Article ID: 1003-6326(2003)01-0135-05

Interface and microstructure characteristics of SiC_p/2024 aluminium alloy composite ①

LUO Shou-jing(罗守靖), JIANG Ju-fu(姜巨福), WANG Ying(王 迎),
TENG Dong-dong(滕东东), ZU Li-jun(祖丽君)
(School of Materials Science and Engineering, Harbin Institute of Technology, Harbin 150001, China)

Abstract: Electron microscope examination of the microstructure, interface and fracture surface of SiC particulate reinforced 2024 aluminium alloy composites produced by powder mixing and semi-solid extrusion process was presented. The microstructure of SiC_p/2024 composites fabricated by the present method is characterized by uniformly distributed SiC particulates in well-densified matrix. Conventional transmission electron microscopy(TEM) reveals the interface between the SiC particulates and the aluminium matrix. It is shown that this interface provides very strong bonding which is further evidenced by the fractographic results, and that there is no apparent chemical reaction. Examination of the fracture surface indicates that the bonding strength between the SiC particulates and the aluminium alloy matrix is stronger than that of the matrix. The dimples and tearing edges on the fracture surface of composites are obviously observed.

Key words: metal-matrix composites; interface; microstructure; semi-solid extrusion
CLC number: TG 146.3 **Document code:** A

1 INTRODUCTION

The importance of particulate reinforced metal matrix composites(PMMCs) due to high specific strength, high specific modulus, better resistance to wear and thermal steadiness has attracted much interest in the development of the manufacturing process for such composites. The forming processes of PMMCs have been mainly conventional casting, stirring casting in semi-solid and powder metallurgy technologies. The casting technique is very promising for manufacturing composite parts at relatively low cost. However, in casting methods, the reactants often appear at the interface between matrix and reinforcement, which deleteriously affects the mechanical properties of final composites[1, 2]. The technology of powder metallurgy can improve the mechanical properties of materials, but increase production cost because of complex working procedures[3]. Compared with the above methods, a powder mixing and semi-solid extrusion process has more advantages in fabricating SiC_p/2024 composites. In this composite there is strong bonding at the interface between the reinforcement and the matrix, and there are no cavities and deleterious reactants at the interface, which is of benefit to the mechanical properties.

The mechanical properties of the metal-matrix composites(MMC) are influenced strongly by the microstructures of the composite and in particular by the nature of the interface between the brittle reinforcing phase and the ductile matrix, as this interface controls the efficiency of the load transfer between the matrix and the reinforcement[4]. By revealing the characteristics of the interface of the composites, it may be possible to provide theoretical basis for the optimization of technological process. The interfacial reaction for an Al/SiC system is very complex. The nature of the resultant interface depends on a variety of factors such as:

1) the nature of the surface of the reinforcement[5-7];

2) the fabrication techniques; and

3) the thermal treatment applied to the composites[8-10].

Since the reaction at the interface is influenced by so many factors, there are many possible interface conditions in the Al/SiC system. In this paper, the microstructure and interface of SiC_p/2024 composites are studied by SEM and TEM.

2 EXPERIMENTAL

The material used for the present investigation was a SiC particulate reinforced 2024 aluminium alloy composite. The composite has reinforcement content of 10% or 20% by volume with an average particle size 14 μm. The powder of 2024 Al alloy used in this experiment was prepared by the argon supersonic atomization, whose chemi-

① **Foundation item:** Project(59675050) supported by the National Natural Science Foundation of China
Received date: 2002-04-04; **Accepted date:** 2002-06-11
Correspondence: Prof. LUO Shou-jing, School of Materials Science and Engineering, Harbin Institute of Technology, Harbin 150001, China; Tel: +86-451-6418723; E-mail: Luosj@hope.hit.edu.cn

cal composition is Al-4.48% Cu-1.45% Mg-0.61% Mn-0.31% Fe-0.15% Si (mass fraction). The composite was produced by a powder mixing and semi-solid extrusion process. The process comprises dry blending the aluminium powder and the SiC particulate material followed by cold compaction, degassing, sealing and semi-solid extrusion. The specific procedures and processing parameters of this technology were described in Refs. [11, 12].

The semi-solid extrusion experiments were carried out in a special hydraulic press, the reduction ratio was 10:1. The exact melting temperature region of the SiC_p/2024Al composites was determined using differential thermal analysis (DTA), as indicated in Fig. 1. It can be seen in Fig. 1 that the incipient melting point of the composites is 507.9 ℃. According to the DTA curve, the temperature of semi-solid extrusion was selected in the range of 550~590 ℃. Before semi-solid extrusion, the stocks were maintained in a furnace at the temperature of semi-solid extrusion for about 30 min, and the temperature difference was controlled in the range of ±2 ℃.

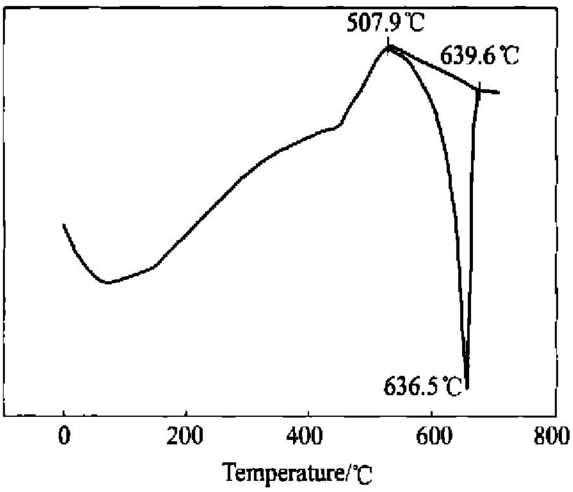

Fig. 1 Differential thermal analysis (DTA) curve of SiC_p/2024Al composites

Tensile tests were carried out on an Instron tensile testing machine. The microstructure and fracture surface of the composites were observed by JSM-T200 SEM. The TEM foils were prepared by a technique consisting of a series of wet grinding stages followed by ion beam thinning until perforation. The interface of composites was examined by Philips EM420 TEM.

3 RESULTS AND DISCUSSION

3.1 Mechanical properties

The mechanical properties of SiC_p/2024 composites fabricated by the powder mixing and semi-solid extrusion technology are shown in Table 1.

The elastic modulus, the ultimate tensile strength and the yield strength of the composites after T6 treatment are much higher than those of the 2024Al alloy after T6 treatment. However, the elongation of the composites decreases obviously, but still maintains at relatively high level. From Table 1, it can be seen that the modulus and the strength of the composite increase with the increase of the SiC volume fraction, the elongation of the composites decreases with the increase of the SiC volume fraction.

3.2 Microstructures of SiC_p/2024 composites

3.2.1 Distribution of SiC particulate

Figs. 2(a)~(d) show the microstructures of SiC_p/2024 composites with 10% or 20% volume fraction SiC (14 μm) after semi-solid extrusion (extrusion ratio of 10:1) for the two directions, perpendicular to and parallel to the extrusion direction, respectively. From Figs. 2(a) and (c) it can be observed that SiC particulates mainly exist at the interface of primary Al grains, and in sequent cold compaction and semi-solid extrusion procedure, the location of the SiC particulates is not changed. The enrichment zone and reorientation of the SiC particulates along the extrusion direction are shown in Figs. 2(b) and (d). Semi-solid extrusion, by reason of the motion of SiC particulates under the action of temperature gradient and flow stress, caused local SiC particulate enrichment[13].

Figs. 2(a)~(d) show that the fracture extent of the SiC particulates during semi-solid extrusion is very small. The argument for this result goes as follows. The extrusion stress of semi-solid extrusion is only 1/5-1/3 times as much as that of the fully solid extrusion. In addition, Higashi, et al[14, 15], using a TEM technique, have directly observed the solute segregation at the interfaces or grain boundaries and the preferential melting of these enriched grain boundaries. Also, the grain boundaries and reinforcement-matrix interfaces (i.e. microstructural heterogeneities) are expected to be preferential sites for these liquid phases. Therefore, there are liquid phases at the grain boundaries of the composites during semi-solid extrusion and the SiC particulates mainly exist at the interface of Al grains. It is easy to harmonize SiC particulates with deformation of the matrix and, consequently, the cracking extent of the SiC particulates is decreased during deformation.

3.2.2 Matrix microstructures

After semi-solid extrusion, it is difficult to distinguish the interface of primary Al grains, and there are no cavities in the aluminium matrix, as shown in Figs. 2(a)~(d). The results indicate that primary Al grains have been combined into the densified matrix and this is further evidenced by fractographic results. The matrix of the

Table 1 Mechanical properties of 2024Al alloy and SiC_p/2024 composites

Material	Fabrication process	$\sigma_{0.2}$/MPa	σ_b/MPa	E/GPa	δ/%
2024 matrix alloy	Hot extrusion + T6	320	480	70	16.8
10% SiC_p(14 μm)/2024	Semi-solid extrusion(10:1) + T6	398	543	88.7	7.8
20% SiC_p(14 μm)/2024	Semi-solid extrusion(10:1) + T6	440	560	105.9	3.8

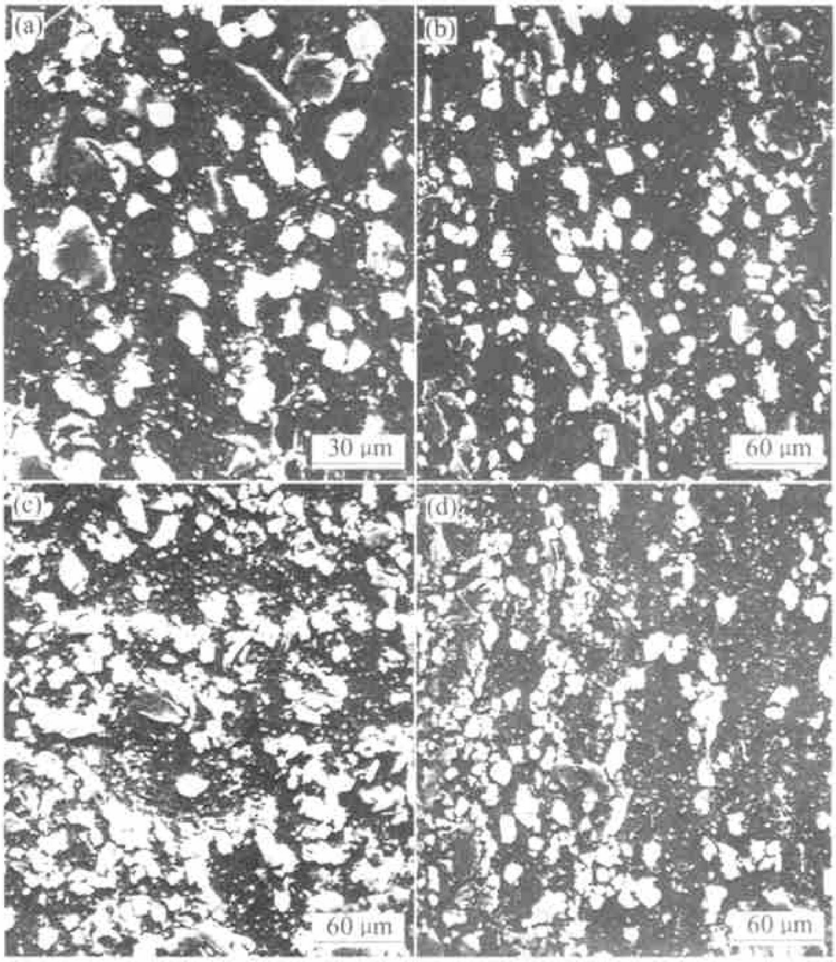

Fig. 2 Microstructures of SiC_p/2024 composites
(a) —10% SiC_p/2024 composite(Transverse); (b) —10% SiC_p/2024 composite(Longitudinal);
(c) —20% SiC_p/2024 composite(Transverse); (d) —20% SiC_p/2024 composite(Longitudinal)

composite experienced large plastic deformation during semi-solid extrusion and original cavities in matrix disappeared during deformation. The oxide film on the surface of Al grains was fragmented under the action of large shearing deformation, which formed the small Al_2O_3 particles of a size about 10 nm that were dispersed homogeneously in the aluminium matrix, as shown in Fig. 3. As the other SiC/Al system, in SiC_p/2024 composites, there are dislocations and dislocation tangles surrounding the SiC particulates, owing to the different coefficients of heat expansion between the SiC particulates and the aluminium matrix.

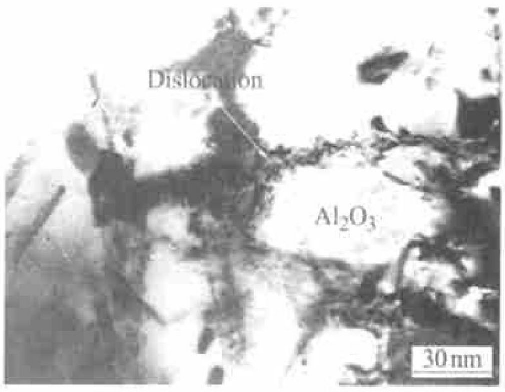

Fig. 3 TEM micrograph of dislocations and Al_2O_3 in SiC_p/2024 matrix

3.3 Interfaces of SiC_p/2024 composites

The SiC_p/2024 composites fabricated by semi-solid extrusion based on powder metallurgy have mainly three kinds of interfaces: (1) the interface of primary Al grains; (2) the interface of Al_2O_3/matrix; (3) the interface of SiC/matrix.

In semi-solid extrusion, the oxide films on the surface of Al grains were fragmented, Al alloy grains were therefore combined into the densified matrix, as shown in

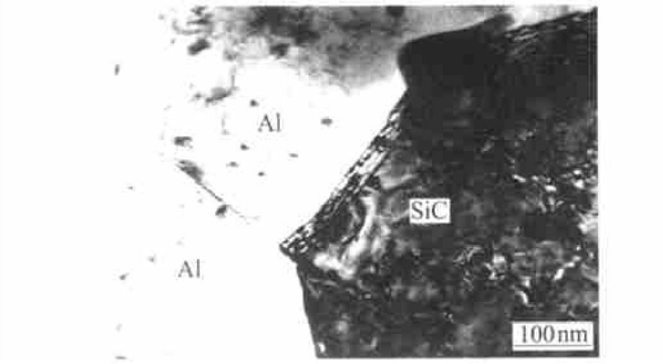

Fig. 4 TEM micrograph of grain boundary of SiC_p/2024 matrix

Fig. 4. There is liquid phase at the grain boundaries of the composites during semi-solid extrusion, which is beneficial to bonding the interface. Fig. 4 shows that the grain boundary of the Al alloy matrix is very thin and there are no cavities and voids at the grain boundaries.

The interface between a SiC particulate and the aluminium matrix is shown in Fig. 5. It is postulated that the interface is clean, smooth and there is no interfacial reaction layer. The pattern of SiC particulate is scarcely changed. TEM pictures show that there are acicular precipitated phases in the aluminium alloy matrix. It is postulated in terms of analysis of phase diagram that the precipitated phase is θ phase ($CuAl_2$), which is primary strengthening phase of 2024 aluminium alloy.

TEM examination of the interface does not detect any Al_4C_3, in spite of the following reaction has been observed by many authors[5, 8, 16]:

$$4Al(l) + 3SiC(s) \rightarrow Al_4C_3(s) + 3Si(s) \qquad (1)$$

The previous studies[16-19] show that the formation of Al_4C_3 is dependent on the concentration of silicon in the alloys. As the latter increases, the extent of reaction (1) will be reduced. In another study, Viala, et al[20, 21] show that for a temperature lower than 627 ℃, no reaction can be observed between solid aluminium and SiC, even when the heat treatment time is very long. In this test, SiC_p/2024 composites were prepared by a powder mixing and semi-solid extrusion process and, in this process, temperature was lower than 600 ℃, Al_4C_3 was therefore not expected to form in the SiC_p/2024 composites. In addition, the purity of the SiC particulates used in the experiment is high and there is a spot of oxides. An X-ray diffraction technique was also used to confirm that there was a very small quantity of oxygen in the composites, no $MgAl_2O_4$ was therefore formed at the interface.

3.4 Fracture surfaces of SiC_p/2024 composites

The fracture surfaces of SiC_p/2024Al composites after T6 treatment are shown in Fig. 6. The dimples and the tearing edges on the fracture surfaces of the composites are

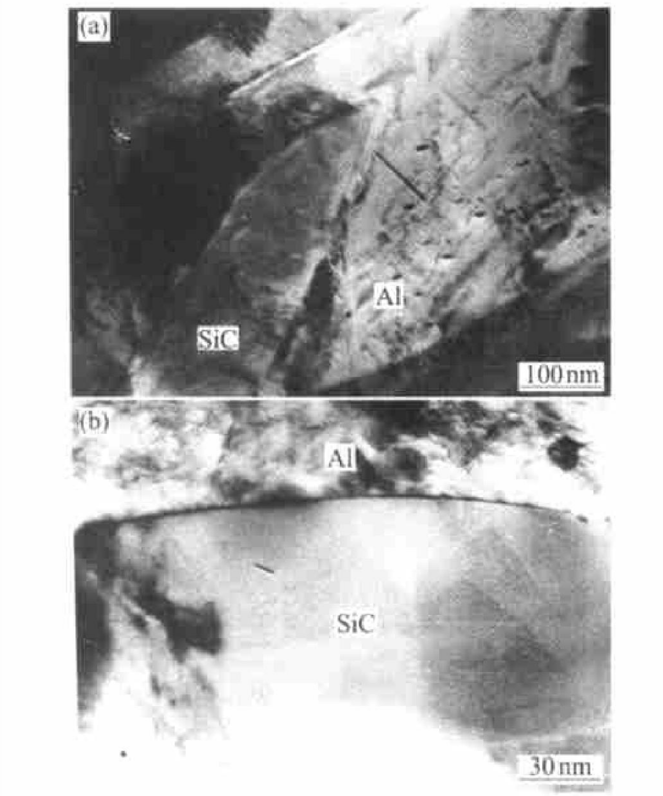

Fig. 5 TEM micrographs showing clean interface in SiC_p/2024 composites
(a) —550 ℃; (b) —570 ℃

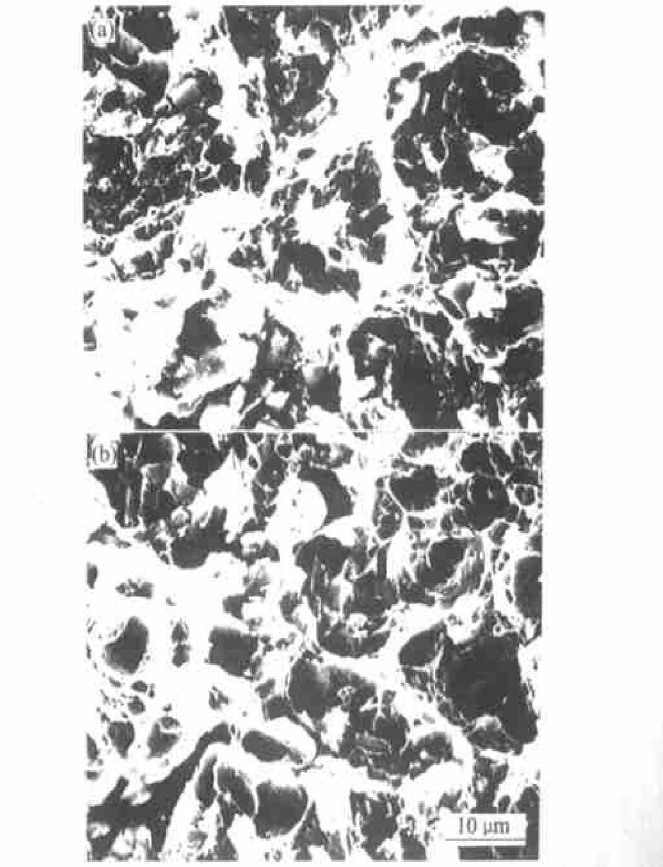

Fig. 6 Fracture surfaces of SiC_p/2024 composites
(a) —10% SiC; (b) —20% SiC

obviously observed from Fig. 6. It is concluded that the composite displays the feature of ductile rupture. In addition, itcan be observed that there is a lack of separation at the interface between SiC particle and the aluminium, indicating a strong bonding between them.

4 CONCLUSIONS

1) The SiC particles are dispersed homogeneously in the aluminium alloy matrix, the fracture extent of the SiC particulates during semi-solid extrusion is very small.

2) Semi-solid extrusion causes local SiC particulate enrichment because of the motion of SiC particulates under the action of gradient of temperature and flow stress.

3) The interface between the SiC particulate and the aluminium matrix is clean, smooth and interfacial reaction layer is very thin. The patterns of the SiC particulates are scarcely changed.

4) There is strong bonding between the reinforcement and the matrix. No Al_4C_3 particles are found.

REFERENCES

[1] Kim Y D, Seo Y H. A continuous fabrication of Al_2O_3 short fiber reinforced aluminum composites by direct rolling process [J]. Scripta Metall Mater, 1995, 32(9): 1387 - 1392.

[2] Maity P C, Chakraborty P N, Panigrahi S C. Processing and properties of Al-Al_2O_3(TiO_2) in situ particle composite[J]. J Mater Proc Tech, 1995, 53: 857 - 870.

[3] Carson S, Masounave J. A literature review on fabrication techniques of particulates reinforced metal matrix composites [A]. Proc of Int Conf Fabrication of Particulates Reinforced Metal Matrix Composites[C]. Materials Park, OH: ASM International, 1990. 79 - 85.

[4] Foo K S, Banks W M, Craven A J, et al. Interface characterization of an SiC particulate/ 6061 aluminum alloy composite[J]. Composite, 1994, 25(7): 677 - 683.

[5] Ribes H, Da Silva R, Suery M, et al. Effect of interfacial oxide layer in Al-SiC particle composites on bond strength and mechanical behaviour[J]. Mater Sci Technol, 1990(6): 621 - 628.

[6] Salvo L, Suery M, Legoux J G, et al. Influence of particle oxidation on age-hardening behavious of as fabricated and remelted SiC reinforced Al-1%Mg alloys[J]. Mater Sci Eng, 1991, A135: 129 - 133.

[7] ZU Li-jun, LUO Shou-jing, ZHANG Hong-yan. Syudy on microstructure' properties and flow analysis of extruding SiC_p/ 2024 composites in semi-solid[J]. Transactions of Harbin Institute of Technology, 2000, 32(5): 69 - 72.

[8] Dignard-Bailey L M, Malis T F, Embury J D. Characterization of interfaces in metal matrix composites[A]. Wilkinson D S. Proc Int Symp on Advanced Structural Material[C]. America, 1988. 87 - 95.

[9] Mahon C J, Howe J M, Vasudevan A K. Microstructural development and the effect of interfacial precipitation on the tensile properties of aluminum/ silicon carbide composites[J]. Acta Metall Mater, 1990, 38(8): 1503 - 1512.

[10] Strangwood M, Hippsley C A, Lewandowski J J. Segregation to SiC/ Al interfaces in Al based metal matrix composites [J]. Scripta Metall Mater, 1990, 24: 1483 - 1487.

[11] SUN Jia-kuan. Mechanical Behavior and Mechanism of SiCp/ 2024 composites in Semi-solid[D]. Harbin: Harbin Institute of Technology, 1999. 62. (in Chinese)

[12] LUO Shou-jing, ZU Li-jun. Study on preparing SiC_p/ 2024 composites by powder mixture and extrusion in semi-solid [J]. The Chinese Journal of Nonferrous Metals, 2000, 10 (S2): 179 - 183.

[13] Nair S V, Tien J K, Bates R C. SiC-reinforced aluminum metal matrix composites[J]. Int Met Rev, 1995, 30(6): 275 - 288.

[14] Higashi K, Nieh T G, Mabuchi M, et al. Effect of liquid phases on the tensile elongation of superplastic aluminum alloys and composites[J]. Scripta Metall Mater, 1995, 32 (7): 1079 - 1084.

[15] LUO Shou-jing, ZU Li-jun. Semi-solid extrusion of SiC_p/ 2024 composites prepared by contact reaction method[J]. Trans Nonferrous Met Soc China, 2000, 10(3): 304 - 308.

[16] Skibo M, Morris P L, Lloyed D J. Structure properties of liquid metal processed SiC reinforced aluminium[A]. Fishman S G, Dhingra A K. Proc ASM Conf on Cast Reinforced Metal Composite[C]. America, 1988. 257 - 259.

[17] Kannikeswaran K, Lin R Y. Element effects on Al-SiC interfaces[J]. JOM, 1987(9): 17 - 19.

[18] Ribes H, Suery M, L'Esperance G, et al. Microscopic examination of the interface region in 6061-Al/ SiC composites reinforced with as received and oxidized Si particles[J]. Metall Trans A, 1990, 21A: 2489 - 2496.

[19] Bonollo F, Guerriero R, Sentimenti E, et al. The effect of quenching on the mechanical properties of powder metallurgically produced Al-SiC(particles) metal matrix composites [J]. Mater Sci Eng, 1991, A144: 303 - 309.

[20] Viala J C, Fortier P, Bouix J. Stable and metastable phase equilibria in the chemical interaction between aluminium and silicon[J]. J Mater Sci, 1990(25): 1842 - 1850.

[21] LUO Shou-jing, ZU Li-jun. Influence on forming force in semi-solid of strain rate, fluid phase volume fraction and fluid viscosity[J]. The Chinese Journal of Nonferrous Metals, 2000, 10(S2): 192 - 197.

(Edited by PENG Chao-qun)