HRTEM study of interfacial structure in SiCp/A390 composites

Zhen Wang, Aiqin Wang, Huihui Han and Jingpei Xie

1 School of Materials Science and Engineering, Henan University of Science and Technology, Luoyang 471023, People’s Republic of China
2 Collaborative Innovation Center of Non-Ferrous Materials of Henan Province, Luoyang 471023, People’s Republic of China

E-mail: aiqin_wang888@163.com

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Abstract

SiCp/A390 composite was fabricated by powder metallurgy method and hot extrusion process. The interfacial microstructure of SiCp/A390 composites after heat treatment was characterized by HRTEM. The results show that the interface between SiC and Al matrix has an orientation relationship of [410]SiC/[011]Al, (1010)SiC/(111)Al and forming a semi-coherent interface. The crystallographic orientation relationship between SiC and Si in the SiCp/A390 composites is [010]SiC/[001]Si, (004)SiC//(220)Si. The interplanar spacing of Al matrix on lattice plane (111) is close to the twice interplanar spacing of the Si phase on lattice plane (400). They keep a semi-coherent crystal plane, with an orientation relationship of [011]Al/[001]Si, (111)Al//(400)Si.

1. Introduction

The SiCp/A390 composites have high specific strength and specific stiffness, elasticity modulus and low thermal expansion coefficient of performance, and low manufacturing costs. It has played a significant role in various areas, such as the aerospace, automotive, electronic instrumentation, and other industry. But in order to get high performance composite materials, it need a suitable interface for composites [1–3]. Due to the load is transferred through the interface of the composites under the action of external force. The interface of the composites is connected bond between the reinforcement phases and matrix and is also a ‘bridge’ to pass the mechanics and other features (such as thermal conductivity, electrical conductivity, damping characteristics). The structure and formation regulation of interface play a vital role on properties of the composite. Therefore it becomes the key to research and development of the composite with a good interface [4, 5].

Since the expansion coefficient, elastic modulus, and organizational structure on both sides of the interface of the composite materials are obviously different. The microstructure of interface is very complex. There are many factors to affect the interface of the composite, such as the type, quantity, size of the reinforcement, chemical composition of the matrix, preparation process and heat treatment [6–9]. He et al [10] studied the interface structure of SiCp/Al-Fe-V-Si composites prepared by spray deposition and found that the coarse Al4C3 brittle phase is formed at the SiC/Al interface, which results in the interface weakening followed by decrease of the composite strength. Fan et al [11] compared the interface of SiCp/Pure Al with that of SiCp/Al-Si composites fabricated using Vacuum-high Pressure infiltration. It is found that the addition of Si to matrix significantly prevents the formation of Al4C3. Luo [12] studied the interface of pre-oxidized SiC reinforced Al-Si alloy composites prepared by pressureless infiltration, and found that MgAl2O4 precipitated phase at the interface, which has four different crystal orientation relationships with SiC and has a semi-coherent relationship with the Al matrix.

Wang et al [13] studied the interface of pre-treated SiC reinforced Al-Si alloy composites prepared by powder metallurgy, and there are 5 nm-thick amorphous layer interface and reaction interface in the composites. However, there are few systematic studies on the interface of SiC/Al-Si composites prepared by powder metallurgy. So it needs further analysis and research for the interface of SiCp/Al-Si composites. In this study, SiCp/A390 composites are prepared by powder metallurgy method and hot extrusion process. The crystal structure of the interface and the crystallographic orientation relationship between SiC particles, Si phase and Al
matrix in the composite are researched by HRTEM. It provides a theoretical and experimental support for interfacial design and improvement of the composites.

2. Experimental

SiC particles are used as the reinforcement with an average particle size of 5 μm in this investigation. A390 aluminum powders fabricated by atomization are used as the matrix alloy. The silicon existed in the A390 alloy powder with Al-Si-Cu-Mg supersaturated solid solution in the α-Al phase. The average particle size of A390 powders is 10 μm. The chemical composition of the matrix alloy is shown in Table 1.

The A390 powders and SiC particles were ball-mixed in a high-speed planetary mill for 8 h with the rotation speed of 200 rpm. The weight ratio of ball and powder was 2:1. The mass fraction of SiC particles in the composites is 25%. The mixed powers were put into the mold with the one-way press and sintered at 560 °C for 4 h. Hot extrusion was conducted in XJ-500 metal profile extrusion machine, extrusion ratio of 15:1. The composite was solid solution at 515 °C for 4 h, water quenched, and then aging at 180 °C for 30 min and 6 h. The samples for TEM were machined into 0.3 mm by wire electrode cutting, and make it less than 50 μm by manually thinning, and then cut into wafer with 3 mm in diameter. To facilitate observation, foils were prepared by argon ion milling on GATAN-691 ion milling instrument, the first wide-angle (9°) thinned to perforation, then a small angle (5°) thinning for one hour. The microstructure of the composites was observed by JEM-2100 HRTEM microscope. The experimental high-resolution images were analyzed and studied by Digital Micrograph software.

3. Results and discussion

3.1. Interface between SiC and Al matrix

TEM images of interfacial morphology between SiC and Al matrix in SiCp/A390 composites are shown in Figure 1. TEM observations indicate that it has a good bonding between SiC and Al alloy. The interface between SiC and Al matrix is clean, straight, and combining closely. The reaction product Al4C3 and MgAl2O4 ad described in literature [10, 12] in the interface. TEM image of the composite is hot extrusion state, as shown in Figure 1(a). TEM image of the composite is heat treatment state, as shown in figures 1(b)–(c). And a high density of dislocations at the interface between SiC particle and Al matrix is shown in figure 1(c). This is because the coefficient of thermal expansion of between SiC particle and Al matrix is significant differences causing thermal mismatch. causing thermal mismatch. When the composite solid solution treatment, the thermal mismatch leads to the base alloy strained near the interface. And then it can generate a high density of dislocations at the interface between SiC particle and Al matrix. The existence of interfacial dislocations is beneficial to improve combination of the interface.
3.1.2. Interfacial microstructure between SiC and Al matrix

Figure 2(a) shows HRTEM image of interface between SiC particle and Al matrix. As it can be seen from the figure, the interfacial atoms between SiC and Al matrix are arranged closely without any other reaction products. Due to the composites is prepared by powder metallurgic method, the temperature throughout is too low to produce interface chemical reaction. So the interfacial binding between SiC and Al belongs to the solid state. Thus, there is no fixed crystallographic orientation relationship, but there appear preferentially parallel relationship between SiC and Al matrix [14]. The electron diffraction calibrated and diffraction spots of the interface between SiC and Al matrix in the composite are shown in figure 2(e). Through the analysis of the composite diffraction spot calibration, it shows that the interface between SiC and Al matrix in this research forms a certain the following crystallographic orientation relationship. The close-packed $\bar{1}11$ plane of the Al phase is parallel to the close-packed basal plane $10\bar{1}0$ of the SiC phase, the following preferential ones are observed:

$$\begin{align*}
\{4510\}_{\text{SiC}} & / / \{10\bar{1}1\}_{\text{Al}}, \\
\{10\bar{1}0\}_{\text{SiC}} & / / \{\bar{1}1\}_{\text{Al}}
\end{align*}$$

Through Digital Micrograph software the choice square areas deal with the noise in figure 2(a). Figures 2(b)–(d) shows the IFFT image of square area 1 ~ 3 in figure 4(a). After Fourier (FFT) and inverse Fourier (IFFT) transform, it can be more clearly observed atomic arrangement structure of the interface between SiC particle and Al matrix. The SiC is along $[4510]$ zone axis in figure 4(b), which is typical close-packed hexagonal structure $\alpha$-SiC (6 H). In the hexagonal $\alpha$-SiC (6 H), the close-packed layer of the stacking is ABCABC (A). After being accurately measured, the interplanar spacing of 6 H $\alpha$-SiC phase on lattice plane (0001) is 0.26 nm. The interplanar spacing of the Al matrix on lattice plane (111) is 0.23 nm. Figure 2(d) shows the interfacial microstructure of between SiC and Al matrix. From the figure 2(d) can be seen that the (10$\bar{1}0$) crystal planes of SiC is parallel to the (111) crystal planes of Al, which further demonstrates crystallographic orientation relationship between SiC and Al matrix. By calculation, the degree of mismatch between SiC and Al matrix is only 0.115. So the interfacial bonding of the (10$\bar{1}0$) crystal planes in 6H $\alpha$-SiC and (111) crystal planes in Al matrix is semi-coherent interface. It is a kind of very favorable crystallographic orientation relationship in terms of the structure of crystallography. It indicates that the interface
between SiC and Al matrix has a lower energy, as well as the interface between SiC particles and the Al matrix combine closely [15].

3.2. Interface features between SiC and Si in SiCp/A390 composites

3.2.1. TEM morphology of SiC/Al interface in SiCp/A390 composites

In this experiment, A390 alloy have high silicon content. So the interface between SiC and Si in the composites is a common interface. The interfacial bonding strength between SiC/Al has an important effect on the properties of SiCp/A390 composites. TEM image of interface between SiC and Si in SiCp/A390 composites is shown in figure 3(a). It can be seen from figure 3(a), SiC/Al interface is clear, there is no obvious interfac reactant generation, the interface with a good, no hole defects. By plotting the black and gray parts in figure 3(a), the diffraction particles were calibrated to be SiC particles and Si particles, as shown in figures 3(b)–(c).

3.2.2. Interfacial microstructure between SiC and Si in SiCp/A390 composites

Figure 4(a) shows an interfacial HRTEM image between SiC and Si. It can be seen from figure 4(a), the atomic arrangement between SiC particles and Si particles is also very close, good combination, no reactants generated. Through the diffraction pattern analysis of SiC/Al interface, the electron diffraction pattern of the composites between SiC and Si is shown in figure 4(e). As can be seen from the diffraction pattern, the interface between SiC and Si has the crystallographic orientation matching relationship, which is determined as follows:

\[ \begin{align*}
010_{\text{SiC}} & \parallel 001_{\text{Si}}, & (004)_{\text{SiC}} & \parallel (220)_{\text{Si}}
\end{align*} \]

Using the Digital Micrograph software, HRTEM image of the original interface is processed in the 1, 2, 3 box region in figure 4(a). After Fourier transform (FFT) and inverse Fourier transform (IFFT), the arrangement of the atomic structure of SiC particles, Si particles and the SiC/Al interface can be more clearly observed, as shown in figures 4(b)–(d). Figure 4(b) shows the crystal structure of SiC particles in the crystal axis [010], which is a hexagonal structure. Figure 4(c) shows the crystal structure of the Si-phase in the crystal axis [001], which is a face-centered cubic structure. The microstructure of SiC/Al interface after Fourier transform (FFT) and inverse Fourier transform (IFFT) is shown in figure 4(d). It can be seen from the figure 4(d) that the (004) crystal plane of SiC particles is parallel to of the (220) crystal plane of Si phases, which further demonstrates the crystallographic orientation relationship of SiC/Al interface. The interplanar spacing of SiC on lattice plane (004) is 0.251 nm, while the interplanar spacing of the Si phase on lattice plane (220) is 0.192 nm. According to the formula of mismatch degree, the lattice mismatch degree between them is 0.23, which also indicates the semi-coherent interface between Si phases and SiC particles. This semi-coherent interface can promote the bonding strength of SiC/Al interface in SiCp/A390 composites, and can effectively transfer the load and improve the mechanical properties of the composites.

Figure 3. TEM image of interface of SiC/Si and corresponding electron diffraction pattern. (a) TEM image of interface of SiC/Si; (b) Electron diffraction pattern of Si phase; (c) Electron diffraction pattern of SiC particle.
3.3. Interface features between Si and Al matrix

3.3.1. Interfacial morphology between Si and Al matrix

In this experiment, the matrix material A390 is a hypereutectic Al-Si alloy, which possesses a high content for silicon. During sintering of the composite, Si elements are precipitated from the supersaturated matrix, forming a large number of silicon particles. So there are many interfaces between Si phase and Al matrix in the composite. Therefore, it is necessary to in-depth study the interfacial structure between the Si phase and Al matrix.

Figure 5 shows TEM image of interface between the Si phase and Al matrix. It is clearly seen at the interface between Si phase and Al matrix, and no reaction product has been found at the interface. It indicates that the interfacial bonding between Si phase and Al is good.

3.3.2. Interfacial microstructure between Si and Al matrix

In this research, HRTEM image of interface between silicon phase and Al matrix in the composite is shown in Figure 4(a). The IFFT images of three square areas 1, 2, and 3 in Figure 6(a) are respectively corresponding to Si phase along [001] zone axis, Al matrix along [011] zone axis and Si/Al interfaces. Figures 6(b)–(d) are respectively the corresponding indexed patterns of Si phase and Al matrix and Si/Al interfaces. It can be seen more clearly observed the conditions of the arrangement of atoms and the interfacial binding from the IFFT image which is shown in Figures 6(b)–(d). Si phase is cubic structure of polycrystalline diamond-like, which has close-packed {220} plane and the close-packed {100} direction, as well as the lattice constant of \( a = b = c = 0.543 \) nm. Al is face-centered cubic structure, which possesses close-packed {111} plane and the close-packed {011} direction, as well as the lattice constant of \( a = b = c = 0.405 \) nm. In the process of silicon particles precipitation from the composite, it is possible for Si particles within the matrix to adjust itself with the lowest energy, and then forming a certain crystallographic orientation relationship between Al matrix and Si particles.

Figure 6(e) shows the diffraction pattern and the corresponding index calibration between Si particles and...
Al matrix. As can be seen from the diffraction pattern, the interface between Si particles and Al have the crystallographic orientation matching relationship, which is determined as follows:

\[
\begin{align*}
01\bar{1} & \parallel 001, \\
(111) & \parallel (400).
\end{align*}
\]

As can be seen from figures 6(a)–(c), it can be clearly observed that the lattice plane (400) of Si particles is parallel to the lattice plane (111) of the Al matrix, which further demonstrates the crystallographic orientation relationship between Si phase and Al matrix under this experimental conditions. As can be seen from

Figure 5. TEM image of interfacial morphology between the Si phase and Al matrix.

Figure 6. HRTEM image of interface between Si phase and Al matrix and SAED pattern of interface after aging at 180 °C for 6 h. (a) Initial HRTEM image of interface; (b)–(d) IFFT images of square areas 1~3 in figure 6(a); (e) Electron diffraction pattern of interface.
the interplanar spacing of the matrix Al on lattice plane (111) is 0.234 nm, while the twice interplanar spacing of the Si phase on lattice plane (400) is 0.275 nm. Thus, the interplanar spacing of the matrix Al on lattice plane (111) is close to the twice interplanar spacing of the Si phase on lattice plane (400). According to the formula of mismatch degree, the lattice mismatch degree between them is 0.149, which also indicates the semi-coherent interface between silicon particles and Al matrix. Such interfacial structure which is beneficial for plastic and corrosion of the composite inhibits the formation and extension of microcracks in the composites. To some extent, it overcomes some inherent disadvantages of the SiCp/A390 composite [18].

4. Conclusions

(1) The interface of SiCp/A390 composite prepared by powder metallurgy is clean, without interfacial reactants and void defects. The interface between SiC and the Al matrix has an orientation relationship of $[4510]\text{SiC}||[011]\text{Al}, (1010)\text{SiC}||\{111\}\text{Al}$ and forming a semi-coherent relationship, which is beneficial to mechanical properties;

(2) The SiC/Si interface in SiCp/A390 composites prepared by powder metallurgy has the following crystal relationship: $[010]_{\text{SiC}}||[001]_{\text{Si}}, (004)_{\text{SiC}}||(220)_{\text{Si}}$;

(3) The interplanar spacing of the matrix Al on lattice plane (111) is close to the twice interplanar spacing of the Si phase on lattice plane (400). The interfacial bonding between silicon particles and Al matrix is semi-coherent interface. The interface between Si phase and Al matrix forms a certain crystallographic orientation matching relationship, which is determined as follows: $[01\bar{1}]_{\text{Al}}//\{001\}_{\text{Si}}$, $(111)_{\text{Al}}//\{400\}_{\text{Si}}$.

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ORCID iDs

Aiqin Wang  @  https://orcid.org/0000-0002-6337-8428

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