



Effect of Thermal Exposure on the Interface Microstructure and Interfacial Shear Strength of the SiC Fiber Reinforced AlFe5Si2 Matrix Composite

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Abstract

The effect of high temperature on the interface microstructure and interfacial shear strength of the continuous SiC fiber reinforced AlFe5Si2 matrix (45 vol.% SiC_f/AlFe5Si2) composite was investigated. The composite was prepared by hot isostatic pressing (HIP), and then thermally exposed at 260/300/350/400/450 °C for 20/50/100 h, respectively. The interfacial shear strength of the composite with and without thermal exposure was measured by push-out test. The interface microstructure and phase compositions of the composite with and without thermal exposure were characterized by scanning electron microscopy, transmission electron microscopy and X-ray diffraction. Results showed that the interfacial reaction took place at the interface between SiC fiber and aluminum matrix and formed Al₄C₃ phase during the preparation process. The activation energy of Al₄C₃ growth at the C/Al interface was about 163 kJ/mol. With increasing the thermal exposure temperature and time, the interfacial shear strength of the composite declined, especially at temperatures above 400 °C. The brittle Al_{3,21}SiO_{0,47} compound formed at temperatures above 400 °C mainly resulted in the severe strength deterioration of the interfacial reaction zone.

Keywords Thermal exposure · SiC_f/AlFe5Si2 composite · Interface microstructure · Interfacial shear strength · Interfacial reaction

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1 Introduction

Continuous SiC_f/Al composite has been considered as a potential material for advanced aircraft and aerospace applications due to its outstanding properties such as high specific stiffness, high specific strength, good high-temperature performance and so on. The preparation methods for SiC_f/Al composite can be divided into two categories, i.e., liquid method and solid method [1–3]. For liquid method, the fibers may severely react with molten Al, deteriorating the mechanical performance of the composites [4–6]. For solid method, the distribution of fibers and interfacial reaction could be well controlled.

The interface bonding behavior plays a critical role in transmitting force and has a substantial effect on the properties of metal matrix composites [7]. The interface bonding strength between fiber and matrix can generally be quantitatively evaluated by interfacial shear strength, which has received great attentions [8–11]. The most commonly used methods to measure the interfacial shear strength of continuous fiber reinforced composites include fiber pull-out, push-out, and fragmentation tests. Among them, the push-out test has some comparative advantages such as convenience and more accurate results, because data can be easily obtained from one specimen from the prepared composite [12, 13]. The thickness of the specimen for push-out test needs to be limited to a value less than 1.0 mm, avoiding experimental problems such as failure of the indenter or fiber damage during loading [14].

Thermal exposure is usually conducted to simulate the high temperature working environment. Chen et al. [15] have found that under aging treatment (oil bath, at 120 °C), the interfacial shear strength of the 0.6 vol.% SiC_f/7075Al composite increased with increasing aging time to 25 h, corresponding to the increasing tendency of the ultimate tensile strength of 7075Al matrix. However, the work by Jiang et al. [16] indicated that the interfacial shear strength of SiC_f/LD-2Al composite decreased from 19.78 MPa to 7.84 MPa after heat treatment. An issue for 7075 and LD-2 aluminum alloys is that their strength generally decreases due to overaging when the temperature exceeds 150 °C. For the applications at temperatures above 200 °C, it is necessary to select other heat-resistant aluminum alloy to act as the matrix of the SiC_f/Al composite.

In this work, the AlFe5Si2 alloy, a novel heat-resistant aluminum alloy, was used as the matrix of the SiC_f/Al composite. Magnetron sputtering was employed to deposit matrix coating on SiC fiber and hot isostatic pressing was applied to prepare the SiC_f/AlFe5Si2 composites. Then, the composites were thermally exposed at 260/300/350/400/450 °C for 20/50/100 h. Push-out test, SEM, TEM and XRD were utilized to study the effect of thermal exposure on the interface microstructure and interfacial shear strength.

2 Materials and Methods

2.1 Preparation of the SiC_f/AlFe5Si2 Composite

The matrix-coated SiC fibers (MCFs) were prepared by Beijing Institute of Aeronautical Materials (BIAM). The CVD SiC fiber of ~100 μm in diameter consists of a tungsten core (~20 μm in diameter), a β-SiC layer (~40 μm in thickness) and a C-rich layer (~1.5 μm in thickness). Aluminum alloy (~25 μm in thickness) was deposited on the outmost surface of the SiC fiber by magnetron sputtering to prepare the MCFs. The nominal chemical composition of the matrix alloy is Al-5wt%Fe-2wt%Si (abbreviated as AlFe5Si2).

The above MCFs were packed into an aluminum capsule (80 mm in length, 5 mm in internal diameter and 12 mm in outside diameter). The capsule was then vacuum-sealed. Hot isostatic pressing was conducted with a mini hip q1h9 equipment at the temperature 580 °C for 4 h under a pressure of 120 MPa to consolidate the MCFs.

The as-prepared SiC_f/AlFe5Si2 composites were cut perpendicular to the fiber axis to a total of 32 slices (0.6 mm in nominal thickness) using wire electrical discharge machining. These slices were divided into 16 groups, each containing 2 slices and one slice for the push-out test and the other for the microstructure observation. Then, except for one group without thermal exposure, the other groups were thermally exposed at 260/300/350/400/450 °C for 20/50/100 h, and cooled to room temperature in an air furnace, respectively.

2.2 Push-out Test

The slices in each group for the push-out test were polished with 1 μm diamond paste. The push-out test was conducted using an Instron testing machine with a WC cone indenter, on the tip of which there was a plateau of ~60 μm in diameter (as shown in Fig. 1a). The SiC fibers in composites were aligned with the cone tip and then were pushed out at a velocity of 1 mm/min. For each specimen, at least five fibers were measured, and the average of the measured results was used to calculate the interfacial shear strength to ensure the reliability of obtained data. Applied force was measured by a force sensor connected to the indenter.

A typical push-out curve of the SiC_f/AlFe5Si2 composite without thermal exposure is shown in Fig. 1b. The process of push-out test could be separated to three stages: elastic deformation (1), interfacial debonding (2) and sliding friction (3). The interfacial shear strength is given by the following equation [17]:

$$\tau = \frac{F_{max}}{2\pi r t} \quad (1)$$

where F_{max} is the peak value of applied force, r is the radius of SiC fiber, t is the thickness of specimen.

2.3 SEM, TEM and X-Ray Diffractometry

The slices in each group for the microstructure observation were ground successively with 400, 600, 1000 and 1200 grit silicon carbide metallographic papers, and polished with 1 μm

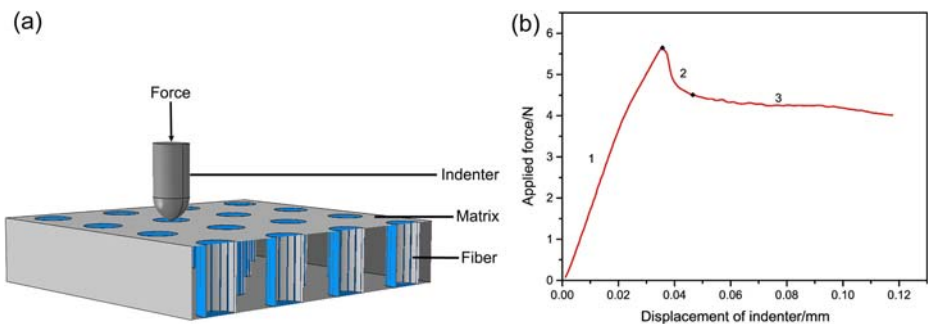


Fig. 1 (a) Schematic of the push-out test and (b) typical push-out curve of the SiC_f/AlFe5Si2 composite without thermal exposure

diamond paste. The microstructures of the $\text{SiC}_f/\text{AlFe5Si2}$ composite with and without exposure were observed by a field emission scanning electron microscope (FE-SEM, Nova NanoSEM 450) with an energy dispersive X-ray spectrometer.

Some slices selected from the above continued to be polished to about 80 μm . The specimens were then polished to 20 μm and ion milled at 5 kV for about 6 h, inclining at about 15 degree to the ion beam. The transmission electron microscope (TEM, JEOL 2100) with an energy dispersive X-ray spectrometer was utilized to analyze the microstructure of interface between SiC fiber and AlFe5Si2 matrix.

The phases in the $\text{SiC}_f/\text{AlFe5Si2}$ composite with and without thermal exposure were characterized by an ADVANCE D8 X-ray diffractometer (XRD) using Cu $K\alpha$ radiation with a scanning speed of 4°min^{-1} (2θ).

3 Results and Discussion

3.1 Microstructure of the $\text{SiC}_f/\text{AlFe5Si2}$ Composite

Figure 2 shows the microstructure of as-prepared $\text{SiC}_f/\text{AlFe5Si2}$ composite. The cross section of the sample is about 4.3 mm in diameter. The volume fraction of the as-prepared $\text{SiC}_f/\text{AlFe5Si2}$ composite is about 45%. The distribution of the SiC fibers in the AlFe5Si2 matrix is quite uniform. The AlFe5Si2 alloy is bonded well and there is no hole or crack defect observed at the interface between SiC fiber and AlFe5Si2 matrix. From the center of SiC fiber to the AlFe5Si2 matrix, the order is W core, SiC, C-rich layer and AlFe5Si2 alloy (as shown in Fig. 2c).

Figure 3 shows the element line-scanning profiles of the interface in the as-prepared $\text{SiC}_f/\text{AlFe5Si2}$ composite. The center part with a thickness of $\sim 1.5 \mu\text{m}$ is the C-rich layer, the left side and right side are the SiC fiber and AlFe5Si2 matrix, respectively. The SiC fiber coated with a C-rich layer contained silicon carbide particles which is not crystalline [18, 19]. So, the

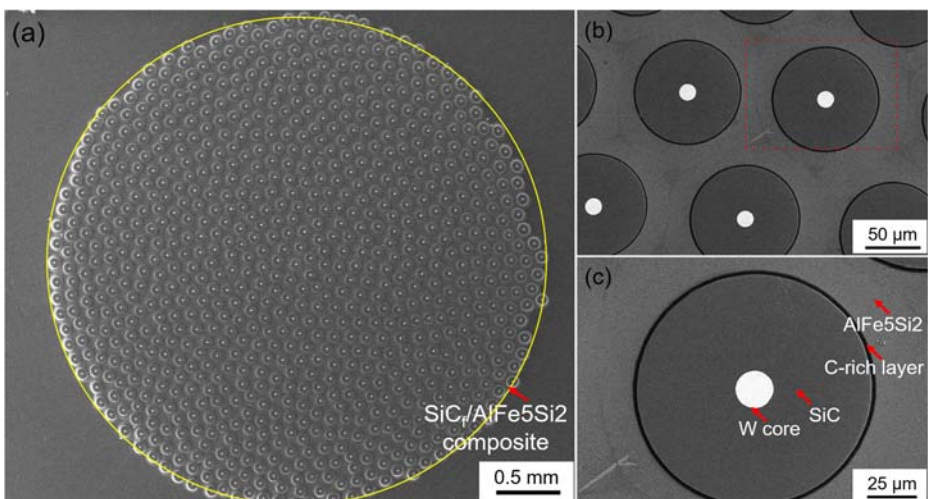


Fig. 2 Microstructure of the $\text{SiC}_f/\text{AlFe5Si2}$ composite: (a) morphology of the cross section; (b) high magnification of (a); (c) major components in the composite of (b)

Si element has been detected to diffuse along the SiC/ C interface from the left side. The C element is the highest at the center of the C-rich layer. The X-ray signal diffracted from carbon atoms with low Z number is weak and would be easily absorbed by the aluminum matrix in the composite. Therefore, the counts per second (cps) of the C element is very low. The Al element has been detected to diffuse along the AlFe5Si2/C interface from the right side.

Figure 4 shows the interface microstructure of the as-prepared SiC_f/AlFe5Si2 composite and energy dispersive spectrum (EDS) analysis of the selected point in the interfacial reaction zone. There exists a thin zone (~43 nm in thickness) containing C, Al and Si elements at the interface between C-rich layer and AlFe5Si2 matrix, which may be resulted from the high temperature (580 °C) for preparing the composite.

During the HIP process, the aluminum atoms may diffuse from the AlFe5Si2 matrix into the C-rich layer, and the carbon atoms diffuse in the opposite direction (as shown in Fig. 3). Therefore, the interfacial reaction as follow may be expected to occur.



The diffraction pattern of the as-prepared SiC_f/AlFe5Si2 composite in the range of 20–90° (2θ) is shown in Fig. 5. The reaction product Al₄C₃ has been detected in the XRD pattern except for Al, SiC, W and C phases. The interfacial reaction in Eq. 2 has been confirmed to occur at the interface between aluminum matrix and C-rich layer, but no coarse Al₄C₃ phase can be found, increasing the interfacial shear strength.

3.2 Effect of Thermal Exposure on the Interfacial Shear Strength

Figure 6 shows the interfacial shear strength versus thermal exposure time curve of the SiC_f/AlFe5Si2 composites exposed at different exposure temperature. It shows that the interfacial shear strength of the SiC_f/AlFe5Si2 composite exposed at temperatures below 350 °C has

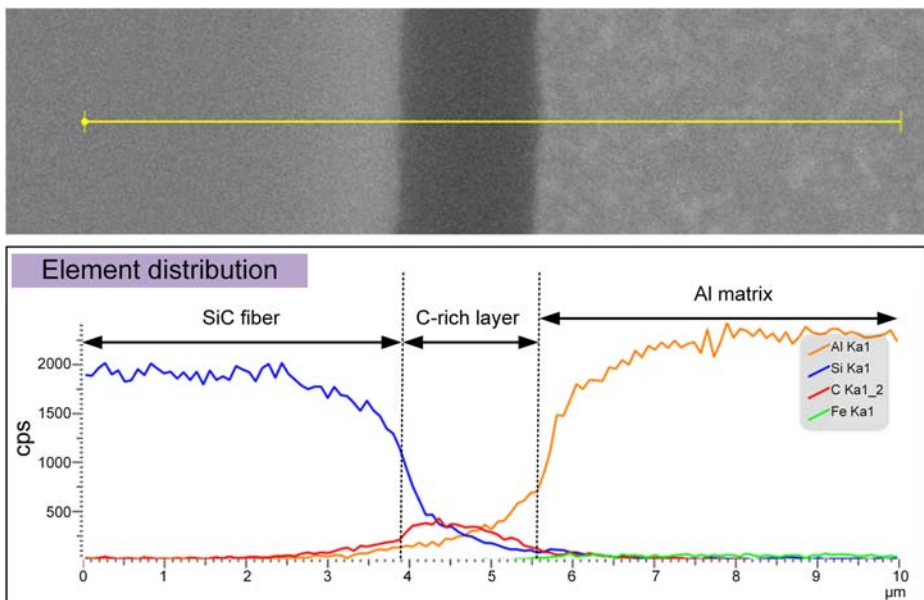


Fig. 3 Element line-scanning profiles of the as-prepared SiC_f/AlFe5Si2 composite

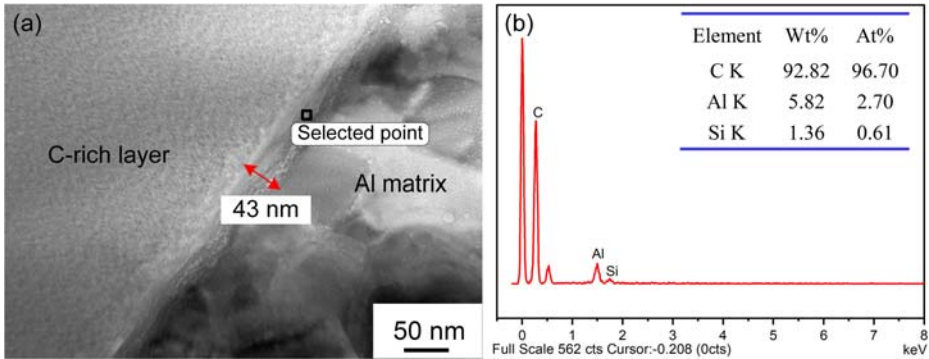


Fig. 4 (a) TEM image of the interface microstructure of the $\text{SiC}_f/\text{AlFe}_5\text{Si}_2$ composite; (b) EDS analysis of the selected point in the interfacial reaction zone

relatively small decrease. The interfacial shear strength at 300 °C and 350 °C for 20 h appeared to be somewhat lower than that for 50 h. This phenomenon may be related with the thickness effect of specimen, which has been observed in $\text{SiC}_f/7075$ composites by Chen et al. [15]. There existed a relatively large difference between the actual thickness and nominal thickness (0.6 mm) for the specimens exposed at 300 °C and 350 °C for 20 h and 50 h, respectively. For example, the actual thickness values of the specimens exposed at 350 °C for 20 h and 50 h were 0.48 mm and 0.73 mm, respectively. The actual thickness for other specimens ranged from 0.55 mm to 0.59 mm. The upper and lower surface regions around SiC fiber are nearly the free surfaces, exhibiting less compressive stress on the fiber in radial direction from AlFe_5Si_2 matrix than the inner region around the fiber. Therefore, the ratio of high radial compressive stress region to the overall surface of SiC fiber for the thin specimen is lower than that for the thick specimen, transforming lower friction force. Thus, the thin specimen exhibits lower interfacial shear strength than the thick specimen. The slope of the interfacial shear

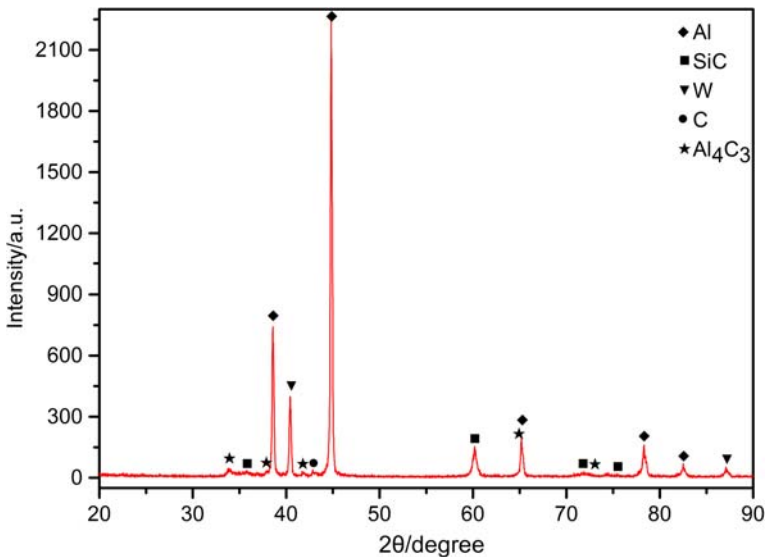


Fig. 5 X-ray diffraction pattern of the as-prepared $\text{SiC}_f/\text{AlFe}_5\text{Si}_2$ composite

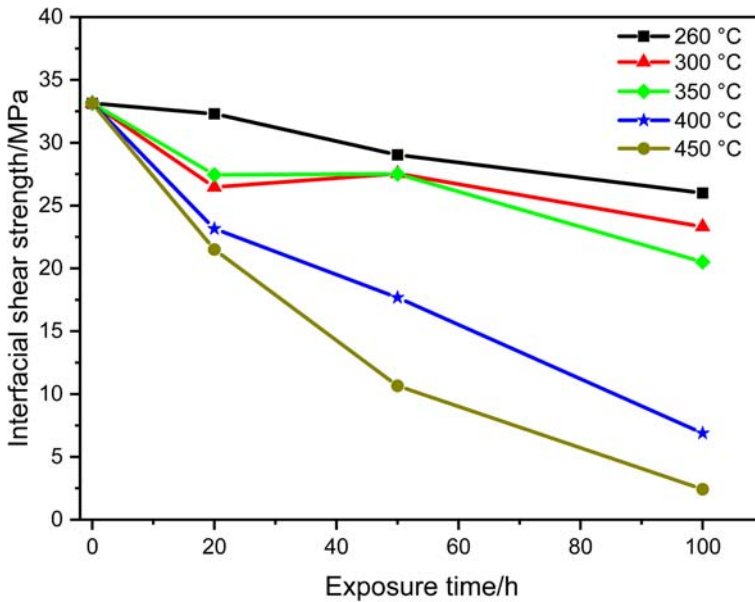


Fig. 6 Interfacial shear strength versus time curve of the SiC_f/AlFe₅Si₂ composites without thermal exposure and exposed at 260/300/350/400/450 °C for 20/50/100 h

strength versus thermal exposure time curve increases markedly when the thermal exposure temperature exceeds 400 °C, indicating that the interfacial shear strength declines dramatically. For the composite exposed at temperatures above 400 °C for over 50 h, the interfacial shear strength dropped more than 50% compared with the one without exposure. It is indicated that the SiC_f/AlFe₅Si₂ composite can be used at temperatures below 350 °C, which is much higher than that of conventional aluminum alloys (~200 °C).

3.3 Effect of Thermal Exposure on the Interface Microstructure

Figure 7 shows the interface microstructures and EDS analysis results of the SiC_f/AlFe₅Si₂ composites exposed at 450 °C for 20 h and 50 h, respectively. The interfacial reaction zone at the interface of the SiC_f/AlFe₅Si₂ composite exposed at 450 °C for 20 h has more content of Si element compared with that without thermal exposure and has been oxidized slightly. The elevated temperature promotes the diffusion of Si and C elements to the interface, and the additional silicon element in the reaction zone comes from amorphous silicon in SiC fiber and AlFe₅Si₂ alloy. There exists a stronger elemental diffusion of C, Al and Si at the interface of the SiC_f/AlFe₅Si₂ composite exposed at 450 °C for 50 h, exhibiting more serious oxidation.

The interfacial reaction zone of the SiC_f/AlFe₅Si₂ composites exposed at 450 °C are much thicker than that without thermal exposure. The thickness of reaction zone in the SiC_f/AlFe₅Si₂ composites increases from initial 43 nm to about 216 nm after thermal exposure at 450 °C for 50 h, and it has been confirmed principally as the Al₄C₃ phase according to the selected area diffraction pattern in Fig. 7c. Additionally, the oxidation phase in the reaction zone (see Fig. 7a) has been identified as the brittle Al_xSiO_y compound in previous studies and can severely deteriorate the mechanical properties of the composites [5, 6]. In the present

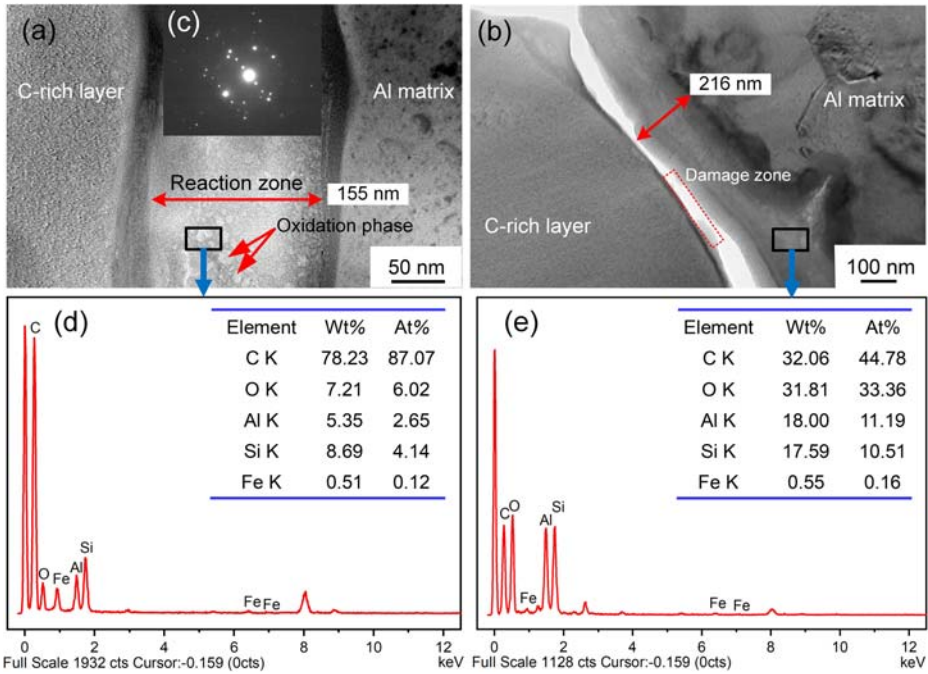


Fig. 7 TEM images of interface microstructures of the $SiC_p/AlFe_5Si_2$ composites exposed at (a) 450 °C for 20 h; (b) 450 °C for 50 h; (c) selected area diffraction pattern of the Al_4C_3 phase in (a); (d) and (e) EDS analysis results of the selected areas in the interfacial reaction zone in (a) and (b), respectively

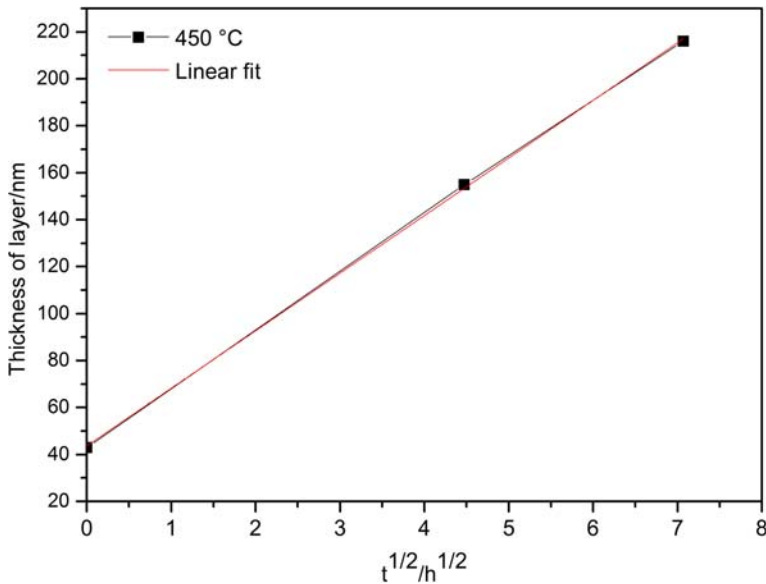


Fig. 8 Interfacial reaction kinetic curves of the $SiC_p/AlFe_5Si_2$ composite exposed at 450 °C

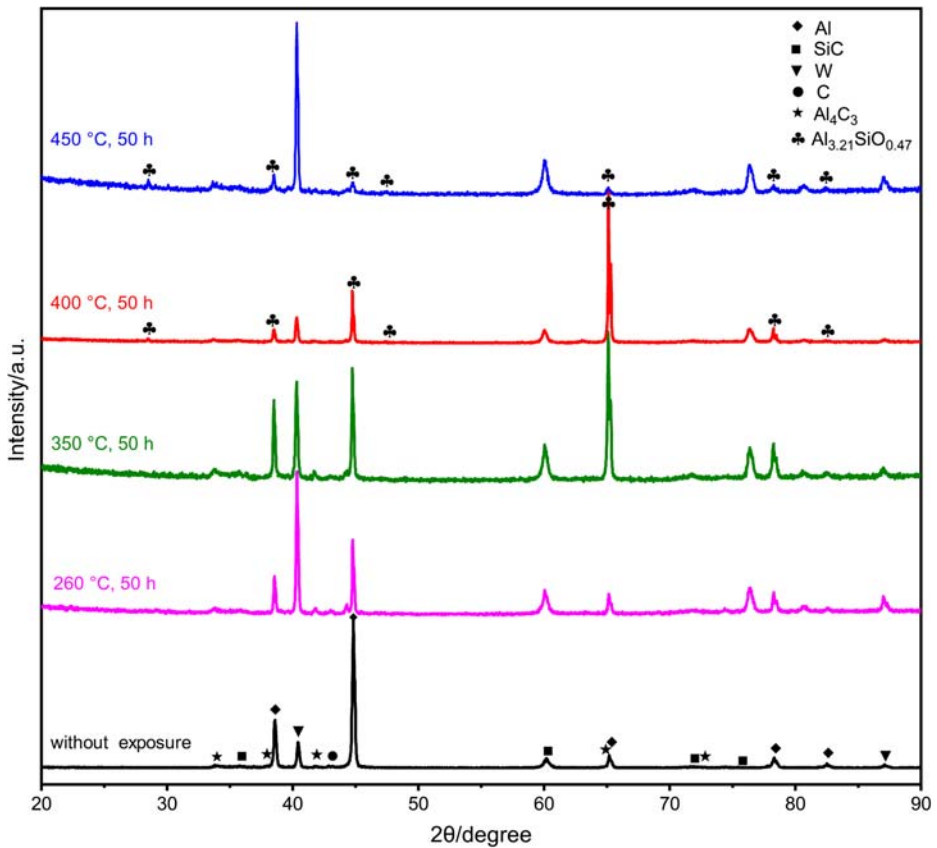


Fig. 9 X-ray diffraction patterns of the SiC/AlFe5Si2 composites with and without thermal exposure

study, the damage zone (see Fig. 7b) may be resulted from ion beam milling process due to the weak strength of the interface.

The interfacial reaction is a reaction-controlled process at its very early stage and then changes to be controlled by diffusion when a thin reaction zone is formed [20]. The growth of the interfacial reaction zone may follow a parabolic law as [19, 21]:

$$l = (kt)^{1/2} + b_0 \tag{3}$$

where l is the thickness of the interfacial reaction zone, k is the rate constant related to the diffusion coefficient of the chemical components and to the thermodynamic properties of the interfacial reaction zones, t is the thermal exposure time and b_0 is the original thickness of the reaction zone in the as-prepared specimen.

The data points for the exposure temperature 450 °C is fitted with a straight line to estimate a generalized reaction rate constant, as shown in Fig. 8. The fitted equation is as follow:

$$l = 24.529t^{1/2} + 43 \tag{4}$$

The thickness l is in nm, and time t is in h for the eq. (4). Consequently, the rate constant k is calculated to be $1.67131 \times 10^{-19} \text{ m}^2\text{s}^{-1}$.

As an important parameter to determine the growth rate of the reaction zone, the expression for k is given by the Arrhenius law as [6]:

$$k = A \exp(-Q/RT) \quad (5)$$

where A is the pre-exponential factor, Q is the activation energy for the interface reaction, R is the gas constant and T is the absolute temperature.

The Arrhenius law is based on the postulation that the activation energy Q is regarded as a constant independent of temperature. Previous research [22] has reported the A value as $1 \times 10^{-7} \text{ m}^2\text{s}^{-1}$, substituting $R = 8.31141 \times 10^{-3} \text{ kJ}\cdot\text{K}^{-1}\cdot\text{mol}^{-1}$, $T = 723 \text{ K}$ and $k = 1.67131 \times 10^{-19} \text{ m}^2\text{s}^{-1}$ in Eq. 5, activation energy $Q = 163 \text{ kJ/mol}$ can be obtained. This derived value is supposed to account for the growth of the reaction zone in Eq. 2. The k value rises with increasing the thermal exposure temperature, which means the interfacial reaction zone grows faster with increasing the exposure temperature.

Figure 9 shows the X-ray diffraction patterns of the $\text{SiC}_f/\text{AlFe5Si2}$ composite with and without thermal exposure. When exposed below $350 \text{ }^\circ\text{C}$ for 50 h, the main phases in the composite are Al, SiC, W, C and Al_4C_3 . When exposed above $400 \text{ }^\circ\text{C}$ for 50 h, the oxidation phase, which was identified as $\text{Al}_{3,21}\text{SiO}_{0,47}$ compound, was formed in the composite.

With increasing the thermal exposure temperature and time, the interfacial reaction zone in the $\text{SiC}_f/\text{AlFe5Si2}$ composite becomes thicker and the interfacial shear strength declines, especially at temperatures above $400 \text{ }^\circ\text{C}$. The brittle $\text{Al}_{3,21}\text{SiO}_{0,47}$ phase formed at temperatures above $400 \text{ }^\circ\text{C}$ mainly results in the severe deterioration of the interfacial shear strength.

4 Conclusions

The 45 vol.% $\text{SiC}_f/\text{AlFe5Si2}$ composite was successfully prepared by consolidating the matrix-coated fibers in this research. The interfacial reaction took place at the interface between C-rich layer and aluminum matrix and formed Al_4C_3 phase during the preparation process (HIP at $580 \text{ }^\circ\text{C}$ for 4 h under a pressure of 120 MPa). The activation energy of Al_4C_3 growth at the C/Al interface was about 163 kJ/mol.

The interfacial shear strength of the $\text{SiC}_f/\text{AlFe5Si2}$ composites declined with increasing the thermal exposure time and temperature, especially at temperatures above $400 \text{ }^\circ\text{C}$. It is indicated that the $\text{SiC}_f/\text{AlFe5Si2}$ composite can be used at temperatures below $350 \text{ }^\circ\text{C}$, which is much higher than that of conventional aluminum alloys ($\sim 200 \text{ }^\circ\text{C}$).

The interfacial reaction zone in the $\text{SiC}_f/\text{AlFe5Si2}$ composite becomes thicker with increasing the thermal exposure temperature and time. Meanwhile, it has been found that the brittle $\text{Al}_{3,21}\text{SiO}_{0,47}$ compound was formed at temperatures above $400 \text{ }^\circ\text{C}$. The existence of the brittle phase mainly results in the severe deterioration of the interfacial shear strength.

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