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Scripta Materialia 53 (2005) 75–80
Scripta MATERIALIA
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Development of ultrafine-grained microstructure and low temperature (0.48 T_m) superplasticity in friction stir processed Al–Mg–Zr

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Received 31 December 2004; received in revised form 6 March 2005; accepted 10 March 2005

Abstract

Friction stir processing (FSP) was applied to extruded Al–4Mg–1Zr to produce fine-grained microstructure with grains sized 0.7–1.6 μm. Low temperature deformation behavior was investigated at 175 °C and initial strain rates of 5×10^{-5}–3×10^{-3} s^{-1}. Low temperature superplasticity was observed in ultrafine-grained material. A maximum superplastic elongation of 240% was obtained in ultrafine-grained (0.7 μm) FSP sample at an initial strain rate of 1×10^{-4} s^{-1} where a maximum strain rate sensitivity of 0.34 was observed.

Keywords: Low temperature superplasticity; Friction stir processing; Aluminum alloy; Ultrafine grains

1. Introduction

Superplastic forming of commercial aluminum alloys has been considered as one of the important fabrication means for unitized components in automotive and aerospace industries. From the viewpoint of practical industrial fabrication, it is highly desirable to perform superplastic forming at higher strain rate and/or lower temperature. A higher forming rate of $>1 \times 10^{-2}$ s^{-1} would satisfy the current industrial fabrication speed [1]. On the other hand, a lower forming temperature would save energy, prevent grain growth and reduce cavitation level and solute loss from surface layer, thereby maintaining superior post-forming properties [2].

Constitutive relationships for superplasticity of fine-grained aluminum alloys predict that a decrease in the grain size results in an increase in optimum superplasticity strain rate and a decrease in optimum superplasticity temperature, which have been verified by a number of experimental investigations [3–5]. In the past few years, numerous research efforts have been focused on development of fine-grained aluminum alloys exhibiting low temperature superplasticity (LTSP) by using thermomechanical treatment (TMT) [6–8], equal channel angular pressing (ECAP) [9–13], torsion under compression [14], multi-axial alternative forging (MAF) [15], and accumulative roll bonding (ARB) [16]. However, previous investigations on LTSP were limited to temperatures of $\geqslant$200 °C (0.51 T_m where T_m is the melting temperature of aluminum expressed in K). No studies on LTSP of aluminum alloys at temperatures of <0.5 T_m have been reported. A scientific curiosity is whether superplasticity can be developed in fine-grained aluminum alloys at temperatures <0.50 T_m and if so, what the operative deformation mechanism is, at such low temperatures?

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doi:10.1016/j.scriptamat.2005.03.018

Friction stir processing (FSP) is a new solid state processing technique for microstructural modification [17,18]. During FSP, the material in the processed zone (nugget zone) undergoes intense plastic deformation, mixing, and thermal exposure, resulting in significant microstructural changes. In general, the nugget zone is characterized by fine recrystallized grains of 1.5–12 μm with 85–92% high-angle grain boundaries [18–23], typical of microstructure for superplasticity. In previous investigations, the development of high strain rate superplasticity (HSRS) has been demonstrated in several FSP aluminum alloys, such as 7075 Al, Al–4Mg–1Zr, 2024 Al, and 5083 Al [19–24].

In several recent investigations, it has been shown that FSP can produce ultrafine-grained microstructure in aluminum alloys by using improved tool design or active cooling [25–27]. An ultrafine-grained FSP aluminum alloy with a high fraction of high-angle grain boundaries is an ideal microstructure to check the low temperature limit for manifestation of superplasticity. In this study, we report the first result for low temperature superplasticity of aluminum alloys at 175 °C (0.48 T_m).

2. Experimental

Al–4Mg–1Zr was obtained as a 10 mm × 20 mm extruded bar. Fabrication of the extruded bar has been described in detail in previous works [28,29]. Two tool geometries were used for FSP. One is a standard tool with pin and shoulder diameters of 8 and 18 mm, respectively. Another is a smaller tool design for producing ultrafine grained material with pin and shoulder diameters of 4 and 12 mm, respectively. Both pins have the same threaded design. Single pass FSP was made on the extruded bars using a tool rotation rate of 350 rpm (revolution per minute) and a tool traverse speed of 203.2 mm/min for the standard tool, and a tool rotation rate of 600 rpm and a tool traverse speed of 25.4 mm/min for the smaller tool. The microstructure of both as-extruded and FSP samples was examined by a Philips EM430 transmission electron microscope (TEM). Thin foils for TEM were prepared by jet polishing techniques. Jet polishing was conducted at −25 °C using a solution of 20% HNO_3 + 80% methanol (by volume). The average grain size in the FSP samples was determined by the mean linear intercept technique.

To evaluate the superplastic behavior of FSP and as-extruded Al–4Mg–1Zr, mini tensile specimens with 1.3 mm gage length were electro-discharge machined from the nugget zone of the FSP samples transverse to the FSP direction, and from the as-extruded material with the tensile axis parallel to the extrusion direction. The tensile specimens were polished to a final thickness of ~0.5 mm using 1 μm polishing paste. Constant cross-head speed tensile tests were conducted using a computer-controlled, custom-built mini tensile tester.

3. Results and discussion

Fig. 1 shows the microstructures of the as-extruded and FSP Al–4Mg–1Zr alloys. For the extruded sample, the microstructure was characterized by predominantly low-angle grain boundaries with grains/subgrains aligned along the extrusion direction [30], and the grain size was nonuniform (Fig. 1a). The average subgrain size was determined to be 1.8 μm [30]. For the FSP samples (Figs. 1b and c), the microstructures were characterized by uniform and equiaxed recrystallized grains with predominantly high-angle grain boundaries [30]. The average grain size in the FSP sample prepared by the standard tool was 1.6 μm (hereafter referred to as micron-grained FSP sample). However, when a smaller tool with reduced shoulder and pin diameters was used, the average grain size was reduced to ~0.7 μm (hereafter referred to as ultrafine-grained FSP sample), though a higher tool rotation rate of 600 rpm and a lower tool traverse speed of 25.4 mm/min were adopted for FSP.

It is well accepted that growth of recrystallized grains in the nugget zone occurs during the FSW/FSP thermal cycle [25,31–34]. Low tool rotation rate or higher ratios of tool traverse-speed/rotation-rate were reported to be beneficial in reducing the grain size in the nugget zone due to low thermal input [20,35,36]. On the other hand, it was reported that an improved tool design, cone-shaped pin with a sharpened tip, significantly reduced the amount of frictional heat generated during FSP, thereby producing ultrafine-grained microstructure of ~0.5 μm in FSP 1050 Al [26,36]. For this study, it is evident that the tool size is the dominant factor that determines the size of recrystallized grains. A reduction in the diameters of the tool pin and shoulder by 50% and 33%, respectively, is believed to reduce the thermal input significantly due to the decrease in the contact area between the tool and workpiece and the decrease in the linear surface velocity of the tool, resulting in generation of submicron-grained microstructure in FSP Al–4Mg–1Zr.

The stress–strain behavior of the ultrafine-grained FSP sample is shown in Fig. 2a as a function of initial strain rate at 175 °C. The optimum strain rate for maximum elongation was $1 \times 10^{-4}\ s^{-1}$. The ultrafine-grained FSP sample exhibited a well-behaved stress–strain curve, where the flow stress remained almost constant during the superplastic flow, at the initial strain rates of 1×10^{-4}–$3 \times 10^{-3}\ s^{-1}$, whereas a continuous strain softening was observed at a lower strain rate of $5 \times 10^{-5}\ s^{-1}$. The stress–strain behavior of the ultrafine-grained FSP sample at 175 °C is quite different from that of micron-grained FSP alloys at higher tempera-

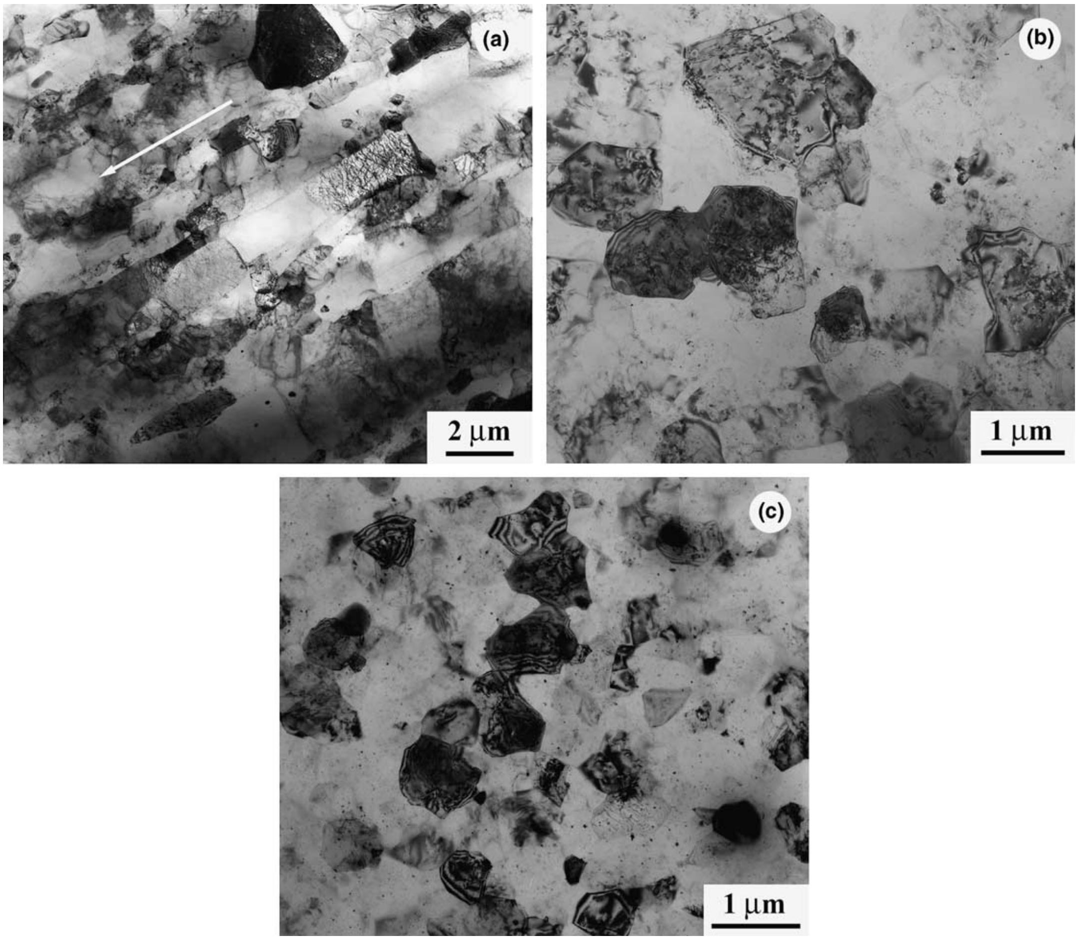

Fig. 1. TEM micrographs of (a) as-extruded Al–4Mg–1Zr (the arrow denotes the extrusion direction), (b) FSP Al–4Mg–1Zr with standard tool, and (c) FSP Al–4Mg–1Zr with smaller tool (the FSP direction is vertical to the page).

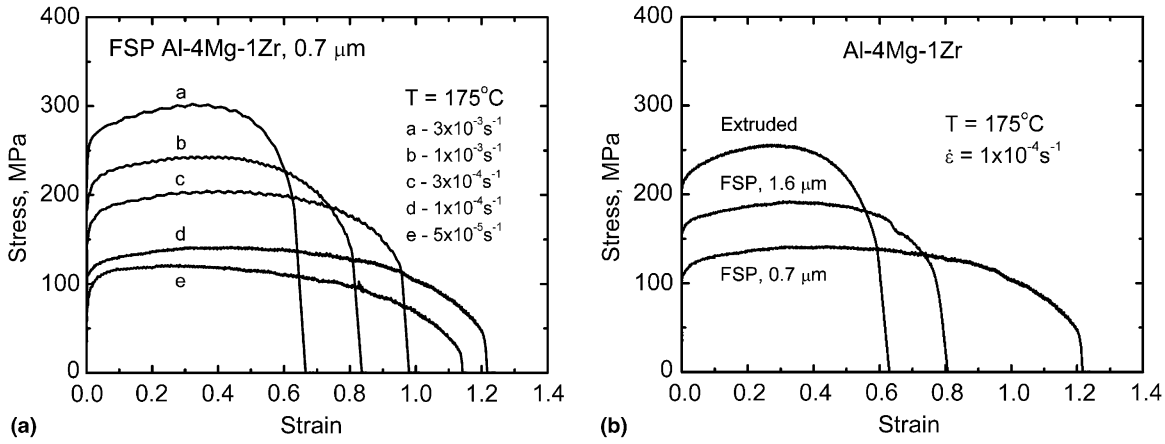

Fig. 2. (a) Stress–strain behavior of ultrafine-grained FSP Al–4Mg–1Zr as a function of initial strain rate at 175 °C and (b) effect of processing condition on stress–strain behavior of Al–4Mg–1Zr at 175 °C and an initial strain rate of $1 \times 10^{-4}\ \mathrm{s}^{-1}$.

tures of 425–570 °C, where continuous strain hardening during superplastic flow was observed due to concurrent grain growth [20,22,37]. Fig. 2b shows the effect of processing condition on the stress–strain behavior of Al–4Mg–1Zr at 175 °C and initial strain rate of $1 \times 10^{-4}\ \mathrm{s}^{-1}$. The elongation of the ultrafine-grained FSP sample was significantly higher than that of micron-grained FSP and extruded samples.

Fig. 3a shows the variation of elongation with initial strain rate for FSP and extruded Al–4Mg–1Zr alloys.

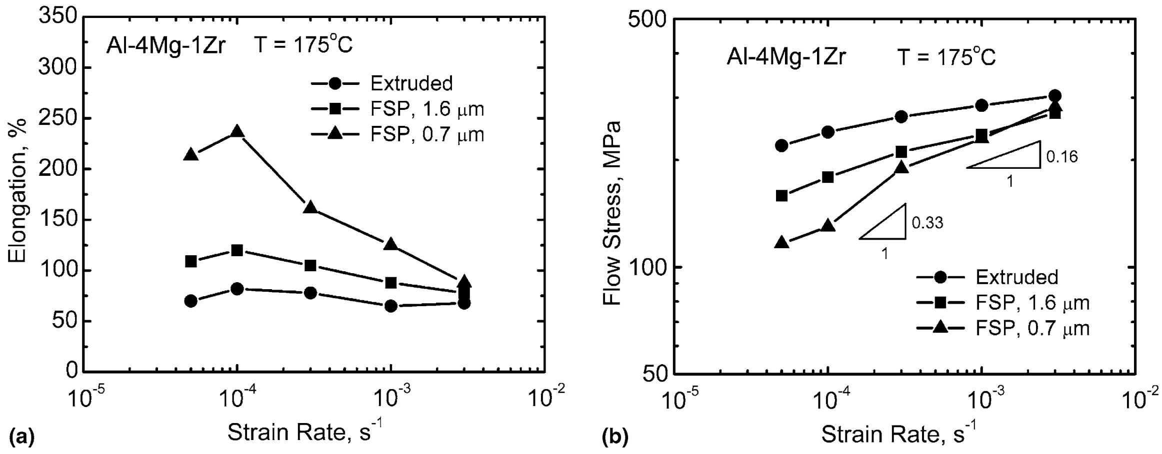

Fig. 3. Variation of (a) elongation and (b) flow stress with initial strain rate for extruded and FSP Al–4Mg–1Zr.

Maximum elongation of 82% and 120% was observed in the extruded and micron-grained FSP samples, respectively, at an initial strain rate of 1×10^{-4} s^{-1}. This means that both extruded and micron-grained FSP samples did not exhibit superplasticity at a low temperature of 175 °C. In a previous study [30], excellent superplastic ductility was obtained in micron-grained FSP Al–4Mg–1Zr at 425–550 °C and in extruded Al–4Mg–1Zr at 550–590 °C. The present study indicates that superplasticity cannot be achieved at 175 °C in a micron-grained aluminum alloy, though FSP aluminum alloys have a higher ratio of high-angled grains [18,19]. For the ultrafine-grained FSP sample, a maximum elongation of 240% was attained at an initial strain rate of 1×10^{-4} s^{-1} for 175 °C. This indicates that low temperature superplasticity at 175 °C is developed in the ultrafine-grained FSP Al–4Mg–1Zr alloy.

Fig. 3b shows the variation of flow stress with initial strain rate for FSP and extruded Al–4Mg–1Zr alloys. The strain rate sensitivity of both extruded and micron-grained FSP samples were consistently lower than ~0.16 throughout the investigated strain rates of 5×10^{-5}–3×10^{-3} s^{-1}. This accounts for the absence of superplasticity in the extruded and micron-grained FSP samples. By comparison, the strain rate sensitivity of the ultrafine-grained FSP sample is 0.16, 0.34, and 0.17 in the initial strain rate ranges of 5×10^{-5}–1×10^{-4} s^{-1}, 1×10^{-4}–3×10^{-4} s^{-1}, and 3×10^{-4}–3×10^{-3} s^{-1}, respectively, i.e., the ultrafine-grained FSP sample exhibited the typical S-type stress–strain rate behavior characteristic of a superplastic material. The maximum strain rate sensitivity of 0.34 at the initial strain rate of 1×10^{-4} s^{-1} corresponds to the maximum elongation of 240%. A strain rate sensitivity of 0.34 indicates that the superplastic deformation mechanism for the ultrafine-grained FSP sample may be associated with solute drag ($m = 0.33$). Furthermore, flow stress of the ultrafine-grained FSP sample is significantly lower than that of the extruded and micron-grained samples at the initial strain rates of 5×10^{-5}–1×10^{-3} s^{-1}. This is attributed to a significantly-refined microstructure.

Fig. 4 shows tested Al–4Mg–1Zr specimens deformed to failure at 175 °C and an initial strain rate of 1×10^{-4} s^{-1}. The ultrafine-grained FSP specimen shows relatively uniform elongation characteristic of superplastic flow.

The lowest reported temperature for LTSP of aluminum alloys is 200 °C [11,15,16]. The processing condition, grain size, and superplastic properties of ultrafine-grained aluminum alloys at 200 °C are summarized in Table 1. For comparison, superplastic data of the present ultrafine-grained Al–4Mg–1Zr at 175 °C is also included in Table 1. Two observations can be made from this table. First, maximum elongation is achieved

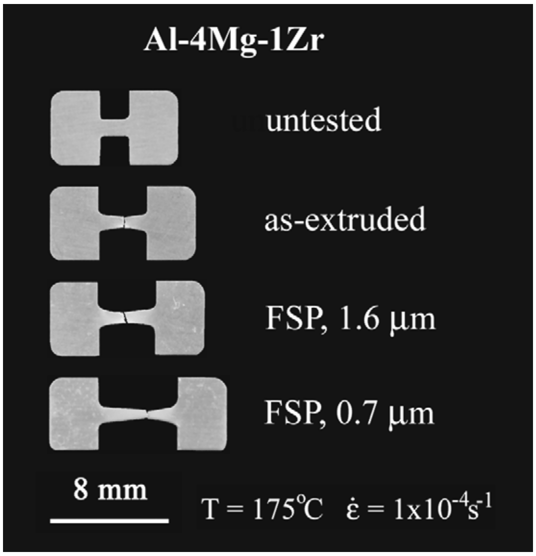

Fig. 4. Appearance of specimens before and after superplastic deformation at 175 °C and an initial strain rate of 1×10^{-4} s^{-1}.

Table 1
A summary of low temperature superplastic properties in various ultrafine-grained aluminum alloys prepared by different processing techniques

Alloy	Processing	Grain size (μm)	Temperature (°C)	Strain rate (s^{-1})	Elongation (%)	*m* value	Ref.
Al–3Mg–0.2Sc	ECAP (8 passes)	0.2	200	3×10^{-4}	420	–	11
5083Al	MAF (10 cycles)	~0.8	200	2.8×10^{-3}	340	0.39	15
5083Al	ARB (5 cycles)	0.28	200	1.7×10^{-3}	230	0.37	16
Al–4Mg–1Zr	FSP	~0.7	175	1×10^{-4}	240	0.34	This study

ECAP: equal channel angular pressing; MAF: multi-axial alternative forging; ARB: accumulative roll bonding; FSP: friction stir processing.

at low strain rates of 10^{-4}–10^{-3} s^{-1}. This is consistent with the prediction of the constitutive relationship for superplasticity of fine-grained aluminum alloys, i.e. a decrease in deformation temperature results in a decrease in optimum strain rate. Second, strain rate sensitivity is consistently between 0.3 and 0.4 for superplastic elongations. This implies that the main deformation mechanism for LTSP of ultrafine-grained aluminum alloys is associated with solute drag considering the fact that the aluminum alloys exhibiting LTSP are Mg-containing alloys. In a recent investigation, Hsiao and Huang [8] reported that the primary deformation mechanisms is solute drag creep plus minor power-law creep during the initial LTSP stage ($\varepsilon \leqslant 0.5$) in a TMT 5083Al and grain boundary sliding gradually controlled the deformation at later stages.

The present study shows that the simple application of FSP with smaller tool design can induce ultrafine-grained microstructure and LTSP in an aluminum alloy at 0.48 T_m. To the best of our knowledge, this is the first result illustrating superplastic behavior of aluminum alloys at temperatures of <0.5 T_m This demonstrates that FSP is a very effective processing technique to create very fine-grained microstructure in aluminum alloys capable of exhibiting high strain rate/low temperature superplasticity. Research is in progress to explore the low temperature limit for superplasticity and understand the deformation mechanism responsible for LTSP in ultrafine-grained aluminum alloys.

4. Conclusions

1. Tool size plays a dominant role in affecting the size of recrystallized grains in FSP aluminum alloys. A decrease in the diameter of the tool pin and shoulder by 50% and 33%, respectively, resulted in a decrease in the grain size of the nugget zone from 1.6 to 0.7 μm in FSP Al–4Mg–1Zr.
2. Maximum superplasticity of 240% was obtained at a low temperature of 175 °C (0.48 T_m) for an initial strain rate of 1×10^{-4} s^{-1} in ultrafine-grained (0.7 μm) FSP Al–4Mg–1Zr, whereas extruded and micron-grained (1.6 μm) FSP samples did not exhibit superplasticity at 175 °C.
3. Ultrafine-grained FSP Al–4Mg–1Zr exhibited an S-type stress–strain rate behavior with a maximum strain rate sensitivity of 0.34 at 175 °C for initial strain rates of 1×10^{-4}–3×10^{-4} s^{-1}. The flow stress of submicron-grained FSP Al–4Mg–1Zr was significantly lower than that of extruded and micron-grained FSP samples.

Acknowledgement

The authors gratefully acknowledge the support of the National Science Foundation through grant DMR-0076433 and the Missouri Research Board for the acquisition of a friction stir welding and processing machine, and the National Science Foundation through grants DMI-0085044 and DMI-0323725, Dr. Jian Cao, Program Manager, for this study. We also want to thank Prof. R. Grimes for providing us the material used in the present study.

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