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Achieving exceptionally high superplasticity at high strain rates in a micrograined Al–Mg–Sc alloy produced by friction stir processing

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Friction stir processing (FSP) was applied to extruded Al–Mg–Sc alloy to produce fine-grained microstructure with 2.6 μm grains. A maximum elongation of 2150% was achieved at 450 °C and a high strain rate of $1 \times 10^{-1}\ s^{-1}$. Although the grains obtained by FSP were much larger than those by other techniques, such as equal-channel angular pressing, approximately the same superplasticity was achieved at an even higher strain rate in the FSP alloy.
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Superplastic forming (SPF) is a well-established industrial process for the fabrication of complex shapes in sheet metals [1]. In practice, however, the widespread use of SPF of aluminum alloys is generally limited to low volumes of components due to the slow optimum strain rate for superplasticity. To use SPF in production-oriented industries, there is a need to develop high strain rate superplasticity (HSR SP) (optimum superplastic strain rate $\geqslant 10^{-2}\ s^{-1}$) to rejuvenate the area of superplasticity with its potential for large-scale applications of SPF. Besides the low strain rate, the complex and expensive thermomechanical processing (TMP), typically necessary to make these materials superplastic also restricts the commercial applications of SPF. A new, easy and inexpensive processing technique is needed for superplastic material production.

Al–Mg–Sc alloys were developed as a class of highly formable aluminum alloys with superior properties (especially yield strength) compared to conventional Al–Mg alloys with the same magnesium content [2]. They have the potential to be used in the fabrication of airframes, stiffened panels and tanks for condensed gas storage. The well-distributed, nanoscale-coherent Al_3Sc precipitates in the alloys are extremely thermodynamically stable and very effective at inhibiting dislocation movement and stabilizing the microstructure [3]. A number of studies have confirmed that Al–Mg–Sc alloys subjected to severe plastic deformation exhibited HSR SP with high ductility [4–10].

Several previous investigations indicated that elongation of more than 2000% could be achieved in ultrafine-grained Al–Mg–Sc alloys with grain sizes 0.2–1 μm [5,6,8]. Langdon et al. [5] reported that an Al–3Mg–0.2Sc alloy with a grain size of 0.2 μm, prepared by equal-channel angular pressing (ECAP), exhibited an elongation of ∼2280% at 400 °C and a strain rate of $3.3 \times 10^{-2}\ s^{-1}$. Similarly, Musin et al. [6] found that an ECAP Al–5.7Mg–0.32Sc–0.3Mn alloy with average grain size of ∼1 μm exhibited a maximum elongation of 2000% at 450 °C and an initial strain rate of $5.6 \times 10^{-2}\ s^{-1}$. More recently, Kaibyshev et al. [8] reported that a superior superplastic ductility of 2300% was achieved at 520 °C and $5.6 \times 10^{-2}\ s^{-1}$ in an Al–5Mg–0.2Sc alloy with a cell size of 0.2–0.5 μm produced by traditional chill casting followed by cold rolling with a total reduction of 80%.

Friction stir processing (FSP) has been developed as a solid-state processing technique [11,12], based on the concepts of friction stir welding (FSW) [13]. FSP causes intense plastic deformation and elevated temperature in the stir zone (SZ), resulting in the generation of fine recrystallized grains of 0.1–12 μm [12]. A number of investigations have been conducted to evaluate the

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superplastic behavior of FSP fine-grained aluminum alloys [11,14–17]. It has been noted that FSP is a very effective means inducing HSR SP and low-temperature superplasticity (LTSP) [14–17]. For example, the optimum strain rate for the FSP 7075Al alloy with a grain size of 3.3 μm was observed to be $1 \times 10^{-2}\ s^{-1}$ and was more than one order of magnitude higher than that for the TMP alloy [11]. Furthermore, FSP Al–4Mg–1Zr alloy with a grain size of 1.5 μm exhibited a maximum superplasticity of 1280% at a high strain rate of $1 \times 10^{-1}\ s^{-1}$ [16]. More recently, Charit and Mishra reported that FSP Al–8.9Zn–2.6Mg–0.09Sc alloy with a grain size of 0.68 μm exhibited a maximum superplasticity of 1165% at a high strain rate of $3 \times 10^{-2}\ s^{-1}$ and a lower temperature of 310 °C [14]. However, to the best of our knowledge, no study on superplasticity of FSP Al–Mg–Sc alloy has been reported so far. Hence we thought that it would be very interesting to check whether a superplasticity of ⩾2000% could be developed in micrograined Al–Mg–Sc alloy via FSP, and if so, whether the optimum strain rate would be higher than that achieved by other processing techniques? In this study, we reported the superplasticity of 2150% in a FSP Al–5.3Mg–0.23Sc alloy with a grain size of 2.6 μm at a high strain rate of $1 \times 10^{-1}\ s^{-1}$.

Al–5.33Mg–0.23Sc–0.49Mn–0.14Fe–0.06Zr (in wt.%) alloy was used in this study. The alloy was initially manufactured by ingot casting. After a homogenization treatment at 430 °C for 24 h, the ingot was extruded into a flat plate of 8 mm × 70 mm at an extrusion ratio of ~16 with a ram speed of 0.5 mm s^{-1}. A single-pass FSP was carried out on the extruded plate at a tool rotation rate of 600 rpm and a traverse speed of 25 mm min^{-1}. A steel tool with a concave shoulder 14 mm in diameter, and a threaded conical pin 5 mm in root diameter, 3.5 mm in tip diameter and 4.5 mm in length was used. Microstructural characterization was performed by optical and transmission electron microscopy (OM and TEM). In order to reveal the grain boundaries clearly, the samples for OM examination were aged at 120 °C for 16 h to decorate the boundaries. The samples were polished in colloidal silica, and then etched in 10% phosphoric acid at 50 °C. Thin foils for TEM were prepared by twin-jet polishing using a solution of 70% methanol and 30% nitric acid at −35 °C and 19 V.

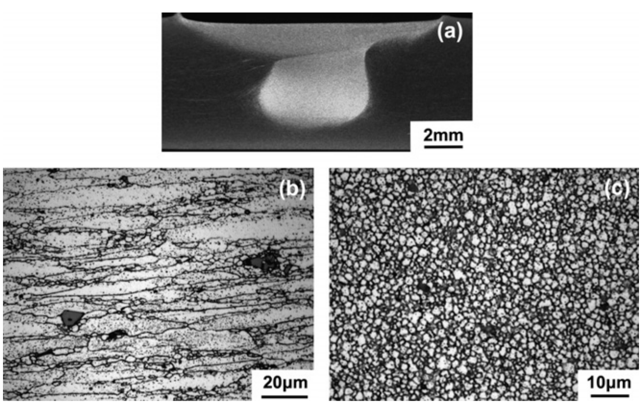

Figure 1. (a) Optical cross-sectional macrograph of FSP Al–Mg–Sc alloy; (b) optical micrograph showing heterogeneous grains in extruded sample; (c) optical micrograph showing fine and equiaxed grains in SZ of FSP sample.

Dog-bone-shaped superplastic tensile specimens (2.5 mm gage length, 1.4 mm gage width and 1.0 mm gage thickness) were electrodischarge machined from the SZ of the FSP sample transverse to the FSP direction. These samples were subsequently ground and polished to a final thickness of ~0.8 mm. Constant crosshead speed tensile tests were conducted using an Instron 5848 microtester. Each sample was held at the testing temperature for about 15 min in order to reach thermal equilibrium. The failed specimens were subjected to scanning electron microscopy (SEM) and OM examinations.

Figure 1a shows the macrograph of the cross-section of the FSP Al–Mg–Sc sample. The SZ consisted of two parts: a dish-shaped upper SZ and an elliptic lower SZ. No onion-ring structure was observed in the SZ, indicating uniform microstructure. The microstructure of the extruded sample was characterized by large elongated grains and a few fine recrystallized grains (Fig. 1b). FSP generated significant frictional heating and extensive plastic deformation, thereby creating fine and equiaxed recrystallized grains in the SZ (Fig. 1c). The average grain size in the SZ, determined by the mean linear intercept technique, was ~2.6 μm. The grain size of the FSP Al–Mg–Sc is much larger than that (0.2–1.0 μm) of ECAP or cold-rolled Al–Mg–Sc.

Figure 2 shows the typical true stress–true strain curves for the FSP Al–Mg–Sc alloy at an initial strain rate of $1 \times 10^{-1}\ s^{-1}$ for different temperatures ranging from 425 to 500 °C (Fig. 2a) and at 450 °C for different initial strain rates ranging from 1×10^{-2} to 1 s^{-1} (Fig. 2b). For a strain rate of $1 \times 10^{-1}\ s^{-1}$, the optimum temperature for maximum elongation was determined to be 450 °C, and at 450 °C, the optimum strain rate was

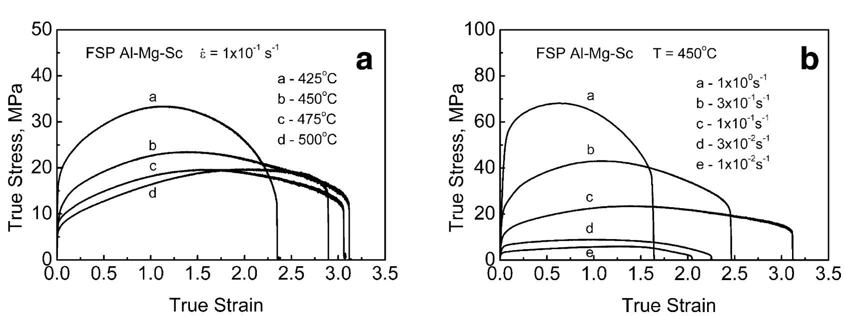

Figure 2. Effect of (a) temperature and (b) strain rate on true stress-true strain curves for FSP Al–Mg–Sc alloy.

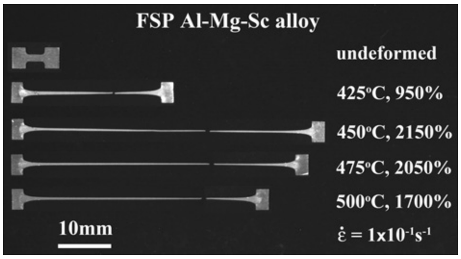

Figure 3. Tensile samples pulled to failure at a strain rate of $1 \times 10^{-1}\ s^{-1}$ for different temperatures.

$1 \times 10^{-1}\ s^{-1}$. No obvious steady-state flow region was observed. Extensive strain hardening took place initially. After reaching a maximum, the flow stress continuously decreased until failure. Increasing temperature led to a shift of the peak stress to a higher strain. A similar phenomenon was found in ECAP 1570Al alloy deformed at $1.4 \times 10^{-2}\ s^{-1}$ [6] and TMP 5083 Al deformed at $2.8 \times 10^{-3}\ s^{-1}$ [18]. All the specimens showed relatively uniform elongation characteristic of superplastic flow (Fig. 3).

Figure 4a shows the variation of elongation with initial strain rate for the FSP and extruded Al–Mg–Sc samples. At temperatures of 450–475 °C, elongations of less than 400% were observed in the extruded sample, with the optimum strain rate being $1 \times 10^{-2}\ s^{-1}$. Compared to the extruded sample, the FSP sample exhibited a significant enhanced superplasticity. A maximum elongation of 2150% was achieved at a high strain rate of $1 \times 10^{-1}\ s^{-1}$. Although the superplasticity of Al–Mg–Sc alloys has been widely studied, HSR SP of larger than 2000% was obtained only in the UFG structure of 0.2–1.0 μm [5,6,8], as summarized in Table 1 in which the present data were included. It is noted that while the grain size of the FSP Al–Mg–Sc was much larger than that of the ECAP or cold-rolled Al–Mg–Sc, the FSP alloy exhibited approximately the same superplasticity at an even higher strain rate. This implies that the grain size of fine-grained structure is not the only factor determing whether HSR SP is achieved. The grain boundary character might exert a significant effect on the superplastic behavior of the fine-grained alloys. It was reported that FSP fine-grained aluminum alloys exhibited predominant high-angle grain boundaries (HABGs) of 85–95% [19,20]. The high ratio of HABGs would be beneficial to the occurrence of grain boundary sliding (GBS). Detailed examination on the grain boundary character of the FSP Al–Mg–Sc is in progress to elucidate the origin of exceptionally high superplasticity in the micrograined structure.

Figure 4b shows the variation in flow stress (at true strain of 0.1) with initial strain rate for the FSP and extruded Al–Mg–Sc samples. The strain rate sensitivity m of both FSP and extruded samples was ~0.38 for the strain rates of 3×10^{-3}–$3 \times 10^{-2}\ s^{-1}$. The m value of the extruded Al–Mg–Sc sample decreased with increasing strain rate, which is consistent with the variation in superplasticity (Fig. 4a). By comparison, the m value of the FSP Al–Mg–Sc sample increased with increasing the strain rate, and reached ~0.62 when the strain rate was higher than $1 \times 10^{-1}\ s^{-1}$. This trend is quite similar to the prediction of Fukuyo et al. [21], which has been

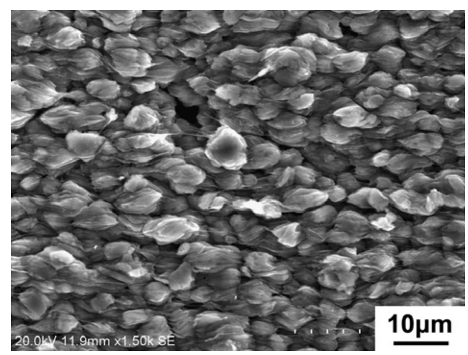

Figure 5. SEM micrograph showing surface morphology of FSP Al–Mg–Sc sample superplastically deformed to failure at 450 °C and $1 \times 10^{-1}\ s^{-1}$.

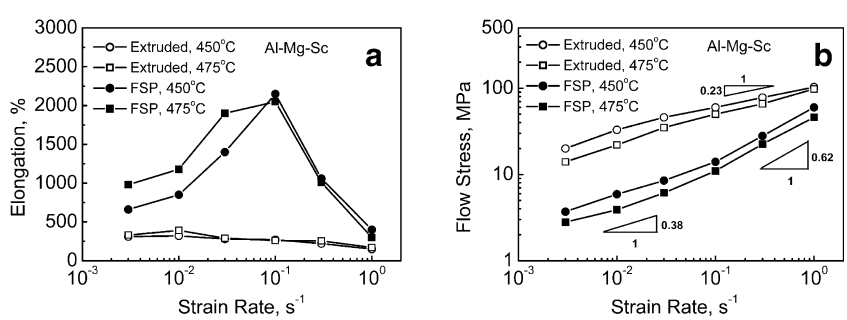

Figure 4. Variation of (a) elongation and (b) flow stress with initial strain rate for extruded and FSP Al–Mg–Sc alloys.

Table 1. A summary of high strain rate superplasticity of larger than 2000% in Al–Mg–Sc alloys prepared by various processing techniques

Alloy	Processing	Grain/cell size, μm	Temperature, °C	Strain rate, s^{-1}	Elongation, %	Ref.
Al–3Mg–0.2Sc	ECAP	0.2	400	3.3×10^{-2}	2280	[5]
Al–5.7Mg–0.32Sc–0.3Mn	ECAP	1	450	5.6×10^{-2}	2000	[6]
Al–5Mg–0.2Sc	Cold-rolled	0.2–0.5	520	5.6×10^{-2}	2300	[8]
Al–5.3Mg–0.23Sc–0.49Mn	FSP	2.6	450	1×10^{-1}	2150	This study

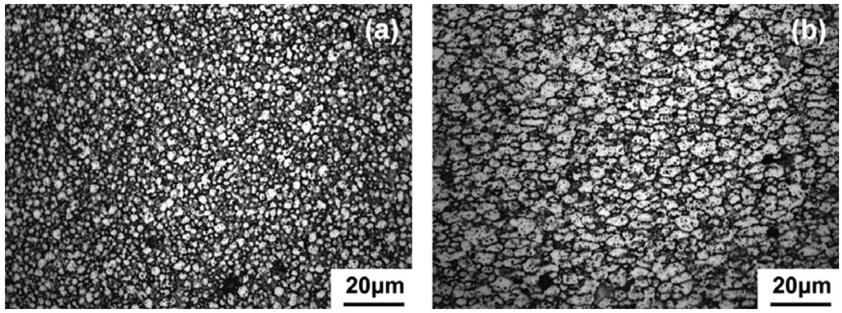

Figure 6. Optical micrograph showing grain structure of FSP Al–Mg–Sc sample superplastically deformed to failure at 450 °C and 1×10^{-1} s^{-1}: (a) grip region and (b) gage region.

confirmed in a number of fine-grained class I solid solution alloys at high temperatures. For class I solid solution alloys, at low strain rates, the GBS is accommodated by dislocation climb and the m value is about 0.5, whereas at high strain rates, the GBS is accommodated by dislocation glide and the m value increases to a value as high as 0.8 with increasing strain rate. These two regions are identified to be within the superplastic deformation region. Thus, the variation in the m values suggested there might be a deformation mechanism transition from the dislocation climb accommodated GBS to the dislocation glide accommodated GBS in the FSP Al–Mg–Sc alloy.

SEM examinations revealed distinct evidence of GBS on the surface of the FSP Al–Mg–Sc sample superplastically deformed at 450 °C and 1×10^{-1} s^{-1} (Fig. 5). The microstructural evolution of the FSP Al–Mg–Sc sample was examined under conditions of static annealing in grip region and dynamic annealing (during superplastic deformation) in the gauge region at 450 °C and 1×10^{-1} s^{-1}. The FSP Al–Mg–Sc alloy exhibited superior stability during static annealing. Annealing at 450 °C for ~1115 s led to a static growth of grains from 2.6 to 3.6 μm (Fig. 6a). The microstructural stability of the fine-grained structure is attributed to the existence of numerous fine Al_3Sc dispersoids that effectively impede the growth of the grains at high temperature. Similarly, Musin et al. [6] reported that ECAP 1570Al alloy also exhibited superior stability during static annealing. Especially when the samples annealed at temperatures lower than 300 °C, no grain growth was observed. However, in the gage region, superplastic deformation led to a remarkable grain growth (Fig. 6b). The grain size grew to 7.5 and 5.2 μm in the longitudinal and traverse directions, respectively. The grain aspect ratio (AR), defined as the ratio of grain sizes along the longitudinal and traverse directions, was ~1.44. This AR value is typical for conventional superplastic alloys, where there is a high contribution of GBS and low contribution of dislocation glide to the total elongation [22,23].

In summary, FSP is an attractive way to generate fine and equiaxed recrystallized grains in Al–Mg–Sc alloy. FSP Al–Mg–Sc alloy with a grain size of 2.6 μm exhibited a maximum elongation of 2150% at 450 °C and a high strain rate of 1×10^{-1} s^{-1}.

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