Available online at www.sciencedirect.com
ScienceDirect
Trans. Nonferrous Met. Soc. China 21(2011) 1911−1916

Transactions of
Nonferrous Metals
Society of China
www.tnmsc.cn

Superplasticity of AZ31 magnesium alloy prepared by friction stir processing

ZHANG Da-tong, XIONG Feng, ZHANG Wei-wen, QIU Cheng, ZHANG Wen

School of Mechanical and Automotive Engineering, South China University of Technology,
Guangzhou 510640, China

Received 28 July 2010; accepted 26 December 2010

Abstract: Microstructure and tensile behaviors of AZ31 magnesium alloy prepared by friction stir processing (FSP) were investigated. The results show that microstructure of the AZ31 hot-rolled plate with an average grain size of 92.0 μm is refined to 11.4 μm after FSP. The FSP AZ31 alloy exhibits excellent plasticity at elevated temperature, with an elongation to failure of 1050% at 723 K and a strain rate of 5×10^{-4} s^{-1}. The elongation of the FSP material is 268% at 723 K and 1×10^{-2} s^{-1}, indicating that high strain rate superplasticity could be achieved. On the other hand, the hot-rolled base material, which has a coarse grain structure, possesses no superplasticity under the experimental conditions.

Key words: friction stir processing; AZ31 magnesium alloy; superplasticity; microstructure

1 Introduction

Magnesium alloys are expected to be widely used in aerospace and automobile industries due to their low densities and high specific strength. However, most of the magnesium alloy parts are produced through casting since the plastic-formability of this HCP metal is limited [1]. Grain refinement is considered an effective method to improve the plasticity of magnesium alloys. Therefore, severe plastic deformation (SPD) techniques, such as equal channel angular pressing (ECAP) and accumulating rolling and bonding (ARB) have been investigated extensively in fine-grained magnesium alloy preparation in the past few years. It was found that the elongation of magnesium alloys, either at room temperature or at elevated temperatures, can be improved to a large extent through grain refinement, and superplasticities of these new materials were reported by many researchers [2−7].

Friction stir processing (FSP) is a novel SPD process proposed by Mishra et al based on friction stir welding (FSW) [8]. Through FSP, CHARIT and MISHRA [9] produced an ultrafine grained Al-Zn-Mg-Sc alloy which possessed excellent ductility at low temperature (220 °C) and high stain rate [9]. LIU et al [10] reported a friction stir processed ultrafine-grained Al-Mg-Sc alloy with a ductility of 620% at 300 °C and 3×10^{-2} s^{-1}. As to magnesium alloys, FSP of AM60 [11], AZ91 [12], AZ31 [13], Mg-Al-Ca [14] and Mg-RE alloys [15] was investigated by different researchers. FSP has been proved effective for grain refinement in these magnesium alloys, and CHANG et al [13] even produced an AZ31 alloy with its grain size as fine as 100−300 nm. CAVALIERE and MACRO [11−12] reported that AM60 and AZ91 magnesium alloys prepared by FSP exhibited superplasticity properties due to the fine and stable microstructures. However, superplastic behaviors of magnesium alloys prepared by FSP are still not fully studied compared with those alloys prepared by other SPD techniques.

AZ31 is a widely used wrought magnesium alloy, and FSW/FSP of this alloy has been studied extensively in the past few years [13, 16−19]. On the other hand, superplasticity of AZ31 alloy prepared by hot extrusion, ECAP and other SPD methods has been investigated thoroughly [4−5]. However, superplasticity of AZ31 alloy prepared by FSP has been rarely reported up to now. HUNG et al [18] compared the tensile properties of AZ31-O and FSP material, and their results showed that the tensile elongation of friction stir processed AZ31 was almost the same as the base material at 373−673 K. LEE

Foundation item: Project (2009Z2-D811) supported by Guangzhou Science and Technology Development Program, China; Project (2009ZM0264) supported by the Fundamental Research Funds for the Central Universities, China

Corresponding author: ZHANG Da-tong; Tel: +86-20-87112272; Fax: +86-20-87112111; E-mail: dtzhang@scut.edu.cn

DOI: 10.1016/S1003-6326(11)60949-7

et al [19] found that FSP could improve the room temperature tensile ductility of a hot-extruded AZ31 alloy. In this study, FSP was used to prepare AZ31 magnesium alloy, and the microstructure and mechanical properties of the experimental materials were investigated. It was found that the ductility of the FSP AZ31 alloys was improved greatly, and superplasticity of this material was achieved.

2 Experimental

FSP was carried out on commercial hot-rolled AZ31 magnesium alloy plate with a thickness of 4 mm, and the chemical composition of the plate was Mg-2.57Al-0.84Zn-0.32Mn (mass fraction, %). The facility used was FSW-3LM-003 welding machine with a cone- threaded pin of 3 mm in diameter, 3 mm in length and a concave shoulder 10 mm in diameter. The FSP experiments were conducted parallel to the rolling direction of the plate, with a tool rotation rate of 1 500 r/min and a traveling speed of 60 mm/min.

Dog-bone shaped tensile specimens with gauge dimensions of 3 mm×2.5 mm×1.5 mm were cut carefully in the transverse direction by electro-discharged machining so that the gauge parts of the specimens consisted of friction stir zone only. High temperature tensile tests were performed on a SANS CMT5105 machine, with the test temperature ranging from 573 K to 723 K and the strain rate ranging form 1×10^{-2} s^{-1} to 5×10^{-4} s^{-1}. For comparison, tensile tests of the base materials (BM) specimens which were machined from the as-received plate along the rolling direction were also performed.

Microstructures of the BM and FSP specimens were observed by KEYENCE VHX−600 optical microscope (OM), a solution of 4.2 g picric acid, 8 mL acetic acid, 10 mL distilled water and 70 mL ethanol was used as the etchant of the specimen. Mean linear intercept technique was used to determine the average grain size. Microstructure near the tensile fracture tip was also examined with the same method. A Quata200 scanning electron microscope (SEM) equipped with an EDX system was used to observe the tensile fracture morphologies.

3 Results and discussion

3.1 Microstructure

Figure 1 shows the optical microstructures of the BM and FSP specimens. The initial microstructure of BM consists of coarse α(Mg) grains with an average grain size of 92 μm. Microstructure of FSP specimen was greatly refined due to dynamic recrystallization, and the average grain size in the stir zone (SZ) is 11.4 μm. It is well known that equiaxed and fine-grained structure (usually ≤10 μm) is a favorable condition for superplastic deformation of metals although some exceptions existed [20]. Therefore, the FSP material is expected to show superplasticity when deformed under suitable conditions.

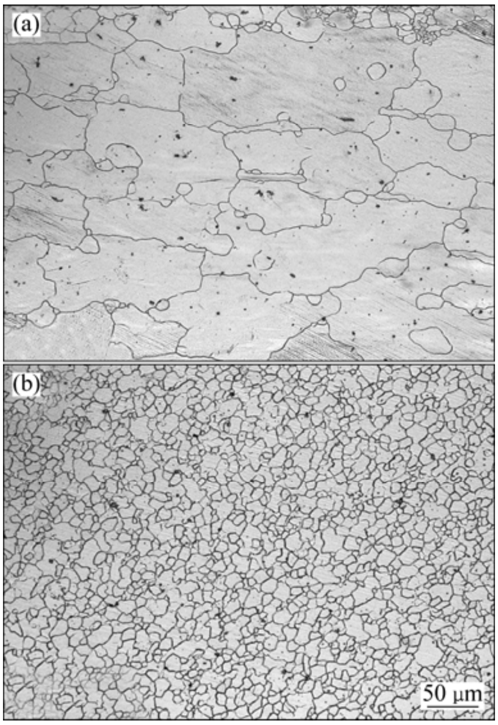

Fig. 1 Optical microstructures of experimental materials: (a) BM; (b) FSP

3.2 Tensile properties

Figure 2 shows the appearances of the BM and FSP specimens after tensile test at a strain rate of 5×10^{-4} s^{-1}. As shown in Fig. 2(b), the elongation to fracture of the FSP specimen is 150% at 573 K, and necking can be seen clearly on the test specimen, indicating that the FSP material does not exhibit superplasticity under this condition. With increasing the tensile temperature, the deformation of the specimen becomes more and more uniform. The elongation to fracture is 489% at 673 K, and the elongation increases to 1050% at 723K, which is the maximum in this study.

Comparison of the elongation to fracture between BM and FSP specimens with a strain rate of 5×10^{-4} s^{-1} is plotted in Fig. 3. It can be seen that at all of the test temperatures, the elongation of the FSP specimen is much higher than that of the BM specimen. Furthermore, the maximum elongation of BM specimen is only 112%, which is obtained at 723 K. Therefore, the BM exhibits no superplasticity even at high temperature. The main reason for the poor ductility of BM is that the grain size is too coarse, as shown in Fig. 1.

Tensile behaviors of the FSP specimens were

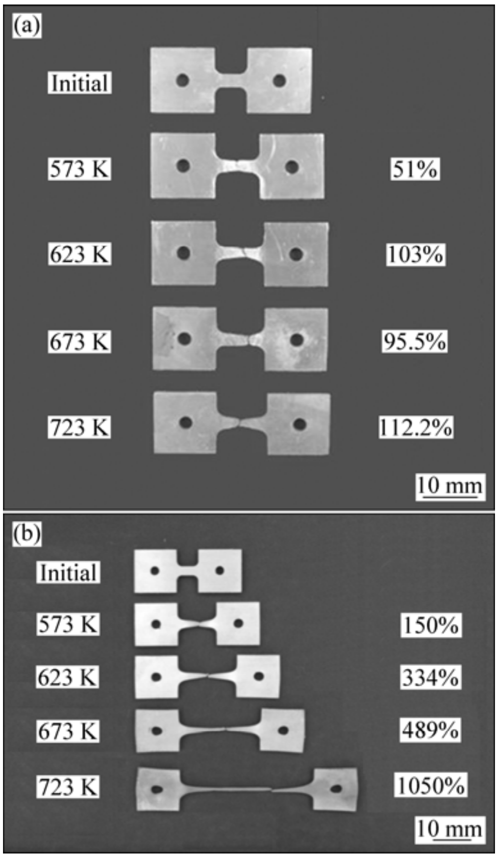

Fig. 2 Appearances of specimens after tensile test at elevated temperature with strain rate of 5×10^{-4} s^{-1}: (a) BM; (b) FSP

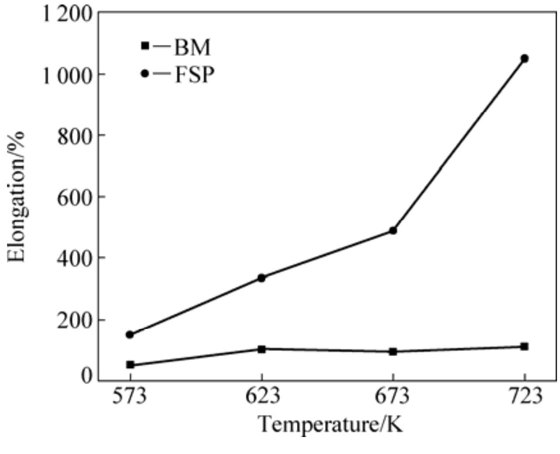

Fig. 3 Elongations of BM and FSP at different tensile temperatures with strain rate of 5×10^{-4} s^{-1}

investigated in a temperature range of 573−723 K and a strain rate range of 1×10^{-2}–5×10^{-4} s^{-1}. Figure 4 shows the effects of temperature and strain rate on the elongation of the FSP AZ31 alloy. Under the test conditions, the elongation to failure decreases with increasing the strain rate. The elongation to failure at 573 K is in a range of 120%−150%, indicating that the strain rate has little effect on elongation at low temperature. On the other hand, the elongation of the FSP specimen increases greatly at 723 K with the decrease of strain rate. The elongation at 723 K strained at 1×10^{-2} s^{-1} is 268%, indicating that high strain rate superplasticity can be achieved in the FSP material.

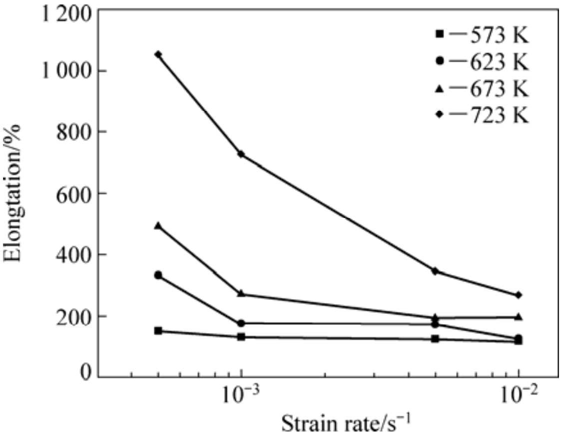

Fig. 4 Elongation of FSP specimen at different temperatures and strain rates

Flow stresses at a true strain of 0.2 during tensile tests were measured. The results are summarized in Fig. 5. As shown in Fig. 5, the flow stress decreases with the temperature increasing and strain rate decreasing. The mean strain rate sensitivity (*m* value) is lower than 0.2 at 573 K, which is consistent with the low elongation, as shown in Fig. 4. When the test temperature is higher than 673 K, most of the strain rate sensitivity *m* values are higher than 0.3, and the highest *m* value is ~0.60, which is observed at 723 K and 1×10^{-2} s^{-1}. A high *m* value (≥0.3) is generally necessary for superplastic alloys to prevent necking occurrence [21], and the *m* value of the FSP AZ31 alloy is in agreement to the elongation measurement result.

Figure 6 shows the microstructures near the fracture tips after tensile test at a strain rate of 5×10^{-4} s^{-1} and different test temperatures. In Fig. 6, the tensile direction lies in horizontal. The initial microstructure of FSP

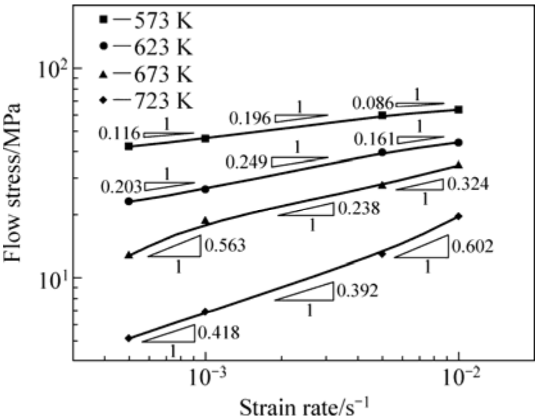

Fig. 5 Flow stress of FSP specimen at different temperatures and strain rates

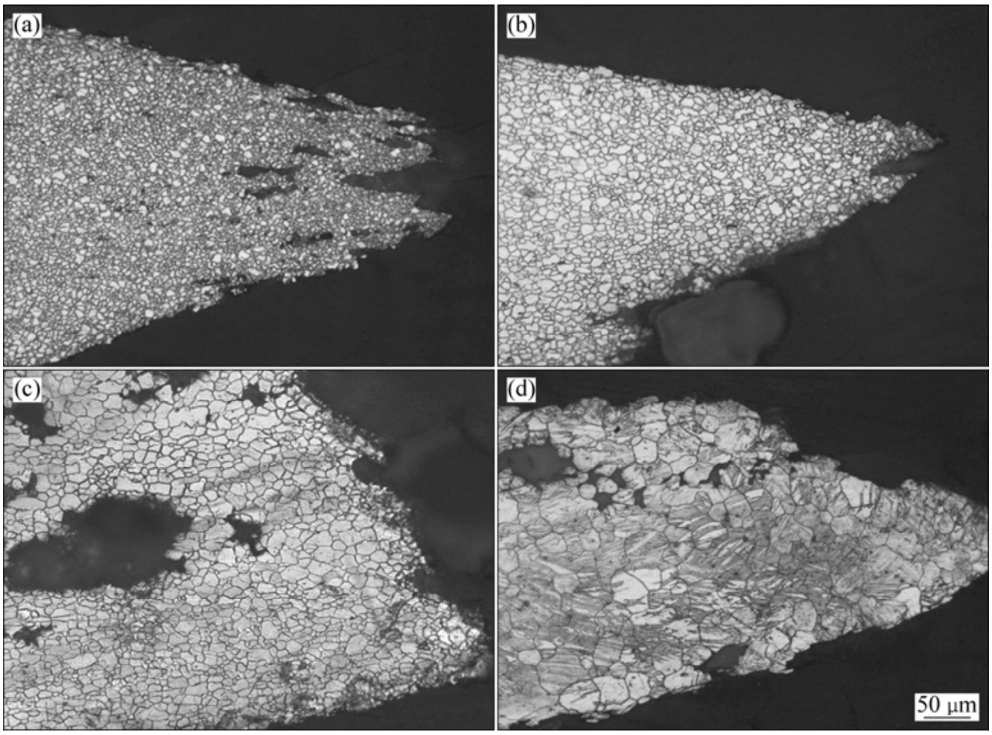

Fig. 6 Optical microstructures of FSP specimen after tensile test at strain rate of 5×10^{-4} s^{-1} and different temperatures: (a) 573 K; (b) 623 K; (c) 673 K; (d) 723 K

specimen has an average grain size of 11.4 μm, as shown in Fig. 1(b). The grain structure after tensile failure remains equiaxed, but the grain size is quite different compared with the initial grain structure. The average grain size of the specimen strained at 573K is ~8 μm, which is finer than that of the initial material (Fig. 6(a)). BUSSIBA et al [22] also found the microstructure refinement near the necking zone when an extruded AZ31 was deformed to failure at 450 K. With the tensile temperature increasing, the grain structure becomes coarse. The average grain sizes of the specimen strained at 623, 673 and 723 K are 11.9, 17.8 and 35.6 μm, respectively. During superplastic deformation, two competitive mechanisms, i.e. dynamic grain growth and dynamic recrystallization will operate in the microstructure evolution of AZ31 alloy, and the final structure may be different according to the initial microstructure and deformation conditions [4]. As to the present material, dynamic recrystallization may play an important role at lower temperatures (573 K), while grain growth is the dominating mechanism for the microstructure evolution during superplastic deformation at high temperature (673 K and 723 K). At 623 K, it seems that the two processes, dynamic recrystallization and grain growth, get into balance since the initial grain size is almost equal to the finial structure. As a comparison, remarkable grain growth was observed in an ECAE AZ31 alloy pulled at 623 K, from initial grain size of 4−5 μm to 30.3 μm at failure [4]. Moreover, cavities can be seen clearly in Figs. 6(c) and (d). A large cavity with a size of ~200 μm is recognized in Fig. 6(c), which is formed by cavity coalescence during superplastic deformation.

Figure 7 shows the fracture morphologies of BM and FSP specimens after tensile test at 673 K with a strain rate of 5×10^{-4} s^{-1}. The BM fails in an intergranular fracture manner, while shallow cavities with different sizes can also be observed on the fracture surface, as shown in Fig. 7(a). On the other hand, the size and shape of cavities on the fracture surface of FSP specimen are quite uniform. It is clear that the FSP specimen fails through cavity coalescence in superplastic deformation.

FIGUEIREDO et al [4] summarized the optimum superplasticity in magnesium alloy prepared by ECAE in a recent research, and the maximum elongation of ECAP AZ31 alloy reported was in the range of 1 000%−1 200%. The maximum elongation of FSP AZ31 alloy in this study is 1070%, which is at the same level of ECAE AZ31 alloy. However, it should be mentioned that the optimum temperature for superplasticity is different. For AZ31 prepared by ECAP, the optimum temperature is 623 K [4], while the largest elongation is obtained at 723 K in our study. A common phenomenon in these two cases is that the grains near the fracture tip grow coarse obviously during deformation at the optimum temperature. By comparing the superplasticity of

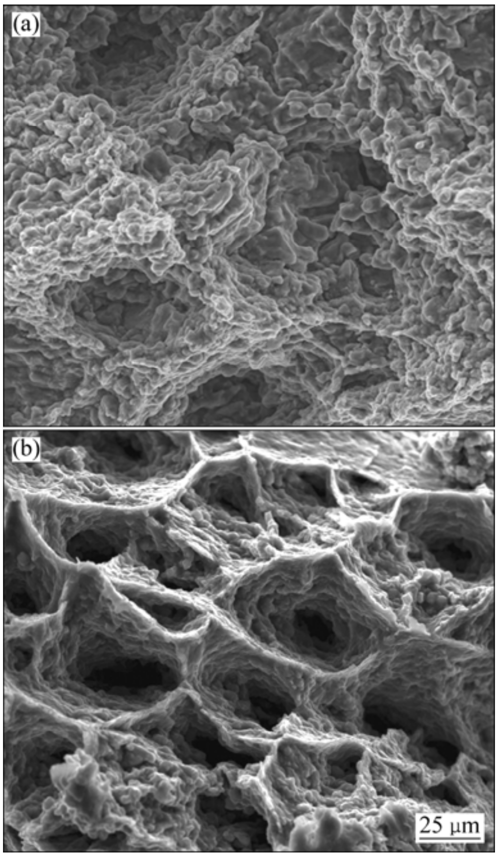

Fig. 7 Surface morphologies of specimens after tensile test at 673 K with strain rate of 5×10^{-4} s^{-1}: (a) BM; (b) FSP

Al-Mg-Sc alloys produced by ECAP and FSP, LIU and MA [23] pointed out that the grain boundary characteristics of the FSP material might have a significant effect on its superplastic behaviors. Moreover, the grain orientation, i.e., texture, is also one of the main reasons responsible for the different deformation behaviors of AZ31 alloys prepared by different SPD methods. Effect of microstructure characteristics on the mechanical behaviors of FSP AZ31 alloy is under research in detail.

4 Conclusions

1) FSP is an effective grain refinement method for AZ31 magnesium alloy. The average grain sizes of the AZ31 BM and FSP specimen are 92 μm and 11.4 μm, respectively.

2) The elongation of the FSP AZ31 alloy increases with increasing the tensile temperature or decreasing the strain rate. The maximum elongation of the FSP material is 1050% at 723 K and 5×10^{-4} s^{-1}. The FSP material also strained at 1×10^{-2} s^{-1} shows a potential to achieve high strain rate superplasticity, with an elongation of 268% at 723 K. The maximum elongation of the BM is only 112%, and no superplasticity properties are detected under the experimental conditions.

3) Grain growth and cavities coalescence play important roles in the deformation behaviors of the FSP AZ31 magnesium alloy at 673 K and 723 K, while dynamic recrystallization dominates the structure evolution at 573 K.

References

[1] MORDIKE B L, EBERT T. Magnesium: Properties-applications-potential [J]. Materials Science and Engineering A, 2001, 302: 37−45.

[2] KOIKE J, KOBAYASHI T, MUKAI T, WATANABE H, SUZUKI M, MARUYAMA K, HIGASHI K. The activity of non-basal slip systems and dynamic recovery at room temperature in fine-grained AZ31B magnesium alloys [J]. Acta Materialia, 2003, 51: 2055−2065.

[3] Del VALLE J A, PÉREZ-PRADO M T, RUANO O A. Accumulative roll bonding of a Mg-based AZ61 alloy [J]. Materials Science and Engineering A, 2005, 410−411: 353−357.

[4] FIGUEIREDO R B, TERZI S, LANGDON T G. Using X-ray microtomography to evaluate cavity formation in a superplastic magnesium alloy processed by equal-channel angular pressing [J]. Acta Materialia, 2010, 58: 5737−5748.

[5] SPIGARELLI S, MEHTEDI M E, REGEV M. Enhanced plasticity and creep in an extruded Mg-Zn-Zr alloy [J]. Scripta Materialia, 2010, 63: 617−620.

[6] KANG Zhi-xin, PENG Yong-hui, LAI Xiao-ming, LI Yuan-yuan, ZHAO Hai-dong, ZHANG Wei-wen. Research status and application prospect of ultrafine grained and/or nano-crystalline metallic materials processed by severe plastic deformation [J]. The Chinese Journal of Nonferrous Metals, 2010, 20(4): 587−598. (in Chinese)

[7] JIAN Wei-wei, KANG Zhi-xin, LI Yuan-yuan. Microstructural evolution and mechanical property of ME20M magnesium alloy processed by multidirectional forging [J]. The Chinese Journal of Nonferrous Metals, 2008, 18(6): 1005−1011. (in Chinese)

[8] MA Zong-yi. Friction stir processing technology: A review [J]. Metallurgical and Materials Transaction A, 2008, 39: 642−658.

[9] CHARIT I, MISHRA R S. Low temperature superplasticity in a friction-stir-processed ultrafine grained Al-Zn-Mg-Sc alloy [J]. Acta Materialia, 2005, 53: 4211−4223.

[10] LIU F C, MA Z Y, CHEN L Q. Low-temperature superplasticity of Al-Mg-Sc alloy produced by friction stir processing [J]. Scripta Materialia, 2009, 60: 968−971.

[11] CAVALIERE P, de MARCO P P. Friction stir processing of AM60B magnesium alloy sheets [J]. Materials Science and Engineering A, 2007, 462: 393−397.

[12] CAVALIERE P, de MARCO P P. Superplastic behaviour of friction stir processed AZ91 magnesium alloy produced by high pressure die cast [J]. Journal of Materials Processing Technology, 2007, 184: 77−83.

[13] CHANG C I, DU X H, HUANG J C. Achieving ultrafine grain size in Mg-Al-Zn alloy by friction stir processing [J]. Scripta Materialia, 2007, 57: 209−212.

[14] ZHANG Da-tong, SUZUKI M, MARUYAMA K. Microstructural evolution of a heat-resistant magnesium alloy due to friction stir welding [J]. Scripta Materialia, 2005, 52: 899−903.

[15] FREENEY T A, MISHRA R S. Effect of friction stir processing on microstructure and mechanical properties of a cast-magnesium-rare earth alloy [J]. Metallurgical and Materials Transaction A, 2010, 41: 73−84.

[16] COMMIN L, DUMONT M, MASSE J E, BARRALLIER L. Friction stir welding of AZ31 magnesium alloy rolled sheets: Influence of

processing parameters [J]. Acta Materialia, 2009, 57: 326−334.

[17] SUHUDDIN U F H R, MIRONOV S, SATO Y S, KOKAWA H, LEE C W. Grain structure evolution during friction-stir welding of AZ31 magnesium alloy [J]. Acta Materialia, 2009, 57: 5406−5418.

[18] HUNG F Y, SHIH C C, CHEN L H, LUI T S. Microstructures and high temperature mechanical properties of friction stirred AZ31-Mg alloy [J]. Journal of Alloys and Compounds, 2007, 428: 106−114.

[19] LEE H W, LUI T S, CHEN L H. Studies on the improvement of tensile ductility of hot-extrusion AZ31 alloy by subsequent friction stir process [J]. Journal of Alloys and Compounds, 2009, 475: 139−144.

[20] WU Xin, LIU Yi. Superplasticity of coarse-grained magnesium alloy [J]. Scripta Materialia, 2002, 46: 269−274.

[21] XU Zhu-yao, LI Peng-xing. An introduction to material science [M]. Shanghai: Shanghai Science and Technology Press, 1986. (in Chinese)

[22] BUSSIBA A, ARTZY A B, SHTECHMAN A, IFERGAN S, KUPIEC M. Grain refinement of AZ31 and ZK60 Mg alloys—Towards superplasticity studies [J]. Materials Science and Engineering A, 2001, 302: 56−62.

[23] LIU F C, MA Z Y. Achieving exceptionally high superplasticity at high strain rates in a micrograined Al-Mg-Sc alloy produced by friction stir processing [J]. Scripta Materialia, 2008, 59: 882−885.

搅拌摩擦加工 AZ31 镁合金的超塑性

张大童，熊 峰，张卫文，邱 诚，张 文

华南理工大学 机械与汽车工程学院，广州 510640

摘　要：对搅拌摩擦加工 AZ31 镁合金的微观组织和拉伸力学行为进行了研究。结果表明，通过搅拌摩擦加工，热轧 AZ31 板材的平均晶粒尺寸由 92.0 μm 细化到 11.4 μm。搅拌摩擦加工板材在高温下具有优异的塑性，伸长率在温度为 723 K 和应变速率为 5×10^{-4} s^{-1} 的条件下达到 1050%。该材料还具有高应变速率超塑性，在 723 K 和 1×10^{-2} s^{-1} 的条件下伸长率达到 268%。在相同实验条件下，母材由于晶粒尺寸粗大，没有显示出超塑性。

关键词：搅拌摩擦加工；AZ31 镁合金；超塑性；微观组织

(Edited by LI Xiang-qun)