Properties investigation and microstructures characterization of SiCp/6061Al composites produced by PM route

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Abstract. In this study, 35 vol.% SiC particles with different sizes reinforced 6061 aluminium alloy matrix composites were prepared by a powder metallurgy method. The Scanning Electron Microscope (SEM) images of composites were observed, the Coefficient of Thermal Expansion (CTE) and tensile strength of composites were examined, and the influences of SiC particle size on microstructures and properties of the composites were analyzed. Furthermore, the SiCp/6061Al composites with SiC particle size of 7.5 µm were selected to investigate the SiCp/Al interface microstructure and precipitated phases by the means of SEM, TEM and HRTEM. The study indicated that, with the increase of SiC particle size, the SiC particles distributed more uniformly in the matrix, the CTE of composites increased, but the tensile strength of composites decreased. The SiCp/Al interface in this experiment is clean and smooth, and the combination mechanism of SiC and Al is the formation of a half coherent interface by closely matching of atoms. Some micron-sized coarse intermetallic particles existed in the hot-pressed composites, such as random-shaped Mg2Si, long stick shaped Al13(Mn, Fe, Cu)3Si2. When the composites were solution treated at 510 °C for 2 h and then aging treated at 190 °C for 9 h, except long stick shaped Al13(Mn, Fe, Cu)3Si2, numerous nano-sized precipitated phases (Mg2Si) with diameters of 50-200 nm dispersively distributed in the matrix. After heat treatment, the tensile strength of composite with SiC particle size of 7.5 µm enhance from 298 MPa to 341 MPa.

1. Introduction
Owing to their high specific strength and elastic modulus, high wear resistance and low CTE values, SiC particles reinforced aluminium matrix composites have received significant attention over the past few decades and are attractive as candidate materials for aerospace and automotive applications [1-5]. The SiCp/Al composites can be prepared by the general fabricating methods, such as powder metallurgy (PM), squeeze casting, pressural or pressureless infiltration and spray forming, etc [6]. Powder metallurgy is considered as a good technique in producing aluminium matrix composite, due to its lower processing temperature compared to melting techniques. Therefore, interaction between the reinforcement and matrix is prevented. Moreover, powder metallurgy technique has the ability to manufacture near net shape product with low cost [7-8].

It is commonly recognized that both the particle amount and particle size have obvious effect on the properties of the composites [9-10]. The voids coexisted with the clustered SiC particles and around the large sized SiC particles can be treated as pre-existed cracks. These particles cannot
transfer any load from soft matrix to the hard reinforcements, and thus lead to the degraded mechanical properties of the composites. Hardness, compression strength and wear resistance of the composites can be improved by proper addition of the reinforcement. For a certain particle volume fraction, there is a close relationship between the particle size and the properties of composite.

As the load transfers from the matrix to the reinforcements during the deformation process, the interfacial microstructures between SiC and Al are critical to control the composite mechanical properties, it is accepted that good bonding interfaces with coherency or semi-coherency are favorable to the mechanical properties, whereas interfaces with incoherency, especially those with the presence of brittle intermetallic phases at the interfaces, degrade these properties [11-12]. Hence, interface microstructures are of great scientific and technological interest. It is important to characterize the interface microstructure in order to understand the interaction between reinforcement and matrix during processing and to establish the relationship between microstructure and mechanical properties.

For 6xxx aluminium alloy matrix composites, the solid solution strengthening and precipitation hardening treatment are imparted to this alloy increase its strength. Mg and Si are the major alloying elements in this alloy, form precipitates through T6 treatment, which increases the strength of this alloy [13]. Edwards et al. [14] reported that the precipitation sequence in Al 6061 alloy is as follows: Supersaturated solid solution $\rightarrow$ GP Zones $\rightarrow$ $\beta''$ $\rightarrow$ $\beta'$ $\rightarrow$ $\beta$ (Mg$_2$Si) phase. In addition, several kinds of constituent phases have been found in 6061Al alloy [15]. These include Al$_{12}$Fe$_3$Mn$_2$Si$_2$ phase (BCC structure with $a$ = 1.23 nm), Al$_{13}$Fe$_4$Si phase (BCC structure with $a$ = 1.25 nm), which were proved that cannot dissolve during the solid solution.

In this study, 35 vol.% different sizes of SiC particles reinforced 6061 aluminium alloy matrix composite were produced using the PM technique. Our paper will be organized as follows. Section 2 will give the experimental materials and process. Section 3.1-3.3 will give a detailed research on the effect of particle sizes on the SiCp distribution, physical and mechanical properties. The microstructure and combination mechanism of the interface between SiCp and Al are investigated by the means of TEM and HRTEM and presented in the section 3.4. Finally, section 3.5 will research the shape, size and phase composition of alloy phases in the composites hot pressed and treated by a proper T6 heat treatment (solution treated at 510 °C for 2 h+ aging treatment at 190 °C for 9 h).

2. Experimental procedures

2.1. Experiment materials

SiC particles (mean particle size 40 µm, 25 µm, 15 µm, 7.5 µm) and gas atomized 6061 aluminium powders with average particle size of 10 µm were used as reinforcement and matrix, respectively. The chemical composition of 6061 aluminium was listed in table 1. The SEM images of SiC particle (mean particle size 7.5 µm) and 6061 aluminium matrix powder are respectively shown in figure 1(a) and figure 1(c). We can see that the SiC particles are angular in shape and 6061Al powders are close to spherical in shape. XRD patterns of SiC particle in figure 1(b) show that the SiC has predominantly the 6Hα poly-type and also contain few cubic β-SiC and 15R-SiC. It also can be obtained from the figure 1(d) that only Al diffraction peak exist in the pattern of 6061Al, which indicates that the alloy elements, like Mg, Si, Fe, are all supersaturated in the Al matrix in the process of gas atomization due to the extremely rapid cooling rate.

| Alloy   | Cu  | Mg  | Si  | Fe  | Al  |
|---------|-----|-----|-----|-----|-----|
| 6061Al  | 0.25| 1.0 | 0.6 | 0.3 | Bal.|

Table 1. Chemical compositions of 6061 aluminium matrix alloy (wt. %).
2.2. Experiment process
In this research, the aluminium matrix composite was manufactured by powder metallurgy technique. The powders (35 vol.% SiC particles) and balls with the ratio of 2:1 were blended in the Y style mixer for 24 h at the revolving speed of 50r/min and then the mixed powders were putted into the mould and hot-pressed up to 580 °C at 8 °C/min in the VDBF-250 experiment machine with the vacuum pressure of 2.3×10⁻³ Pa. The stress of 80 MPa was applied in the powders for 3 h at 580 °C and then the powders were cooled in the furnace with the protection of vacuum. When the temperature drops to room temperature, the stress was removed and composite was acquired.

The tensile tests of composites were conducted using the Shimadzu AG-I250KN precision universal testing machine at a constant crosshead speed of 1 mm/min. The tensile property data for each condition was the average from the observations of four specimens. Figure 2 shows the sizes of tensile specimens. The CTE tests were carried out on a PCY-III dilatometer system from 25 to 100 °C with a heating rate of 5 °C/ min. To eliminate system errors, the dilatometer was calibrated by measuring a standard alumina specimen under identical conditions. The metallographic samples were ground and polished following standard metallographic practices, then were etched using Keller’s reagent. The microstructure was examined in scanning electron microscope (JSM-5610LV). The samples for TEM observations were machined into 0.5 mm by wire-electrode cutting and ground to 50 μm by Mechanical thinning, and then were cut into 3 mm diameter foils. After that, foils were prepared by argon ion milling using Gatan 691 Precision Ion Polishing System. Transmission electron microscopy (TEM) investigations were performed on JEM-2100 TEM microscope operated at 200 kV.

3. Experimental results
3.1. SEM microstructure of composite with different particle sizes
Figure 3 shows the SEM images of composites containing 35 vol.% SiC with different particle sizes of 40 μm, 25 μm, 15 μm, 7.5 μm. Grey regions imply Al matrix and dark grey and cornered particles...
imply the reinforcement SiC. It can be observed that the SiC particles with 40 μm are well dispersed and uniformly distributed in Al matrix, but the quantity of SiC in the composites become more and more as the decrease of SiC particles sizes and the distribution of SiC become more nonuniform. Slight SiC particle clustering can be observed in the composites with the particle size of 7.5 μm. It is well known that if the ratio of matrix/reinforcement particle sizes is much smaller, the reinforcements can cluster very easily in the matrix and result in an inhomogeneous distribution of reinforcements.

![Figure 3](image)

**Figure 3.** SEM images of SiC/6061Al composites with different SiC particle size: (a) 40 μm SiC; (b) 25 μm SiC; (c) 15 μm SiC; (d) 7.5 μm SiC.

3.2. *Thermal expansion analysis of composite with different particle sizes*

Figure 4 is the CTEs of SiCp/6061Al composite with four types of particle size measured between 25 °C and 100 °C, it is seen that all the linear expansion coefficients of composites rise with the enhancement of temperature. Meanwhile, the decrease of SiC particle size can significantly reduce the CTE of composite. Furthermore, with the increase of temperature, composites with smaller particle size possess the smaller increase speed of linear coefficient expansion.

![Figure 4](image)

**Figure 4.** The CTEs of SiCp/6061Al with different particle sizes tested between 25 °C and 100 °C.
it will relax in the form of matrix plastic flow and the particle dislocation density around the particle will thus increase. Arsenault and Shi [16] establish a dislocation density prediction model:

\[
\rho = \frac{6f(\alpha_p - \alpha_m)\Delta T}{b(1-f)d}
\]

Where \( b \) is the burgers vector of matrix, \( d \) and \( f \) is respectively the diameter and volume fraction of reinforcement, \( \alpha_p \) and \( \alpha_M \) is the CTE of reinforcement and matrix, respectively. We can obtain the inverse relationship between dislocation density and particle size from equation (1).

According to Taylor dislocation strengthening relations [17], the increase of dislocation density would cause the in-situ yield strength of matrix in the composite is greater than that of matrix without the reinforcement. The in-situ yield strength of matrix in the composite can be described as follows:

\[
\sigma_{my} = \sigma_{m0} + \alpha \mu_m b \sqrt{\rho CTE}
\]

Where \( \sigma_{my} \) is the in-situ yield strength of matrix in the composite, \( \sigma_{m0} \) is the yield strength of pure matrix. \( \alpha \) is a constant which is 1.25 for Al, \( \mu_m \) is the shear modulus of matrix. From equation (2), we can obtain that the dislocation density of matrix would gradually increase with the decrease of the particle size, and then the in-situ yield strength of matrix would increase. The thermal expansion nature of composites can be viewed as increases of average distance between lattice structure with temperature rise and the micro plastic relaxation in the matrix during heating process. So there will be the following equation:

\[
\Delta L = \Delta L_a + \Delta L_p
\]

Where \( \Delta L \) is the total elongation of composite during the process of heating, \( \Delta L_a \) is the natural temperatures change volume, \( \Delta L_p \) is the elongation caused by the plastic relaxation.

As we discussed above, the in-situ yield strength of matrix would increase as the decrease of the particle size, and thus the thermal mismatch stress is not easy to loose. As a result, the coefficient thermal expansion of composite decreases as the particle size decreases. When the particle is at a certain volume, the smaller the particle size is, the more the number of particle and the smaller the particle spacing are, and thus the expansion of the matrix can be effectively prevented under high temperature. So the smaller particle size can significantly reduce the increase rate of thermal expansion coefficient.

### 3.3. The mechanical properties analysis of composite with different particle sizes

The effect of the SiC particle size on the tensile strength of composites is shown in the figure 5. Considering this figure, by increasing the particle size of SiC from 7.5 μm to 40 μm, the tensile strength of composites decreased from 298 MPa to 212 MPa. Although the particle distribution of composites with the 40 μm SiC particle is the most uniformly, it is regretful that the tensile strength is the lowest. Apparently, the tensile strength of SiCp/6061Al composites has a close relationship with particle size.

Strengthening mechanisms of the metal matrix composites could be classified into direct and indirect mechanisms [10]. Direct strengthening is obtained by the load being transferred from the weaker matrix to the hard reinforcement during deformation, while indirect strengthening results from the variation of the matrix microstructures (such as Orowan strengthening mechanism, grain refinement, dislocation multiplication, etc.) in the composites by the addition of the reinforcements. In view of the above analysis, the influence mechanisms of particle size on the tensile strength of composites can be summed up as follows: Firstly, at a certain volume fraction of reinforcements, smaller size of SiC particle can bring a larger quantity of interface area, which can significantly enhance the capacity of transferring load from matrix to particle. Secondly, we have discussed in section 3.2 that composite with smaller particle size would possess a higher dislocation density, which can effectively strengthen the matrix; thereby the tensile strength is improved. Thirdly, the particle with larger size is more susceptible to contain defects. The cracked particles in the composites cannot transfer any load since the stress is released along the cracking surfaces. Furthermore, the cracked
particles will act as the micro-crack initiators during deformation, leading to the decrease in the strength of the composites.

![Graph showing tensile strength vs SiC particle size](image)

**Figure 5.** The tensile strength of SiCp/6061Al composites with four types of particle sizes.

### 3.4. Microstructure of the interface between SiC and Al

#### 3.4.1. TEM characterization of SiC/Al interface.

Figure 6(a) shows the TEM images of SiC/Al interfaces of the composites. A typical SiC/Al interface has been obtained. We can see from the figure that the interface is clean and smooth; there are no interface reactants and phenomenon of SiC particles dissolved. Previous studies [18] have shown that SiC particles are easy to react with Al to generate Al₄C₃ in the SiC particles reinforced aluminium alloy matrix composites. But we didn't find Al₄C₃ reactants in the composite materials in this work. It is mainly because the hot-press sintering technology was used in this experiment, the temperature throughout of which is too low to produce interface chemical reaction. So the interface of the composite prepared is clean, and the combining is good. It means that the external load can transfer from matrix to reinforcements and provides the prerequisites for the composite material with excellent performance. Moreover, figure 6(a) also displays the feature of high-density dislocations in the Al matrix adjacent to a very pointed SiC particle. The difference in the thermal expansion coefficients between the matrix and SiC particles necessitates the generation of dislocations in the matrix vicinal to SiC particles to accommodate thermal strain when change temperature. Figure 6(b) and figure 6(c) are the diffraction pattern of 6H-SiC in the zone axis [1210] and the diffraction pattern of Al in the zone axis [112].

![TEM images of the interface between SiC and Al](image)

**Figure 6.** TEM images of the interface between SiC and Al: (a) the interface morphology of SiC/Al composites; (b) the diffraction pattern of SiC; (c) the diffraction pattern of Al.

#### 3.4.2. HRTEM characterization of SiC/Al interface.

Figure 7(a) shows an initial high resolution image of the interface between the reinforcement particle and the matrix. Figure 7(b) and (c) are
corresponding SAED and indexed patterns. In figure 7(c), the circles represent the reflections from the matrix and the dots represent those from SiC particles. The incident beam is parallel with [121] Al and [1210] 6Hα-SiC, the (111) plane of the matrix Al is parallel to the plane (0006) of the SiC phase. Thus, their orientation relationship is determined as follows: [1210] SiC || [121] Al, (0006) SiC || (111) Al.

We can intuitively and clearly observe the atomic arrangement structure by the Inverse Discrete Fourier Transform (IFFT) of initial HRTEM image. The IFFT images of 6H SiC phase along [1210] zone axis and Al along [121] zone axis are respectively shown in figure 7(d) and figure 7(e). We can see from figure 7(d) that the interplanar spacing of (0001) plane in 6Hα-SiC phase is 1.51 nm and each (0001) plane consist of six layers. Figure 7(f) is the IFFT image of square area 3 in figure 7(a) and we can intuitively and clearly observe the atomic arrangement of the interface between SiC and Al. The (111) plane of Al is parallel to the plane (0006) of SiC phase, the interplanar spacing of (111) planes in the matrix is 0.234 nm, while the interplanar spacing of (0006) planes in α-SiC phase is 0.251 nm. The lattice misfit between them is 0.07 and it is obviously that the combination mechanism of SiC and Al is the formation of a semi-coherent interface by closely matching of atoms. This semi-coherent interface can effectively transfer the load from matrix to SiC ceramic particles and provides the prerequisites for the composite material with excellent performance.

Figure 7. HREM images, corresponding SAED and IFFT images of the interface between SiC and Al: (a) The initial HREM image of interface between SiC and Al; (b) Corresponding SAED patterns of interface; (c) Indexed patterns; (d) IFFT image of square area 1 in Figure 6(a); (e) IFFT image of square area 2 in Figure 6(a); (f) IFFT image of square area 3 in Figure 6(a).

3.5. The evolution of alloy phases in the composite during heat treatment

3.5.1. Characterization of alloy phases in hot-pressed composites. When composites were prepared by vacuum hot pressing, the microstructure of alloy phases are especially complex by the reason that different kinds of alloying elements contained in the aluminium matrix can form various kinds of coarse precipitated phases at the coupled action of heat and stress. Considering the important role alloy
phases played in the strengthening of composites, it is of practical importance to understand the microstructure of alloy phases in the hot-pressed and the evolution in the process of heat treatment. It can be seen from figure 8(a) that there is a considerable volume fraction of micron coarse intermetallic phases in the hot-pressed composite. The high brightness of these particles is due to the higher atomic number of Mg and Cu in comparison with that of Al, Si and C.

In order to clearly understand the morphological characteristics and phase composition of coarse intermetallic phases in the hot-pressed composites, TEM analysis was conducted, the TEM morphologies and corresponding diffraction patterns of coarse intermetallic particles were shown in figure 8(b)-(f). It can be found that two kinds of coarse intermetallic particle existed in the hot-pressed composites. The shape of coarse intermetallic particle in figure 8(b) is less regular and the size is about 3-5µm. Diffraction patterns from the coarse particle (see figure 8c) was indexed to be consistent with Mg2Si(Space Group: Fm-3m, lattice parameters: a= 6.350 Å), which is considered the main strengthening phase in the 6000 series of Al alloys.

As shown in the figure 8(d), a spinel phase in the form of 100-300 nm size is present in the matrix, corresponding EDS, chemical composition and indexed patterns are presented in the figure 8(e) and figure 8(f). The EDS spectrum and chemical composition in figure 8(e) of the spinel phase reveal that they are Al-, Fe-, Mn-, Cu- and Si-rich with an atomic ration of Al;(Fe, Mn, Cu):Si≈15.1:3.26:2. By ignoring the experimental uncertainty, the phase can be written as Al15(Mn, Fe, Cu)3Si2, which was confirmed to have a BCC structure with lattice parameter a=1.28 nm. This result was consisted with the research of Wang and Cheng [19-20].

3.5.2. Characterization of alloy phases in composites after T6 heat treatment. When the composites were treated by a proper T6 heat treatment (solution treated at 510 °C for 2 h+ aging treatment at 190 °C for 9 h), the TEM images of alloy phases is shown in the figure 8. It can be seen from the figure 9(a) that a spinel phase about 200 nm appeared in the matrix, which is analogous to the phase shown in the figure 8(d). Further calibration of diffraction pattern shown in figure 9(b) confirmed the phase is
Al$_{15}$(Mn, Fe, Cu)$_3$Si$_2$. The shape and composition of phase in figure 9(a) are consonant with the result shown in figure 8(d). It can be obtained that the Al$_{15}$(Mn, Fe, Cu)$_3$Si$_2$ is mainly formed during the hot pressed sintering process and cannot dissolve in the solution process, due to the fairly low solubility of Mn and Fe elements in Al matrix. Figure 9(c) shows a TEM image of numerous nano-sized β′-Mg$_2$Si precipitated phases with diameters of 50~200 nm homogeneously distributed in the matrix.

As discussed above, the alloy phases in the hot-pressed composites are mainly micron coarse intermetallic particles, while in the aged composites are nano-sized precipitated phases. Compared with the coarse micron intermetallic phases in the hot-pressed composites, the precipitated phases in the aged composites are smaller in size and more in the quantity. Because the dislocation can be more effectively block by numerous nano-sized precipitated particles than slight micron coarse intermetallic particles, heat treatment is an effective way to enhance the mechanical properties of SiCp/6061Al composites. The strengthening effect is testified in this experiment that the tensile strength of composite with 7.5 μm particle size enhanced from 298 MPa to 341 MPa after heat treatment.

![Figure 9](image)

**Figure 9.** TEM analysis of alloy phases in the SiCp/6061Al composites after T6 heat treatment: (a) TEM image of long stick phase; (b) Diffraction pattern of long stick phase; (c) TEM image of dispersed phase.

4. **Conclusions**

Through this study, following conclusion could be achieved:

1. The SiC particles distribute more uniformly in the matrix by increasing the particle size, but there is an inverse relation between tensile strength and particle size. The tensile strength of composites with 7.5 μm exhibited highest tensile strength 298 MPa.

2. All the linear expansion coefficients of composites rise with the enhancement of temperature. Meanwhile, the decrease of SiC particle size can significantly reduce the expansion coefficients of composites. Furthermore, with the increase of temperature, composites with smaller particle size possess the lower increase speed of linear expansion coefficients.

3. The SiCp/Al interface in this experiment is clean and smooth, and SiC particles are well combined with Al matrix. The combination mechanism of SiC and Al is the formation of a half coherent interface by closely matching of atoms.

4. Some micron-sized coarse intermetallic particles existed in the hot-pressed composites, such as random-shaped Mg$_2$Si, long stick shaped Al$_{15}$(Mn, Fe, Cu)$_3$Si$_2$. After heat treatment, except long stick shaped Al$_{15}$(Mn, Fe, Cu)$_3$Si$_2$, numerous nano-sized precipitated phases (Mg$_2$Si) homogeneously distributed in the matrix. After heat treatment, the tensile strength of composite with SiC particle size of 7.5 μm enhance from 298 MPa to 341 MPa.

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