



One-step molten-salt-mediated preparation and luminescent properties of ultra-long SiC/SiO₂ core–shell nanowires

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Abstract

Ultra-long SiC/SiO₂ core–shell nanowires were successfully prepared on a graphite felt with Ni(NO₃)₂ coating by a straightforward molten-salt-mediated carbothermal reduction route using silica fume and phenolic resin as starting materials at 1400 °C. The as-synthesized nanowires, 150–500 nm in diameter and up to several hundred microns in length, comprised a crystalline 3C–SiC core with a thin amorphous SiO₂ shell (about 30 nm). The growth mechanism of the as-synthesized nanowires was most likely controlled by the combination of vapor–liquid–solid and the vapor–solid mechanism. The photoluminescence spectrum of the ultra-long core–shell SiC/SiO₂ nanowires exhibited a significant blue-shift, indicating a potential application in optoelectronic devices.

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1. Introduction

One-dimensional (1D) SiC nanoscale materials (nanorods and nanowires) have attracted intense interests owing to their high mechanical strength, good abrasion resistance, excellent chemical and thermal stability, wide energy band gap and breakdown field [1–3]. Among these 1D nanoscale materials, SiC nanowires (NWs) have been identified as a potential material for use in microwave absorbers, high temperature ceramics, field emitters, sensors, nanoelectromechanical devices and supercapacitors [4–9]. Apart from these unique and outstanding properties, the luminescent and field emission properties of SiC NWs can be greatly enhanced by the quasi-1D SiC/SiO_x nanostructured materials (e.g. SiC/SiO₂ core–shell NWs and nanochains) due to the combination of the merits of SiC and SiO₂ nanostructure [10–12].

To date, various methods/techniques have been attempted to synthesise the 1D SiC/SiO_x nanomaterials, including chemical vapor reaction [13,14], microwave method [15], pyrolysis of polymeric precursors [16], template method [17], arc-discharge [18], emulsion electrospinning [19], carbothermal reduction (CR) [20–22], sol–gel [23] and two-step route [24]. Unfortunately, most of these reported methods suffered from expensive materials precursors, complex equipments and relatively lower yield. Apart from these, procedures of further purification (e.g. removing the residual carbon in CR and sol–gel process) and separation of NWs and bulk particles were normally required. To overcome the above-mentioned disadvantages, significant efforts are still required to make the overall preparation more economical and feasible.

We have found that NaCl/NaF molten salts can accelerate the transformation of SiO₂ to SiC in the CR process and reduce the synthesis temperature [25]. In response to these issues, a combination of a molten-salt-mediated CR synthesis procedure with the assistance of a catalyst (Ni(NO₃)₂), we demonstrated a novel, straightforward and cost-effective method of preparing ultra-long SiC/SiO₂ core–shell NWs without any purification/separation procedures of NWs and bulk particles. Silica fume

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and phenolic resin were chosen as starting materials. Meanwhile, a graphite felt with $\text{Ni}(\text{NO}_3)_2$ coating was employed as an initial nucleation and growth site of NWs. Morphologies and crystalline structures of the resultant NWs were characterized in detail. In addition, the photoluminescence (PL) property of the as-synthesized products was examined. The growth mechanism of SiC/SiO_2 core-shell NWs was also discussed.

2. Experimental

A typical synthesis procedure of SiC/SiO_2 core-shell NWs was carried out in a tube furnace. The whole experimental apparatus is shown in Fig. 1. Silica fume ($\geq 97.0\%$, $\leq 1 \mu\text{m}$) and phenolic resin powder ($\geq 99.0\%$, $\leq 80 \mu\text{m}$) were used respectively as silicon and carbon sources, and their molar ratio is 1:3. NaCl and NaF were employed to form a molten salt medium according to previous work [25]. Silica fume, phenolic resin and molten salts were firstly mixed, and then loaded into a corundum boat. A graphite felt was immersed in a 0.05 M $\text{Ni}(\text{NO}_3)_2$ solution for 4 h, dried in an oven at 80°C for 12 h, after that employed to cover the corundum boat. The whole set-up was placed in the center of a long corundum tube. Prior to heating, the tube was evacuated by a vacuum pump and purged with Ar flow (99.99%) to reduce the oxygen to as low a level as possible. The prepared system was heated to 1400°C at 5°C min^{-1} and maintained at this temperature for 4 h in a flowing Ar atmosphere. After heating, the furnace was naturally cooled to room temperature, and the thick, light green layer of products was seen on the underside of the graphite felt.

The phases and crystalline structures of as-collected samples were analyzed by X-ray diffraction (XRD, Panalytical X'pert, Netherlands) with $\text{CuK}\alpha$ radiation at 40 kV and 40 mA. The microstructure and morphology of as-synthesized samples were examined by a field emission scanning electron microscopy (FESEM, JEOL JSM-7600F, Japan), equipped with energy-dispersive X-ray spectroscopy (EDS, INCA) and high-resolution transmission electron microscope (HRTEM, JEOL JEM-2010, accelerating voltage 200 kV). Fourier transform infrared (FT-IR), Raman shift spectrum and photoluminescence (PL)

spectra at room temperature were recorded by a FT-IR spectrometer (Bruker Tensor-27, Germany), a Raman spectrometer (Horiba, HR800, France) and a fluorescence spectrophotometer (Hitachi, F-4610, Japan) respectively.

3. Results and discussion

The XRD pattern (Fig. 2a) of the samples collected from the graphite felt shows that five major diffraction peaks can be indexed as the (111), (200), (220), (311) and (222) crystal planes of 3C-SiC (JCPDS Card no. 73-1665). Apart from 3C-SiC, a broad diffraction peak at 22° was detected, which was attributed to the presence of amorphous SiO_2 phase [26]. Furthermore, the strongest (111) peak implies the predominant growth along [111] direction of 3C-SiC. Low-magnification SEM images (Fig. 2b and c) show that the morphology of as-prepared products is 1D fiber. The fibers are 150–500 nm in diameter and hundreds of microns in length. High-magnification SEM images (Fig. 2c–e) further reveal that the NW has a smooth surface, and many of the NWs are highly curved, suggesting good flexibility. EDS (Fig. 2g and h) detects Si, C, O and no Ni in their bodies and tips, suggesting that amorphous SiO_2 may exist on the surfaces of the NWs.

As shown in Fig. 3a, the bands from Si–O stretching vibrations (1100 cm^{-1}), Si–O–Si bending vibrations (480 cm^{-1}) and transversal optic mode Si–C vibrations (790 cm^{-1}) were detected by FT-IR, indicating that the as-achieved NWs are composed of SiC and amorphous SiO_2 [27]. Similarly, the Raman shift spectrum (Fig. 3b) demonstrates that the peak at 790 cm^{-1} conforms to 3C-SiC zone center transverse optical (TO) phonon modes, and an amorphous bulge at the center of 918 cm^{-1} corresponds to the amorphous SiO_2 [14]. Furthermore, a down shift appears compared to the previous result (TO mode at 796 cm^{-1} obtained from bulk 3C-SiC [28]). This may be attributed to the confinement effect, inner stress from the heterostructure or structural defect of achieved NWs [14,29]. However, the longitudinal optic (LO) phonon at about 960 cm^{-1} of β -SiC as reported [28,30,31] in the Raman spectrum was not detected, which may be caused by the LO shift to the low number due to the existence of amorphous SiO_2 on the β -SiC surface.

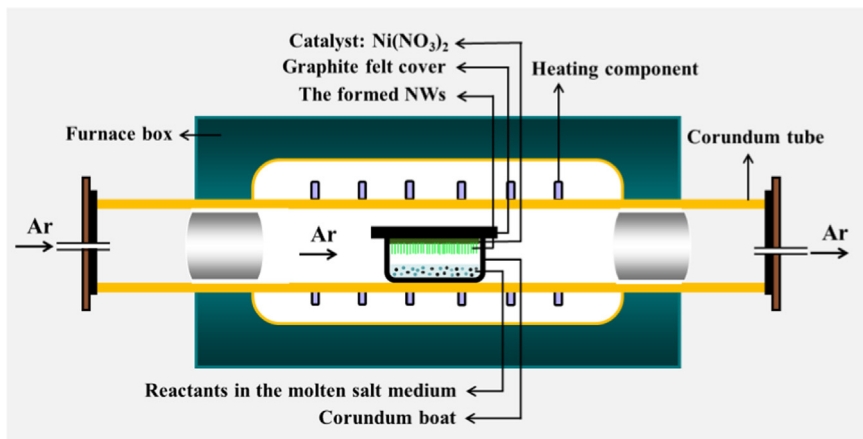


Fig. 1. Schematic experimental setup for the synthesis of SiC/SiO_2 core-shell NWs.

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