

Growth of SiC nanowires on wooden template surface using molten salt media



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ABSTRACT

This paper examines the growth of SiC nanowires on a wooden template surface through the reaction of wooden template/silicon composites in static argon atmosphere, using molten salt media. The effects of temperature and salt/Si ratio on the growth of wooden template were investigated. Morphology and structure of the biomorphic SiC/C ceramics were characterized by X-ray diffraction (XRD), transmission electron microscopy (TEM), scanning electron microscopy (SEM) and thermogravimetric analysis (TGA). The pore size distribution within the porous SiC/C ceramics was investigated using automatic mercury porosimetry. The results show that the biomorphic cellular morphology of wooden template was remained in the porous SiC ceramic with high precision that consists of β -SiC with traces of α -SiC. SiC in the wooden template exists in the cellular pores in the form of nanowires. The SiC nanowires were formed at about 1250 °C by molten salt reaction between Si and C during the wooden-to-ceramic conversion.

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1. Introduction

Traditionally, the study for new and improved materials has been guided by inspiration and prediction from physics and chemistry. Design of novel ceramic materials with specific structures and functional properties by mimicking the hierarchical cellular structure of wood has recently attained particular interest [1–3]. Due to the unique characteristics of plant structure, scientists have taken advantage of them as bio-templates to produce advanced ceramic materials. The original structure of the plant fibers is retained in the final products, but the chemical composition is found to be substituted with other components. Biomimetic ceramics might be of interest for high-temperature exhaust gas filters, catalyst carriers, advanced microreactor systems, immobilization supports for living cells, microbes or enzymes, and waste water treatment, as well as acoustic and heat insulation structures [4–8].

In the previous works, the fabrication of porous carbide ceramics with wooden templates has been reported before [9–13]. For example, Qian et al. [5,6] produced a SiC ceramic by carbothermal reduction of oak wood charcoal. Vogli et al. [9] produced cellular silicon carbide ceramic by gas phase reaction with silicon monoxide of oak.

Some advanced SiC materials such as nanowires, nanorods, and nanometer-sized powders or whiskers, have been prepared by different methods including laser ablation [14], chemical vapor deposition (CVD) [15], thermal evaporation process with iron as a catalyst [16,17], vapor–liquid–solid (VLS) growth mechanism [18], vapor–solid (VS) growth mechanism [19], solid–liquid–solid (SLS) growth mechanism [20], magnetron sputtering method [21], etc. Also, many interesting works have been carried out that are focused on the growth of SiC nanostructures [22–24].

However, most of the methods are complex, expensive and environmentally unfriendly. In response to this, a low temperature and relatively low cost molten salt synthesis (MSS) technique has been developed recently [25,26]. Recently, some advanced materials such as nanofibers, nanorods, and whiskers, have been prepared by MSS [27–32].

So far, few people have attempted to prepare these porous materials using wooden templates and a molten salt synthesis (MSS) technique. In this work, a porous SiC/C composite ceramic was synthesized by MSS. The morphology changes and synthetic mechanism were investigated by XRD, SEM, TEM and TGA techniques.

2. Experimental materials and procedure

Phoenix wood was utilized as the plant template. After drying at 110 °C for 24 h, the phoenix was converted to a porous carbon

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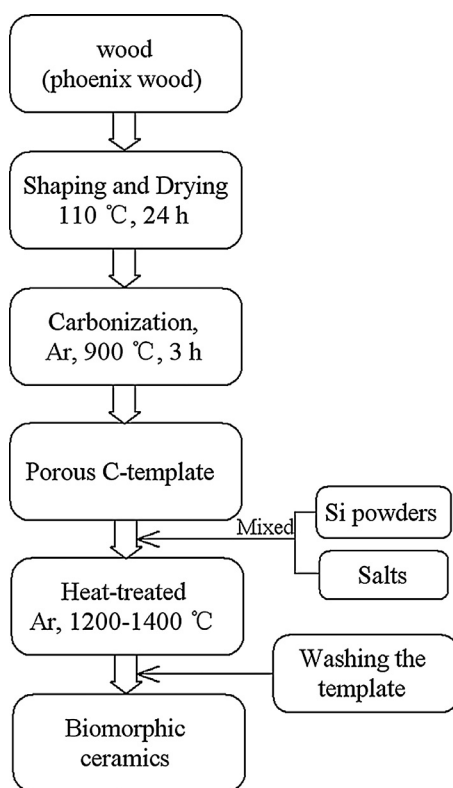


Fig. 1. Flow chart for the preparation of porous biomorphic SiC/C ceramics.

template by heating at 650 °C for 3 h and then heating at 900 °C for 3 h in an argon furnace. The carbon template was used as the carbon source for the synthesis of a SiC nanowires by reaction with Silicon powder (purity $\geq 99\%$ (w/w), particle size $\leq 10 \mu\text{m}$) in a molten salt mixture composed of KCl (purity $\geq 99\%$, w/w) and KF (purity $\geq 99\%$, w/w). The molar ratios of salts to Si were 1/1, 2/1, 3/1, and 4/1, the weight ratio of KCl to KF was 10/1. Ten porous carbon templates with each size of 3 mm cubes, was placed in an alumina crucible and covered by the powder mixture of 8 g, heated for 3 h at 1250–1400 °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. This process was repeated several times until no Cl^- was detected in the filtrate by an AgNO_3 solution (i.e.,

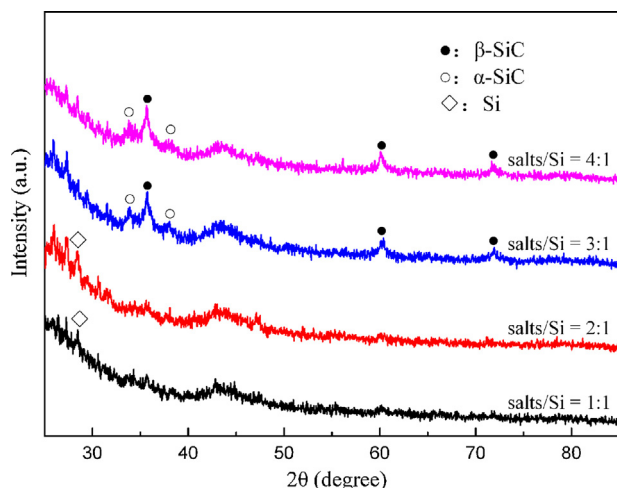


Fig. 2. XRD patterns of samples at different ratios heated at 1250 °C for 3 h.

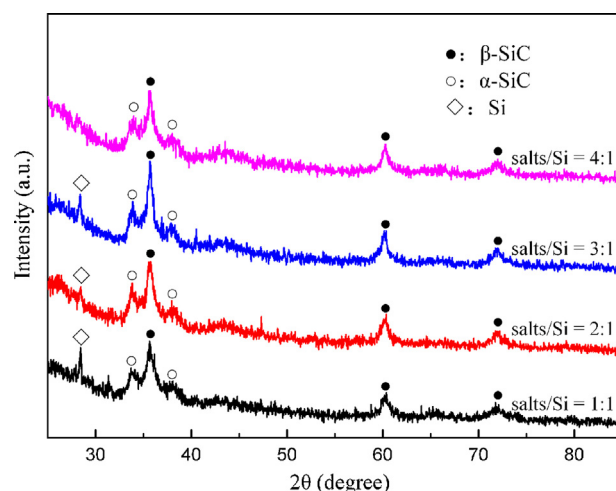


Fig. 3. XRD patterns of samples at different ratios heated at 1300 °C for 3 h.

no AgCl precipitation was observed when an AgNO_3 solution was added dropwise to the filtrate). The resulting bioceramics were oven-dried overnight at 110 °C before further characterization. The processing scheme for the preparation of porous biomorphic SiC/C ceramics is described in Fig. 1.

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, respectively. The pore size distribution within the porous SiC/C ceramics was determined by automatic mercury porosimetry (AutoPore IV 9500). To elucidate the synthesis and oxidation mechanism of the powders, thermogravimetric analysis (TGA) was performed at temperatures up to 1300 °C with a heating rate of 10 °C/min in air by simultaneous thermal analyzer (NETZSCH, STA 449C).

3. Results and discussion

3.1. XRD analysis

A series of experiments were also performed in which the molten salts/Si ratio was varied. Again, SiC peaks appeared in the resulting XRD patterns of the samples heated, in these cases, under a flowing argon atmosphere at 1350 or 1400 °C for 3 h, as shown in Figs. 4 and 5. It is seen that heat-treated temperature of 1350 and

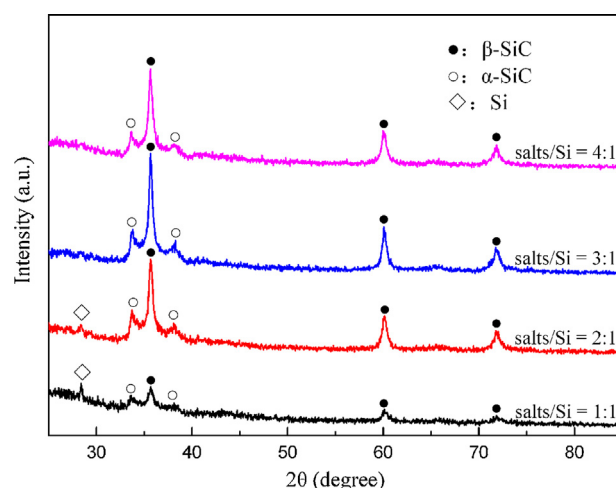


Fig. 4. XRD patterns of samples at different ratios heated at 1350 °C for 3 h.

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