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Combustion synthesis of Si-related crystalline nanostructures



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ABSTRACT

The unique self propagated high temperature synthesis (SHS technique) allows for an effective, energetically autothermal formation of different novel materials, including nanostructural, non-stoichiometric and bearing new phases, this all during fast reaction in a mixture strong oxidant/strong reducer. This all results from specific characteristics of the process: high temperatures/pressures, short reaction times and very fast quenching of gaseous reaction products during their expansion from combustion zone towards cooling zone. Silicon carbide as so-called refractory special ceramics possesses very special physical and chemical properties, especially in case of its nanostructural morphology this including 1-D (nanofibres). The results of the exploratory runs regarding the application of SiCNFs as polymer modifier, super-hard ceramic composites (SiCNFs/nano-SiC mixtures) or electron emitter are very encouraging indeed. The results will provide the information regarding the possible reaction channels. The fundamental parametric studies concentrate on the relationship between many process variables and SiCNFs formation efficiency. Relatively costly commercial reactants (Aldrich) were previously used which are now replaced by waste Tarflen (as oxidant). Their cost is a few orders of magnitude (!) lower. It will drastically reduce the costs of SiCNFs production since the operational costs of energetically autogenic SSW technique are obviously very low.

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1. Introduction

The limited control over nanomaterials formation and the lack of the knowledge of its growth mechanism definitely hamper the further progress of nanotechnology. With more than 1000 'nano' products already on the market, the high expectations regarding its development in the XXI century definitely encourage for more-in-depth studies of nanomaterials formation. Thus, new and fundamental aspects of the nanomaterials growth science are, evidently, worth of further exploration, especially considering the inherent limitations of existing production techniques. Among them, a search for new procedures of fabrication of the 1-D materials and the exploratory research on its characterization and applications are undoubtedly well justified.

Because of excellent various properties and specific characteristics due to 'nano' size dimensionality, the silicon carbide has long been sought as extremely interesting nanomaterial [1–3]. Its 1-D morphology (nanofibres, nanowires, nanotubes) is even more promising, mostly because of electronic and mechanical properties [4–7]. Thus, there have been many attempts to produce 1-D SiC [8–14].

Combustion synthesis (SHS) is a well-known method [15] to produce many useful carbon-related compounds, such as SiC

nanowires (SiCNWs), active carbons [16], and magnetic carbon nanoencapsulates [17]. High temperatures/pressures of extremely fast redox reactions provide favorable conditions for the processing of high-temperature melting materials. Moreover, these reactions are thermally autogeneous and usually yield nanosized products. Essentially, there are also no limitations regarding the processed reactants as long as coupled strong reducers/oxidants are considered [18]. To the best of our knowledge there were, however, no efforts to intentionally produce these novel 1-D nanostructures so far while the lengthy experience of the authors shows that through the careful selection of combustion synthesis reagents the sought product can be obtained.

2. Experimental

2.1. Synthesis

Synthesis of SiC nanofibres in a typical system uses a mixture of silicon powder (< 43 μm, Sigma Aldrich, 36 wt%) and PTFE (100 μm, Sigma Aldrich, 64 wt%) and can be carried out under several different atmospheres. We previously described the various attempts to increase process efficiency and to get knowledge of the mechanism of SiC nanowires growth [19,20]. The studies focus on gaining the full knowledge of the formation

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mechanism, which will allow to optimize the synthesis and enable to maximize the effectiveness of the synthesis. Regarding the process economic characteristics, it is obvious that the cost of nanomaterials is always one of the barriers to the development of nanotechnology. The commercial reagents (Sigma-Aldrich[®]), previously used in the synthesis of pure SiCNWs, are relatively expensive. Thus, the presented study attempts to use waste materials as initial reactants (instead of expensive “catalogue” starting materials) in order to reduce costs. The main idea is to use re-ground PTFE powder. It is a secondary PTFE, which is obtained as a leftover of finished products—plates, rods. It has only slightly different properties comparing to the ‘virgin’ PTFE.

The synthesis was performed by combustion of mixed reactants in a reactor which is a modified calorimetric bomb described elsewhere [19]. The syntheses were carried out either in argon or in air. The processing parameters are summarized in Table 1. After the synthesis, the system was flushed with buffer gas and the solid products were collected. The crude product was purified by boiling for 0.5 h in 30% solution of KOH (to remove un-reacted Si and SiO₂) and calcinated in air at 600 °C for 3.5 h (amorphous carbon removal).

2.2. Characterization

The resulting solid product had a spongy morphology (for argon—more loose resembling carbon black), but the product from the ignition tape had a substantially different morphology of a dense greyish hard particles (Fig. 1). The differences in the morphology of the products were more pronounced for tests performed in Ar atmosphere. The “dense fragments” made up 12 wt% of the entire product for air and 17 wt% for argon. Analysis of the resulting products allowed for the mass balance and the degree of conversion of Si to SiC determination as a key parameter regarding the efficiency of the process. The composition of solid products of reaction was confirmed by performing XRD analysis (Powder Diffractometer, Bruker D8 Discover, Cu K α radiation). The elemental analysis (CHNS analyzer mode VARIO ELIII, Elementor) confirmed the effectiveness of isolation of SiC fibers.

Table 1
Operating parameters.

Atmosphere	Sample weight [g]	Initial pressure [MPa]	Peak pressure [MPa]	Final pressure [MPa]	Product weight [g]
Air	8.973 ± 0.184	1.02 ± 0.03	5.03 ± 0.50	1.37 ± 0.05	2.395 ± 0.029
Argon	9.133 ± 0.250	1.01 ± 0.02	4.88 ± 0.32	1.29 ± 0.03	2.886 ± 0.178



Fig. 1. The morphology of the products; Left—typical fibers in the spongy form of the product, right—dense fragments of different morphology at the nano level.

3. Results and discussion

Fig. 1 shows the different morphologies of products both at the macroscopic level and at the nano level. This type of product has not yet been obtained in any of the earlier experiments carried out by the authors, where the interior of the bomb was typically filled with SiC nanowires. Material balance was performed to estimate the degree of conversion of silicon—Table 2. During the synthesis Si present in starting materials is transformed to such solid compounds as SiC and SiO₂, with the remaining un-reacted Si. Gaseous products of the synthesis such as SiF₄ are not included in this balance. The purity of the obtained product was confirmed by the results of the elemental analysis shown in Table 3. They are

Table 2
Conversion rates for silicon performed experiments (A) and the comparison to a typical system (B).

	Substraty	Atmosphere	Total Si	Si [%]	SiO ₂	SiC
(A)	Si/waste tPTFE	Air	84	21.9	6.6	12.6
	Siwaste PTFE	Argon	82	20.5	2.8	15.1
(B)	Si/PTFE	Air	92	10	40	20
	Si/PTFE	Ar	75	30	7	16

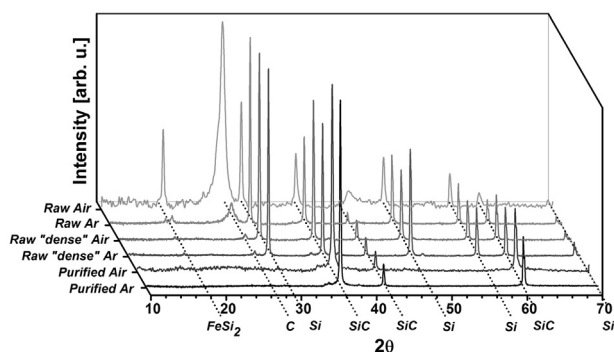


Fig. 2. XRD spectra of products resulting from runs with waste PTFE in air and Ar atmosphere.

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