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Improved Mechanical Properties of C_{sf}/SiC Composite by Rapid Hot Press Sintering

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Abstract

In this work , Csf/SiC composite was hot pressed by adding short carbon fiber and nano carbon black and nano SiC powder into SiC powder by rapid hot press sintering in order to improve the fracture toughness and other mechanical properties .The density, hardness, phase composition, mechanical properties of Csf/SiC composite were studied, the toughening principle was also discussed. The results indicated that: the Csf/SiC composite had a high density of above 98% in less than 30vol.% short carbon fiber . The hardness was decreased with the increase of the amount of Csf fibe. The composite were mainly composed of 6H-SiC and C, the high temperature graphitization of short carbon fiber resulted the diffraction peaks of C in crystalline by XRD. Short carbon fibers were distributed uniformly and no aggregation in the matrix when contents of fiber were less 30vol.% by SEM test. The bending strength of the composite reached 423MPa at optimal conditions. Too many fibers in the matrix led to decline of bending strength sharply. The fracture toughness reached 4.9MPa·$m^{1/2}$ of maximum with more obvious toughening effect. The debonding, pullout and breakage behaviors of the Csf fiber and crack propagation were identified as the dominant toughing mechanism in such composites.

Keywords: *Csf/SiC Composite; SiC; Csf; nano C ;hot pressing sintering;toughening*

1. Introduction

Silicon carbide (SiC) ceramic has lots of excellent properties because of its strong covalent bond, such as high strength, hardness, wear resistance, oxidation resistance and low density[1-4]. Consequently, it was widely used in the field of high temperature structure and wear resistance, such as high-temperature nozzle, bearings, seals, wear parts, valves and other parts[5]. But, lower Self diffusion coefficients of C and Si in SiC results in its harder sintering. Some sintering aids are added in high temperature conditions. Aids themselves will form glass phase, which lead to poor performance of composite[6-9]. In addition, the inherent brittleness also limits its development. Therefore, to improve the brittleness of SiC ceramic materials, realize the wide application of SiC ceramic, some novel technology conditions are inevitable to realize toughening[10-14]. At present, the general toughening method is adding fibers to SiC ceramics matrix. Carbon fiber is widely used to toughen SiC because of its excellent strength, elastic modulus and low cos t[15]. And the short fiber can be randomly and evenly distributed in the matrix by controlling the type, length and content of the fiber, three-dimensional toughening can be achieved on a macro level[16,17]. In the paper, short carbon fiber was selected as reinforcing material. To ensure densification and excellent properties, the hot press sintering was used to prepare Csf/SiC ceramic composite with filler of 40nm carbon black and no any aids. The effect of the fiber volume fraction on the dispersion of short fiberand mechanical properties and toughening in the SiC ceramic matrix were studied.

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2. Experimental

In the work, firstly, surface treatment of short carbon fibers was carried out based on the air oxidation method and the liquid oxidation method. Then, 0.5μm SiC Powder was used as matrix material, 40nm carbon black and 40nm SiC Powder were used as filling material. The short carbon fiber of 5mm length was used as reinforcer. 0.5μ m SiC, 40nmSiC and carbon black were filled into ball mill tank by a certain percentage of 7:2:1. In order to ensure that the matrix and reinforcing material were mixed evenly, were added into tank kept stirring for 48 h in the anhydrous alcohol with wet-milled process. The resulting slurry was dried in the drying chamber, and then the composite powder was obtained by milling. A certain mass of powder in the graphite mold were sintered to prepare Csf/SiC ceramic composite in hot press at 2000℃ and 30MPa pressure and 20-60mminutes sintering time by rapid hot press sintering stove. Bulk densities of the composites were measured based on Archimedes principles, using distilled water as the immersing medium. Indentation method was also used to the hardness of the material with measured at load of 5000gf. strips of 3mm*4mm*36mm were cut from Composite, the bending strength and fracture toughness of the composite were measured by WDW-2000 electronic universal testing machine. The XRD was used to analyze phase composition of the composite. The surface and fracture morphology of the composite were characterized by metallographic microscopy and SEM, the dispersion of fiber in matrix and toughening mechanism were studied.

3. Results and Discussion

3.1. Effects of Fiber Content on Relative Density of Composite

Figure 3-1 showed the curve of relative density of the composite with various short carbon fiber volume fractions.

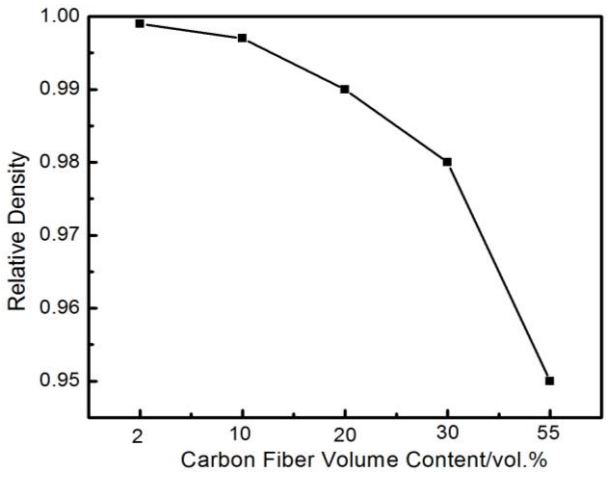

Figure 3-1. Relative Density of the Composite with Various Short Carbon Fiber Volume Fractions

As shown in Figure, the relative density of the composite decreased but kept more than 95% with the increasing of the fiber volume fraction. The density of carbon fiber and carbon black were lower than SiC. In addition, the bridging effect of carbon fiber made the porosity of the composite increase, so the density of the composite was reduced. Relative density of composite decreased slowly relatively when the fiber content was less than 30%. The pores in the sintering process were

filled by nano carbon powder, carbon black and carbon fiber expanded easily in high temperature, which would fill the voids, realize the composite densification and slow down the relative density. But fiber bridging probability was increased, too much carbon fibers made composite appear more voids when the carbon fiber content was higher than 30%, so that the density of composite decreased significantly.

3.2. Effects of Fiber Content on Hardness of Composite

Figure 3-2 represented the change curve of composite hardness with the short carbon fiber volume fraction. The results showed that the hardness of the composite decreased with the increasing of the fiber fraction. The indentation was easy to be produced on the matrix in testing because of less fiber. So the hardness of composite and pure SiC were small differences when the fiber content was below 10%. But the indentation was easy to produced on fiber because of more fibers. Consequently, the hardness of composite was decreased seriously.

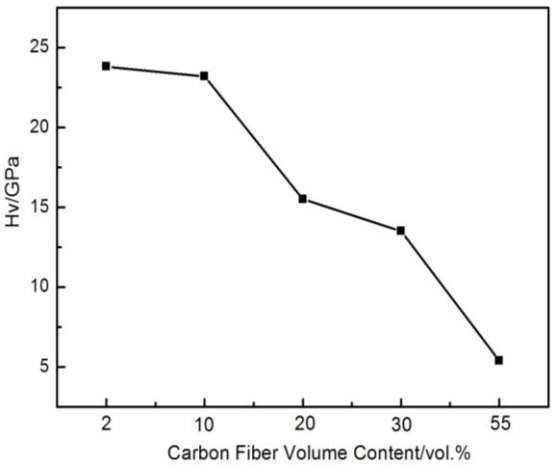

Figure 3-2 Vickers Hardness of The Composites with Various Carbon Fiber Volume Fraction.

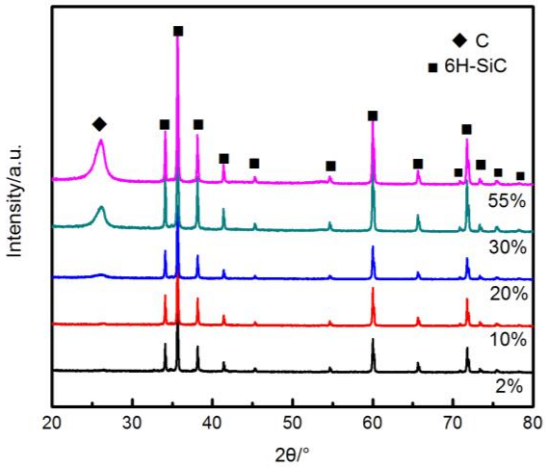

Figure 3-3 XRD Patterns of Csf/Sic Composite

3.3. Phase Analysis of Composite of Different Short Carbon Fiber Content

Figure 3-3 showed XRD patterns of Csf/SiC composite. The composite were mainly composed of C and 6H-SiC(α-SiC) wurtzite structure, more perfect crystallization, which was most stable in all SiC. and β-SiC was translated into 6H-SiC at high temperature. Diffraction peak intensity of C gradually increased with the increasing of carbon fiber content. Fibers in the matrix were more dispersed due to the lack of carbon fiber, it was difficult to detect C, which resulted in unconspicuous diffraction peak. But more fibers were opposite. In addition, the diffraction peaks of C showed crystalline that the high temperature graphitization of carbon fiber resulted [18].

3.4. Microstructure and Fiber Dispersion of Composite

Figure 3-4 showed the surface microstructure of the composite from metallographic microscopy. In the Figure, the gray area was SiC, and the white strips were short carbon fiber. The distribution of carbon fibers in the matrix was more obvious with the increasing of carbon fiber content. There was no pore in composite and it was more compact. Short

carbon fibers were dispersed better and no agglomeration, which reached the ideal state that random distribution of isotropy, so that it could improve the macroscopic properties of composite.

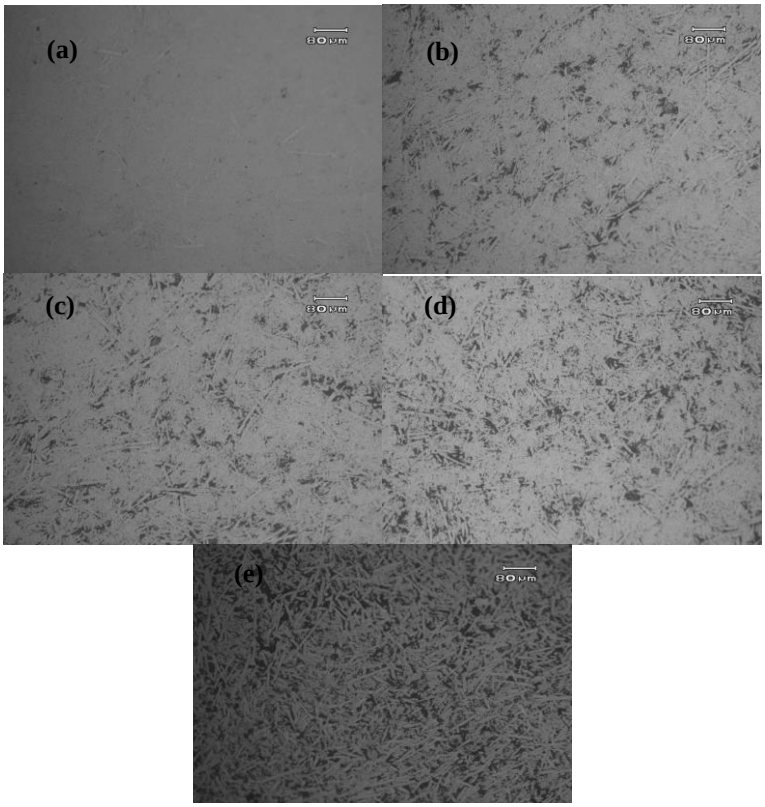

Figure 3-4. The Surface Microstructure of the Composite in Different Volume Fraction of Short Carbon Fiber (A) 2% Csf (B) 10%Csf (C) 20%Csf (D) 30% (E) 55%Csf

3.5. Effects of Fiber Content on Mechanical Properties of Composite

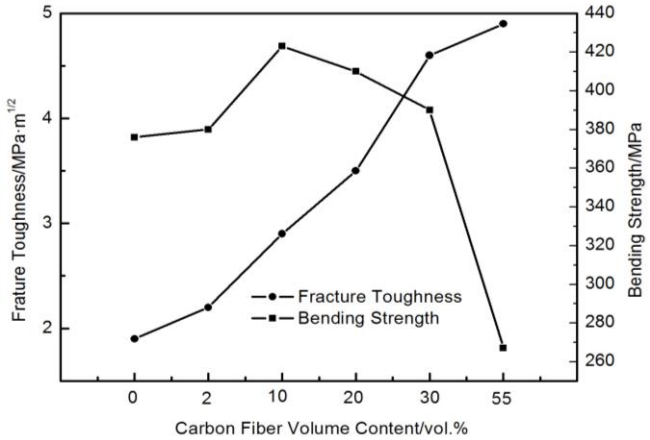

Figure 3-5. The Bending Strength and Fracture Toughness of Csf/Sic Composite

Figure 3-5 showed the bending strength and fracture toughness of Csf/SiC composite. The bending strength of composite with no carbon fiber was 376MPa, the fracture toughness was 1.9MPa·$m^{1/2}$ from the Figure. The bending strength of composite were increased first and then decreased with the increasing of fiber content, and the maximum value was 423MPa with an increasing of 12.5%, which was the content of 10%Csf. Less fiber resulted in no obvious bridging effect and low porosity rate. The fibers were able to withstand a certain load when a load were applied to the composite, which could increase the bending strength of composite. But too much fibers would increase the difficulty of densification and porosity rate, so that the loose structure decreased bending strength of composite. And the fracture toughness of the composite were increased gradually with the increasing of fibers, and the toughening effect was more obvious, which reached the maximum of 4.9MPa·$m^{1/2}$. It indicated that short carbon fiber could reduce the brittleness of SiC and increase the toughness of composite greatly[19].

3.6. Toughening Principle

Because of the brittleness of ceramic materials, it was necessary to overcome the fatal weakness of the brittleness to meet corresponding demands of SiC products. Short carbon fiber had a reinforcing effect, adding short carbon fiber to SiC matrix could improve the toughness of SiC ceramic greatly. Carbon fibers were randomly distributed in the SiC matrix by using short carbon fiber to reinforce SiC composite. The toughening mechanism was composed of fiber breakage, fiber pullout, fiber debonding and crack propagation. the energy absorption mechanism was mainly the fiber debonding and fiber pullout when the composite were destroyed[20]. Composite toughening mechanism was showed in Figure 3-6.

Figure 3-6a showed fiber debonding. Extra energy was needed to obtain a new surface after fiber debonding. Although surface energy of per unit area was very small, but the total surface energy of fiber debonding were very large. The debonding energy of unit area could be calculated by formula (1):

$$Q_d = \left(\sigma_f{}^2 l_c V_f\right) \Big/ 12E_f \tag{1}$$

Where σ_f was tensile strength of fiber, l_c was critical length of fiber, E_f was elastic modulus of fiber, V_f was fiber volume. Therefore, the toughening effect from fiber debonding could be increased by enlarging fiber volume and critical length .

Figure 3-6 b showed fiber pullout. It was the phenomenon that the short carbon fibers near the crack tip are were slid along the interface between fibers and matrix. Obviously, fibers were pull out after fiber debonding, fiber pullout could make the stress relaxation of crack tip, which could slow the propagation of crack. The external working force of fiber pullout played an important role in toughening. The fiber pullout energy was more than fiber debonding, so the effect of the fiber pullout was more obvious than fiber debonding. Consequently, the fiber pullout was more important in the toughening mechanism. The pullout energy was presented with the product of the resistance times its distance, following the formula (2):

$$Q_p = \left(\pi \frac{d}{2} l \bullet T\right) \bullet l \tag{2}$$

Where d was fiber diameter, l was fiber length, T was shear strength. When the fiber was broken, the effective length was half of l_c. The formula (2) was changed to (3):

$$Q_p = \pi d l_c{}^2 \bullet T \Big/ 8 \tag{3}$$

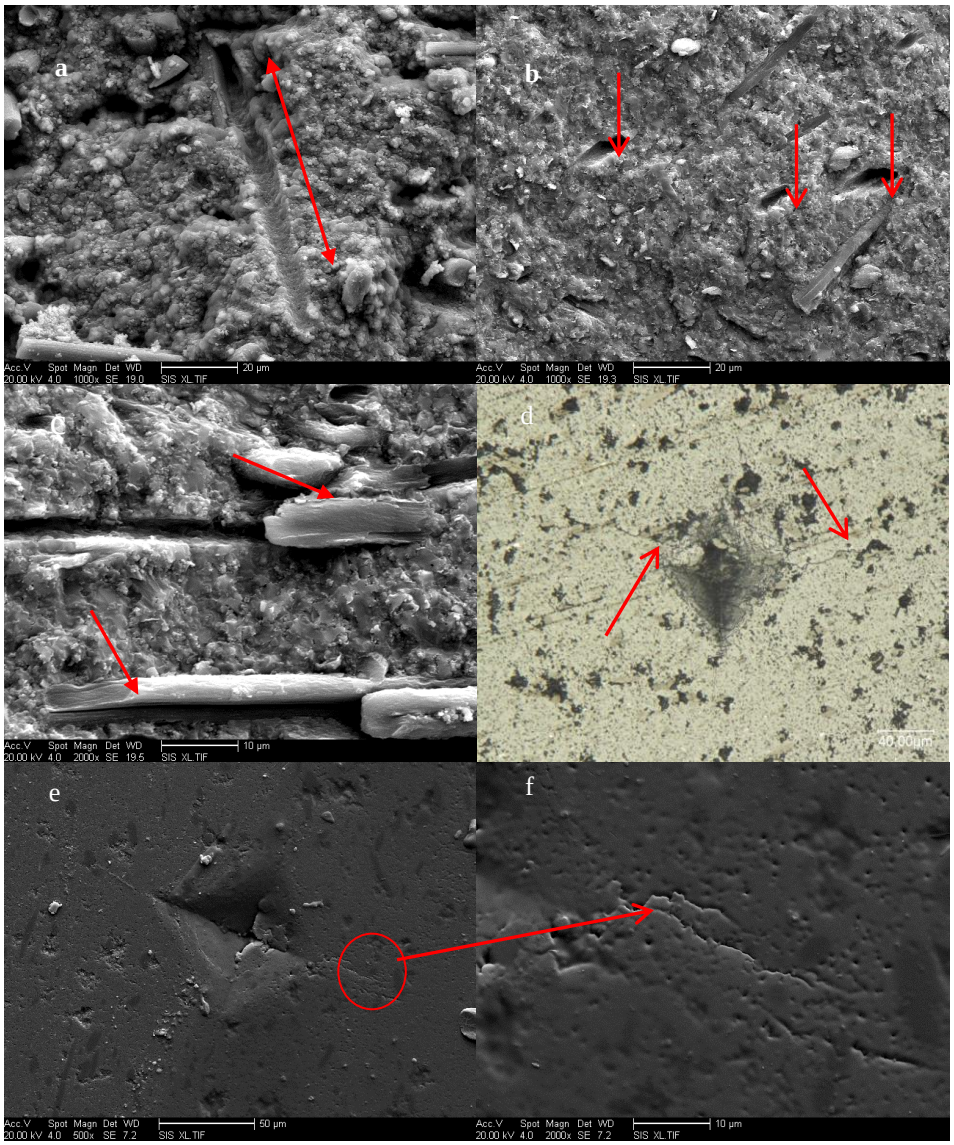

Figure 3-6. Toughening Mechanism Of Csf/Sic Composite (A) Fiber Debonding (B)Fiber Pullout (C) Fiber Breakage (D), (E)×500, (F)×2000 Crack Propagation

Figure 3-7c showed fiber breakage. Fiber of crack tip was extended and generated dislocation relative to matrix in crack opening in fiber breakage process. Finally, fibers were ruptured due to too large stress. The broken fibers receded in the matrix, and the dislocation disappeared, elastic deformation energy was released. The possibility of fiber broken was small for the shorter fiber, which resulted in lesser energy absorbed in breakage. The fiber breakage energy was calculated similarly by formula (4):

$$Q_b = \pi d^2 l_c \sigma_f^2 / 2E_f \quad (4)$$

SiC ceramics were brittle materials, almost no plastic deformation. The fracture of the composite was that micro cracks were produced in matrix when composite were subjected to stress. Micro cracks propagated in matrix, as shown in figure 3-7(d, microscope),(e, SEM),(f,SEM). Part of the energy belong to cracks was absorbed by interface to prevent the propagation of crack, another part of the energy was absorbed by fiber debonding and fiber pullout, the remaining energy made composite fracture. Interface and fiber were the

main consumption among three parts energy in crack propagation. The crack propagation must be realized in the condition of fiber breakage and pullout. The crack needed to detour the fiber to continue to expand because of high elastic modulus of the fiber, paths of the crack propagation were changed by that. Most of crack energy was consumed in that process. But the crack continued to propagate along interface in weak binding force between short fiber and matrix due to smaller energy at this moment. A large amount of propagation paths for cracks were provided by fiber, the energy was absorbed ceaselessly, which achieved the purpose of toughening[21,22].

4. Conclusion

Csf/SiC composite were prepared by rapid hot press sintering at 2000℃ and pressure of 30MPa and 20 -60mminutes sintering time, and following conclusions were obtained, the Csf/SiC composite had a high density of above 98% in less than 30vol.% of Csf. The hardness was decreased with the increasing of fiber. The composite were mainly composed of 6H-SiC and C, the high temperature graphitization of carbon fiber resulted the diffraction peaks of C in crystalline. Fibers were distributed uniformly in the matrix and no aggregation with less content. The bending strength of the composite reached 423MPa of maximum. Too many fibers in the matrix led to decline of bending strength sharply. The fracture toughness reached 4.9MPa·$m^{1/2}$ of maximum with more obvious toughening effect. The toughening mechanism was composed of fiber debonding, fiber pullout, fiber breakage and crack propagation.

Acknowledgements

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