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Ultra-fast efficient synthesis of one-dimensional nanostructures

Agnieszka Dąbrowska^{*1}, Andrzej Huczko¹, Michał Soszyński¹, Badis Bendjemil², Federico Micciulla³, Immacolata Sacco³, Laura Coderoni³, and Stefano Bellucci³

¹Department of Chemistry, Warsaw University, 1 Pasteur str., 02-093 Warsaw, Poland

²Department of Physics, University of Badji-Mokhtar, LEREC, BP. 12, 23000 Annaba, Algeria

³Laboratori Nazionali di Frascati, Via E. Fermi 40, 00044 Frascati, Roma, Italy

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* Corresponding author: e-mail aggy-dab@wp.pl, Phone +48-22-8222375

Self-propagating high-temperature synthesis (SHS) can be regarded as an efficient method to obtain new nanomaterials. Different starting mixtures of magnesium powder with various carbonates (Li_2CO_3 , Na_2CO_3 , CaCO_3 , FeCO_3 , $(\text{NH}_4)_2\text{CO}_3$) were tried and the auto-thermal reactions were carried out under both reactive (air) and neutral atmosphere (argon) with an initial

pressure of 1 or 10 atm to yield novel nanomaterials. Both SiC nanofibres and novel branched SiC nanostructures were also obtained from Si/polytetrafluoroethylene (PTFE) mixtures and their synthesis and purification have been optimized. The application of those one-dimensional (1-D) SiC nanostructures as a composite filler is presented.

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1 Introduction In a search for new methods to produce novel nanomaterials we propose the combustion synthesis [self-propagating high-temperature synthesis (SHS)], a thermal-explosion, autogenous mode of fast redox reaction between the strong reducing agent and oxidant. A careful selection of powdered reactants, which are very basic chemicals, can result in the efficient formation of novel nanostructures. The process takes place usually under far-from-equilibrium conditions so it may lead to products with a new morphology and stoichiometry. It is known that the one-dimensional (1-D) nanostructures (nanowires, nanotubes) often show distinct properties from their bulk counterparts because of the radial confinement. Here, we present a fast, simple, easy to operate and one-step chemical synthesis of branched 1-D SiC nanostructures, SiC nanofibres and 1-D nanocarbons from carbonates via an SHS route. The process can be easily escalated by using a bigger reactor chamber. Finally, a possible application of those nanomaterials in nanocomposites is proposed.

2 Experimental The combustion was carried out in a high-pressure reactor, the modified calorimetric bomb provided with a polycarbonate window (Fig. 1) to perform *in situ* spectral registration of the emitted light. The details of the experimental procedure have been outlined elsewhere [1]. The effect of process parameters, such as: reactant

composition (powdered Mg/carbonate, Si/PTFE or Si/PTFE/ NaN_3 mixture), initial combustion pressure (1–20 atm) and atmosphere (air, argon, nitrogen) was studied. The products were characterized using X-ray diffraction (XRD), scanning electron microscopy (SEM), transmission electron microscopy (TEM) and Raman spectroscopy [2]. The protocol for the chemical purification of sought products was proposed. The powder mixture of the reactants was placed in a quartz crucible (with the immersed carbon tape), the reaction was initiated by Ohmic heating and terminated usually within less than 2 s.

3 SHS process in carbonate systems Different starting homogenous mixtures of Mg powder (Sigma–Aldrich, >99%) with various carbonates (Li_2CO_3 from ‘POCh’, Na_2CO_3 from Sigma–Aldrich, >99%, CaCO_3 from Sigma–Aldrich, >99%, FeCO_3 from Polish Institut of Geology, $(\text{NH}_4)_2\text{CO}_3$ from Sigma–Aldrich, >99%), as a source of elemental carbon, were tried. Reactions were carried out under reactive (air) or neutral atmosphere (argon) at an initial pressure of 1 or 10 atm. Fe, Co and Ni powders were used as catalysts (~325 mesh).

3.1 Product characterization Under the applied conditions the presence of crystalline MgO in products confirmed by XRD analysis (not presented here), even for