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Superplastic deformation mechanism of an ultrafine-grained aluminum alloy produced by friction stir processing

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Abstract

An ultrafine-grained (UFG) Al–4Mg–1Zr alloy with a grain size of ∼0.7 μm with predominantly high-angle boundaries of 97% was produced by friction stir processing (FSP). The UFG Al–4Mg–1Zr retained submicrometer grains even after static annealing at 425 °C, and exhibited excellent superplasticity at 175–425 °C. High strain rate and low-temperature superplasticity of >1200% were observed at 1×10^{-2}–1×10^{-1} s^{-1} and 300–350 °C. Even at 425 °C, a superplasticity of 1400% was achieved at 1 s^{-1}. A linear relationship between $\log \dot{\varepsilon}_{opti}$ and T was observed (where $\dot{\varepsilon}_{opti}$ is the optimum strain rate, and T is the temperature). The analyses on the superplastic data revealed the presence of threshold stress, a stress exponent of 2, an inverse grain size dependence of 2, and an activation energy of 142 kJ mol^{-1}. This indicated that the dominant deformation mechanism was grain boundary sliding, which was controlled by lattice diffusion. Based on this notion, a constitutive equation has been developed. A new superplastic deformation mechanism map for FSP aluminum alloys is proposed.

Keywords: Superplasticity; Friction stir processing; Aluminum alloys; Ultrafine-grained microstructure

1. Introduction

Superplasticity refers to the ability of materials to exhibit high uniform elongation when pulled in tension. From the viewpoint of practical industrial fabrication, it is highly desirable to perform superplastic forming at higher strain rates and/or lower temperatures. A higher forming rate of $>1 \times 10^{-2}$ s^{-1} is very attractive for current industrial fabrication techniques because one of the drawbacks with existing superplastic forming technology is its low forming rate, typically 10^{-5}–10^{-3} s^{-1} [1]. On the other hand, a lower forming temperature would save energy, prevent grain growth, and reduce cavitation level and solute loss from the surface layer, thereby maintaining superior post-forming properties [2].

In the past few years, much research has been devoted to producing fine-grained aluminum alloys exhibiting high strain rate superplasticity (HSRS) and/or low-temperature superplasticity (LTSP), by using thermomechanical treatment (TMT) [3–5], equal channel angular pressing (ECAP) [6–9], high-pressure torsion (HPT) [10], multiaxial alternative forging (MAF) [11], and accumulative roll bonding (ARB) [12].

Friction stir processing (FSP) is a relatively new processing technique for producing fine-grained aluminum alloys exhibiting HSRS and/or LTSP. This has led to considerable research interest in superplastic behavior of FSP aluminum alloys. In the past few years, a number of aluminum alloys, such as 7075Al, 7050Al, 2024Al, 5083Al, A356,

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Al–4Mg–1Zr, Al–Mg–Sc and Al–Zn–Mg–Sc, have been subjected to FSP and superplasticity investigations [13–23].

It is important to develop a constitutive equation of superplastic flow for FSP aluminum alloys. The steady-state deformation of polycrystalline materials at elevated temperatures is usually analyzed through the equation [25–28]:

$$\dot{\varepsilon} = A\frac{D_{o}Eb}{kT}\exp\left(-\frac{Q}{RT}\right)\left(\frac{b}{d}\right)^{p}\left(\frac{\sigma-\sigma_{o}}{E}\right)^{n}, \quad (1)$$

where $\dot{\varepsilon}$ is the strain rate, A is a constant, D_o is the pre-exponential factor for diffusion, E is the Young's modulus, b is the Burger's vector, k is the Boltzmann's constant, T is the absolute temperature, Q is the activation energy dependent on the rate-controlling process, R is the gas constant, d is the grain size, σ is the applied stress, and σ_o is the threshold stress. Three variables, n, p and Q, are the most important for determining the deformation mechanism.

A number of fine-grained FSP aluminum alloys (grain sizes 1–10 μm) exhibit HSRS [13–19]. Surface observations have revealed evidence of GBS. The constitutive equations have been described in the form of [14,16,18]:

$$\dot{\varepsilon} = A\frac{D_{o}Eb}{kT}\exp\left(-\frac{84000}{RT}\right)\left(\frac{b}{d}\right)^{2}\left(\frac{\sigma-\sigma_{o}}{E}\right)^{2}. \quad (2)$$

The coefficient A is in the range of 700–1400, which suggests that the kinetics of grain boundary sliding (GBS), which dominate the deformation in the FSP materials, is higher than that in conventionally processed materials [27]. The deformation mechanism is GBS controlled by grain boundary diffusion.

LTSP has been obtained in several FSP ultrafine-grained (UFG) aluminum alloys, such as 7075Al, Al–4Mg–1Zr, Al–Mg–Sc and Al–Zn–Mg–Sc [20–24]. The optimum strain rate, maximum elongation and strain rate sensitivity shifts to higher values with increasing temperature. Abnormal grain growth (AGG) usually occurs in the UFG alloys at elevated temperatures, resulting in the disappearance of superplasticity. Surface observation of the deformed specimens indicates that GBS occurs during LTSP. Marker line offset measurements have shown that the contribution of GBS to the total strain for FSP UFG Al–Mg–Sc exceeded 50% even at 175 °C [24]. Despite a number of LTSP studies on the FSP UFG aluminum alloys, fundamental understanding of the deformation mechanism is still poor. Stress exponent, grain size exponent and activation energy should be characterized to clarify the deformation mechanism. A constitutive equation for the UFG alloys is also needed for superplastic forming in practical industrial fabrication.

Recently, a UFG Al–4Mg–1Zr alloy with a grain size of 0.7 μm was produced via FSP under a low heat input using a small tool [22]. It was reported that this UFG Al–4Mg–1Zr exhibited superplasticity of 240% at a low temperature of 175 °C, corresponding to $0.48T_m$, where T_m is the absolute melting temperature of aluminum. This was the first report of superplasticity at temperatures below $0.5T_m$ for aluminum alloys. Irrespective of good superplasticity at lower temperatures, superplastic tensile tests over a wide temperature range, detailed microstructural examinations and superplastic data analyses on the FSP UFG Al–4Mg–1Zr are still lacking.

In this work, the superplastic behavior of the FSP UFG Al–4Mg–1Zr was investigated over a wide temperature range of 175–425 °C and strain rates of 5×10^{-5}–$3\ s^{-1}$. The purpose of this study is: (i) to examine the boundary characteristics and thermal stability of the FSP UFG Al–4Mg–1Zr; (ii) to evaluate the superplastic behavior of the FSP UFG Al–4Mg–1Zr at elevated temperatures; and (iii) last, but most importantly, through clarifying the stress exponent, grain size exponent and activation energy, to develop a constitutive equation for UFG aluminum alloys and identify the deformation mechanism.

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 [29,30]. Single-pass FSP was conducted on the extruded bar along the extrusion direction at a tool rotation rate of 600 rpm and a tool traverse speed of 25.4 mm min^{-1}. A tool with a shoulder 12 mm in diameter and a threaded cylindrical pin 4 mm in diameter and 4 mm in length was used.

The specimens for microstructural examination were cross-sectioned perpendicular to the FSP direction. Microstructural characterization and analysis were carried out using transmission electron microscopy (TEM) and scanning electron microscopy (SEM, Hitachi S-3400N). Thin foils for TEM were prepared using jet polishing techniques. Jet polishing was conducted at −25 °C using a solution of 20% HNO_3 + 80% methanol (by vol.). The average grain size in the FSP sample was determined by the mean linear intercept technique. The specimens for SEM were lightly electropolished to produce a strain-free surface. Electron backscatter diffraction (EBSD) orientation maps (with a resolution of 80 nm) were obtained using a Zeiss Supra 35, operated at 20 kV, and interfaced to an HKL Channel EBSD system. The indexing rate was 91%. A standard noise reduction technique was applied before calculating the misorientation angles. Owing to the limited angular resolution, misorientations less than 2° were not considered.

To check the thermal stability of the ultrafine grains produced by FSP, small specimens with a dimension of 10 × 10 × 10 mm^3 cut from the FSP sample were statically annealed for 20 min at temperatures ranging from 175 to 425 °C, and then water quenched to provide microstructure information of the FSP sample just before tensile tests at various temperatures.

Mini tensile specimens with a gauge length of 1.3 mm were electrodischarge-machined from the FSP region in the transverse direction, ground and polished to a final thickness of ~0.5 mm using a 1 μm polishing paste. Constant crosshead