding (GBS) mechanism [20].

All the tensile specimens showed relatively uniform elongation characteristic of superplastic flow (Fig. 4a). Figure 4b shows the OM image of the grip region of the specimen deformed to failure at 450 °C and $1 \times 10^{-2}\ s^{-1}$. Clearly, the FSP Mg–Zn–Y–Zr exhibited superior thermal stability, as the grain size was funda-

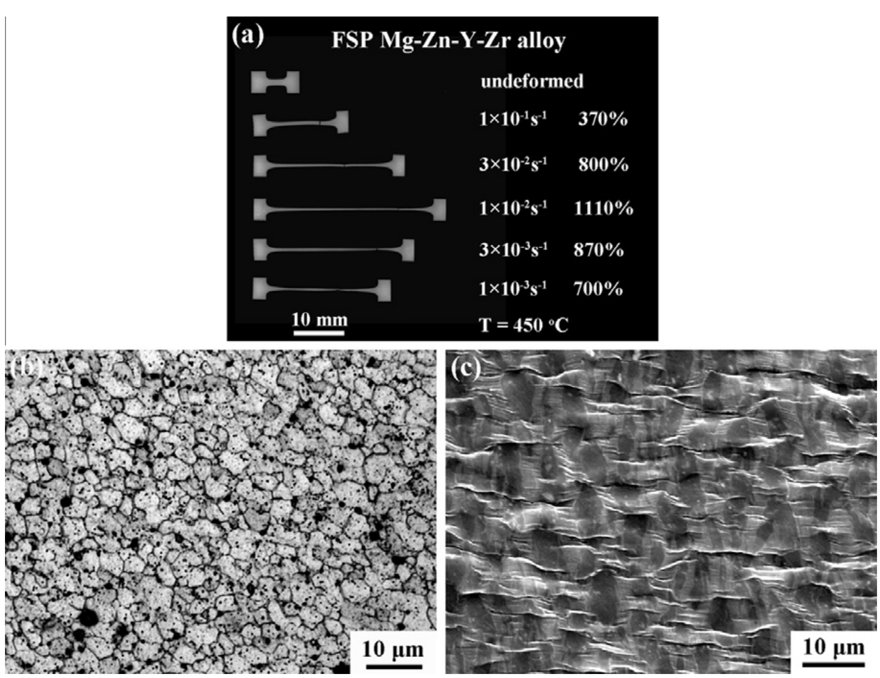

Figure 4. (a) Tensile specimens pulled to failure; (b) microstructures in the grip region after being deformed to failure at 450 °C and $1 \times 10^{-2}\ s^{-1}$; (c) surface morphology of the specimen deformed at a strain of 1.1 at 450 °C and $1 \times 10^{-2}\ s^{-1}$ (tensile axis is horizontal).

mentally unchanged after high-temperature annealing (Figs. 1b and 4b). Furthermore, the W phase particles did not exhibit noticeable growth. This confirms that the fine, uniformly distributed W particles were very thermally stable and, therefore, very effective in pinning the grain boundaries at higher temperatures. Thus, greater superplasticity could be achieved at higher temperatures and higher strain rates. The surface morphology of the FSP Mg–Zn–Y–Zr deformed to a true strain of 1.1 at 450 °C and a strain rate of $1 \times 10^{-2}\ s^{-1}$ is shown in Figure 4c. GBS was clearly observed, and this was attributed to the predominance of HAGBs produced via FSP (Fig. 2b). It is well documented that HAGBs are beneficial to GBS [21].

Although the coherent I phase was reported to favor superplastic deformation, as it prevents cavity formation at the interface during high-temperature deformation [9,11], the superplastic investigations in this study and in Ref. [19] suggest that, for the Mg–Zn–Y–Zr alloys containing incoherent W phase, enhanced superplasticity could also be achieved. Therefore, it seems that the interface coherency between the second phase and the matrix is not a dominant factor affecting the superplastic behavior of the Mg–Zn–Y–Zr alloys.

It is well known that fine grains and secondary particles are beneficial to enhancing superplasticity [20]. For the Mg–Zn–Y–Zr alloys deformed at much higher temperatures than conventional magnesium alloys, relatively coarse grains and thermally stable fine particles were critical to ensure the thermal stability required for superplastic deformation. The coarse grains show better thermal stability and the dense, fine particles exert a strong pinning effect on the grain growth. The reported alloys containing the I phase exhibited a structure of either fine grains with fine particles or coarse grains with large particles [11,12,22]. Such microstructures are not thermally stable, thus the optimum superplastic temperature was restricted to lower temperatures and the superplastic elongation was limited.

The superior superplasticity at the high strain rate in the present FSP Mg–Zn–Y–Zr with only W phase particles is attributed to the following three factors. First, the fine, uniformly distributed W particles could exert a strong pinning effect on the grain boundaries. Thus, the FSP Mg–Zn–Y–Zr with relatively large grains and densely distributed fine particles exhibited good thermal stability. Second, the eutectic temperature of the W phase is higher than that of the phase I [19], thus higher deformation temperature could be applied for superplastic deformation in the Mg–Zn–Y–Zr with the W phase. Third, it has been reported that HAGBs can facilitate GBS, resulting in an acceleration of superplastic deformation [14,21]. The microstructure of the present FSP Mg–Zn–Y–Zr consisted predominantly of HAGBs (Fig. 2b). Therefore, the FSP Mg–Zn–Y–Zr exhibited superplasticity at a higher strain rate than those alloys produced by conventional processing methods.

It is noted that although the optimum superplastic temperature in the present FSP Mg–Zn–Y–Zr is the same as the FSP Mg–Zn–Y–Zr reported by Xie et al. [19], the present alloy exhibited significantly enhanced superplasticity at a higher strain rate and lower flow stress. There are two possible explanations for this enhanced superplasticity. First, the present alloy exhibited finer grains and W particles, which resulted in an enhancement in superplasticity and a shift in the optimum strain rate to a higher value when tested at the same temperature. Second, the number of large W particles in the present alloy was much lower than that in Ref. [19]. This reduced the possibility of cavitation at the particles [23], thereby enhancing superplastic elongation.

In summary, fine-grained Mg–Zn–Y–Zr with a grain size of 4.5 μm and fine, uniformly distributed W phase particles as well as predominant HAGBs was produced via FSP at a high tool rotation rate of 1500 rpm. The FSP Mg–Zn–Y–Zr exhibited a maximum elongation of 1110% at a high strain rate of $1 \times 10^{-2}\ s^{-1}$ and 450 °C.

This work was supported by the National Natural Science Foundation of China under Grant Nos. 50901075 and 50890171 and the National Outstanding Young Scientist Foundation under Grant No. 50525103.

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