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Combustion synthesis of one-dimensional nanocrystalline silicon carbide

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Beta-SiC (cubic phase) nanowires (SiCNWs) have been grown spontaneously during the autothermal self-propagating high-temperature synthesis (SHS) from elemental silicon and poly(tetrafluoroethylene) (PTFE) powder mixtures in oxygen-enriched atmosphere. The combustion process was on-line monitored using high-speed photography in order to estimate the reaction processing time which was well below 1 s. From the emission spectroscopy the averaged combustion temperature was evaluated to be close to 2000 K. The products were characterized by wet chemical analysis, X-ray diffraction, scanning and transmission microscopy, and Raman spectroscopy. The raw products were processed by wet chemistry to obtain pure (above 90%) well-crystallized one-dimensional single crystals of SiCNWs.

1 Introduction

Silicon carbide (SiC) nanostructures have attracted recently a broad interest because of their novel morphologies and resulting specific properties, such as quantum-confined blue photoluminescence, electron emission, extreme hardness, chemical resistance and high thermal conductivity [1,2]. Nanowire-based novel numerous applications demand nanowires with controlled characteristics and high production yield [3]. Hence, the interest in one-dimensional SiC nanostructures which find their potential applications in materials science [4] and electronics [5]. Several high-temperature synthesis methods have been proposed to produce SiC nanowires, such as CVD and template growth, catalytic carbothermal reduction of silica, and plasma growth [1]. However, they virtually all suffer from low productivity, long processing times and specific equipment required. Here we propose a versatile, fast, one-step autothermal reaction to produce SiCNWs via SHS route which is known to produce many useful compounds [6]. We have already reported the formation of one-dimensional SiC nanometric structures through the reductive de-fluorination of PTFE with silicon-bearing compounds [7,8]. In this paper we present the results shedding light on the SiCNWs growth mechanism according to the general reaction scheme represented by

$$2\ Si_{(s)} + 2\ (CF_2)_{n(s)} \rightarrow C_{(s)} + SiF_{4(g)} + SiC_{(s)}. \qquad (1)$$

2 Experimental

The reactants (the stoichiometric mixture of powdered Si and PTFE, ≈ 5 g) were placed in a stainless-steel high-pressure reaction vessel constructed from the bomb calorimeter (Fig. 1). It was provided with the observation port to measure the emitted radiation from the combustion zone. During combustion pressure variations were monitored by using quartz pressure transducers. The experimental protocol and product characterization techniques have been outlined in details elsewhere [8]. The reaction was initiated by the resistant heating with the Kanthal wire. The emission photos were taken using a Canon digital camera. The light emission was recorded by the high-resolution spectrograph (Acton Research Corp.). After the reaction was completed, the off-gases were vented and a very voluminous, sponge-like grey solids were collected for further analyses.

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