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Realization of exceptionally high elongation at high strain rate in a friction stir processed Al–Zn–Mg–Cu alloy with the presence of liquid phase

K. Wang,[a] F.C. Liu,[b] Z.Y. Ma[a,*] and F.C. Zhang[b]

[a]*Shenyang National Laboratory for Materials Science, Institute of Metal Research, Chinese Academy of Sciences, 72 Wenhua Road, Shenyang 110016, China*

[b]*State Key Laboratory of Metastable Materials Science and Technology, Yanshan University, Qinhuangdao 066004, China*

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Friction stir processed Al–Zn–Mg–Cu alloy with grain size 6.2 μm was subjected to superplastic investigation at 500–535 °C and a high strain rate of $1 \times 10^{-2}\ s^{-1}$. A maximum elongation of 3250% was achieved at 535 °C, which was just above the incipient melting temperature of the sample. Exceptionally high elongation was attributed to the presence of a liquid phase, which served both to relax the stress concentration and to suppress the appearance of cavities during deformation.

Keywords: Superplasticity; Friction stir processing; Aluminum alloys; Liquid phase; Cavitation

Superplastic forming (SPF) is widely used to fabricate complex parts in sheet metals [1]. However, the expansion of SPF into the fabrication of high-volume components in the automotive and consumer product industries is currently limited because of the relatively low strain rate. Therefore, there is an urgent requirement to increase the deformation rate. Besides high strain rate, a large elongation is also required in the forming of extraordinarily complex components.

Superplastic failure by necking and/or cavitation is often observed in the most highly superplastic aluminum alloys [2–5]. However, for the aluminum alloys with extensive grain boundary sliding (GBS) and high strain rate sensitivities ($m \geqslant 0.5$), superplastic failure is controlled mainly by the formation and/or interlinkage of the cavitations [2,3,5]. Therefore, restraining cavitation during superplastic deformation is beneficial for enhancing the tensile elongation for these materials.

Many reports [6–8] revealed that cavitation would be minimized when both grain and particle sizes were small. Ma and Mishra [6] showed that a decrease in grain size from 7.5 to 3.8 μm in 7075Al alloy resulted in a significant decrease in the density, size and volume fraction of cavities at $1 \times 10^{-2}\ s^{-1}$ and 480 °C. In contrast, Kaibyshev et al. [9] observed that the cavitation level in a superplastic Al–Mg alloy increased with an increase in temperature from 500 to 550 °C and then reduced with a continual increase in temperature to 570 °C, a temperature close to the solidus temperature of the alloy. The cavitation reduction at higher temperatures was attributed mainly to the presence of a small amount of liquid phase at grain boundaries, which can assist GBS accommodation and thus delay the appearance of internal cavitation and subsequent failure [10].

The strain rate of superplastic flow varies inversely with the grain size to a power of ∼2, and this was evidenced by the fact that the superplastic regime was moved to faster strain rates when the grain size of the material was reduced [11]. In previous reports, in order to obtain high strain rate superplasticity (HSRS), great efforts have been made to refine the grain size of the alloys to submicron level [12]. However, the fine-grained microstructure is usually unstable at elevated temperatures, thus resulting in degraded superplasticity [13]. Therefore, in order to maintain the microstructural stability at the elevated temperatures required for superplastic flow, not the finest, but a properly sized gain structure is desired.

Friction stir processing (FSP) [14,15] has been used to produce fine recrystallized grains with predominant high-angle grain boundaries (HAGB), features that are

* Corresponding author. Fax: +86 24 83978908; e-mail: zyma@imr.ac.cn

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important for enhanced superplasticity [11,16]. Liu and Ma [3] showed that FSP Al–Mg–Sc alloy with grain size ~2.6 μm exhibited approximately the same superplasticity at an even higher strain rate compared with the ultrafine-grained Al–Mg–Sc produced by other techniques, owing to the high ratio of HAGB in the FSP alloy, which would be beneficial to the occurrence of GBS.

Based on the analyses mentioned above, it seems that, if a fine-grained aluminum alloy produced by FSP is reasonably stable at a temperature near the incipient melting temperature, it is possible to achieve exceptionally high elongation at high strain rates. Therefore, in this study, a fine-grained 7075Al alloy was specially produced by FSP. The aim is to provide a rigorous appraisal of the potential for achieving HSRS with large elongation at high temperatures.

Commercial 7075Al-T651 rolled plate with nominal composition 5.85Zn–2.56Mg–1.89Cu–0.22Cr (in wt.%) was used. A single-pass FSP was carried out at a tool rotation rate of 1200 rpm and a traverse speed of 25 mm min^{-1}. The tool was manufactured from M42 steel 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 4.0 mm in length, with a standard pitch of 0.8 mm for the M5 screw thread. The processed samples were cut in the transverse direction, lightly electropolished to produce a strain-free surface. Electron backscatter diffraction (EBSD) orientation maps (with resolution 0.2 μm) were obtained using a ZEISS SUPRA 35, operated at 20 kV, and interfaced to an HKL Channel EBSD system. Metallographic examination was completed by optical microscopy. Grain sizes were measured manually on printed micrographs by the linear intercept method.

To evaluate the superplastic behavior of FSP 7075Al, mini tensile specimens (2.5 mm gage length, 1.4 mm gage width and 1.0 mm gage thickness) were electrodischarge machined perpendicular to the FSP direction, with the gage length centered in the SZ. These 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. All specimens were held at the selected temperature for 20 min to establish thermal equilibrium prior to the tensile test. Constant crosshead speed tensile tests were conducted using an Instron 5848 microtester. A differential scanning calorimetry (DSC) experiment was performed on a 26.78-mg specimen at a heating rate of 5 °C min^{-1} in Ar atmosphere via a SETSYS Evolution 18 analyzer. Tensile specimen deformed to failure, or a fixed strain was examined to determine the changes in cavity volume fraction and grain size. Local true strain was used to relate to the volume fraction of cavities because of the strain gradient along the gage length of the deformed specimens.

Figure 1a shows that the microstructure of the as-FSP 7075Al was characterized by equiaxed recrystallized grains with predominantly HAGB. The average grain size of the as-FSP 7075Al was determined as ~6.2 μm and increased to 6.9 μm after the sample was annealed at 535 °C for 20 min (Fig. 1b). This indicates that the fine-grained microstructure in this FSP 7075Al is relatively stable. The fractions of HAGB in the as-

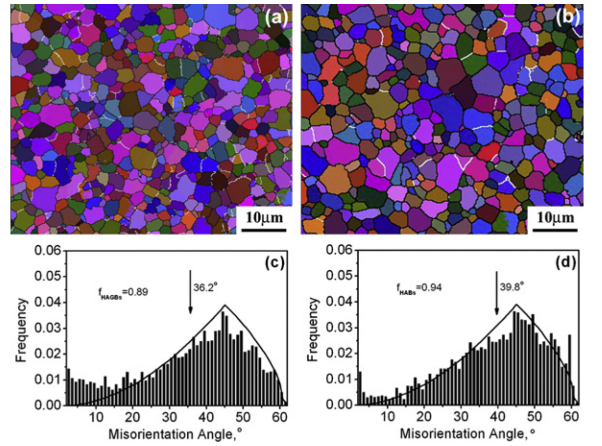

Figure 1. Microstructure of FSP 7075Al: EBSD maps under (a) as-FSP, (b) annealing at 535 °C for 20 min; boundary misorientation angle distribution under (c) as-FSP, (d) annealing at 535 °C for 20 min.

FSP and annealed samples were measured as 89% and 94%, respectively (Fig. 1c and d).

Figure 2a shows the true stress–true strain curves of the FSP 7075Al deformed at 500 and 535 °C and an initial strain rate of 1×10^{-2} s^{-1}. At 500 °C, the FSP 7075Al exhibited continuous strain hardening up to a large strain and, after reaching a maximum stress, the flow stress continuously decreased until fracture. At 535 °C, the FSP 7075Al displayed a slight strain hardening at the initial stage, and then a significant strain hardening occurred until the sample fractured abruptly. The maximum stress of the sample deformed at 535 °C is higher than that at 500 °C. Figure 2b shows the failed tensile specimens which experienced the maximum elongation at 1×10^{-2} s^{-1} for different temperatures. The deformed specimens showed uniform elongation, characteristic of superplastic flow. A maximum elongation of 3250% was achieved at 535 °C and 1×10^{-2} s^{-1}.

The superplastic behavior of fine-grained 7075Al alloys produced by FSP and thermo-mechanical process (TMP) have been widely investigated [11,14,17]. In these studies, the grain sizes of the alloys are in the range 3–10 μm, and optimum elongation of 1000–2100% was achieved at 480–510 °C and strain rates 3×10^{-3}–3×10^{-2} s^{-1}. The present FSP 7075Al exhibited exceptionally high elongation at even higher temperature. Two reasons may be responsible for the enhanced thermal stability of the present alloy. First, the large grain-sized 7075Al is relatively stable at high temperature, owing to effective pinning of a high density of Cr-bearing dispersoids to the migrating boundaries [18,19]. For example, Ma and Mishra [11] reported that FSP 7.5-μm 7075Al was thermally stable even after annealing at 490 °C for 1 h and, further, the superplasticity of this sample did not show a noticeable decrease at a high temperature of 530 °C. Second, it was reported that rapid heating led to the simultaneous growth of the fine grains in a fine-grained FSP 7010Al, thus benefiting by avoiding abnormal grain growth, and a relatively stable and uniform grain structure remained [20]. In this study, tensile specimens were fastened in the testing apparatus when the temperature reached the preset

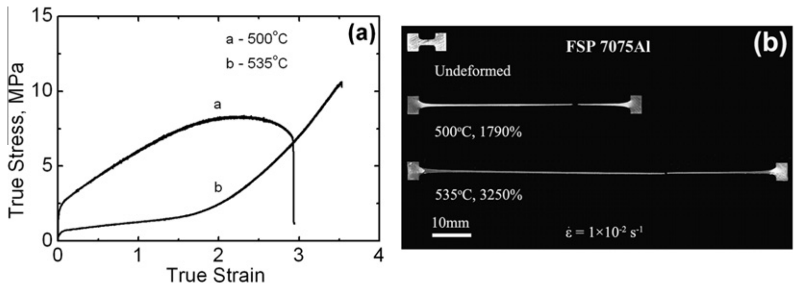

Figure 2. (a) True stress–true strain curves of FSP 7075Al at an initial strain rate of $1 \times 10^{-2}\ s^{-1}$ and (b) tensile specimens pulled to failure.

value. This would produce a faster heating rate, thereby benefiting by retaining the fine-grained structure.

The DSC curve in the temperature range 300–690 °C is shown in Figure 3. In addition to a continuous endothermic peak attributable to the melting of the alloy, an endothermic peak with an incipient temperature of 527 °C was detected. This endothermic peak implies that partial melting started at 527 °C for the FSP 7075Al, which is lower than the deformation temperature of 535 °C. In this case, some precipitates with lower melting temperatures, such as $MgZn_2$, $Mg_3Zn_3Al_2$ and $CuMgAl_2$ [21], melted at 535 °C. These incipient liquid phases, which were distributed at the grain boundaries, could promote GBS and delay cavitations, thereby enhancing the superplastic deformation ability.

Figure 4 shows typical microstructures of FSP 7075Al deformed at 500 and 535 °C, and the variation in cavity volume fraction and grain size with true strain are summarized in Table 1. Figure 4 and Table 1 reveal the following observations. First, the specimen deformed at 500 °C showed a higher cavity volume fraction at a given strain and a higher increasing rate of cavitation with the strain than that deformed at 535 °C. Further, the coalescence of the cavities developed along the tensile direction at 500 °C, leading to a significant increase in the cavitation. This suggested that a plasticity-controlled cavity growth mechanism dominated at 500 °C. At 535 °C, the cavitation level hardly increased with strain. The maximum volume fraction of cavities was as low as 0.1% even at failure, and the cavities were fine and nearly equiaxed. These suggested that diffusion-controlled cavity growth might dominate at 535 °C. The reduced cavitations at 535 °C should be attributed to the existence of liquid phase at the gain boundaries, which prevented the occurrence of cavitations, which is well consistent with previous studies [9,22]. Second, a significant grain growth developed during superplastic deformation with increasing strain. The specimen deformed at 535 °C showed a higher grain size at a given strain.

It is well documented that the grain size increased with increasing strain during superplastic deformation at elevated temperatures, which resulted in the increase in flow stress with strain [23]. This phenomenon was observed in the two specimens during the early stage of straining. A relaxation of the tensile stress before failure occurred in the specimen deformed at 500 °C, due to the significant increase in the volume fraction of cavities (Fig. 4 and Table 1). By comparison, a continuous strain hardening was observed in the specimen deformed at 535 °C because of its low cavitation level. In addition, the intensive strain hardening before failure was related to significant grain growth at higher strains.

For comparison, typical superplastic elongations >2000% obtained in various aluminum alloys are summarized in Table 2. It is noted that, for the aluminum alloys with thermally stable dispersoids such as Al_3Sc [2,3,13,24,25], fine-grained microstructure with grain sizes ⩽3 μm exhibited excellent superplasticity at high strain rates $>2.8 \times 10^{-2}\ s^{-1}$. However, for 7xxx aluminum alloys, superplastic elongations >2000% were obtained only in coarse-grained structure (⩾10 μm) at very high temperatures and very low strain rates, owing to coarse initial grains. A high deformation temperature was supposed to restrain cavitations, enhancing the superplasticity [26,27].

The present FSP 7075Al had excellent thermal stability up to the incipient melting temperature. During superplastic deformation, the grains slide until unfavorable grains obstruct the process. The resultant stress concentration should be relieved by accommodation processes so that further sliding can occur. Previous studies [11,28] suggested that part of the melting regions at the boundaries served both to relax the stress concentration and to limit the appearance of cavities. Therefore, an exceptionally high elongation of 3250% was developed in the FSP 7075Al. Furthermore, this FSP 7075Al had a moderate grain size smaller than that in Refs. [26] and

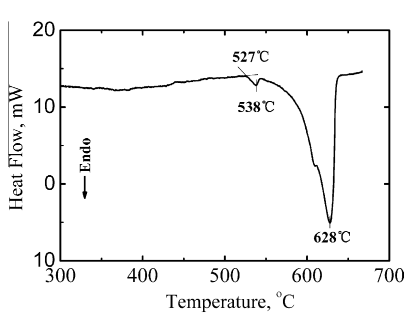

Figure 3. DSC curve of FSP 7075Al.

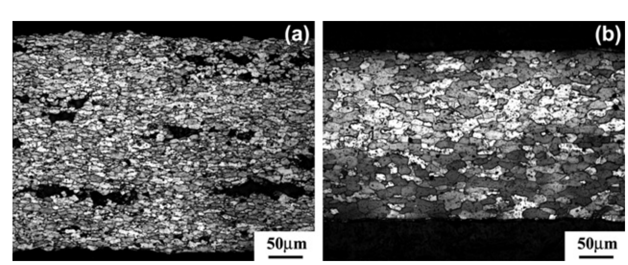

Figure 4. Optical microstructure of tensile specimens deformed to failure at $1 \times 10^{-2}\ s^{-1}$ for (a) 500 °C and (b) 535 °C.

Table 1. Cavitation (area fraction %) and grain size in the gauge section until failure at $\dot{\varepsilon} = 1 \times 10^{-2}\ s^{-1}$.

True strain		1	2	2.5	3	Failure
500 °C	Cavitation area fraction (%)	0.08 ± 0.012	0.26 ± 0.122	0.88 ± 0.193	–	5.13 ± 0.744
	L_d (μm)[a]	11.92/8.31	17.28/9.67	20.04/10.60	–	26.87/13.08
535 °C	Cavitation area fraction (%)	0.06 ± 0.008	0.05 ± 0.004	0.10 ± 0.010	0.08 ± 0.018	0.10 ± 0.013
	L_d (μm)[a]	14.20/8.90	20.92/10.47	29.34/15.65	35.50/18.71	38.73/21.78

[a] The numerator and denominator are the grain sizes measured in the tension and transverse directions, respectively.

Table 2. Summary of HSRS >2000% in aluminum alloys prepared by various processing techniques.

Material	Processing	Grain size (μm)	Temperature (°C)	Strain rate (s^{-1})	m value	Elongation (%)	Reference
Al–Mg–Sc	FSP	2.6	450	1×10^{-1}	0.62	2150	[3]
Al–5Mg–0.2Sc	Cold rolling	0.2–0.5	520	5.6×10^{-2}	0.53	2300	[24]
Al–Mg–Sc	ECAP	1	450	5.6×10^{-2}	–	2000	[25]
Al–3Mg–0.2Sc	ECAP	0.2	400	3.3×10^{-2}	0.5	2280	[13]
Al–Li–Mg	ECAE	2.6	450	2.8×10^{-2}	0.6	3000	[2]
7475	Ingot metallurgy	19.8	533	4×10^{-5}	0.5	2400	[26]
7075 Alloy	TMP	10	510	8.33×10^{-4}	–	2100	[27]
7075 Alloy	FSP	6.2	535	1×10^{-2}	0.52	3250	This study

[27] and a higher ratio of HAGB than that in traditionally processed alloys [24,29–32]. As mentioned above, grain refinement was beneficial for increasing the optimum strain rate [12]. Further, increasing deformation temperature also increased the optimum strain rate of the stable fine-grained materials, because GBS is usually rate controlled by either diffusion or dislocation motion [33]. In addition, it is generally believed that GBS occurs only along the HAGB [34]. A high ratio of HAGB in the present FSP 7075Al made GBS take place easily, thereby increasing the strain rate for superplasticity. For these reasons, an HSRS with a large elongation of 3250% was developed in the moderate grain-sized 7075Al at 535 °C.

In summary, the FSP 7075Al with a grain size of 6.2 μm exhibited good thermal stability up to incipient melting temperature, and a maximum elongation of 3250% was obtained at 535 °C and $1 \times 10^{-2}\ s^{-1}$, with a very low cavitation level until fracture. This is attributed to the appearance of the liquid phase at the grain boundaries, which relaxed the stress concentration and limited the development of cavities.

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