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Low-temperature superplasticity of friction stir processed Al–Zn–Mg–Cu alloy

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Friction stir processing was used to produce ultrafine-grained Al–Zn–Mg–Cu alloy. Low-temperature superplasticity of 350–540% was achieved at 200–350 °C. Increasing the temperature resulted in an increased optimum strain rate for maximum elongation. At 350 °C, the optimum strain rate shifted to a high strain rate of $1 \times 10^{-2}\ s^{-1}$. A strain rate sensitivity of 0.33–0.42 was observed for maximum elongation at the investigated temperatures. Grain boundary sliding was observed even at 200 °C.
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It has been well documented that grain refinement down to the submicrometer level results, in some experimental or commercial aluminum alloys, in high-strain-rate and/or low-temperature superplasticity [1–3]. This phenomenon is not only of an academic interest, but also has considerable practical value, because it is highly desirable to achieve superplastic forming at high strain rates and low temperatures in industrial fabrication. High-strain-rate superplasticity (HSRS), defined as superplasticity occurring at strain rates at or above $10^{-2}\ s^{-1}$ [4], is of great interest because it is expected to result in economically viable, near-net-shape forming techniques. On the other hand, a lower forming temperature would save energy, improve the surface quality of the formed component, prevent severe grain growth, and reduce the cavitation level and solute loss from the surface layer, thus maintaining better post-forming properties [5].

Ideal low-temperature superplasticity (LTSP) should be obtained at temperatures of below 0.5 T_m (where T_m is the melting temperature of the matrix alloy expressed in K). For aluminum alloys, the corresponding temperature would be about 190 °C or less. However, in previous studies, LTSP of aluminum alloys has focused on the temperature range of 200–350 °C [5–11]. This may be because it is usually difficult, if not impossible, to achieve superplastic deformation at lower temperatures. Hence, the investigation of superplasticity at temperatures of $\leqslant$350 °C is needed to develop understanding of LTSP.

In the past few years, many efforts have been made to produce ultrafine-grained (UFG) metallic materials for LTSP. Conventionally, thermomechanical processing (TMP) is used to produce an UFG microstructure in commercial aluminum alloys [12–14]. Typical TMP for heat-treatable aluminum alloys consists of solution treatment, overaging, multiple pass warm rolling with intermittent reheating, and a recrystallization treatment [14]. Clearly, TMP is complex and time consuming and results in increased material cost. Multi-pass equal-channel angular pressing (ECAP) can significantly reduce grain size [6,7,11], but at least 6–8 passes of ECAP are required to achieve UFG microstructure and homogenization. Furthermore, this technique produces relatively small quantities of material and is very difficult to scale up.

Based on the concepts of friction stir welding (FSW) [15,16], a new solid-state processing technique, friction stir processing (FSP), has been developed by Mishra and coworkers [17–19]. FSP causes intense plastic deformation and elevated temperatures in the stir zone (SZ), resulting in the generation of fine recrystallized grains with predominantly high-angle grain boundaries, features that are important for enhanced superplastic properties. A previous study by Ma et al. [18] has shown that FSP 7075Al alloy with a grain size of 3.8 μm exhibited

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superplastic elongations of >1250% at 480 °C in the strain rate range of 3×10^{-3}–3×10^{-2} s^{-1}. The optimum strain rate for FSP 7075Al alloy is more than one order of magnitude higher than the previous best TMP effort on a 7xxx aluminum alloy [14]. Furthermore, LTSP was obtained at 175 °C (0.48 T_m) in an UFG Al–4Mg–1Zr prepared via FSP [20]. These results clearly show the effectiveness of FSP for producing fine-grained materials that are amenable to HRSP or LTSP.

Most LTSP efforts to date have focused on solution-strengthened Al–Mg alloys [7–10]. Little attention has been paid to precipitation-strengthened Al–Zn–Mg 7000 series alloys, which are widely used for high-strength structural applications such as aircraft and sporting goods. Especially for commercial 7075Al alloy, to the best of our knowledge, no study on superplasticity at temperatures of ⩽350 °C has been reported. In this study, an UFG 7075Al was prepared by FSP and subjected to superplastic investigation at temperatures of ⩽350 °C. The aim is (i) to identify the possibility of achieving LTSP in 7075Al via FSP and (ii) to elucidate the deformation mechanism of FSP aluminum alloys at low-temperature.

Commercial 7075Al–T651 rolled plates 8 mm thick, 80 mm wide and 400 mm long with a composition of 5.85Zn–2.56Mg–1.89Cu–0.22Cr (in wt.%) were used. A single-pass FSP with a length of about 360 mm was carried out at a tool rotation rate of 400 rpm and a traverse speed of 100 mm min^{-1}. The tool was manufactured from M42 steel with a concave shoulder of 14 mm in diameter, and a threaded conical pin (0.8 mm pitch) with a root diameter of 5 mm, a tip diameter of 3.5 mm and a length of 4.5 mm. Room-temperature water was used to quench the plate immediately behind the FSP tool to prohibit the growth of the recrystallized grains. Microstructure characterization was performed by optical and transmission electron microscopy (OM and TEM). 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. Grain size were measured by the mean linear intercept technique. To evaluate the superplastic behavior of FSP 7075Al, dog-bone shaped 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 being centered in the SZ. These samples were subsequently ground and polished to a final thickness of ~0.8 mm. Constant cross-head speed tensile tests were conducted using an Instron 5848 microtester. The failed specimens were subjected to SEM examinations.

Figure 1a shows the macrograph of the cross-section of the FSP 7075Al sample. A basin-shaped SZ was observed with a widened upper surface due to the extreme deformation and frictional heating resulting from the contact with the shoulder during FSP. As-received 7075Al consisted of large, elongated, pancake-shaped grains typical of a hot-rolled structure (Fig. 1b). After FSP with water cooling, an UFG microstructure was obtained in the SZ and the average grain size was estimated to be ~0.8 μm (Fig. 1c). The grain size of the FSP 7075Al in this study is significantly smaller than that obtained by a common FSP without cooling [17,18]. This indicates that water cooling could effectively restrain the growth of the grain during the FSP thermal cycle. Previously, Su et al. [21] produced an UFG 7075Al of 100–200 nm by FSP with an active cooling using a mixture of water, methanol and dry ice. Figure 1d shows that a high density of dispersoids (50–150 nm in diameter) was randomly distributed both within the grain interiors and at the grain boundaries. Although these particles are much finer than the large second-phase particles (1.0–5.0 μm in diameter) in Al–Mg alloy [22], they are remarkably larger than the Al_3Sc dispersoids in FSP Al–Zn–Mg–Sc alloy [23]. Large particles, in particular those at the grain boundaries, tend to promote cavity development during superplastic deformation [22,24].

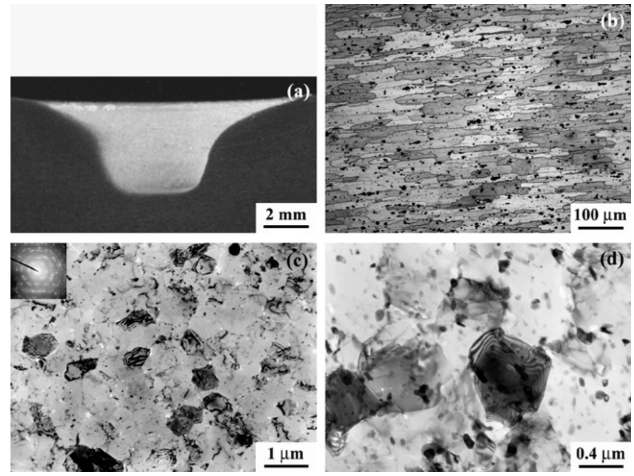

Figure 1. (a) Optical cross-sectional macrograph of FSP 7075Al; (b) OM micrograph showing elongated grains in as-rolled parent material; (c) TEM image showing ultrafine grains in SZ; (d) TEM image showing high density of precipitates in SZ.

Figure 2a shows the classical well-behaved true stress–true strain (σ–ε) curves for the UFG FSP 7075Al at 250 °C for various initial strain rates. The flow stress remains nearly constant during the superplastic flow, followed by a decrease in flow stress before failure, i.e. a type "F" true stress–true strain curve as defined in Ref. [25]. However, at 350 °C, σ vs. ε curves exhibited a slight strain hardening at the initial stage (Fig. 2b). After reaching the maximum, the flow stress decreases continuously until failure. At a low initial strain rate of 3×10^{-4} s^{-1}, the strain hardening appears to dominate the whole deformation process. The explanation for the strain hardening is typically based on grain coarsen-

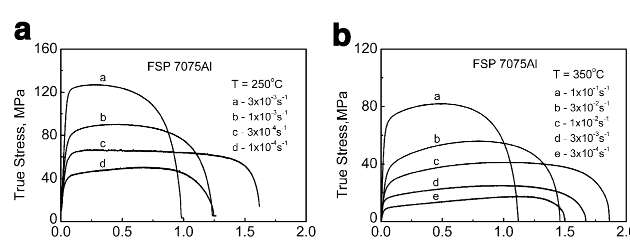

Figure 2. True stress–strain curves of UFG FSP 7075Al at (a) 250 °C and (b) 350 °C.

ing, which is obviously enhanced for the tensile specimens undergoing superplastic deformation at a higher temperature for a longer time [24,25].

Figure 3a shows the variation of elongation as a function of the initial strain rate at different temperatures. Superplastic ductility was achieved over a wide low-temperature range of 200–350 °C. At 200 °C (0.51 T_m), superplasticity was obtained at lower strain rates of 1×10^{-5}–$1 \times 10^{-4}\ s^{-1}$, and a ductility of 350% was observed at a strain rate of $1 \times 10^{-5}\ s^{-1}$. A temperature increase from 200 to 350 °C resulted in an increase in the optimum strain rate for superplasticity as well as the maximum elongation. At 350 °C, a maximum ductility of 540% was obtained at a high strain rate of $1 \times 10^{-2}\ s^{-1}$. This result is attractive because a single-pass FSP could induce the occurrence of high-strain-rate and low-temperature superplasticity in commercial 7075Al alloy.

Figure 3b shows the effect of temperature on the superplastic ductility of the FSP 7075Al at an initial strain rate of $1 \times 10^{-2}\ s^{-1}$. The FSP alloy exhibited superplasticity at a high strain rate of $1 \times 10^{-2}\ s^{-1}$ within the lower temperature range of 275–350 °C. Ductility increased with an increase in the testing temperature until it reached the maximum value at 350 °C, and then decreased with increasing temperature. For the UFG FSP 7075Al, superplasticity disappeared at 400 °C because of abnormal grain growth. Similarly, in a previous study [23], it was observed that FSP Al–Zn–Mg–Sc alloy with a grain size of 0.68 μm did not exhibit superplastic elongation at 420 °C due to microstructural instability. The better thermal stability of FSP Al–Zn–Mg–Sc alloy than that of this FSP 7075Al was attributed to the effective pinning effect of fine Al_3Sc particles in the Al–Zn–Mg–Sc alloy [23].

Figure 4 shows the failed tensile specimens which experienced the maximum elongation at different testing temperatures. All the specimens show relatively uniform elongation, characteristic of superplastic flow. Although the flow localization at the grip region adjacent to the shoulder of specimen was limited [19], the superplastic ductility obtained from the minimum-tension specimens with a gage length of 2.5 mm is greater than would be expected for a longer gage sample.

The flow stress taken at a true strain of 0.1 is plotted as a function of the initial strain rate on double logarithmic scales in Figure 5. The strain rate sensitivity (m) of the FSP sample ranged from 0.12 to 0.42 at various temperatures with variation in the initial strain rate. Maximum superplasticity at various temperatures was associated with m values of 0.33–0.42. At higher temperatures, grain boundary sliding (GBS) is the dominant superplastic deformation mechanism in fine-grained materials and is characterized by a strain rate sensitivity of ~0.5. However, for superplastic deformation at lower temperatures, the strain rate sensitivity was typically ~0.3–0.4 [5,7–11,20,23]. A strain rate sensitivity of 0.33 usually indicates that the superplastic deformation mechanism is associated with solute drag for Al–Mg alloys. However, a previous study showed that "Rachinger GBS" might be a plausible mode of deformation for FSP Al–Zn–Mg–Sc alloy with a strain rate sensitivity of 0.33 [23]. Furthermore, Islamgaliev et al. suggested that the GBS is an important deformation process in 1421 aluminum alloy at a temperature of 400 °C, though the measured strain rate sensitivity of ~0.2–0.3 is lower

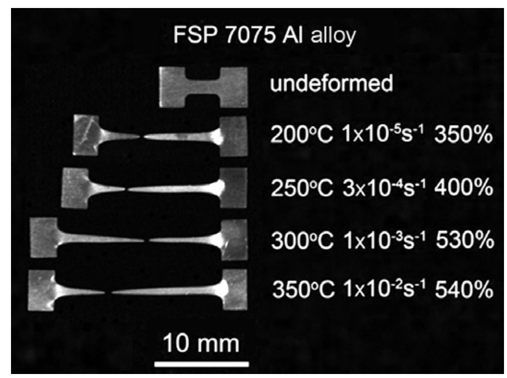

Figure 4. Appearance of specimens before and after maximum superplastic deformation at different temperatures.

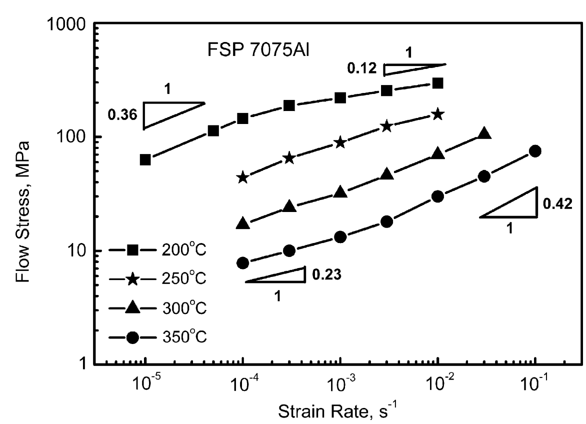

Figure 5. Variation of flow stress with initial strain rate at different temperatures for FSP 7075Al.

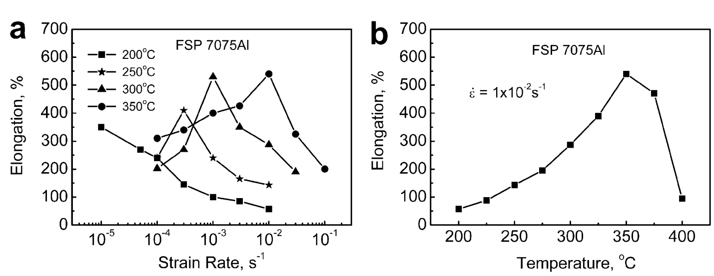

Figure 3. Variation of ductility with (a) initial strain rate for different temperatures and (b) temperature at an initial strain rate of $1 \times 10^{-2}\ s^{-1}$ for FSP 7075Al.

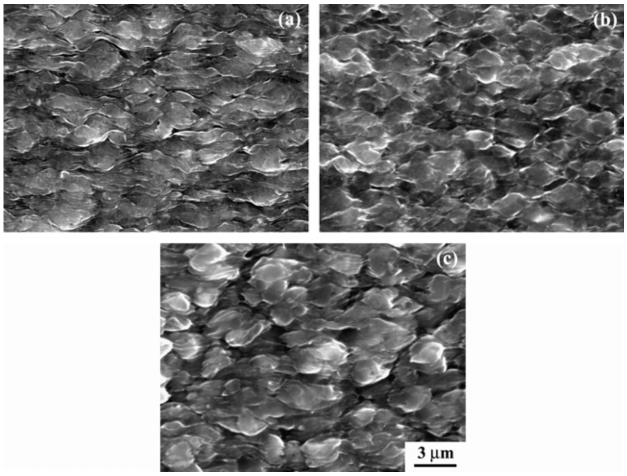

Figure 6. The surface topography of tensile specimens near the fracture tip superplastically deformed to failure at (a) 200 °C and 1×10^{-5} s^{-1}, (b) 250 °C and 3×10^{-4} s^{-1}, (c) 350 °C and 1×10^{-2} s^{-1}.

than the anticipated value for conventional superplasticity [26].

In this study, SEM examinations revealed the distinct evidence of the GBS on the surface of the tensile specimens superplastically deformed over the temperature range 200–350 °C (Fig. 6). Therefore, GBS can be dominant in the superplastic deformation process of UFG aluminum alloys at lower temperatures with lower strain rate sensitivities. It is expected that with refining the grain size further, the GBS might extend its operation below 0.5 T_m.

Fiber (whisker) formation is generally thought to be the evidence of the existence of liquid phases along grain boundaries at high temperatures, and the quantity and radius of the fibers increased as the testing temperature increased [27–30]. However, some fibers were detected between sliding grains in the FSP 7075Al deformed at low temperatures of 200–350 °C (Fig. 6), which are much lower than the melting points of the phases [31,32]. Previously, the fibers were also observed by Zelin in Al–5% Mg–2% Li at 250 °C [33]. These observations indicate that the existence of a liquid phase might not be a prerequisite for fiber formation. Further study is needed to elucidate the origin of the fibers at low-temperature.

In summary, the following conclusions are reached:

1. Fine grains of 0.8 μm in size were obtained in commercial 7075Al rolled plate by FSP with water cooling behind the pin.
2. Superplasticity of 350–540% was achieved at low temperatures of 200–350 °C. The optimum strain rate for superplasticity as well as maximum elongation increased with increasing superplastic deformation temperature. A maximum ductility of 540% was obtained at 350 °C and a high strain rate of 1×10^{-2} s^{-1}.
3. A strain rate sensitivity of 0.33–0.42 was observed for maximum superplasticity at various temperatures. Grain boundary sliding was observed even at temperatures as low as 200 °C.

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