Synthesis of SiC nanowhiskers by microwave heating: effect of size of graphite

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Abstract. Silicon carbide is a promising material for various applications, especially for applications in high frequency, high power and high temperature and harsh conditions such as highly oxidative and corrosive environment. Currently, silicon carbide is produced by conventional heating process. For silicon carbide nanomaterials, various methods have been attempted. In this study, SiC nanowhiskers were synthesized by microwave heating of blends of graphite and silica in the ratio of 3:1. Graphite of two different size, namely micro graphite and nano graphite were used. The blends firstly prepared by ultrasonic mixing graphite and silica in ethanol followed by drying. The blends were then heated by using laboratory microwaves furnace to 1450 °C at heating rate of 20 °C/min for 20 minutes. Finally, the SiC nanowhiskers was characterized by using scanning electron microscope.

1 Introduction

Silicon carbide (SiC) is one of the promising and attractive ceramic due to its fascinating properties such as high melting point, excellent oxidation resistance, excellent thermal stability, high mechanical strength, chemical inertness, wide energy band gap, and unique optical properties. Acheson process is the most widely used method for the production of SiC. This process involves carbothermal reduction of SiO2 by coke at 2200-2500 °C for about one week [1]. However, this method required high temperature and long heating duration, which render the process time consuming and costly. For this reason, numerous other synthesis route have been studied and developed for production of SiC including mechanical milling [2], sol-gel processes [3], chemical vapor deposition (CVD) [4], rapid carbothermal synthesis [5] and thermal plasma [6]. However, these methods have similar limitations and drawbacks such as presence of impurities in the product, large variation of grain size and time-consuming process. In recent years, various nanomaterials were extensively synthesized and studied for their unique and novel properties. SiC nanomaterial is one of the most important nanomaterials for current and future research and applications for their unique properties.

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Comparing to conventional heating which heats from outside to the inside of the materials, microwave heating can volumetrically heat materials with favourable dielectric properties. Therefore, microwave heating is more uniform and efficient comparing to conventional heating which causes temperature gradient across the materials. Furthermore, microwave heating is also more energy efficient than conventional heating due to higher heating rate and lower heat loss to the surrounding. Previous studies have shown that graphite is an excellent microwave absorber [7] and 1:3 for silicon dioxide to carbon source (carbon nanotube) was the suitable ratio for the preparation of high purity SiC nanomaterials [8]. Thus, the aim of this study is to produce silicon carbide nanowhiskers (SiCNWs) by microwave heating the blends of silicon dioxide and graphite in the ratio of 1:3 at 1400 °C with heating rate of 20 °C/min for 20 minutes. Besides, the effect of size of graphite, namely micro graphite and nano graphite was also studied.

2 Experimental

2.1 Sample preparation

Blend of SiO₂ powder (particle size 4 μm, purity: >99.9%) and micro graphite powder (particle size 4 μm, purity: >99.9%) were used as starting material. Both SiO₂ powder and micro graphite powder were obtained from Sigma Aldrich while nano graphite powder was obtained from Guangzhou Hongwu Materials Technology Co., Ltd. The ratio of the amounts of silica to graphite was fixed at 3:1 with total of 1 gram of blend was acquired. Ethanol was used as liquid medium for the mixing of the raw material. Ultrasonic mixing bath was conducted for 1 hour generate vibration in the mixture of silica and graphite for the homogeneous mixing of the raw material. Mixture of silica and graphite was dried using the hot plate to vaporize ethanol. The blend of SiO₂ and micro graphite were compressed to become a pellet to prevent the escape of SiO gas during the heating process. The same procedure was repeated for nano graphite powder (particle size 100 nm, purity: >99.9%).

2.2 Synthesis of SiCNWs by microwave irradiation

Microwave heating was performed in microwave furnace with multimode cavity in which 2.45 GHz microwave irradiation was brought out through a waveguide. The set up for the microwave heating process was reported previously [9]. Blend of micro graphite and SiO₂ was placed in silica crucible and the crucible was placed in microwave cavity. Silica sand was used as a heat insulator to prevent heat lost. SiC susceptors was used as microwave absorber to absorb and convert electromagnetic energy to heat because SiC susceptor is a good microwave absorber material. This allowed rapid heating of the blend of micro graphite and SiO₂. The blend of SiO₂ and micro graphite was heated to 1450°C with heating rate of 20 °C/min and soaked for 20 minutes. The blend was then allowed to cool down to room temperature. The same procedure was repeated for blend of nano graphite and SiO₂.

2.3 Characterization of SiC nanowhiskers

After the SiCNWs were synthesized, characterization was conducted using scanning electron microscopy (SEM). The morphology of the SiCNWs was observed by scanning electron microscopy (SEM JEOL JSM6010LV) at magnification of 30000X and accelerating voltage of 20 kV.
3 Result and discussion

3.1 Characterization of graphite using SEM

SEM images of micro graphite and nano graphite were shown in Fig. 1. As shown in Fig. 1 (a), the particle size of micro graphite ranges from 1 to 4 \( \mu \text{m} \). The shape of the micro graphite is irregular polygonal, as shown in Fig. 1 (b). Fig. 1 (c) shows the SEM image of nano graphite in which nano graphite are agglomerated to form larger particles. Fig. 1 (d) shows the SEM image of nano graphite at higher magnification and it is observed that the particle size of nano graphite ranged from 100 – 200 nm.

![SEM images of micro graphite and nano graphite.](image)

Fig. 1. SEM images of (a) and (b) micro graphite and (c) and (d) nano graphite.

3.2 Characterization of SiCNWs using SEM

SEM images of SiCNWs synthesized from micro graphite and nano graphite were shown in Fig. 2. As shown in Fig. 2 (a), SiCNWs were successfully synthesized when micro graphite was used. SiCNWs grew randomly on the surface of micro graphite particles. SEM images of higher magnification in Fig. 2 (b) shows the SiCNWs are straight and have uniform diameter along the length. The diameter of the SiCNWs ranges from 100-300 nm. Although SiCNWs were successfully synthesized, the reaction is incomplete with unreacted micro graphite. Fig. 2 (c) shows that very few SiCNWs were synthesized when nano graphite was used. Higher magnification in Fig. 2 (d) shows that the diameter of SiCNW ranges from 50 - 100 nm. Comparing to the SiCNWs synthesized from micro graphite, the amount of SiCNWs synthesized by using nano graphite as carbon source is very small. Further study is required.
to understand the mechanism of formation of SiCNWs from both micro graphite and nano graphite.

Fig. 2. SEM images of SiCNWs synthesized from (a) and (b) micro graphite and (c) and (d) nano graphite.

4 Conclusions

In this study, synthesis of SiCNWs was achieved by using microwave heating of blends of SiO$_2$ and graphite at 1450°C for 20 minutes. It is found that SiCNWs were formed when both micro graphite and nano graphite were used as carbon source to react with silica. However, the amount of synthesized SiCNWs were significantly larger when micro graphite was used, compared to when nano graphite was used.

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