



High strain rate superplasticity of a fine-grained AZ91 magnesium alloy prepared by submerged friction stir processing

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ABSTRACT

The as-cast AZ91 plate was subjected to normal friction stir processing (processed in air) and submerged friction stir processing (processed in water, SFSP), and microstructure and superplastic tensile behavior of the experimental alloys were investigated. SFSP results in remarkable grain refinement due to the enhanced cooling rate compared with normal FSP, with an average grain size of 1.2 μm and 7.8 μm . The SFSP AZ91 specimen exhibits considerably enhanced superplastic ductility with reduced flow stress and higher optimum strain rate, as compared to the normal FSP specimen. The optimum superplastic deformation temperature is found to be 623 K for both the normal FSP and SFSP AZ91 specimens. An elongation of 990% is obtained at $2 \times 10^{-2} \text{ s}^{-1}$ and 623 K for the SFSP specimen, indicating that excellent high strain rate superplasticity could be achieved. By comparison, maximum ductility of the normal FSP specimen strained at high strain rate is 158%. Grain boundary sliding is the main mechanism for the superplastic deformation of the normal FSP and SFSP specimens. The excellent high strain rate superplasticity of the SFSP alloy is attributed to its finer grain structure and higher fraction of grain boundary.

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1. Introduction

As the lightest materials among constructional alloys, magnesium alloys are expected to be widely-used in transportation and aerospace industries [1–3]. However, one of the main limitations for the application of magnesium alloy is its poor formability due to its intrinsic HCP crystal structure. Aiming at improving the ductility of magnesium alloys, there are extensive works on the development of fine-grained magnesium alloys, which are produced through techniques such as spray forming, powder metallurgy, and severe plastic deformation techniques [4–6].

Superplasticity is the capacity of a crystalline material to undergo large plastic deformation prior to failure. In general, a tensile elongation higher than 500% can be achieved in superplastic materials [7]. Superplastic forming is a near-net shape forming process which has considerable industrial applications. However, typical strain rates for superplastic deformation are among 10^{-4} to 10^{-3} s^{-1} , which are not cost effective for large volume productions [8,9]. Moreover, low strain rates may lead to undesirable oxidation and grain growth during superplastic forming at elevated temperatures. Therefore, high strain rate superplasticity (HSRS), which refers to the ability of a material to sustain plastic deformation at strain rates not lower than 10^{-2} s^{-1} with a total ductility > 200%

[8,10,11], is of great commercial importance to increase the utilization of superplastic forming. Due to HSRS, components with complex shape can be produced within relatively short forming time. However, magnesium alloys possessing HSRS are rarely reported, although excellent superplasticity is achieved in many fine-grained magnesium alloys [12–14]. Consequently, development of materials with HSRS is of great significance to expand the application of magnesium alloys.

The constitutive law for superplasticity flow is expressed as [15]

$$\dot{\varepsilon} = \frac{ADGb}{KT} \left(\frac{b}{d}\right)^p \left(\frac{\sigma - \sigma_{th}}{G}\right)^n \quad (1)$$

where $\dot{\varepsilon}$ is the strain rate, A is a constant, D is the diffusion coefficient, G is the shear modulus, b is Burgers vector, K is Boltzmann's constant, T is the absolute temperature, d is the grain size, σ is flow stress, σ_{th} is the threshold stress, p is the grain size exponent, and n is the stress exponent. Eq. (1) demonstrates that a decrease in the grain size will result in an increase in the strain rate associated with optimum superplastic flow at a constant temperature. Therefore, grain refinement is one of the main ideas to develop magnesium alloys with HSRS.

Friction stir processing (FSP) is a novel grain refinement technique developed by Mishra et al. based on the basic principles of friction stir welding (FSW) [16,17]. To present, many researchers have shown that normal FSP, which is done in air without any additional cooling, is an effective method to refine grain size and

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improve ductility of metallic materials. Typical researches showing HSRS in magnesium and aluminum alloys prepared by normal FSP are summarized in Table 1 [8,18–24]. From Table 1, it can be found that the grain sizes of aluminum alloys prepared by normal FSP are much finer than those of the magnesium alloys. Moreover, the normal FSP aluminum alloys exhibit excellent HSRS, and elongations higher than 1000% can be achieved at a rather high strain rate. On the other hand, HSRS of the normal FSP magnesium alloys is not so good since most of the elongations are below 200% when strained at a strain rate higher than $1 \times 10^{-2} \text{ s}^{-1}$. In our previous works, we have prepared fine-grained AZ31 and AZ91 magnesium alloys through normal FSP, and the maximum elongation was 1050% for the AZ31 alloys when strained at 723 K and $5 \times 10^{-4} \text{ s}^{-1}$ and 1604% for the AZ91 alloys when strained at 573 K and $4 \times 10^{-4} \text{ s}^{-1}$, respectively [22,23]. However, the excellent superplasticities were both gained at a relatively low strain rate, which greatly limits the application of superplastic forming. In order to produce HSRS material, magnesium alloys with much finer grain size should be prepared.

Mishra et al. suggested that materials with much finer structure could be prepared by means of enhancing cooling rate during FSP [16]. Based on this consideration, submerged friction stir processing (SFSP) comes up as a further variation to normal FSP, which means that the entire processing is carried out underwater. Some researchers have demonstrated that SFSP has great potential in the preparation of ultrafine-grained aluminum alloys [25,26]. However, researches on the SFSP magnesium alloys are still limited. To the best of the authors' knowledge, superplastic behaviors of magnesium alloys prepared by SFSP are not reported until now.

In this study, AZ91 magnesium alloys were subjected to normal FSP and SFSP, and microstructure and tensile properties at elevated temperature were examined. The aim is to demonstrate that SFSP can be used as a potential grain refinement technique for AZ91 alloy, and to evaluate the possibility of obtaining HSRS in the SFSP AZ91 alloy.

2. Experimental

Cast AZ91 magnesium alloy billets with a composition of 9.08Al–0.60Zn–0.27Mn–0.014Si–0.002Fe–0.012Ce (wt%) were used

in this study. Plates with a thickness of 6 mm were machined from the cast billets and subjected to normal FSP and SFSP at a rotation speed of 800 rpm and a traverse speed of 60 mm/min. The FSP experiments were carried out on FSW-3LM-003 welding machine with a 5.6 mm diameter, 5 mm length cone-threaded pin and a concave shoulder 16 mm in diameter. SFSP was conducted in a tank with the plate completely submerged in water, and the flow rate of water was 29 mL/s in the experiment.

Specimens used for microstructural examinations were cross-sectioned perpendicular to the transverse direction. Microstructures of the normal FSP and SFSP specimens were observed by scanning electron microscopy (SEM, Nova Nano 430, FEI, USA). A solution of 5 g picric acid, 10 mL acetic acid, 10 mL distilled water and 80 mL ethanol was used as the etchant of the specimens. Mean linear intercept method was used to estimate the average grain size. The normal FSP and SFSP tensile specimens with a gauge dimension of 3.5 mm \times 1.5 mm \times 5 mm were cut by electro-discharged machining in the transverse direction so that the gauge parts consisted of friction stir zone only. High temperature tensile tests were performed on computer-controlled universal testing machine (CMT5105, SANS Co. Ltd, China), with the test temperatures ranging from 523 K to 648 K and the strain rates ranging from $2 \times 10^{-2} \text{ s}^{-1}$ to $4 \times 10^{-4} \text{ s}^{-1}$. Tensile fracture morphologies were observed by SEM mentioned above. Microstructures of the specimens after tensile test were also observed by optical microscopy (OM, Keyence, VHX-600, Japan).

3. Results

3.1. Microstructure characteristics

Fig. 1(a) and (b) shows the optical macrographs of the stirred zone (SZ) of the normal FSP and SFSP AZ91 alloys in the transverse cross-section, respectively, along with thermo-mechanically affected zone (TMAZ) and heat-affected zone (HAZ). In each macrograph, onion-ring patterns which are formed by the flow of the material during FSP can be found inside the SZ. No defects are observed in the normal FSP and SFSP specimens, indicating that good processing quality could be achieved under the given conditions.

Table 1
A comparison of HSRS of magnesium and aluminum alloys prepared by normal FSP.

Alloy	Processing parameter ($\omega-v$) (rpm mm/min)	Grain size (μm)	Temperature (K)	Strain rate (s^{-1})	Elongation (%)	Reference
2024Al	300–25.4	3.9	703	1×10^{-2}	525	[8]
Al–4Mg–1Zr	–	1.5	798	1×10^{-1}	1280	[18]
Al–5.3Mg–0.23Sc	600–25	2.6	723	1×10^{-1}	2150	[19]
Al–8.9Zn–2.6Mg–0.09Sc	400–25.4	0.68	583	3×10^{-2}	1165	[20]
AZ91	700–150	4	498	1×10^{-2}	< 100	[21]
AZ91	400–60	3	573	2×10^{-2}	188	[22]
AZ31	1500–60	11.4	573	1×10^{-2}	150	[23]
Mg–Zn–Y–Zr	800–100	5.2	698	1×10^{-2}	~300	[24]

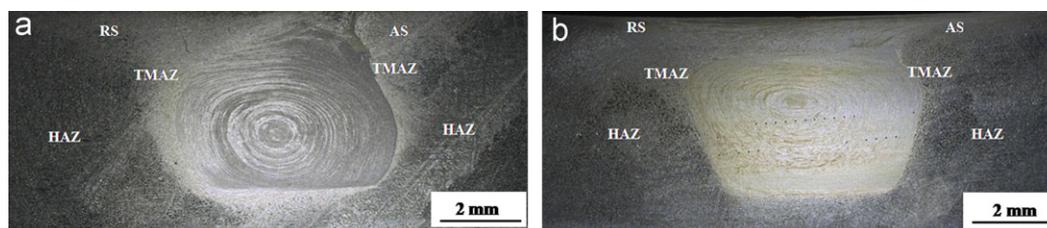


Fig. 1. Macrographs of the FSP specimens in transverse section (a) normal FSP and (b) SFSP.

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