Available online at www.sciencedirect.com
ScienceDirect
Trans. Nonferrous Met. Soc. China 22(2012) 2080−2086
Transactions of Nonferrous Metals Society of China
www.tnmsc.cn

Effect of heat treatment on microstructure and mechanical property of extruded 7090/SiC$_p$ composite

YUAN Wu-hua, AN Bai-ling

College of Materials Science and Engineering, Hunan University, Changsha 410082, China

Received 9 September 2011; accepted 16 January 2012

Abstract: The ultra high strength SiC particles (SiC$_p$) reinforced Al−10%Zn−3.6%Mg−1.8%Cu−0.36%Zr−0.15% Ni composite was prepared by spray co-deposition. Microstructures of the extruded and different heat-treated bars were analyzed by transmission electron microscopy (TEM) and energy dispersive spectrometry (EDS). Grain size of the composites prepared by two-stage solution is smaller than that by single-stage solution. After single-stage solution aging treatment, fine precipitates of both η and AlZnMgCu-rich phase can be found both intragranularly and intergranularly. While after the two-stage solution, an amorphous Si−Cu−Al−O (5 nm) layer appears at the interface. The addition of Ni and Zr modified the influence of the two-stage solution and inhibited the growth of the 7090/SiC$_p$ composite grain size. Heat treatments can significantly improve the fracture toughness of the composite. The fracture toughness first decreases then increases with the elongation of the aging time.

Key words: Al matrix composite; spray co-deposition; heat treatment; solid solution; aging; microstructure; interface; fracture toughness

1 Introduction

Metal matrix composites are widely used because of their high specific strength, low density, good hot workability, low thermal expansion coefficient and good dimensional stability [1]. The 7090/SiC$_p$ composites were widely used in the area of aerospace, military, car and electronic instrument. The SiC reinforcement can increase the elastic modulus and yield stress but decrease the ductility and toughness of the composites [2].

SiC reinforced Al composites can be fabricated in these different ways, such as ordinary metal metallurgy, mechanical alloying, powder metallurgy and spray co-deposition, friction stir processing (FSP) [3]. In this work, the 7090/SiC$_p$ composites were prepared by spray co-deposition, and then extruded and heat treated. Many researches have been dedicated to the influences of SiC particles over the microstructures and properties of composites. The incorporation of SiC reinforcement into aluminum matrix can enhance the plastic flow particulate (PFS) in macro-plastic stage while slightly decrease PFS in micro-plastic stage [4]. High residual thermal misfit stress and dislocation density in the matrix are mainly ascribed to the large difference in thermal expansion coefficient between the SiC and matrix [5]. Fracture toughness of the composites decreases with the increment in volume fraction of SiC particles [6]. Heat treatment is a primary process for aluminum alloy, during which the soluble second phase can be dissolved into the matrix. Moreover, the second phase can re-dissolve more adequately at a higher solution temperature. The application of aging treatment before the diffusion bonding of Al/SiC$_p$ composite to pure Al, increased Cu concentration at the interface which treats as the insert alloy [7]. The alloys subjected to promotively-solutionizing treatment always can exhibit increment in the yield strength and fracture toughness because of the remelt [8,9]. However, the evolvement of the microstructures and precipitation during solution and aging treatment in the aluminum matrix composites has been rarely reported. This work is a deep investigation on the basis of the previous one [10]. In our work, microstructures and precipitation evolvement in the extruded 7090/SiC$_p$ composite after different heat treatments were investigated. Effect of different heat treatments on fracture toughness and mechanical properties was analyzed.

Foundation item: Project (02Gky2004) supported by Hunan Provincial Science and Technology Department, China
Corresponding author: AN Bai-ling; Tel: +86-13786152493; E-mail: jingtingshan11@163.com
DOI: 10.1016/S1003-6326(11)61431-3

2 Experimental

The aluminum matrix composite billets reinforced with SiC particles with the diameter of 163 mm were produced by spray co-deposition. Nominal composition of the preform (mass fraction) was Al−10%Zn−3.6%Mg−1.8%Cu−0.36%Zr−0.15%Ni. Volume fraction and average size of the SiC particle were 20% and 5 μm, respectively. After homogenization, the deposited billet was then extruded to 18 mm at 420 °C with the extrusion coefficient of 20.0. The solution temperature was chosen preliminarily depending on differential scanning calorimetry (DSC), hardness measurement and microstructure analysis. Aging hardening-curve was obtained by hardness measurement of different duration between 0 and 36 h at 120 °C, as recorded in Ref. [10]. All the heat treatment technologies are listed in Table 1. The mechanical properties of the samples (standard samples with 10 mm in diameter) in different states, such as the single-stage solution +peak aging and the two-step solution + peak aging, were tested on a Instron 8032 tensile machine.

Table 1 Heat treatment technologies

Sample No.	Solution treatment
1	Hot extrusion
2	(490 °C, 1 h)
3	(470 °C, 1 h)+(490 °C, 1 h)
4	(490 °C, 1 h)+(120 °C, 28 h)
5	(470 °C, 1 h)+(490 °C, 1 h)+(120 °C, 24 h)
6	(470 °C, 1 h)+(490 °C, 1 h)+(120 °C, 28 h)
7	(470 °C, 1 h)+(490 °C, 1 h)+(120 °C, 32 h)

Foils for TEM observation were prepared by twin-jet polishing with an electrolyte solution consisting of 30% HNO_3 and 70% methanol (volume fraction) below −20 °C. The foils were examined on a JEM−3010 electron microscope. Grain size was obtained from the TEM image of the composite experiencing different heat treatments.

For the fracture toughness test, the specimens with dimensions of 60 mm × 10 mm × 5 mm obtained by wire-electrode cutting were selected parallel to the jet axis from the extruded bar (*d*18 mm). Seven different kinds of heat treatments shown in Table 1 were carried out to investigate the effect of solution aging treatment on the fracture toughness. Every specimen was embedded a 3 mm notch, and then introduced a fatigue precrack with the size 1.5−2.0 mm on the Instron 8032 fatigue test machine. Three-point bending tests were then carried out on the Instron 8032. The fatigue test for each heat treatment state was repeated 3 times to acquire an accurate result.

3 Results and discussion

3.1 Microstructure of 7090/SiC$_p$ composites after solution treatment

TEM images of 7090/SiC$_p$ composites after solution treatment at 490 °C for 1 h are shown in Fig. 1. Compared with microstructures of the extruded composites without heat treatment in the previous work [10], most of the second phase particles are dissolved into the matrix, but some finer particles still exist both intragranularly and intergranularly after single-stage solution treatment (Technology 1).

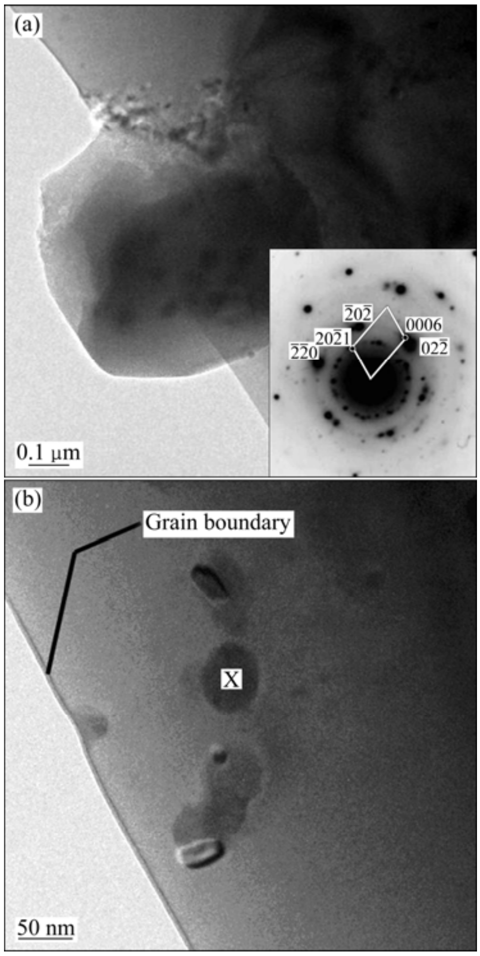

Fig. 1 TEM images of 7090/SiC$_p$ composites after being solution treated at 490 °C for 1 h: (a) Particle existing intergranularly and its corresponding electron diffraction spot patterns; (b) Precipitates existing intragranularly

The electron diffraction spot patterns (as shown in Fig. 1(a)) show that some bright and sharp η phase can be found, so η phase is one of the strengthening phases along the grain boundary. Some finer particles exist both intragranularly and intergranularly, while more precipitates are observed intergranularly, a few intragranular precipitates can be observed. EDS analysis to the oval precipitates ("X" in Fig. 1(b)) is shown in Fig. 2. It can be obviously seen that most of the phases revealed in the system is α(Al) matrix, meanwhile a few Cu-rich precipitates still exist. As a result of the solution treatment, most of the $MgZn_2$ phase dissolves into the α(Al) matrix, thus little $MgZn_2$ can be observed [11]. According to their shape and EDS result, the second phase particles undissolved are determined to be AlZnMgCu-rich precipitates which can decrease fracture toughness of the alloy [12].

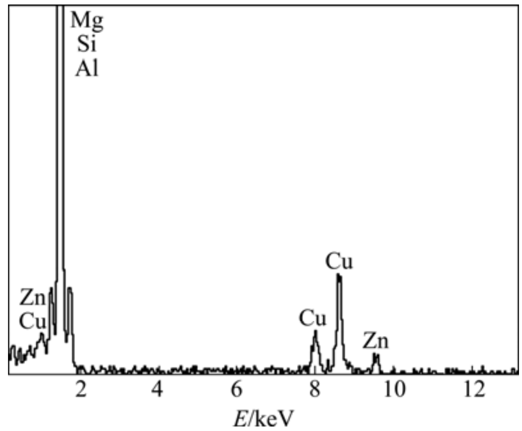

Fig. 2 EDS analysis of precipitates (shown as "X" in Fig. 1(b)) after single-stage solution treatment

3.2 Microstructure of 7090/SiC_p composites after solution aging treatment

TEM images of the extruded 7090/SiC_p composites after single-stage solution and peak aging treatment (Technology 2) are shown in Fig. 3. It can be seen that after single-stage solution + aging treatment, the grain size of the matrix is 1.5−2.0 μm (Fig. 3(a)). Grain boundaries of the matrix are clear, moreover most of the precipitates distribute intergranularly (as shown in Figs. 3(a), (b)), while some fine precipitates also appear intragranularly (as shown in Fig. 3(b)). Alloying element precipitated gradually from the oversaturated matrix during the process of aging treatment (as shown in Figs. 3(a), (b)). Main chemical composition of the composite matrix is Al−10%Zn−3.6%Mg−1.8%Cu−0.36%Zr−0.15%Ni (in mass fraction). So according to the existing literature, we got the conclusion that the strengthening phases in the matrix are mainly lots of η($MgZn_2$) phase and some Cu-rich phase [13].

Microstructures of the composite after two-stage solution and aging treatment (Technology 3) are shown in Fig. 4. Only a few precipitates with the size of 20 nm dispersively distribute in the grains, more of them appear along the grain boundaries with the size of 1.0−1.5 μm (as shown in Figs. 4(a), (b)). The grain size of the

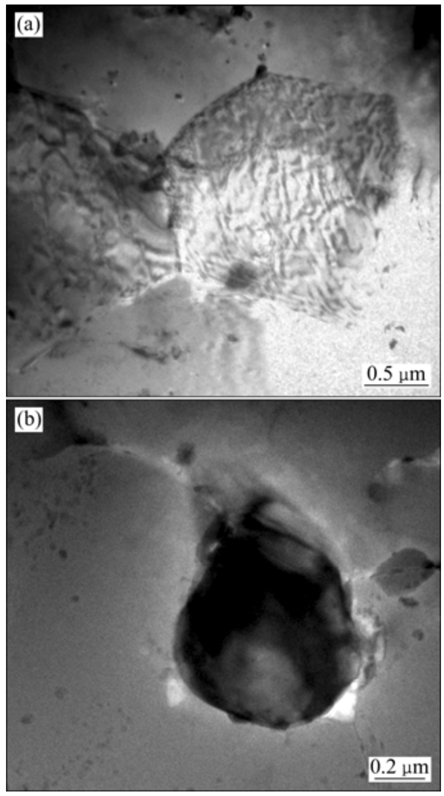

Fig. 3 TEM images of 7090/SiC_p composite after single solution and peaking: (a) Particles appearing intergranularly; (b) Particles appearing both intergranularly and intragranularly

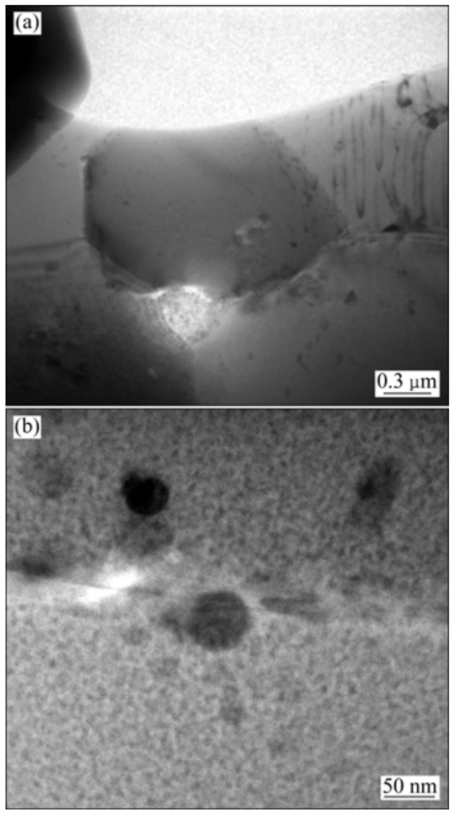

Fig. 4 TEM images of 7090/SiC_p composites after two-stage solution and aging treatment: (a) Overview; (b) Precipitates along grain boundary

matrix is 1.0−1.5 μm, which is much smaller than that of the composite subject to single-stage solution treatment. Moreover, as a result of the addition of alloying element Ni, Zr, Mn, the main strengthening phase also contains $\eta(MgZn_2)$ and $T(Al2Mg_3Zn_3)$ [14,15].

It can be seen that microstructures of the two different heat treated composites by technologies 2 and 3 were different, which mainly due to the melting of the second phase and the addition of microalloying element Ni, Zr that can inhibit the growth of grain [16,17]. Constituents of the eutectic phase in the high-Zn-containing ultra high strength aluminum alloy are complex. As a result of the fact that individual solution rate of different crystalline phases is discrepant, the eutectic phases were dissolved in a certain sequence during the solution treatment. Generally speaking, the Mg/Zn-rich phase has priority to be dissolved, so the $\eta(MgZn_2)$ phase was first dissolved, followed by the T(AlZnMgCu) phase, and then the $T(Al_2Mg_3Zn_3)$ was dissolved [18,19]. The eutectic temperature will be elevated after full solution of one of the eutectic phases. The fact that stepped solution could both avoid the eutectic melting and elevate solution temperature to realize the full solution was verified.

The occurrences of the recovery and recrystallization were drove by the high storage energy in cold-rolled deformation microstructure in the first-stage of the two-step solution treatment. The processes of recovery and recrystallization are competitive in the first-stage solution treatment. When the recrystallization occurs, no more recovery will go on. On the other hand, as lots of deformation storage energy was consumed, the happening of recovery will decrease the driving force of recrystallization. Meanwhile, the low melting point eutectic will first dissolve into the matrix, other higher melting point eutectic will be left in the first-stage and dissolved into the matrix in the second-stage without over-sintering. For the reasons given above, the happening of recrystallization in the second-stage of the solution treatment will be well inhibited as mass deformation storage energy was used in the first stage. The low temperature solution in this work mainly aims to make the Zr and Ni atoms distribute uniformly and finely along the grain boundaries, which can well restrain the recrystallization and growth of the grains in the subsequent second-stage [20]. While in the second-stage, the higher temperature solution can make the residual phase re-dissolve into the matrix, thus the solubility limit was extended. Meanwhile, the aging strength was intensified [21]. Comparatively, during the process of single-solution treatment, Zr and Ni atoms have insufficient time to diffuse uniformly, so it has a weaker effect on the growth of grains [21,22].

3.3 Interface combination condition of 7090/SiC$_p$ composites after aging treatment

There is about 5 nm non-crystalline layer that can be seen at the interface between SiC particles and the matrix (as shown in Fig. 5) in the composite subject to the two-stage solution and aging treatment. The components of the non-crystalline layer which can affect the sticking tendency of the interface are Si, Cu, Al, O (as shown in Fig. 6). However, researches on the interfacial oxide of sprayed depositions are less reported in the existing literature [21].

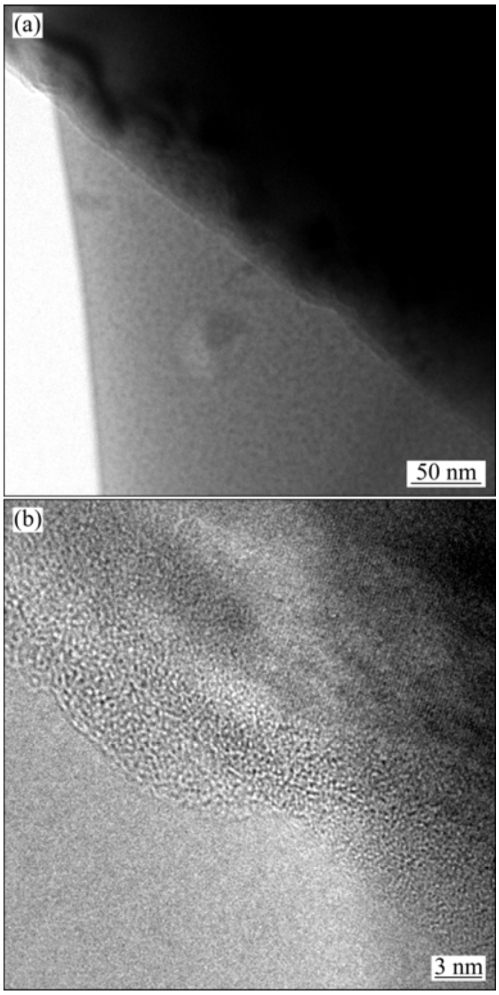

Fig. 5 TEM images of interface between SiC particle and matrix: (a) Overview; (b) Non-crystalline layer

3.4 Mechanical properties of 7090/SiC$_p$ composites after aging treatment

Heat treatment has great influence on the yield stress, stress concentration, strain concentration and fracture toughness of the composites. It can be seen that heat treatment can significantly improve the fracture toughness of the composites from Table 2. There is no significant difference in fracture toughness between the composites experiencing the two different solution

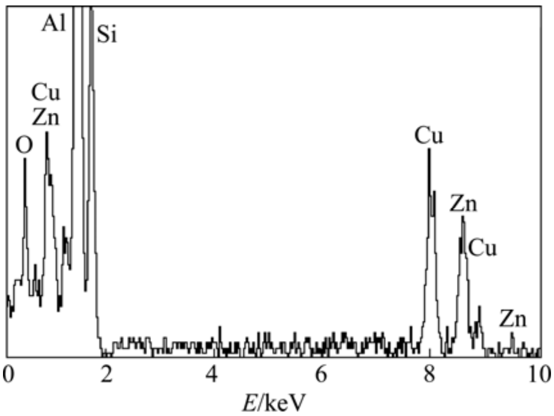

Fig. 6 EDS analysis of interface oxide layer

Table 2 Fracture toughness of 7090/SiC_p composites

Sample No.	K_{IC}/(MPa·m$^{1/2}$)	σ_b/MPa	$\sigma_{0.2}$/MPa	δ/%
1	20.56	525	500	−
2	25.01	−	−	−
3	24.69	−	−	−
4	33.92	755	725	1.5
5	39.49	761	−	1.2
6	34.28	785	−	1.4
7	38.16	753	728	1.1

treatments ((490 °C, 1 h) and (470 °C, 1 h)+(490 °C, 1 h), see Table 2). Moreover, significant increment in the fracture toughness of the composites can be seen in the specimens exposed to aging treatments (10−15 MPa·m$^{1/2}$). The fracture toughness first decreases then increases with the elongation of the aging time.

Solution aging treatment can effectively improve the tensile strength of the Al/SiC_p composites. The strength of the composites first increased then decreased with the extending of aging time. The aging peak was observed when the composite was solution and aging treated at (470 °C, 1 h)+(490 °C, 1 h)+(120 °C, 28 h). It was reported that the evolution of the fracture toughness during aging is almost inversely proportional to the evolution of the yield stress [6]. A similar conclusion was gotten in this work.

4 Discussion

From Fig. 7, it can be seen that there is no significant fracture morphology difference among the composites after different aging treatments. More intergranular fracture was observed on the fracture surface than the intragranular ones, only a few complete particles can be seen on the fracture surface. The fracture mode is concluded to be brittle rupture from the dimples and shearing slices exposed on the fracture surface (as shown in Figs. 7(b−d)).

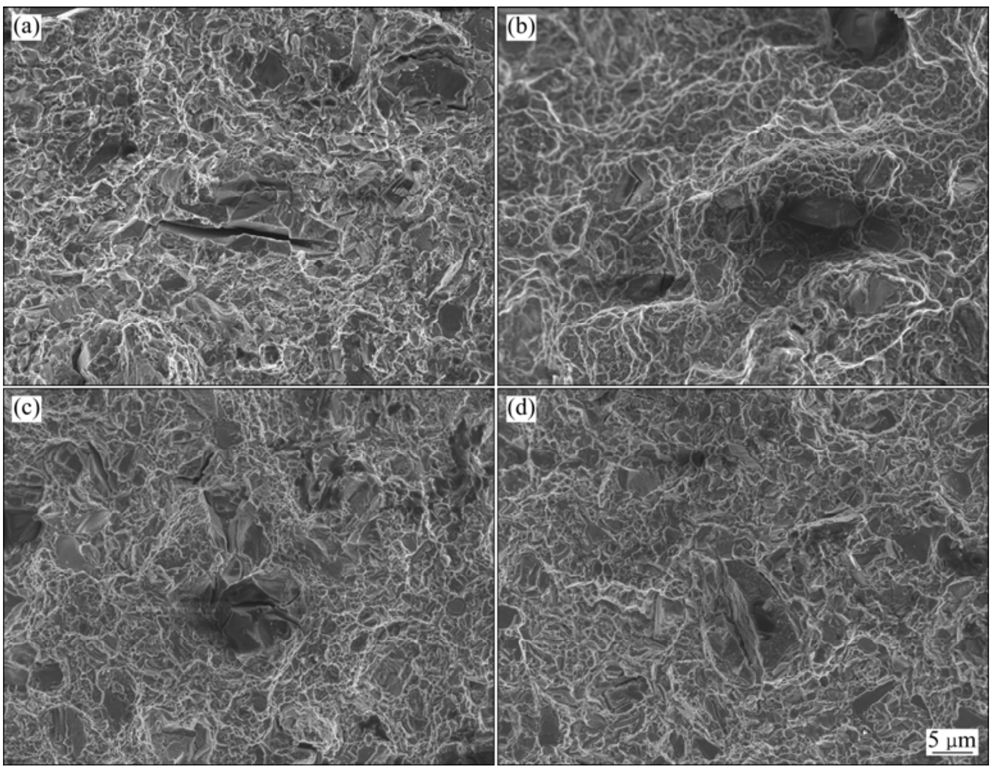

Fig. 7 TEM images showing fractography of 7090/SiC_p composites: (a) As-extruded; (b) Treated under (470 °C, 1 h)+(490 °C, 1 h)+(120 °C, 24 h); (c) Treated under (470 °C, 1 h)+(490 °C, 1 h)+(120 °C, 28 h); (d) Treated under (470 °C, 1 h)+(490 °C, 1 h)+(120 °C, 32 h)

Some fracture particles can be seen in the dimples. In the case of the high strength of the matrix, the brittle particle reinforcement has a great fracturing tendency as the load on particle increases with the increment of matrix strength. The presence of flaws in SiC particles improves their fracture possibility when all of them subjected to stress. Meanwhile, the smaller particles can be drawn from the matrix when the tensile stress acting on them exceeds a critical value. Figure 7 shows that the SiC particles are hardly drawn from the matrix, even with size of about 1 μm. As a result, it can be inferred that bond strength at the interface between SiC_p and matrix is high, in other words, the Si−Cu−Al−O non-crystalline layer plays a positive role on anti-fracture. The SiO_2 layer between the SiC reinforcement and the matrix was reported in solid powder technology. Although rare, some interfacial debonding still can be observed. Thus, the bond strength of the layer is high but still lower than the strength of the matrix. The Si−Cu−Al−O was inferred to be formed in the progress of spray co-deposition because of its high degree of supercooling in the process.

The number of the precipitates increases gradually in the prescription process, some even coarsen, which greatly improves the breaking strength of the composites. Because a major fraction of dislocation was annihilated at the interface, the strength of the composite was improved. On the other hand, more exsolution nucleation position was supplied as a result of the high dislocation density and grain boundary defects. Then, the strength was improved just because much more and finer precipitates had been generated, and the toughness of the composite was also improved resulting from the softening effect of the coarse precipitation.

5 Conclusions

1) After single solution treatment, most of the second phase particles are dissolved into the matrix, but there are still some finer particles, both intragranular and intergranular, most of which are AlZnMgCu phase.

2) Some Cu-rich phases precipitate while the main precipitates are $\eta(MgZn_2)$ after solution aging treatment.

3) After two-stage solution aging treatment, intragranular precipitates become fewer and more dispersive, while more precipitates appear intergranularly. The size of all the precipitates is much smaller than that experiencing single-stage solution treatment. As a result of the addition of Ni, Zr, Mn, the main strengthening phase also contains $\eta(MgZn_2)$ and $T(Al_2Mg_3Zn_3)$. After two-stage solution and aging treatment, about 5 nm non-crystalline layer with the chemical composition of Si, Cu, Al, O appears at the interface between SiC particles and the matrix.

4) The solution aging treatment can significantly improve the tensile strength and fracture toughness of the composite. However, the evolution of the fracture toughness with the elongation of aging time is almost inversely proportional to that of the yield stress.

References

[1] FAN Jian-zhong, SANG Ji-mei, SHI Li-kai. The fabrication, application and development of particle-reinforced aluminum matrix Composites [J]. The Chinese Journal of Materials Review, 2001, 15(10): 55−57. (in Chinese)

[2] MILLER W S, HUMPHREYS F J. Strengthening mechanisms in particulate metal matrix composite [J]. Scripta Metallurgical Materialia, 1991, 25: 33−38.

[3] WANG Wei, SHI Qing-yu, LIU Peng, LI Hong-ke, LI Ting. A novel way to produce bulk SiC_p reinforced aluminum metal matrix composites by friction stir processing [J]. Journal of Materials Processing Technology, 2009, 209: 2099−2103.

[4] ZHANG Fan, SUN Peng-fei, LI Xiao-cui, ZHANG Guo-ding. An experimental study on deformation behavior below 0.2% offset yield stress in some SiC_p:Al composites and their unreinforced matrix alloys [J]. Materials Science and Engineering A, 2001, 300: 12−21.

[5] LORENTZEN T, CLARKE A P. Thermomechanically induced residual strains in Al/SiC_p metal-matrix composites [J]. Composites Science and Technology, 1998, 58: 345−353.

[6] SONG Min, HUANG Bai-yun. Effects of particle size on the fracture toughness of SiC_p/Al alloy metal matrix composites [J]. Materials Science and Engineering A, 2008, 488: 601−607.

[7] MURATOGLU M, YILMAZ O, AKSOY M. Investigation on diffusion bonding characteristics of aluminum metal matrix composites (Al/SiC_p) with pure aluminum for different heat treatments [J]. Journal of Materials Processing Technology, 2006, 178: 211−217.

[8] WU Y L, FROES F H, ALVAREZ A, LI C G, LIU J. Microstructure and properties of a new super-high-strength Al–Zn–Mg–Cu alloy C912 [J]. Materials & Design, 1997, 18: 211−215.

[9] LI Zhi-hui, XIONG Bai-qing, ZHANG Yong-an, ZHU Bao-hong, WANG Feng, LIU Hong-wei. Investigation on strength, toughness and microstructure of an Al−Zn−Mg−Cu alloy pre-stretched thick plates in various ageing tempers [J]. Journal of Materials Processing Technology, 2008, 209: 2021−2027.

[10] YUAN Wu-hua, ZHANG Jian, ZHANG Chen-chen, CHEN Zhen-hua. Processing of ultra-high strength SiC_p/Al−Zn−Mg−Cu composites [J]. Journal of Materials Processing Technology, 2009, 209: 3251−3255.

[11] LI Hai, ZHENG Zi-qiao, WANG Zhi-xiu. Investigation of secondary ageing characteristics of 7055 aluminum alloy—(Ⅱ) Microstructures and fractography [J]. The Chinese Journal of Rare Metal Materials and Engineering, 2005, 34(8): 1230−1234. (in Chinese)

[12] EVANS R D, BOYD J D. Near-interface microstructure in a SiC/Al composite [J]. Scripta Materialia, 2003, 49: 59−63.

[13] HAO Guang-rui, WANG Hong-bin, HUANG Jin-feng, YANG Bin, ZHANG Yong-an, XIONG Bai-qing, ZHANG Ji-shan. Effect of heat treatment on microstructure and mechanical properties of spray deposited Al−Zn−Mg−Cu aluminum alloy [J]. The Chinese Journal of University of Science and Technology Beijing, 2004, 26(5): 502−506. (in Chinese)

[14] LI Hai, ZHENG Zi-qiao, WANG Zhi-xiu. Investigation of secondary ageing characteristics of 7055 aluminum alloy—I. Mechanical properties [J]. The Chinese Journal of Rare Metal Materials and Engineering, 2005, 34(7): 1029−1032. (in Chinese)

[15] WANG Hong-bin, LIU Hui-min, HUANG Jin-feng, ZHANG Ji-shan, ZHANG Yong-an, XIONG Bai-qing. Effects of heat-treatment on microstructure and mechanical properties of ultra-high strength spray deposited Al−Zn−Mg−Cu aluminum alloy [J]. The Chinese Journal of Nonferrous Metals, 2004, 14(3): 398−404. (in Chinese)

[16] MORERE B, SHAHANI R, MAURICE C, DRIVER J. The influence of Al3Zr dispersoids on the recrystallization of hot-deformed AA7010 alloys [J]. Metallurgical and Materials Transactions A, 2001, 32: 625−632.

[17] WANG S H, UAN J Y, LUI T S, CHEN L H. Examination on the aging and tensile properties of Al−Zn−Mg/Al3Ni eutectic composite[J]. Metallurgical and Materials Transactions A, 2002, 33: 707−711.

[18] LUDTKA G M, LAUGHLIN D E. The influence of microstructure and strength on the fracture mode and toughness of 7xxx series aluminum alloys [J]. Metallurgical Transactions A, 1982, 13(3): 411−418.

[19] ZHANG Kun, LIU Zhi-yi, YE Cheng-wu, XU Xiao-chang, ZHENG Qing-chun. Study on mechanical property of a new super-high-strength aluminum alloy [J]. Rare Metals and Cemented Carbides, 2004, 32: 11−14.

[20] ZHANG Xin-ming, HUANG Zhen-bao, LIU Sheng-dan, LIU Wen-hui, ZHANG Chong, DU Yu-xuan. Effects of two-stage solution on microstructures and mechanical properties of 7A55 aluminum alloy [J]. The Chinese Journal of Nonferrous Metals, 2006, 16(9): 1527−1533. (in Chinese)

[21] GENG Lin, YAO Zhong-kai, LI Yi-chun. Microstructure and interface of SiC_p/Al composites [J]. The Chinese Journal of High-tech Communication, 1994(4): 17−20. (in Chinese)

[22] SRITHARANA T, CHANA L S, TANA L K, HUNG N P. A feature of the reaction between Al and SiC particles in an MMC [J]. Materials Characterization, 2001, 47: 75−77.

热处理对挤压态 7090/SiC_p 复合材料组织和力学性能的影响

袁武华，安佰玲

湖南大学 材料科学与工程学院，长沙 410082

摘　要：采用喷射沉积的方法制备 SiC_p 增强超高强 Al−10%Zn−3.6%Mg−1.8%Cu−0.36%Zr−0.15%Ni 复合材料。使用透射电镜(TEM)和能谱仪(EDS)对挤压并热处理过的合金棒进行微观组织取样观察。双级时效后的复合材料的晶粒比单级时效合金的晶粒更小。经单级时效处理，在晶界上和晶粒内部都可以观察到细小的 η 相和富 AlZnMgCu 相的析出颗粒。双级时效后在 SiC 颗粒与基体的界面上发现了一层 5 nm 厚的 Si−Cu−Al−O 非晶层。Ni、Zr 的添加可以改善双级时效的效果，并且抑制 7090/SiC_p 复合材料晶粒的生长。固溶时效处理可以显著提高复合材料的断裂韧性，而断裂韧性随着时效时间的延长而先减小后增加。

关键词：铝基复合材料；喷射沉积；热处理；固溶处理；时效；微观组织；界面；断裂韧性

(Edited by YANG Hua)