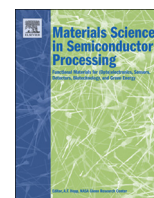




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Materials Science in Semiconductor Processing

journal homepage: www.elsevier.com/locate/mssp

Post-synthesis treatment of silicon carbide nanowires obtained in combustion synthesis

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ARTICLE INFO

Article history:

Received 22 July 2015

Received in revised form

7 October 2015

Accepted 30 October 2015

Available online 14 November 2015

Keywords:

Silicon carbide

Nanowires

Combustion synthesis

ABSTRACT

The paper describes the effects of thermal, mechanical and chemical treatment of silicon carbide nanowires (SiCNWs) obtained *via* combustion synthesis from Si and PTFE micropowders. The bundles of the combustion product are contaminated with various fractions of SiC and impurities like carbon, silicon and silica. Attempts have been made to debundle the wires and to remove impurities from SiCNWs samples, in order to obtain pure and shape-controlled material. The morphology of SiCNWs was examined as the material was physically and chemically modified.

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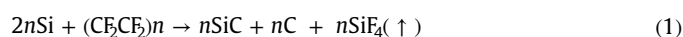
1. Introduction

Silicon carbide exhibits high mechanical strength, high hardness, low density, low thermal expansion coefficient, large band gap and excellent oxidation and corrosion resistance [1–4]. High thermal conductivity, high electric field breakdown strength and high maximum current density of SiC make it a promising semiconductor material for high-powered electronic devices.

SiC nanowires have very promising mechanical, electrical and optical properties due to their low dimensionality and quantum confinement effects. The material has potential applications in high frequency, high temperature and high power nanoscale devices. Other anticipated applications include nanosensors, blue or UV-light emitting diodes, electron emitters and nano-field effect transistors [5–14]. The addition of SiC whiskers has a beneficial effect on the fracture toughness, friction and wears resistance of ceramics [15–17]. SiCNWs may serve as a reinforcing phase in SiC/SiC composites with silicon carbide matrix and fibrous SiC reinforcement [18]. To reinforce bulk materials, SiCNWs do not have to be defined at a single-wire level. However, highly advanced electronic and optical applications require purified, debundled and precisely fractionated nanowires.

Over the past years, various methods have been employed to synthesize nanowires: *e.g.* direct combustion synthesis [19,20], carbothermal reduction [21–24] chemical vapor deposition [25–27], thermal evaporation method [28–30], template-based method [12,31] or using microwave heating [32].

It has been reported that 1D SiC nanostructures may be obtained using defluorination of poly(tetrafluoroethylene) – PTFE, using energetically autothermal SHS route (Self-Sustainable High-Temperature Synthesis, also known as thermolysis or combustion synthesis) [33–41]. The reaction produces voluminous material, so it has an advantage over small-scale methods of SiCNWs synthesis. The process proceeds as shown in Eq. (1), though, the exact reaction mechanism is much more complex [39]:



The combustion gaseous atmosphere and its initial pressure in a bomb calorimeter strongly affect the composition and the morphology of the product. Air, CO, CO₂, N₂, O₂, Ar and He atmospheres can be used to synthesize SiCNWs. Different products are obtained near the ignition tape and in the interior of a bomb calorimeter [33,34].

The effective post-synthesis treatment of SiCNWs obtained *via* combustion method is the key factor to prepare the material suitable for applications. The aim of the present study is to assess the usefulness of the post-synthesis treatment of the as-synthesized SiCNWs in order to obtain purified, debundled and fractionated material.

2. Experiment

2.1. Materials

The SiCNWs materials were synthesized from Si and PTFE micropowders (Sigma-Aldrich). The substrates were mixed in a

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weight ratio of 36:64 and put into a bomb calorimeter filled with air or carbon dioxide. The initial pressure of the gas was 1.0 MPa.

The mixture is then ignited by means of an electrically heated carbon filament. The process yields plenty of entangled SiC nanowires mixed with bulk SiC crystallites and a considerable amount of heterogeneous impurities. The details of the reaction are described elsewhere [33–41].

2.2. Experimental techniques

Chemical etching was carried out using 40 wt% HF_{aq.} – 24 h of soaking at room temperature and alternatively – 30 wt% of NaOH – 1 h of boiling (both chemicals from Avantor Performance Materials). These solutions proved their efficiency in removing of silica coating from silicon carbide nanowires. Free sedimentation and centrifugation techniques were used to separate the wires from dissolved silica and the etching solutions.

In order to crush the wires without supplying an excess mechanical energy, the SiCNWs were pressed with a manual hydraulic press under the force of 1 Tonn/0.2 cm² at room temperature to form a pellet ~5 mm in diameter and ~0.5 mm in height.

In order to debundle and fractionate the nanowires a planetary ball mill (*PM 100* by Retsch), ultrasonic washer (*Elmasonic S 10* by Elma) and a laboratory centrifuge (*MPW-350R, High Speed Brushless Centrifuge* by MPW Med. Instruments) and PTFE syringe filters by ROTH with a pore size of 1.0 μm were used.

SEM analysis of SiCNWs was conducted on carbon-coated samples using *Ultra Plus* instrument from Zeiss.

TG/DSC analysis of raw SiCNWs (synthesized in air) was carried out by using of STA 449 F1 Jupiter by Netzsch coupled with Netzsch Quadrupole Mass Spectrometer QMS 403D Aeolus. The analysis was performed with a heating rate of 15.0 °C/min; the top temperature was 1450 °C. The measurement was performed in the constant flow of N₂/O₂ (synthetic air): 40.0 ml/min and He (protective gas): 20.0 ml/min.

X-ray diffraction patterns of SiCNWs samples were measured on *X'Pert PRO, PANalytical* diffractometer equipped with a copper anode (CuKα₁) and an ultra-fast *PIXcel^{1D}* detector.

3. Results and discussion

Purification, debundling and fractionation steps of SiCNWs were realized by several physical and chemical routes (Fig. 1).

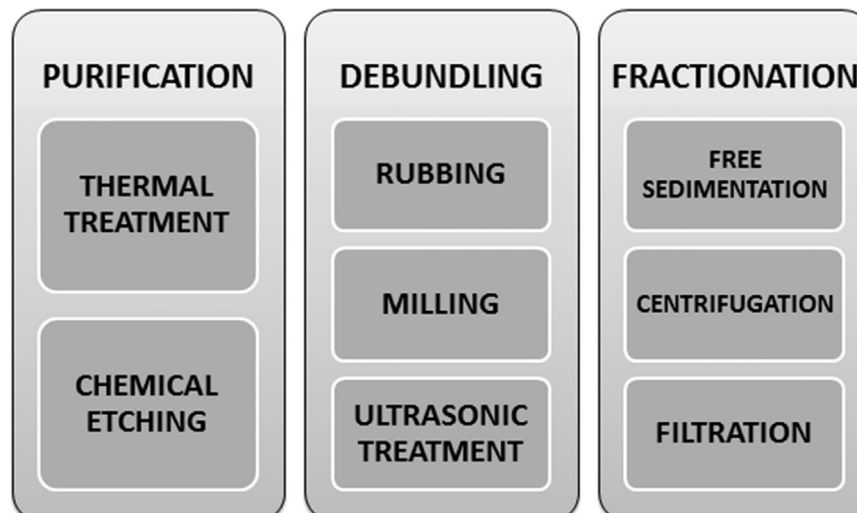


Fig. 1. Purification, debundling and fractionation methods of as-synthesized SiCNWs.

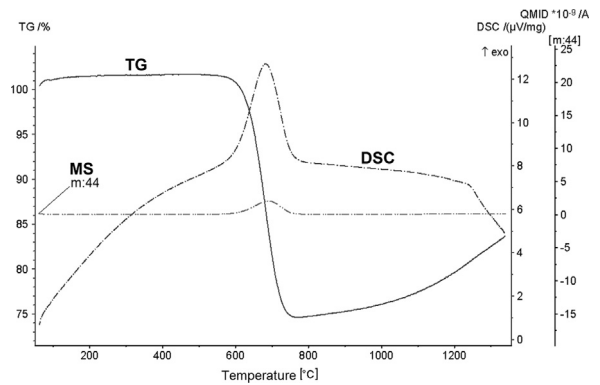


Fig. 2. TG/DSC analysis of raw SiCNWs material coupled with its thermal decomposition studies on MS.

The first stage of the SiCNWs post-synthesis treatment was the material purification by its annealing. The annealing conditions were determined on the basis of TG/DSC analysis. The results of the analysis of raw SiCNWs are presented in Fig. 2. The TG curve shows ca. 25% of mass loss up to ca. 750 °C coupled with an exothermic effect of ca. 4.5 μV/mg (DSC curve in Fig. 2). The effects result from the carbon burning – the raw material contains significant amount of soot. That is confirmed by mass analysis (MS curve in Fig. 2) which shows a peak of $m/z=44$ that can be attributed to CO₂ molecules release. Carbon dioxide is released between 650 and 750 °C therefore the soot is entirely burnt out during 750 °C temperature treatment.

According to the thermogram in Fig. 2 – the optimal temperature for the carbon removal ranges from 750 to 800 °C. Above the temperature of carbon burning, the mass increase of ca. 10% is observed – that is due to residual silicon oxidation (TG curve in Fig. 2). The endothermic effect of ca. 2 μV/mg, which started above 1200 °C, can be connected with crystalline SiO₂ polymorphs transformations (DSC curve in Fig. 2).

In order to avoid an excess silica contamination and to effectively remove the soot, 750 °C seems to be the proper temperature of raw SiCNWs annealing. However, there are some doubts if the treatment at the soot removal temperature could pose a threat to the thinnest wires due to surface oxidation. It is still not unequivocal if the carbon dioxide release originates from the soot removal or if some residues of SiC are burnt out too. The possibility of the SiCNWs loss could be diminished by lowering the burning temperature down to 600 °C – the temperature at which the soot oxidation just begins. Therefore in the

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