Synthesis of several millimeters long SiC–SiO₂ nanowires by a catalyst-free technique

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Abstract:

In situ synthesis of ultra-long SiC–SiO₂ nanowires were successfully conducted with the raw materials of silicon and phenolic resin by an effective and catalyst-free technique. Several millimeters long SiC–SiO₂ nanowires with the diameters in the range of 50–200 nm were mainly composed of Si, C and a small amount of O, and the formation of several millimeters long SiC–SiO₂ nanowires was attributed to a low flow rate and carbon sources supplied continuously by the pyrolysis of phenolic resin. A catalyst-free vapor–solid (VS) growth mechanism was proposed to illustrate the growth process of ultra-long SiC–SiO₂ nanowires in present experiment, which provides a promising method for in situ fabrication of SiC–SiO₂ nanowires as reinforcements into composites.

Keywords: SiC; Nanowires; Millimeter; Mechanism.¹

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1. Introduction

SiC as one of the most widely used non-oxide ceramics has many excellent properties (i.e., high thermal stability, good mechanical behavior and chemical inertness), which make it can be operated at high power and temperature, and under harsh environments [1–5]. In addition to owning the above properties, one dimensional (1-D) SiC nanomaterials (i.e., nanowires and nanorods) exhibit unique mechanical, electrical and optical properties, and show fruitful potential applications in composites and nanodevices [6–8]. Among these 1-D nanostructures, SiC nanowires have attracted considerable interest because of their potential applications in nanocomposites, microelectronic, and nanodevices [9,10]. According to the recent delicate measurement by Wong et al. using atomic force microscopy and lithography techniques, the yield strength of SiC nanowires is over 50 GPa indicating that it has great potential in composite as reinforcement with high strength and toughness [11,12]. However, to the best of our knowledge, the excellent reinforcement efficiency of SiC nanowires in composites was rarely reported. The critical challenges of improving the reinforcement efficiency of SiC nanowires in composites are the way of introducing SiC nanowires into the composites (i.e., the content and distribution of SiC nanowires) and the interface between nanowires and the matrix [12]. According to the previous literature with high reinforcement efficiency of SiC nanowires into SiC matrix by Wen Yang in 2005, in situ process provides a promising solution to improve the reinforcement efficiency of SiC nanowires in composites [12]. Furthermore, ultra-long SiC nanowires (the lengths up to several millimeters or even centimeters) may be more suitable to study the properties of nanostructures on a macroscopic scale compared with the short ones [13–17], which could open up a new opportunity in the application of integrated nanoelectronics and nanocomposites. In

addition, a simple and effective way of in situ preparation of ultra-long SiC nanowires is still needed to develop.

This paper attempts to adopt an effective and low-cost method for in situ synthesis of several millimeters long SiC nanowires, in which phenolic resin and silicon powder were chosen as the raw materials. Although similar results have been reported [18], the novelty of this paper is to show the in situ formation of several millimeters long SiC-SiO₂ nanowires with remarkably different growth model compared with previous literature. This way has inherent advantages with an effective and simple procedure, which provides a promising approach to in situ fabrication of ultra-long SiC-SiO₂ nanowires in composites with excellent properties [19–21].

2. Experimental

2.1. In situ preparation of several millimeters long nanowires

Commercial phenolic resin and silicon were used as raw materials to synthesize several millimeters long SiC nanowires. A detailed synthesis process was described as follows. Firstly, the yield of the resin carbon was about 44.2 wt% as shown in Fig.1 according to the thermogravimetry curve of phenolic resin. Based on the yield of the resin carbon, the mass ratio of phenolic resin to silicon was calculated, in which the mole ratio of carbon to silicon was kept at 1.67:1. The composite-powder, including phenolic resin and silicon, were homogeneously mixed by a high frequency mixer for 30 min and placed into a ceramic crucible, and then the crucible was sent into a tube corundum furnace. The argon gas (99.999%) was introduced into the furnace at a flow rate of 200 ml/min for ten minutes and then changed the flowing speed to 70 ml/min before heating, while the argon was kept flowing during the experimental process. The furnace was heated up to 200 °C by 40 min and maintained for 10 min, and heated up to 800 °C at 5 °C /min and maintained for 120 min, and then heated up to 1300–1400 °C at 5 °C /min and maintained for 2 h. After the heating was terminated,

the furnace was cooled to 500 °C with 5 °C /min and then naturally cooled to room temperature. Finally, the white cotton–wool products were both obtained on the surface of composite–powder, namely in situ growth, and the inner walls of ceramic crucible.

2.2. Characterization

The phase identification and chemical composition of the white wools were analyzed by X–ray diffraction (XRD, X'PERT PRO MPD, Holland). Scanning electron microscopy (SEM, HELIOS NanoLab 600i, America) equipped with energy dispersive spectroscopy (EDX) was used to observe the morphology and analyze the elemental composition of the obtained products. Transmission electron microscopy and high–resolution transmission electron microscopy (TEM and HRTEM, Tecnai G²–F30, America) were used to identify the microstructure of the samples. Fourier transform infrared spectroscopy (FTIR, Spectrum Two, America) was also conducted to confirm the composition of the product.

3. Results and Discussion

3.1 Pyrolysis process of phenolic resins

The pyrolysis process of phenolic resins played an important role in the preparation of several millimeters long SiC–SiO₂ nanowires, and the thermogravimetry (TG) curve in an inert atmosphere with a heating rate of 10 °C/min was used to investigate the pyrolysis process of phenolic resin as shown in Fig. 1. It can be seen that the complete pyrolysis temperature of phenolic resin was about 1500 °C, and the TG curve included three temperature ranges of apparent weight loss.

The first one was from room temperature to 140 °C due to the elimination of water and residual solvent in phenolic resin [22], and the second one was from 300 to 600 °C caused by the release of different gases generated by the decomposition of phenolic resin, which includes CO, CH₄ and H₂. H₂ was the dominant product evolved during the third stage (from 900 to 1500 °C) and to form a carbonaceous char [22,23].

3.2 Preparation and characterization of several millimeters long nanowires

A suitable preparation temperature as one of the most important process parameters should be selected carefully to synthesize ultra-long SiC nanowires, which has been studied frequently, and the photographs of products obtained at different preparation temperatures and holding times were shown in Fig. 2 [24]. At 1300 °C for 2h, almost no obvious white cotton-wool products were found suggesting that the nanowires could not be greatly produced below or even at this temperature (Fig. 2a). The surface of composite-powder and the inner walls of ceramic crucible were covered by a quantity of white cotton-wool products at the preparation temperature of 1350 °C for 2h (Fig. 2b), while only a small amount of white cotton-wool products were obtained both on the surface of composite-powder and the inner walls of ceramic crucible (Fig. 2c) when the temperature reached up to 1400 °C for 2h. Therefore, it is reasonable to believe that 1350 °C was a suitable and relatively low preparation temperature for in situ preparation of nanowires, and the yield of nanowires could be improved when the holding time was prolonged to 4h as shown in Fig. 2d.

SEM images and EDX spectrum of as-prepared products collected from the inner walls of ceramic crucible were shown in Fig.3. From Fig. 3a–b SEM images and macroscopic morphology of the achieved cotton-wool products, it can be seen that products consist of many randomly oriented nanowires with the length up to several thousand micrometers, and the diameters of these nanowires range from 50 to 200 nm.

These nanowires exhibited both straight and curved morphologies, and the EDX spectrum collected from the stem of nanowire demonstrated that the nanowire was composed of Si, C, and only a little amount of O element as shown in Fig. 3c. In addition, there were no droplets found at the tips of the nanowires suggesting that the growth mechanism of nanowires was reasonable attributed to the VS mechanism as reported by other literature [25,26].

XRD was employed to confirm the crystalline phase of products, and a typical XRD pattern of the nanowires powders grounded from the white cotton–wool was shown in Fig. 4 indicating that the positions and relative intensities of these diffraction peaks were in good agreement with the standard values of β -SiC, in which major strong peaks centering at about 35.55° , 41.50° , 59.89° and 71.68° could be attributed to the (111), (200), (220), and (311) lattice planes of β -SiC, respectively. A low-intensity peak at a lower diffraction angle than that of the strong (111) peak was marked by SF, which could be ascribed to the stacking faults [27,28].

A more detailed microstructure characterization was carried out by TEM. From the typical TEM images as shown in Fig. 5, it can be found that the nanowire with a diameter of 80 nm consists of a crystalline core coated with an ultra thin shell layer (less than 2 nm thickness). There are some stacking faults and twins along the growth direction of the nanowire. The insert in Fig. 5 shows that the inter-planar spacing perpendicular to SiC nanowire axes is about 0.25 nm, which is consistent with the (111) plane space of 3C-SiC, suggesting that the growth direction of SiC nanowire is along [111] [29, 30]. FTIR measurement was performed to further confirm the composition of nanowires and a typical FTIR transmittance spectrum was shown in Fig. 6. The strong absorption peak centering at 844 cm^{-1} can be attributed to the transverse optical (TO) phonon vibration mode of the Si–C bonds and the absorption peak at 484 cm^{-1} belongs to the Si–O stretching vibration, which indicated that the

products were mainly composed of SiC and a small amount of SiO₂ [31,32].

Based on the EDX spectrum (Fig. 3c), XRD pattern (Fig. 4), TEM observation (Fig. 5) and FTIR spectrum (Fig. 6), it is reasonable to believe that the core of these nanowire is crystalline 3C-SiC and that the shell is an ultra thin amorphous SiO₂ layer.

3.3 Growth mechanism of several millimeters long nanowires

Four fundamental growth mechanisms were proposed to explain the growth model of nanowires, including VS, VLS, solid-liquid-solid (SLS) and oxide assisted growth (OAG), in which VS and VLS mechanisms were most commonly involved [33–35]. On the basis of experimental results, a catalyst-free VS growth mechanism was proposed to disclose the growth model of several millimeters long SiC-SiO₂ nanowires. Since just two raw materials were employed to synthesise SiC-SiO₂ nanowires, silicon atoms can be supplied only by silicon powders, while carbon atoms could be provided by raw material of phenolic resin. Moreover, four products contain carbon atoms, namely CO₂, CO, CH₄ and resin-derived carbon, in which CO₂ could be formed during the reactions and the last three products were produced by the pyrolysis of phenolic resin, methane-derived carbon and H₂ could also be released by the pyrolysis of CH₄ above 900 °C. Silicon and SiO₂ a thin layer coated on the surface of the original silicon due to the oxidation at room temperature could provide silicon atoms in present experiment. Therefore, three questions could be used to illustrate the growth process of several millimeters long SiC-SiO₂ nanowires. The first one is the ways of introducing carbon atoms, the second one is the ways of introducing silicon atoms and the reactions between carbon atoms, and silicon atoms are the last one [36]. The following detailed analysis is used to answer the above questions.

The first one is the ways of introducing carbon atoms in present experiment. CO₂, CO, CH₄ and resin-derived carbon can act as carbon sources to supply carbon atoms

in our experiment, and the following reactions may take place. The change in Gibbs free energy of reaction (1) is greater than zero at low temperature, while it would decrease when temperature rises up to 1000K and the change in Gibbs free energy of reaction (2) is always lower than zero from the thermodynamic point of view without taking the effect of nanosize into account. It should be noted that the carbon as a reactant of reaction (2) can be produced by three ways with the pyrolysis of phenolic resin, resin-derived carbon and methane-derived carbon. Although Ar was filled into the tube furnace and most of oxygen could be eliminated, there was still leaked or residual oxygen due to the weak air tightness of the tube system, and the concentration of the residual oxygen could meet the requirement on O₂ partial pressure to support the following reactions to take place [36]. The carbon atoms can be supplied with the ways of CO₂, CO and C solid to react with silicon atoms, in which CO₂ should be generated by the following reactions.

The second one is the ways of introducing silicon atoms. Si, SiO₂ coated on the surface of the original silicon and SiO vapor generated by following reactions can supply silicon atoms. In the view of thermodynamics, the change in Gibbs free energy of reaction (3) is always lower than zero, while the change in Gibbs free energy of reaction (4) is greater than zero at low temperature without taking account of the effect of nanosize. Although a low than zero change in Gibbs free energy of reaction (3), reaction (4) maybe take place firstly due to the presence of SiO₂ coated on the surfaces of silicon powders.

The reactions between carbon atoms and silicon atoms are the last question, and

the following reactions may carry out to produce the gas reactants (SiO, CO and CO₂) or SiC. According to the calculated thermodynamics, the changes in Gibbs free energy of reactions (5) and (6) are always greater than zero without taking account of the effect of nanosize, while the changes in Gibbs free energy of reactions (7), (8) and (10) are always lower than zero and the change in Gibbs free energy of reaction (9) is lower than zero until temperature rises up to 1700K.

The growth process of SiC–SiO₂ nanowires can be concluded with three stages, which includes incubation, nucleation and growth, and a possible growth model of SiC–SiO₂ nanowires was illustrated as shown in Fig. 7. In the early stage of heating process, large amount of gases would be generated by phenolic resin decomposition, including CO, CH₄ and H₂ as shown in Fig. 7I. As the temperature rises, reactions (1) and (2) would take place in sequence to produce CO, and then reactions of (3)–(6) and (11) would carry out very slowly and only a small amount of gases would be produced to form a suitable atmosphere for nucleation, including CO, SiO, and CO₂. It should be pointed out that the concentration of CO should be supersaturated firstly compared with the concentration of SiO, and reaction (9) might carry out to form SiC nuclei at first as well as providing the reactant gas CO₂ for the subsequent reaction (11) as shown in Fig. 7II. Furthermore, the reactions of (7)–(10) might take place under the right conditions to form SiC nuclei with the temperature increasing. The last stage is

the subsequent growth of SiC nanowires, which may grow subsequently along a fixed axis as shown in Fig. 7III. In the view of crystallographic, the growth direction of SiC nanowires is preferentially normal to the (111) plane of β -SiC [37,38], and SiC nanowires would grow subsequently along the fixed direction as long as the gases reactant continuously produced, and then large-scale and several millimeters long SiC nanowires would be formed. It is also worth being pointed out that reactions of (7) and (8) may be responsible for the growth of SiC nanowires, and the reaction (10) can take place preferentially during the cooling stage from the perspective of thermodynamic consideration. SiO₂ would deposit on the surface of SiC, and then a core-shell structure of SiC-SiO₂ nanowires would be generated due to a lower melting point and slower solidification rate compared with those of SiC [39].

Why several millimeters long SiC-SiO₂ nanowires can be obtained in present experiment and the reasons can be concluded from the following reports. J. Wei et al. attributed the main reason for the formation of ultra-long SiC nanowires to the low flow rate in reaction, which was similar to the results of G.Y. Li and L.W. Lin [40–42]. In these literatures, the flow rates mainly ranged from 20 to 80 sccm, and the flow rate of this experiment was 70 sccm within the reported range, which could be the first reason for the formation of several millimeters long SiC-SiO₂ nanowires. The low flow rate leading to a high concentration of gas reactants can be easily up to a supersaturated condition to support the generation of nanowires. In addition, carbon sources supplied continuously by the pyrolysis of phenolic resin would be the other reason for the formation of several millimeters long SiC-SiO₂ nanowires. A comparison experiment was carried out with the raw materials of resin-derived carbon and silicon, while other experimental parameters were kept constant. However, a few several micrometers long nanowires were obtained as shown in Fig. 8 suggesting that the pyrolysis process of phenolic resin played an important role in the

formation of several millimeters long SiC–SiO₂ nanowires. During the reaction process, phenolic resin can first release CO, CH₄ at the temperature from 300 to 600 °C to form a suitable reaction atmosphere for SiC nucleation. Furthermore, methane–derived carbon and H₂ could also be released by the pyrolysis of CH₄ above 900 °C to continuously supplied carbon sources. Moreover, it should be noted that the concentrations of CO and other containing carbon gas would be easily saturated in local due to the presence of H₂ with a lower density compared with those of gases containing carbon, which would be favor of SiC nanowires growth to several millimeters long. Therefore, the low flow rate and carbon sources supplied continuously by the pyrolysis of phenolic resin made the reactions have sufficient time to supply gas reactants to promote the nanowires longer and longer.

4. Conclusions

Several millimeters long SiC–SiO₂ nanowires were successfully synthesized by a simple and catalyst–free method using silicon and phenolic resin as raw materials at 1350 °C. The nanowires exhibited straight–line and curved shapes with diameters in the range of 50–200 nm, and the main composition of nanowires was 3C–SiC. Phenolic resin played an important role in supplying the carbon sources, and the formation of several millimeters long SiC–SiO₂ nanowires can be attributed to the low flow rate and carbon sources supplied continuously by the pyrolysis of phenolic resin. A catalyst–free VS growth mechanism was proposed to disclose the growth model of several millimeters long SiC–SiO₂ nanowires offering great potential for in situ fabrication of ultra–long SiC–SiO₂ nanowires in composites.

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Figure Captions:

Fig.1 Thermogravimetry curve of phenolic resin in Ar atmosphere.

Fig.2 Photographs of as-prepared products obtained at different preparation temperatures and holding times. (a) at 1300 °C for 2h, (b) at 1350 °C 2h, (c) at 1400 °C 2h and (d) at 1350 °C for 4h.

Fig.3 SEM images and EDX spectrum of as-prepared products formed at 1350 °C for 2 h in the ceramic crucible. SEM images collected from (a) low-magnification with macroscopic morphology and (b) high-magnification of the achieved cotton-wool products. (c) EDX spectrum obtained from the stem of nanowire.

Fig.4 XRD pattern of products obtained at 1350 °C for 2 h.

Fig.5 Typical TEM images of nanowire obtained at 1350 °C for 2 h.

Fig.6 Typical FT-IR transmittance spectrum of several millimeters long SiC-SiO₂ nanowires obtained at 1350 °C for 2 h.

Fig.7 Illustrations of possible growth process of SiC-SiO₂ nanowires.

Fig.8 SEM image and macroscopic morphology of the products with the raw material of resin-derived carbon and silicon.

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