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Novel synthesis and characterization of silicon carbide nanowires on graphite flakes

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Abstract

Silicon carbide nanowires were synthesized on the surface of graphite by partially reacting with silicon powders in NaF–NaCl based salt at 1150–1400 °C in argon. The effects of temperature and time of heat treatment as well as Si/graphite ratio on synthesis of SiC nanowires were studied. The results showed that the formation of SiC nanowires started at about 1200 °C, and the amounts of SiC nanowires increased in the resultant powders with increasing temperature. Their morphologies were characterized by scanning electron microscopy and high-resolution transmission electron microscopy. It was found that β-SiC nanowires with diameter of 10–50 nm and various lengths grew along their preferred direction perpendicular to (111). The zeta potential of graphite was also increased after coating with silicon carbide nanowires. SiC nanowires that formed on the graphite surface acted as an anti-oxidant to a certain extent, and they protected the inner graphite from oxidation.

Keywords: Silicon carbide nanowires; Graphite; Molten salt; Synthesis

1. Introduction

Silicon carbide (SiC) nanowires have attracted considerable attention, because of their excellent mechanical and chemical properties, high thermal conductivity, and low thermal expansion coefficient [1–3]. They have been widely applied in the electronics, optics, machinery, advanced engineering, and metallurgy industries [4,5]. To date, several techniques have been explored for the synthesis of one-dimensional (nanotube, nanowire, and nanowhisker) SiC nanostructures [6–12], including laser ablation [13], chemical vapor deposition (CVD) [14], thermal evaporation process with iron as a catalyst [15], vapor–liquid–solid (VLS) growth mechanism [16], vapor–solid (VS) growth mechanism [17], solid–liquid–solid (SLS) growth mechanism [18], *etc*.

Graphite is used as a raw material in the metallurgy, advanced engineering, and coatings industries because of its low thermal expansion coefficient and high thermal conductivity. One of its major features includes non-wettability which is mostly beneficial when used in the context of liquid metal or liquid slag applications [19]. A drawback on the use of graphite is its low oxidation resistance, its increased porosity after drying, and concomitantly reduced mechanical strength. On the other hand, due to the non-wettability of graphite, the resulting castables flow poorly and a considerable water content is required [19]. Such a drawback could be overcome by surface treatment of the graphite which acts as wetting and oxidation resistance between the surrounding environment and the graphite's surface.

At present, graphite is modified by oxides, carbides, or metals, which have better water-wettability and dispersion abilities. Yilmaz et al. [20] used a sol–gel method, to form boehmitic alumina on the graphite's surface, but the treatment initiating this process' required materials is complicated. Chen et al. [4] produced SiC nanowires on a graphite substrate by thermal evaporation of silicon powders at high temperature. Li et al. [21] fabricated nickel-coated graphite nanosheets by an oxidation and reduction process and in MgO–C refractories, often added antioxidants to overcome these inherent drawbacks in the graphite [22]. However, most of the methods require to be manipulated in a complex, expensive and environmentally unfriendly way. Ceramic powders with whisker-, needle-, or plate-like morphologies could be prepared by molten salt synthesis (MSS) [23–27]. It was reported that a titanium carbide coating was synthesized on graphite flakes by MSS [19].

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In this work, molten salt synthesis (MSS) technique was used to synthesize silicon carbide nanowires on graphite flakes. The synthesis mechanism and the change in the structure of silicon carbide nanowires at different temperatures were also studied. Microstructure and growth mechanism of as-prepared SiC nanowires were investigated.

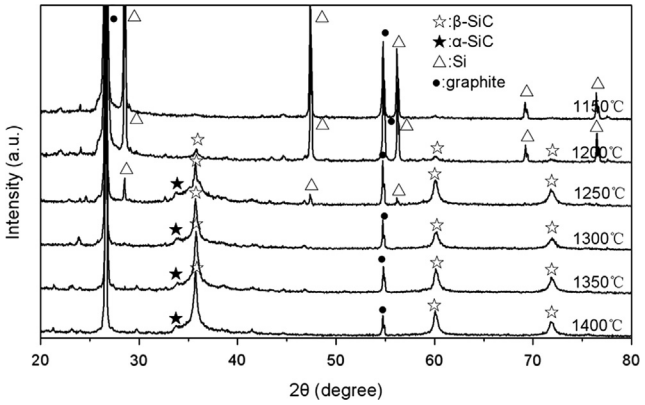

Fig. 1. XRD patterns of samples with a molar ratio of Si/graphite = 1/2 heat-treated for 3 h at different temperatures.

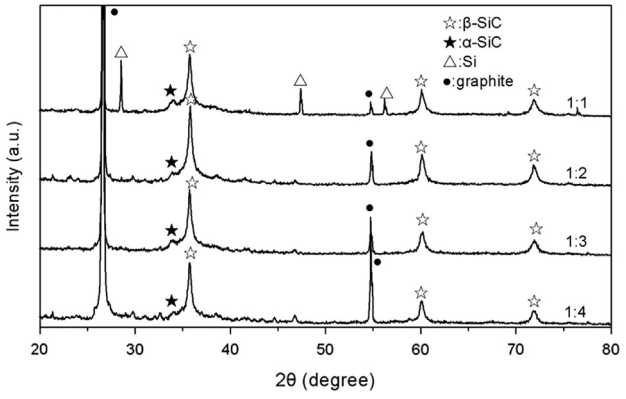

Fig. 2. XRD patterns of samples with a molar ratio of Si/graphite = 1/2 heat-treated at 1400 °C for different times.

2. Experimental materials and procedure

Silicon powder (purity ≥ 99% w/w, particle size ≤ 10 μm), graphite (purity ≥ 97% w/w, particle size 200–300 μm), NaF (purity ≥ 99% w/w) and NaCl (purity ≥ 99% w/w) were used as the raw materials. The molar ratios of Si to graphite were 1:1, 1:2, 1:3, and 1:4, and the weight ratio of NaCl to NaF was 10:1. Mixed salt combined with appropriate amounts of graphite and Si powder was then added. The powder mixture was placed in an alumina crucible and heated for 1, 3, and 5 h at 1150–1400 °C in argon using an alumina-tube furnace. After cooling to room temperature, the solidified mass was repeatedly washed with hot distilled water and filtered several times to remove the residual salt.

Phases in the resultant powders were studied by X-ray diffraction (XRD, Philips, X'Pert Pro), scanning electron microscope (SEM, FEI, Nova 400 Nano) and high-resolution transmission electron microscope (HRTEM, JEOL, JEM-2000F) equipped with energy dispersive X-ray spectroscopy (EDS), respectively. The amount of SiC phase present was calculated using a reference intensity ratio (RIR) from software X'Pert HighScore Plus [28]. The zeta potential of graphite and the treated graphite was determined by a zeta potential analyzer (Zeta Probe, Colloidal Dynamics). The powders and distilled water were confected into a 1% w/w suspension, and then the suspension was subjected to the test for its zeta potential. To elucidate the synthesis and oxidation mechanism of the powders, differential scanning calorimetry (DSC) and thermogravimetric analysis (TGA) were performed at temperatures up to 1200 °C with a heating rate of 10 °C/min in air by a simultaneous thermal analyzer (NETZSCH, STA 449C).

Table 1
Relative content of obtained SiC with a molar ratio of Si/graphite = 1/2 heat-treated for 3 h at different temperatures.

	Temperature of heat treatment (°C)			
	1250	1300	1350	1400
Relative content of SiC (wt%)	2	3	9	12

Table 2
Relative content of obtained SiC with a molar ratio of Si/graphite = 1/2 heat-treated at 1400 °C for different times.

	Heat treatment time (h)		
	1	3	5
Relative content of SiC (wt%)	3	12	9