Rare Metal Materials and Engineering
Volume 45, Issue 11, November 2016
Online English edition of the Chinese language journal

Available online at www.sciencedirect.com
ScienceDirect

Cite this article as: Rare Metal Materials and Engineering, 2016, 45(11): 2798-2803.

ARTICLE

High Strain Rate Superplasticity of in situ Al_3Zr/6063Al Composites

Jiao Lei[1], Wang Xiaolu[1], Li Hui[2], Zhao Yutao[1], Yang Yonggang[1], Chen Jianchao[1]

[1] Jiangsu University, ZhenJiang 212013, [2] Jiangsu University of Science and Technology, Zhenjiang 212003, China

Abstract: In situ 5wt%Al_3Zr/6063Al composites were fabricate by a direct melt reaction method (DMR) and then processed by deformation pretreatment. Process of the pretreatment was forging of 70% deformation at 450 ℃ and friction stir processing (FSP). The high strain rate superplasticity of the composites was studied by modern analytic determination methods. The results show that the average grain size of the composites is less than 10 μm after forging and FSP. The composites exhibit superplasticity at the temperature from 350 to 500 ℃ and initial strain rate from 1.0×10^{-3} s^{-1} to 1.0×10^{-1} s^{-1}. The elongation reaches 330% and the sensitive index (*m* value) is 0.45 at the initial strain rate of 1.0×10^{-2} s^{-1} and the temperature of 500 ℃. The dominant mechanisms of superplastic deformation are grain boundary sliding and dislocation slip which is synergized with moderate grain growth and dynamic continuous recrystallization.

Key words: in situ reaction; friction stir processing (FSP); superplasticity; high strain rate; deformation mechanism

To date, it has been demonstrated that many aluminum based materials exhibit superplasticity. In particular, high strain rate ($\geq10^{-2}$ s^{-1}) superplasticity present possibilities of technological breakthrough for superplastic forming because conventional superplastic forming rates are very low（～10^{-3} s^{-1}）[1,2]. Although in China, there were several reports related to aluminum matrix composites conventional strain rate [3,4], the reports are insufficient concerning the high strain rate superplasticity of in situ composites[5]. A series of new methods have been developed in order to get high strain rate superplasticity, which are called severe plastic deformation (SPD)[6,7]. Better superplasticity of materials can be obtained by these methods than traditional methods. However this process has a lot of disadvantages such as high preparation cost, low work efficiency, high energy resources consumption and poor performance [8].Therefore it is still the research trend to explore new crafts and methods.

In 1999, Mishra at the University of Missouri proposed friction stir processing technology and prepared fine grain superplastic alloy[9]. Ma Zongyi, Institute of Metal Research, Chinese Academy of Sciences, has made a series of important progress in high strain rate and low temperature superplasticity of super fine crystal aluminum alloy by FSP[10-14].At present, FSP is applied in microstructure refinement of metal, preparation of superplastic materials, surface composites, nano phase reinforced metal matrix composites, etc.[15] In the present paper , the superfine crystal Al_3Zr/6063Al composites with tiny particles were prepared by DMR and the composites exhibit the superplasticity after FSP.

1 Experiment

Industrial 6063Al and industrial purity K_2ZrF_6 powder were used for this experiment to fabricate the in situ composites by DMR. Firstly 6063Al was melt at 900 ℃. K_2ZrF_6 was heated at 300 ℃ for 30 min after grinding and mixing and added into the aluminum alloy melt slowly. The product was agitated using a mechanical stirrer for 30 min at 900 ℃ and poured into a metal mould. Thus 5wt%Al_3Zr/6063Al composites were prepared. Before FSP the thickness of the composite samples was forged from 18 mm to 5 mm at 450 ℃. Then they were processed by

Received date: December 16, 2015
Foundation item: National Natural Science Foundation of China (51174098，51605206); Jiangsu University Senior Talent Project Foundation (15JDG077); The Doctor Research Foundation of the Education Ministry of China (20133227110023)
Corresponding author: Jiao Lei, Ph. D., Associate Professor, School of Materials Science and Engineering, Jiangsu University, Zhenjiang 212013, P. R. China, Tel: 0086-511-88790769, E-mail: jiaolei@sina.com

FSP using the special friction stir welding equipment (FSW-3LM-002). The stirring process parameters were set as the rotational speed of 1000 r/min and the processing speed of 70 mm/min. The hot ductility experiment was also conducted on the composite using a high temperature tensile machine at the tensile deformation temperature of 350~500 ℃ and the initial tensile strain rate of 1×10^{-3}~1×10^{-1} s^{-1}.

2 Results and Discussion

2.1 Microscopic microstructure and phase analysis

Fig.1 shows microstructure of composites in different states observed by SEM and the EDS spectrum. It can be seen that particles include Al and Zr elements from EDS analysis. And the peak height ratio Al to Zr element is 1:3 approximately. So it is demonstrated further that the Al_3Zr particles are produced by in situ reaction and the Al_3Zr/6063Al composites are prepared.

As shown in Fig.1a, 1c and 1d, the size of Al_3Zr particle in casting structures varies from 10 μm to 30 μm and the average is 20 μm, while it is about 10 μm on average at casting state. Compared with them, the size varies from 1 μm to 2 μm in the friction stir state. Therefore, a conclusion can be drawn that forging and friction stir processing can refine grains. In addition as-cast particles present a long strip shape and become obtuse after forging and FSP. We can also find that the agglomeration of reinforcing particles is improved greatly from the figure. It is mainly attributed to that heat exposure and severe plastic deformation generated by FSP break thick long reinforcing. Meantime, the broken reinforcement distributes in the matrix more uniformly because the material flows at high temperature caused by FSP.

2.2 Superplastic deformation

Fig.2 shows the relationship between elongation of the composite material with the temperature and initial strain rate. Fig.2a reveals that the elongation is less than 200% at 400 ℃ and increases with the increase of deformation temperature as the deformation temperature is higher than 400 ℃ at the same strain rate. It shows that the composites exhibit a maximum elongation of 330% at 500 ℃ and the initial strain rate of 1×10^{-2} s^{-1}. As can be seen from Fig.2b, elongation decreases firstly and then increases with the increase of the initial strain rate.

The tensile behavior of the studied material is plotted in Fig.3. As shown in Fig.3a, a decrease in flow stress is observed with the increasing in deformation temperature. This is because as the temperature increases, thermal activation enhanced, average kinetic energy of metal atoms increases, and the atomic vibration amplitude increases, thus increasing the activity performance of dislocations and vacancies, the possibility of dislocation climbing and softening degree provided by the sliding and climbing. Therefore it enhances the dynamic recovery of composites and reduces the flow stress. In addition, another reason to lower the deformation resistance is that the weakening action of the grain boundary, as temperature increases, is more and more notables resulting in that the grain boundary is too weaker to block the

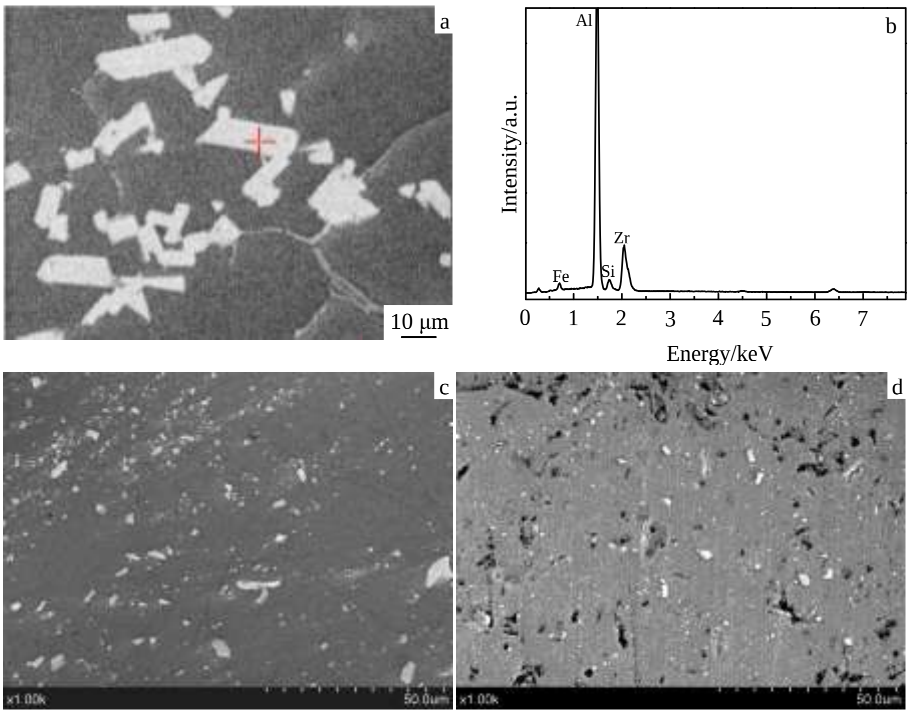

Fig.1 Microstructure of composites in different states and EDS spectrum under casting state: (a) casting states, (b) EDS of casting states, (c) forging states, and (d) FSP

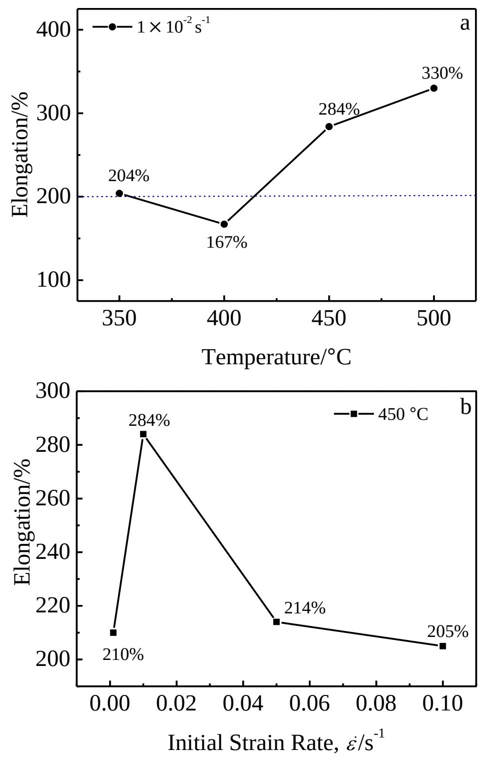

Fig.2 Effects of test temperature (a) and initial strain rate (b) on elongation of composites

deformation; as a result grain boundaries slide together. Fig.3b shows that flow stress is the largest when the initial strain rate is $5.0\times10^{-2}\ s^{-1}$.

2.3 Microstructure before and after superplastic deformation

Fig.4 shows clearly that the matrix grain size increases significantly from 1 μm to 10 μm after high-temperature tension. Similarly, this change is the result of dynamic recrystallization and grains grow up under the high temperature tensile. Therefore, the deformation temperature should be controlled strictly and the dwell time at high temperature should be shortened in the molding process to avoid adverse effect caused by excessive grain coarsening. The grain size is not the same in different parts of the high temperature tensile piece, as shown in Fig.4b and 4c. The size in the middle of stretching pieces is small (less than 10 μm) and less than that of the holding position (20 μm). The grains are refined and isometric state appears in the superplastic deformation process, which creates favorable conditions for further deforming. A large number of holes in material can be also observed after superplastic tension, illustrating that the grain boundary sliding appears in the process of superplastic deformation resulting in the gap and overlap in the grain boundary and

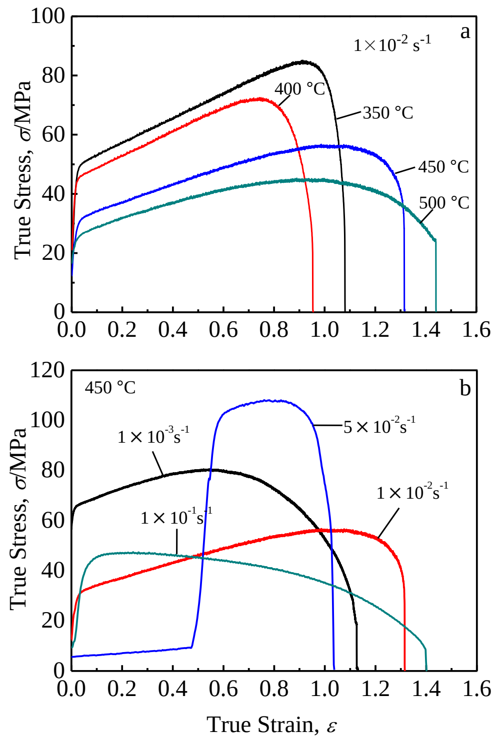

Fig.3 True stress-true strain curves of composite under different conditions: (a) $\dot{\varepsilon}=1\times10^{-2}\ s^{-1}$ and (b) T=450 ℃

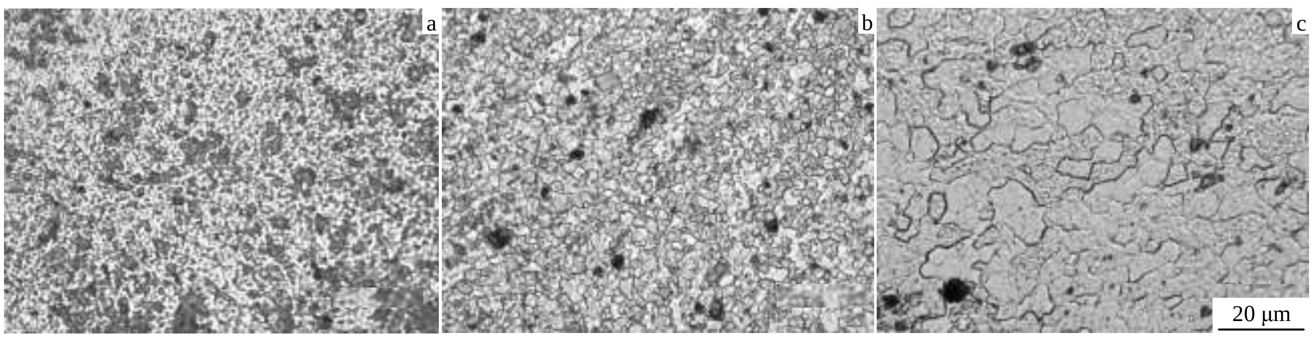

Fig.4 Microscopic images contrast of matrix grains with high temperature tension: (a) FSP before high temperature tensile experiment; (b) the drawing parts after high temperature tensile experiment; (c) the blessing part after high temperature tensile experiment

holes are induced. With the degree of deformation deepening, these holes are connected each other into a whole body and lead to fracture of materials eventually.

Fig.5 is SEM images for fracture of high temperature tensile when elongation is 182% and 330%. It shows the two kinds of fracture with different elongation are composed of pit groups and a small amount of tear ridge. Moreover, fine and uniform-sized granular bumps are distributed in a pit group. The reason is that these bumps are grains generated by recrystallization at high temperature resulting in that the grain boundary between grains is not solid enough where the region is first to disconnect because of the low intensity. Some other phenomena can be observed that the density of pits with low elongation is small, the quantity of bumps is less and the tear ridge is long. On the contrary the density of pits with high elongation is large, the quantity of bumps is large and the tear ridge is short. It is obvious from these phenomena that material plastic ability is higher in the high temperature tension under the circumstances that the formation of recrystallization grain is easy and the dynamic softening ability is strong to cause high elongation.

Composites processed by forging and FSP were analyzed by TEM to study the cause of the superplasticity further. Fig.6 shows that dislocation is regular and unidirectional in forging issue but is staggered and unordered in the friction stir issue. The contrast illustrates the FSP increases the dislocation density and complicates the dislocation relationships, which can illustrate the existence of superplasticity depends on the followings: grain boundary sliding and grain rotation will cause stress concentration at the grain boundary during superplastic deformation and dislocations pile up at grain boundary, providing conditions for the recrystallization nucleation which can increase the number of high-angle grain boundaries, and thus the material exhibits superplasticity.

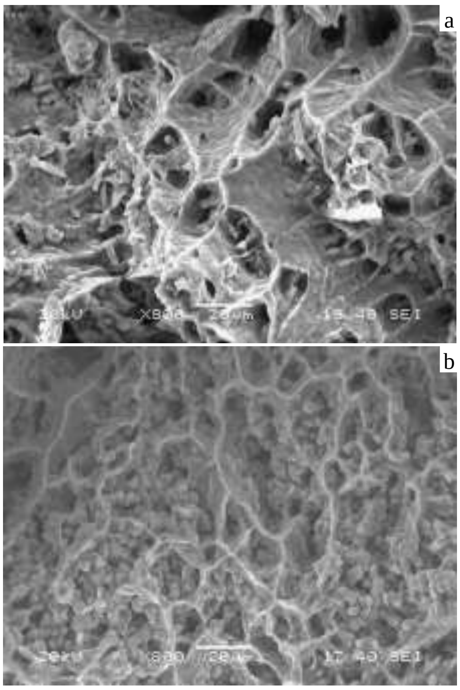

Fig.5 SEM images of high temperature tensile fracture with extension rate of 182% (a) and 330% (b)

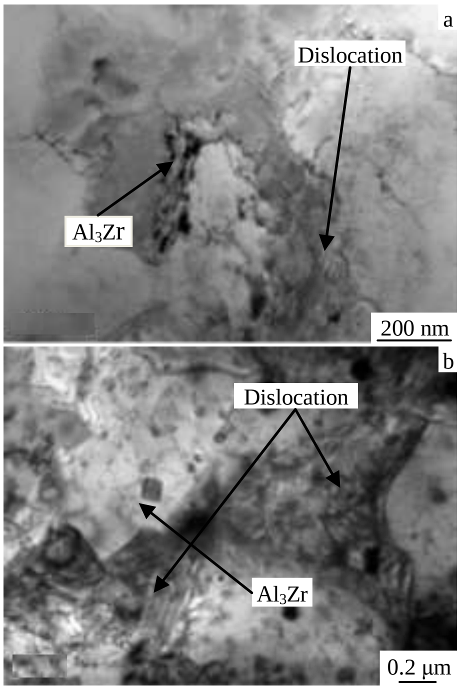

Fig.6 Dislocation of forged (a) and friction stirring state (b) of Al_3Zr/6063Al composites

2.4 Strain rate sensitivity index (*m*) and activation energy (*Q*)

Fig.7 is line chart of strain rate sensitivity index (m) of Al_3Zr/ 6063Al composites at different temperatures and initial strain rates. The chart shows the value of m decreases firstly and then increases as the forming temperature changes from 350~500 ℃ at the initial strain rate of 1×10^{-2} s^{-1}. On the contrary the value of m increases firstly and then decreases as the initial strain rate changes from 1.0×10^{-3} s^{-1} to 1×10^{-1} s^{-1} at the deformation temperature of 450 ℃ and reaches maximum of 0.49 eventually. The change of m above with temperature and initial strain rate approximates with the change of the elongation. The tensile elongation of composites is bigger in the range of deformation temperature and strain rate with high strain rate sensitivity index.

According to the thermal activation energy (Q) formula:

$$Q = nR\frac{\partial \ln \sigma}{\partial(1/T)} \tag{1}$$

where, n=1/m. We can calculate the value of Q=116~202 kJ/mol at the initial strain rate of 1×10^{-2} s^{-1} and the stretching temperature of 350~500 ℃, which is greater than lattice diffusion activation energy of the pure aluminum 142 kJ/mol.

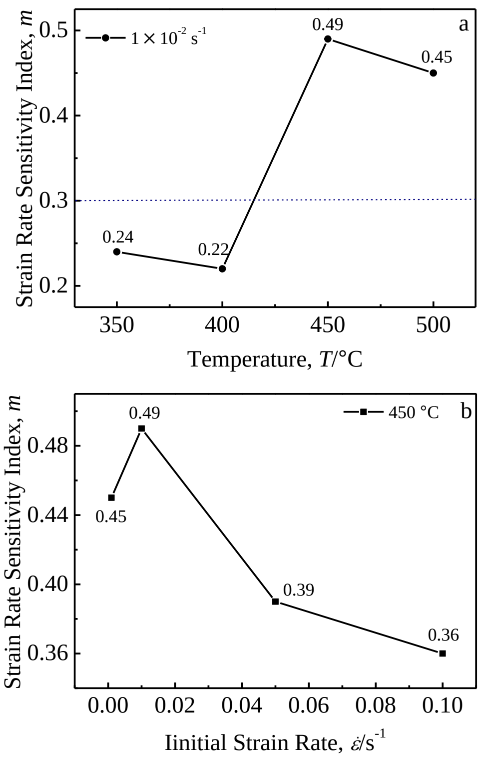

Fig.7 Line chart of the strain rate sensitivity index (m) at different temperatures (a) and initial strain rates (b)

This can be explained by the pinning effect, generated by the reinforcement Al_3Zr in composites, which hinder dislocation motion and increase deformation activation energy. So the dominant mechanism of superplastic deformation is grain boundary sliding[16]. Furthermore, Fig.7 shows that grains grow up only in the holding part during superplastic processes and dynamic recrystallization occurs near the fracture position. Dynamic recrystallization mechanism is the important auxiliary mechanism in superplastic deformation[17]. It can refine grains and improve superplastic property especially for metallic materials with coarse grains at a certain temperature and appropriate deformation rate.

3 Conclusions

1) The 5wt%Al_3Zr/6063Al composites exhibit superplasticity after forging and FSP at the temperature of 350~500 ℃ and initial strain rate of 1×10^{-3}~1×10^{-1} s^{-1}. The elongation reaches maximum 330% and the sensitive index (m) is 0.45 at temperature of 500 ℃ and the initial strain rate of 1×10^{-2} s^{-1}.

2) There are some kinds of phenomena within composites during superplastic deformation such as grain growth, dynamic recrystallization, grain boundary sliding and dislocation.

3) The dominant mechanism of superplastic deformation for Al_3Zr/6063Al composites is grain boundary sliding and dislocation slips, which is synergized by a moderate grain growth and dynamic continuous recrystallization.

References

1 Xu Xiaojing, Chen Minkang, Dai Feng *et al*. *Acta Metallurgica Sinica*[J], 2002, 38(5): 544 (in Chinese)

2 Lin Bin, Ding Weimin. *Transactions of Nonferrous Metals Society of China*[J] , 1996, 6(2): 129

3 Tang Cairong, Li Miaoquan, Li Zelin *et al*. *Materials Engineering*[J], 1996(3): 17

4 Wei Zheng, Zhang Baoliang *et al*. *Acta Materiae Compositae Sinica*[J], 1992(3): 27

5 Xu Xiaojing, Zhang Di, Shi Zhongliang *et al*. *Transactions of Nonferrous Metals Society of China*[J], 1999, 9(2): 231

6 Azushima A, Kopp R, Yang D. *CIRP Annals-Manufacturing Technology*[J], 2008, 57(3): 716

7 Guo Wei, Wang Qudong. *Forging & Stamping Technology*[J], 2010, 35(1): 4

8 Ren Shurong, Ma Zongyi, Chen Liqing. *Materials Reviews*[J], 2007, 21(1): 86 (in Chinese)

9 Mishra R S, Mahoney M W, McFadden S X. *Scr Mater*[J], 1999, 42(2): 163

10 Jiao Lei, Zhao Yutao, Wang Xiaolu. *Rare Metal Materials and Engineering*[J], 2014, 43(7): 1769 (in Chinese)

11 Ma Z Y, Mishra R S, Mahoney M W. *Materials Science and Engineering*[J], 2003 (A351): 148

12 Ma Z Y, Mishra R S. *Scripta Materialia*[J], 2005, 53(1): 75

13 Liu F C, Ma Z Y. *Scripta Materialia*[J], 2008, 58(8): 667

14 Ma Z Y, Liu F C, Mishra R S. *Acta Materialia*[J], 2010, 58(14): 4693

15 Huang Chunping, Ke Liming, Xing Li *et al*. *Rare Metal Materials and Engineering*[J], 2011, 40(1): 183 (in Chinese)

16 Ma Z Y. *Friction Stir Processing Technology: A Review*[J], 2014, 39(3): 1

17 Jian Jinxin, Zheng Linbin, Zhang Kun *et al*. *Journal of Aeronautical Materials*[J], 2011, 31(3): 35

原位生成 Al_3Zr/6063Al 复合材料的高应变速率超塑性

焦　雷[1]，王晓璐[1]，李　惠[2]，赵玉涛[1]，杨永刚[1]，陈建超[1]

(1. 江苏大学，江苏 镇江 212013)

(2. 江苏科技大学，江苏 镇江 212003)

摘　要：采用熔体直接反应法，原位制备 5%Al_3Zr/6063Al 质量分数复合材料。在 450 ℃进行 70%变形量锻造预处理，然后进行搅拌摩擦大塑性加工，通过 XRD、SEM、EDS、超景深及 TEM 等分析测试方法研究其高应变速率超塑性。结果表明，通过锻造和搅拌摩擦加工处理后，复合材料的平均晶粒尺寸小于 10 μm。在 350~500 ℃，初始应变速率为 1.0×10^{-3}~1.0×10^{-1} s^{-1} 范围内，复合材料均呈现超塑性。在 500 ℃，初始应变速率为 1.0×10^{-2} s^{-1}，延伸率达到最大值 330%，反应敏感指数 m 值为 0.45。分析超塑性变形的主要机制是动态连续再结晶与晶界、位错滑移共同协调完成。

关键词：原位反应；搅拌摩擦加工；超塑性；高应变速率；变形机理

作者简介：焦　雷，男，1979 年生，博士，副教授，江苏大学材料科学与工程学院，江苏 镇江 212013，电话：0511-88790769，E-mail: jiaolei@sina.com