Effect of Reinforcement Size on Mechanical Behavior of SiC-Nanowires-Reinforced 6061Al Composites

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Abstract: In the present study, the effects of SiC nanowires (SiCnws) with diameters of 100 nm, 250 nm and 450 nm on the microstructure and mechanical behavior of 20 vol.% SiCnws/6061Al composites prepared by pressure infiltration were studied. It was found that the interface between SiCnws and Al matrix was well bonded, and no interface product was found. The thicker SiCnws are beneficial to improve the density. In addition, the bamboo-like and bone-like morphologies of SiCnws produce a strong interlocking effect between SiCnws and Al, which helps to improve the strength and plasticity of the material. The tensile strength of the composite prepared by SiCnws with a diameter of 450 nm reached 544 MPa. With a decrease in the diameter of SiCnws, the strengthening effect of SiCnws increases. The yield strength of SiCnws/6061Al composites prepared by 100 nm is 13.4% and 28.5% higher than that of 250 nm and 450 nm, respectively. This shows that, in nano-reinforced composites, the small-size reinforcement has an excellent improvement effect on the properties of the composites. This result has a guiding effect on the subsequent composite structure design.

Keywords: SiC nanowires; Al matrix composite; mechanical characterization; interface

1. Introduction

SiC nanowires have the advantages of high strength, strong oxidation resistance, good wear resistance, wide band gap, excellent electromagnetic wave absorption performance, and high temperature stability. They have potential applications in field emitters, electromagnetic wave absorbers, microelectronics and light emitters, ceramics and metal composites [1–4]. Studies have shown that polymer [5,6], ceramic [7–10] and C/C-based [11,12] composites reinforced by SiC nanowires (SiCnws) exhibit higher strength, toughness and thermal shock resistance.

Currently, the use of SiCnws-reinforced metal matrix composites has also been reported. Li et al. [13] prepared 0.3 wt.% SiCnws/Mg-2Zn-0.1Y composite, and the yield strength and tensile strength of the material reached 495.53 MPa and 514.18 MPa, respectively. Zhang et al. [14] added SiCnws with a diameter of 100–600 nm to AZ91D. The results showed that SiCnws had a good combination with the Mg matrix, and the yield strength of the material increased by 33%. Liu et al. [15] reported that the addition of 0.5 wt.% SiCnws (100–200 nm in diameter) to the Ti matrix increased the tensile strength by 52% due to grain refinement and the bridging, drawing and fracture of SiCnws. Li et al. [16] found that the fracture toughness of 20 wt.% SiC whisker (diameter 1.5 µm) reinforced SiC composites increased by 35.5% compared with the matrix. K. Komai et al. [17] prepared SiCnws/7075Al composites (SiCnws diameter range and average size are 0.1–1 µm, 0.5 µm, respectively) by powder metallurgy method. The results show that the properties of...
SiCnws/7075Al composites are higher than those of the matrix aluminum alloy except for the elongation of fatigue fracture. Jintakosol et al. [18] prepared 5–15 vol.% SiCnws/Al composites, indicating that the wear resistance of the material increases with the increase in SiCnws content.

Our group reported in detail the microstructure and mechanical behavior of SiCnws (diameter distribution 50~800 nm, average diameter ~220 nm) reinforced pure aluminum [19,20], 6061Al [21–23] and 2024Al [24] matrix composites. The results show that the interface between SiCnws and Al exhibits good interfacial bonding, and there are no Al₄C₃, Al₂O₃ and MgAl₂O₄ products at the interface [20,25]. The composites show good machinability, the grain of Al matrix in 15 vol.% SiCnws/6061Al composites is obviously refined, and the material shows high strength while retaining the plasticity of aluminum [23]. Adin et al. [26,27] found that the type, content and morphology of reinforcements have an important influence on the properties of composites. When the average size of SiCnws is the same and the content of bamboo-like SiCnws increases from 14.6% to 45.3%, the tensile strength and elongation increase by 8% and 57%, respectively [22]. It is well known that the strengthening effect of particle-reinforced composites is closely related to the size of the reinforcing phase. The smaller the particle size, the more obvious the strengthening effect. In nano-reinforced composites, when the size of the nano-reinforcement is close to the microstructure, such as dislocations and grain boundaries in the matrix, a new phenomenon that is rare in traditional micron reinforcements can be produced [28–33]. However, the influence of diameter size on the strengthening behavior of nanowires-reinforced metal matrix composites is not clear, and the structural design and preparation of composites with different diameters lack theoretical guidance. At the same time, for the reinforced phase with a size of 1–100 nm, there are few reports on the preparation of composites with a volume fraction greater than 10% [13,15,34–37].

In this study, SiCnws with an average diameter of 100 nm, 250 nm and 450 nm were used as reinforcements to study the effect of nanowire size on the microstructure and mechanical behavior of 20 vol.% SiCnws/6061Al composites prepared by pressure infiltration.

2. Materials and Methods

2.1. Materials and Composite Fabrication Process

In this experiment, SiC nanowires with average diameters of 100 nm, 250 nm and 450 nm were used as raw materials, abbreviated as 100D, 250D and 450D (where ‘D’ represents diameter). SiCnws of 100D were prepared using the thermal evaporation of silicon powder method, in which silicon powder (≥99.0%, <5 μm, Beijing Xing Rong Yuan Technology Co., Ltd., Beijing, China) was used as the silicon source, high-purity graphite substrate was used as the carbon source, and SiCnws were obtained by heating to 1350 °C under Ar protective atmosphere. SiCnws of 250D and 450D were provided by Changsha Snett Advanced Materials Co., Ltd. in Changsha, China. The 6061 aluminum alloy was provided by Northeast Light Alloy Co., Ltd., in Harbin, China. The chemical composition of 6061Al alloy is 0.24 wt.% Cu, 1.01 wt.% Mg, 0.15 wt.% Mn, 0.70 wt.% Fe, 0.64 wt.% Si, 0.25 wt.% Zn, 0.29 wt.% Cr and else Al.

The manufacturing process of SiCnws/6061Al composites is similar to that of previous composites. Composites are prepared by pressure infiltration technology [24]. As shown in Figure 1, the composites obtained by using 100D, 250D and 450D SiCnws are named 100D SiCnws/6061Al, 250D SiCnws/6061Al and 450D SiCnws/6061Al, respectively. The SiCnws was put into a steel mold and further pressed to prepare the preforms, then was heated to 550 °C at a rate of 5 °C/min. The volume content of the milled particles in the mold was about 60 vol%. The preheating temperatures of preform and graphite indenter are 550 °C and 780 °C, respectively. The 10 MPa pressure was maintained for 7 min during the infiltration process. Prior to mechanical testing, the specimens were subjected to peak-aging treatment (530 °C/1.5 h solution, quenched in water, 175 °C/6 h aging).
2.2. Microstructure Characterization and Mechanical Properties Testing

The microstructure and morphology of the raw material SiCnws were characterized by a field emission scanning electron microscope (SEM, HELIOS NanoLab 600i). Field emission transmission electron microscopy (TEM, JEM-2010F, JEOL, Tokyo, Japan) was used to observe the microstructure of the composites. X-ray diffractometer (XRD, Rigaku D/max-rB) to detect phase composition, using Cu-Ka radiation for identification, scanning angle of 25° to 90°, scanning speed of 3°/min, tube voltage 40 Kv, tube current 40 mA. The polished samples were etched with 10 vol.% NaoH for 60 s and then observed by SUPRA55 scanning electron microscope (SEM, Zeiss Corporation, Oberkochen, Germany).

At the same time, local composition analysis was performed by energy dispersive spectroscopy (EDX). The sample density was measured using the Archimedes principle. The mass of the samples placed in air and water was measured by a precision balance. The formula for calculating the density is as follows:

\[ \rho = \frac{m_{\text{Air}}}{(m_{\text{Air}} - m_{\text{H}_2\text{O}})/\rho_{\text{H}_2\text{O}}} \]

In order to improve the significance of the statistical results, five samples were tested each. By comparing the measured true density and the theoretical density of the sample, the relative density of the composite material is calculated. Tensile samples were tested on Instron 5569 universal electrical tensile testing machine, and the size of the tensile samples was shown in Figure 2. Finally, SEM was used to observe the fracture of the composites.

Figure 2. Structure diagram of tensile samples.

3. Results and Discussion

The morphology of the SiCnw is characterized by SEM, as shown in Figure 3. The length and diameter of the 100D SiC nanowires range from 50 to 100 μm (Figure 3a) and 40 to 250 nm (Figure 3b), respectively, with an average diameter of 100 nm (Figure 3c), exhibiting a curved morphology. The length and diameter distribution of 250D SiC nanowires are 10–80 μm (Figure 3d) and 50–700 μm (Figure 3e), respectively, and the average diameter...
is 250 nm (Figure 3f). The length and diameter distributions of the 450D SiC nanowires used are 5–50 µm (Figure 3d) and 50–1100 µm (Figure 3e), respectively, with an average diameter of 450 nm (Figure 3f).

Figure 3. SEM and diameter distribution images of SiCnws. (a,b,d,e,g,h) Morphology of 100D, 250D and 450D SiCnws, respectively; (c,f,i) Diameter distribution of 100D, 250D and 450D SiCnws, respectively.

Three kinds of SiC morphologies were observed. They are cylindrical with uniform diameter and a smooth surface (as shown by the white arrows in Figure 3b), bamboo-like with a rough surface of diameter fluctuation (yellow arrows in Figure 3e) and bone-like with a smooth surface of diameter fluctuation (orange arrows in Figure 3h). The surface roughness is related to the size of the nanowires; the larger the surface, the rougher it is. The surface of 100D is smoother, while the surfaces of 250D and 450D nanowires have more protrusions, which are bamboo-like and bone-like. Although nanowires have different morphologies, studies have shown that they all have the effect of inducing stacking faults [38]. It is worth noting that the proportion of each morphology is different. Figure 4 shows the statistics of the three morphologies in SiC nanowires with different diameters. In the nanowires with an average diameter of 100 nm, the proportion of cylindrical, bamboo and bone rod are 97.4%, 0.8% and 1.8%; the ratio of three morphologies of 250 nm is 22.4%, 45.5% and 32.1%; the morphology of 450 nm nanowires accounted for 15.1%, 30.1% and 54.8%, respectively. It is worth noting that there is a certain correlation between the morphology and diameter of SiCnws. When the diameter is small, it exhibits a smooth cylindrical surface, and when the nanowire diameter is large, it is easier to exhibit bamboo-like and bone-like surfaces [39–41].
Si3N4 whiskers can improve the relative density of (220), (311) and (222) crystal planes of a = 4.359 Å, and space group F-43 m) [42]. According to previous research [43], the diffraction peak at 33.7° is caused by stacking faults on the (111) surface of 3C-SiC. Chen et al. [44] evaluated the stacking fault density (X) by the intensity ratio of the SFs peak to the SiC (200) peak, as shown in Equation (1):

$$X = \frac{I_{SFs}}{I_{SiC \ (200)}}$$  \hspace{1cm} (1)

Figure 5 is the XRD pattern of SiC nanowires. There are diffraction peaks at 33.7°, 35.7°, 41.4°, 60.0°, 71.8° and 75.6°. Using the standard powder XRD pattern (JCPDS card), 35.7°, 41.4°, 60.0°, 71.8° and 75.6° correspond to 3C-SiC (JCPDS No.29-1129; (111), (200), (220), (311) and (222) crystal planes of a = 4.359 Å, and space group F-43 m) [42]. According to previous research [43], the diffraction peak at 33.7° is caused by stacking faults on the (111) surface of 3C-SiC. Chen et al. [44] evaluated the stacking fault density (X) by the intensity ratio of the SFs peak to the SiC (200) peak, as shown in Equation (1):

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The microstructure of SiCnws/6061Al composites is shown in Figure 6. A large number of small holes were found in 100D SiCnws/6061Al (orange dotted box in Figure 6a), which was due to poor wettability between SiC and Al [45]. Yin et al. [45] found that the amount of added β-Si3N4 whiskers and sintering temperature had a significant effect on the density of the composites. Zhang et al. [46] found that the surface modification of β-Si3N4 whiskers can improve the relative density of β-Si3N4w/Al composites. Feng et al. [47] electroless plated Cu on the surface of SiCp, which could greatly improve the wettability of SiCp and Al-Si alloys, and then the density of the composites was greatly improved. These results can be used to further improve the densification results of the material.
worth noting that, after NaOH etching, more aluminum adheres to the nanowires with rough surfaces (Figure 6d), while almost no residual aluminum is observed on the smooth nanowires (Figure 6f), which can be attributed to the difference in interface bonding and interface area [22].

The XRD patterns of typical SiCnws/6061Al composites are shown in Figure 7. In addition to the characteristic peak of SiC, there are diffraction peaks at 38.5°, 44.74°, 65.13°, 78.23° and 82.44°, corresponding to Al (JCPDS No.04-0787 (111), (200), (220), (311) and (222) planes of a = 4.0494 Å, and space group F-3 m).

Figure 6. The representative SEM microstructure of SiCnw/6061Al composites. (a,b) 100D SiCnws/6061Al, (c,d) 250D SiCnws/6061Al, (e,f) 450D SiCnws/6061Al.

In this study, as the diameter of the nanowires increases to 250 nm, the number of holes in the composite decreases (Figure 6c, orange virtual coil). When the diameter of SiCnws further increases to 450 nm, no holes are found (Figure 6e), indicating that the increase in the diameter of SiCnws increases the density of the composite. It can be observed in Figure 6b that nanowires are uniformly distributed in the composite, and the protrusions are uncorroded Al matrix (red box in Figure 6b) by EDX analysis. It is worth noting that, after NaOH etching, more aluminum adheres to the nanowires with rough surfaces (Figure 6d), while almost no residual aluminum is observed on the smooth nanowires (Figure 6f), which can be attributed to the difference in interface bonding and interface area [22].

The microstructure of SiCnws/6061Al composites was further observed by TEM, as shown in Figure 8. SiCnws with a diameter of 100 nm are uniformly and randomly distributed in Al (Figure 8a), and most of the SiCnws are located inside the Al grains (Figure 8b, yellow virtual coil). In the process of SiCnws/Al composites prepared by...
pressure infiltration, SiCnws are mainly distributed in the Al grains due to the rapid solidification of the Al matrix [24]. At the same time, the composite has a smaller grain size of about 200 nm to 1 µm, and there are a large number of tangled dislocations in the aluminum grains (Figure 8b). The nanophase (such as nanotubes, nanowires and nanoplates) inhibits the growth of the matrix grains and greatly improves the fine-grain strengthening effect of the composites [13,15,48,49]. Due to the large difference in thermal expansion coefficient between SiCnws and Al, a large number of dislocations are generated, and the pinning effect of SiCnws on the interface refines the grains of the Al matrix. At the same time, in the composites, smooth (Figure 8c), bamboo-like (Figure 8d) and bone-like (Figure 8e) SiCnws have good bonding with Al, the interface is clean and no interfacial reaction products are found.

![Figure 8. The TEM microstructure of SiCnws/6061Al composite. (a) Low magnification image of 100D SiCnws/6061Al, (b) SiC nanowires located within the Al grains in the 100D SiCnws/6061Al, (c) Smooth SiC nanowires in the 100D SiCnws/6061Al, (d) Bamboo-like SiC nanowires in the 250D SiCnws/6061Al, (e) Bone-like SiC nanowires in the 450D SiCnws/6061Al.](image)

The interfacial microstructure between SiC nanowires and the Al matrix was further observed by HRTEM, as shown in Figure 9. Selective electron diffraction patterns and their calibration results at the SiC/Al interface were given in Figure 9b,c, respectively. The calibration results showed that there were only diffraction spots of SiC and Al, which were similar to the results of micron SiC particles [50], nano SiC particles [51] and graphene [48] reinforced aluminum matrix composites prepared by pressure infiltration, and no interface products (such as Al₄C₃, SiO₂ or Al₂O₃) were found [52–55], which showed that SiC nanowires were not damaged during the preparation process.
The tensile curves of representative annealed and aged 20 vol.% 100D, 250D and 450D SiCnws/6061Al composites are shown in Figure 10. Compared with the 6061Al matrix, the strength of the composites has been greatly improved. With the increase in the diameter of the reinforced phase, the strength and plasticity of SiCnws/6061Al composites increase. SiCnws/6061Al composites after annealing show better plastic deformation than peak aging. The specific tensile properties and relative density of the composites are shown in Figure 11. Without considering the heat treatment, the fracture strength (Figure 11a), relative density and elongation (Figure 11b) increase with the increase in nanowire diameter. The hardness test results are shown in Figure 11c. It can be seen that the hardness of the composites increases with the increase in SiCnws diameter. With the decrease in the nanowire size, the infiltration of the composite material becomes more difficult, which inevitably leads to an increase in defects in the composite material. Since the peak-aged 100D SiCnws/6061Al composites fractured before yielding, the yield strength was not obtained. However, in the annealed state, the yield strength decreased with the increase in nanowire diameter.

The fracture morphology of 20 vol.% 100D, 250D and 450D SiCnws/6061Al composites is shown in Figure 12. Holes can be found in the fracture of 100D SiCnws/6061Al composites (Figure 12a, orange box). The fracture of nanowires is mainly divided into sites is shown in Figure 12. Holes can be found in the fracture of 100D SiCnws/6061Al composites (Figure 12a, orange box). The fracture of nanowires is mainly divided into sites is shown in Figure 12. Holes can be found in the fracture of 100D SiCnws/6061Al composites (Figure 12a, orange box). The fracture of nanowires is mainly divided into sites is shown in Figure 12.
composites (Figure 12a, orange box). The fracture of nanowires is mainly divided into three characteristics: negligible pull-out fracture (Figure 12b, orange circle), obvious pull-out fracture (Figure 12b, yellow box) and nanowire/Al debonding (Figure 12b, red oval), in which the pull-out fracture is mainly neglected. At the same time, a small amount of the Al matrix is observed on the smooth surface of SiCnws, and the fracture diagram is shown in Figure 12c. There are a few pores in the fracture of 250D SiCnw/6061Al composites (Figure 12d), and the fracture characteristics of nanowires in 100D SiCnw/6061Al are divided into three types (Figure 12e). However, due to the high content of bamboo-like and rod-like nanowires (45.5% and 32.1%, respectively), the fracture morphology of SiCnws is mainly characterized by the obvious pull-out fracture (Figure 12e, yellow box) and nanowire/Al debonding (Figure 12e, red ellipse). It is worth noting that there is still a large amount of adhesive Al on the surface of bamboo-like SiCnws, and the fracture diagram is shown in Figure 12f. No obvious holes were found in the fracture of 450D SiCnw/6061Al. 30.1% bamboo-like and 54.8% bone rod-like in the raw materials, making the fracture morphology of SiCnws mainly obvious pull-out fracture (Figure 12h, yellow box) and nanowire/Al debonding (Figure 12h, red ellipse). Similarly, a large amount of Al matrix is also bonded on the surface of bone rod-like SiCnws, and the fracture diagram is shown in Figure 12i.

**Figure 12.** Fracture morphology of SiCnw/6061Al composites prepared by SiCnws with different diameters. (a,b) 100 nm, (c) Schematic corresponding to (a–d) 250 nm, (f) Schematic corresponding to (d,e), (g,h) 450 nm, (i) Schematic corresponding to (g,h).

The properties of composites are closely related to density and interfacial bonding strength [37]. It can be seen that the 100D SiCnw/6061Al composite has the lowest density (93.6%). Therefore, in the mechanical properties test, more holes make the crack initiate rapidly. At the same time, because the interface between SiCnws and Al is mechanically bonded (Figure 9a), the bonding strength between SiCnws with a smooth surface and the aluminum matrix is weak, resulting in rapid instability and early fracture of the material (Figure 12c), showing low strength and low plasticity in mechanical properties. The density of 450D SiCnw/6061Al composites is the highest, reaching 98.9%. At the same time, due to the higher interlocking effect of the bamboo-like morphology than nanowires with a smooth surface, the average interface strength of 450D SiCnw/6061Al composites is higher than that of 100D SiCnw/6061Al composites, and the crack is difficult to deflect at the interface. Therefore, the former has a similar plastic shape to the latter, but has higher
strength. The improvement in plasticity and strength of 250D SiCnw/6061Al composites compared with 100D SiCnw/6061Al composites can be attributed to the improvement in interface bonding and the increase in density.

Previous studies have shown that SiCnWs with non-uniform diameters are more uniform than nanowires with uniform diameters, which can produce a strong interlocking effect with the matrix. SiCnws are not easy to pull out, which can make more effective use of the bearing effect of the reinforcement and help to improve the strength and deformation of the matrix [22,56]. For ceramic-reinforced composites, when the reinforcement size is large, cracks at the reinforcement or interface are easily initiated and propagated, resulting in a significant reduction in the ductility and toughness of the material [33,57]. As a result, 250D SiCnws/6061Al composite has better plasticity than 450D SiCnw/6061Al composite, which can be attributed to the higher content of bamboo-like nanowires in 250D SiCnws/6061Al composite. The interlocking effect at the interface is enhanced, and the smaller size makes the crack at the interface not easy to initiate and propagate.

The yield strength is usually less dependent on stress concentration and is more representative for discussing strengthening behavior. The improved shear-lag model can explain the strengthening behavior of SiCnws/Al composites well [20,24]. When the length of nanowires (l) is greater than the critical length (lc), where lc is usually 5–10 times the diameter of nanowires (d), the yield strength (σcy) of SiCnws/Al composites can be expressed as Equation (2):

\[ σ_{cy} = σ_{my} + 2ckg\frac{1}{d} \left(2 - \frac{l}{l_c}\right)τ_{max}V_r \]

(2)

σmy is the yield strength of the matrix, c is the modified empirical constant, k is the matrix-reinforcement interface bonding performance, the geometric factor g is the surface volume ratio, the alignment factor ψ is the average angle between the loading direction and the nanowires, τmax is the maximum shear stress, and Vr is the volume fraction of the reinforcing phase. In the as-cast composite, SiCnws are randomly distributed (ψ is the same). The increase in the specific surface area g of the reinforcement effectively improves the yield strength σcy.

According to Equation (2), in order to further utilize the strengthening effect of SiCnws, in addition to reducing the size of nanowires, further research can be carried out in the following fields: (1) Optimizing the interface structure to further enhance the interface bonding strength, such as adding alloying elements [38], interface modification [46,59] and increasing the bamboo-like content [15,22]. (2) When the reinforcements are randomly distributed and aligned, the orientation optimization of SiCnws shows that the alignment factor ψ is 0.56~0.66 and 1, respectively, [37]. By extrusion, quasi-aligned nanowires can be obtained. However, traditional extrusion leads to severe fracture of nanowires, which offsets the strengthening effect. For this study, the low density of the composite material seriously limits the strengthening effect of SiCnws. Therefore, further improvements to the density of the material and the quasi-directional arrangement of the nanowires while ensuring the integrity of the nanowires will become the focus of the next research.

4. Conclusions

In the present work, the effects of SiCnws with diameters of 100 nm, 250 nm and 450 nm on the microstructure and mechanical behavior of 20 vol.% SiCnws/6061Al composites prepared by pressure infiltration were studied. The following conclusions were obtained:

1. SEM and TEM analysis showed that SiCnws were uniformly distributed in the SiCnws/6061Al composite. TEM observation showed that the interface between SiCnws and the Al matrix was well bonded and no interface product was found in the composite.

2. With the diameter of the reinforcement increased, the density of the composite increased, and the composite with 450 nm SiCnws reached 98.9%. The tensile strength and plasticity of SiCnws/6061Al composites increased with an improvement in the average diameter of SiCnws. The tensile strength of the composites prepared by 450 nm SiCnws
reached 544 MPa and the elongation reached 1.25%, which could be attributed to the increase in density and the improvement in interface bonding.

3. With the decrease in the diameter of the nanowires, the effective atoms involved in the interface load transfer increased, and the strengthening effect of SiCnws increased. Under the annealing state, the yield strength of the SiCnws/6061Al composite prepared by 100 nm reached 212 MPa, which was 13.4% and 28.5% higher than that of 250 nm and 450 nm, respectively.

4. The fracture analysis showed that the fracture characteristics of nanowires in smooth SiCnws-reinforced composites were dominated by negligible pull-out fractures. In bamboo-like and rod-like SiCnws-reinforced composites, the characteristics of nanowires were dominated by obvious pull-out fractures and nanowire/Al debonding.

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