PRELIMINARY STUDIES ON NANOCOMPOSITE BASED ON HIGH QUALITY SILICON CARBIDE NANOFIBERS

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Abstract–*Nanocomposites are nowadays the most promising new materials due to their unique properties (such as high mechanical strength, chemical and thermal resistance). The nanocomposite matrix is blended with a nanostructured filler. In this study, Silicon Carbide nanofibers (NFSiC) and their bundles were tested as a reinforcement of two epoxy resins: EPIKOTE 828 and EL 20. PAP-4 (33 phr) and P-900 (40 phr) were used as hardeners in the two cases, respectively. Several samples were prepared in the range between 0.1 and 5 % wt for both types of resins and fillers (NFSiC and NFSiC bundles). Mechanical and electrical properties were tested. The fillers were obtained using a new simple, fast, low-cost and efficient method to synthesize nanomaterials: Self-propagating High-Temperature Synthesis (SHS). The produced nanofillers were analyzed by Scanning Electron Microscope (SEM), Trasmission Electron Microscope (TEM). They were purified, as well. The nanocomposites obtained using such nanofillers were assayed by SEM and TEM techniques.*

Keywords*: Nanocomposites, Silicon Carbide nanofibers, Mechanical, properties.*

1. INTRODUCTION

Many potential applications have been proposed for silicon carbide (SiC) nanostructures, including high-strength composites, nanosensors and nanodevices.

Limitations in processing are an important barrier that has to be overcome in order to develop these applications. Silicon carbide (SiC) ceramics are being used for high temperature materials, because they have several characteristic properties, such as high elastic modulus and hardness, excellent thermal and chemical stabilities, low thermal and electrical conductivities, and relatively low thermal expansion coefficients. The silicon-carbon bond is thermodynamically nearly as strong as a single C-C bond. The value for dissociation energy of the silicon-carbon bond has been reported as 318 kJ/mol and for the C-C bond as 345.6 kJ/mol [1,2]. Silicon carbide, the simplest silicon-carbon compound, is especially stable. The best known allotropic form of SiC is ß-SiC. This compound has a diamond structure, that is, each Si atom is surrounded by four C atoms, and each carbon atom is surrounded by four Si atoms [3]. In this paper it is showed a fast method to synthesize SiC nanofibers using the Self-propagating High-Temperature Synthesis (SHS). This method is simple, fast, low-cost and efficient. The exothermic, self-sustainable process is initiated by the resistive heating and terminated within few seconds.

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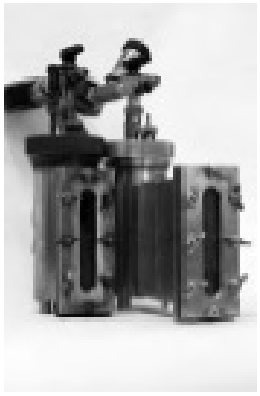
2. SYNTHESIS PROCESS

The starting homogenous mixture for NFSiC production consisted of Si (<43 μm) and polytetrafluoroethylene (PTFE) (1μm) powders with NaN_3 added in case of the NFSiC bundles synthesis. Initial pressure was equal to 1 MPa and the reaction was carried out under the air atmosphere, as seen in figure 1. Using the SHS process Si/PTFE/NaN_3 systems Si (Sigma–Aldrich, <43 mm, 99%), PTFE(Sigma–Aldrich, 1 mm, powder) NaN_3 (Sigma–Aldrich, >99.5%) in different proportions, a variety of interesting nanomaterials was obtained. Mainly, SiC nanofibres have been efficiently produced and during the parametric studies the optimal

synthesis conditions were found: the stoichiometric composition of reactants (36 wt% Si and 64 wt% PTFE), air atmosphere (as the oxygen seems to play an important role in SiC growth), and the initial pressure equal to 10 atm. Optimal NaN_3 content was found to be 55 wt% of reactants. In the second experiment the basic composition of reactants was modified by the addition of NaN_3. Its content varied between 0.1 and 85 wt% in Si/PTFE mixture. Under initial air pressure equal to 10 atm the efficient formation of novel nanostructures, the bundles of SiC fibres, was observed.

a)



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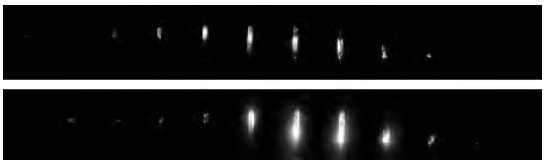
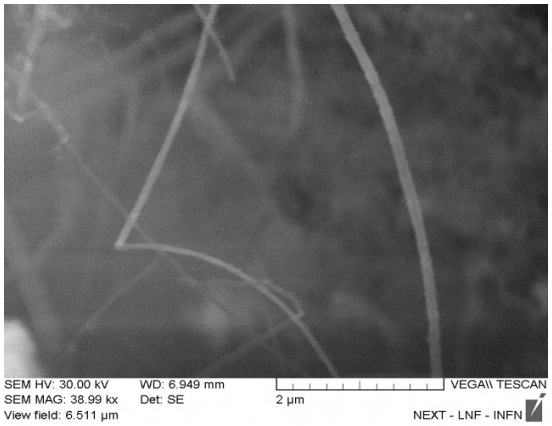


c)

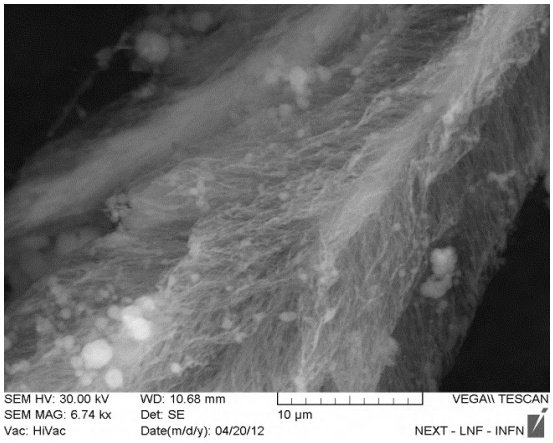
Fig. 1. a) and b) Images of the Synthesis Chamber; **c)** a photo registration of combustion wave propagation in Si/PTFE/NaN3 system (air, 0.1 MPa) for $\lambda > 720$ nm (upper row) and for a visible range (second row).

The smaller crystallite outgrowths were found on fibres surfaces. The content of those nanostructures increases with decreasing the initial pressure. The XRD characterization allowed us to identify the following compounds: SiC, SiO_2, Na_2SiF_6 and Si_3N_4[4].

The following figure 2 shows Scanning Electronic Microscope images of two kinds of synthesized nanofibers.



a)



b)

Fig. 2. a) SiC nanofibers b) Bubbles of SiC nanofibers.

The Trasmission Electron Microscope micrographs show that the nanofibers diameter ranges between 20-200 nm in the micron length scale. Large crystalline domains of 10nm appear in the analysis, as seen in figure 3.

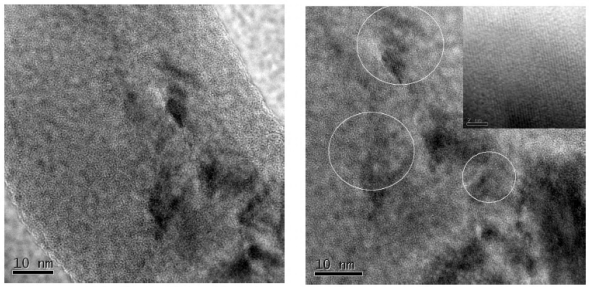


Fig. 3. Pictures of SiC nanofibers taken by Transmission Electronic Microscope.

3. EXPERIMENTAL

A series of composite samples, realized in different thicknesses, using an epoxy resin, i.e. Epikote 828, a curing agent called PAP4 (i.e. a modified TEPA). EPIKOTE™Resin 828, performed by MomentiVe Specialty Chemicals Inc., known as Hexion, was used for composites fabrication. EPIKOTE Resin 828 is a medium viscosity liquid epoxy resin produced from bisphenol A resin and epichlorohydrin. It contains no diluent. EPIKOTE 828 provides good pigment wetting and good resistance to filler settling and a high level of mechanical and chemical resistance properties in cured state [5, 6]. Modified Epoxy resin EL-20 is produced by bisphenol F. It is a resin at low viscosity and high wettability. P-900 is the commercial hardener used as curing agent. Several samples were made with different loading of fillers, as seen in table 1 and figure 4.

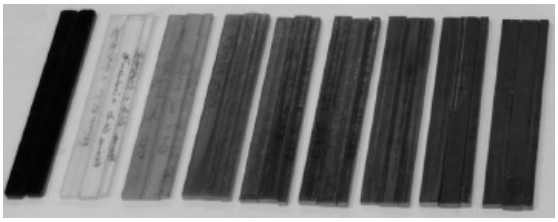


Fig. 4. Picture of nanocomposite samples.

Table 1. Matrixes and Materials used as Fillers

Matrix	Filler	Filler content [phr]
Epikote 828 & PAP4	SiCNFs	0.1, 0.2,03,0.4,0.5,0.6,0.7,0.8,0.9,1.0,1.5. 2.0,3.0
	SiCNFs Bundles	0.1, 0.2,03,0.4,0.5,0.6,0.7,0.8,0.9,1.0,1.5. 2.0,3.0
EL-20 & P-900	SiCNFs	0.1, 0.2,0.3,0.4,05,0.6,0.7,0.75,0.8, 1.0,2.0,3.0,4.0,6.0
	SiCNFs Bundles	0.1, 0.2,0.3,0.4,0.5,0.6,0.7,0.8

The results show that already at less than 1wt%, the nanofiller changes the mechanical properties of the matrix. For the majority of samples, an increase in Young modulus was observed (up to a 45% increase for 4 phr content of SiC). The enhancement of ultimate tensile strength was observed for the matrix with 0.25 phr content of SiC bundles, figure 5.

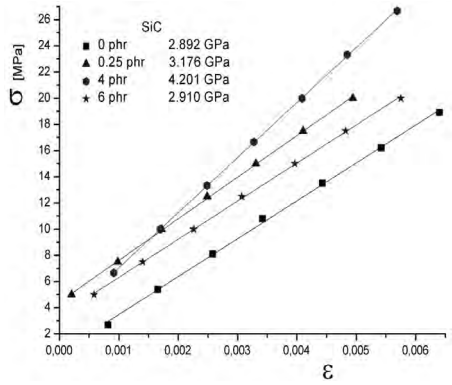


Fig. 5. Young modulus for different fillers content in El 20 epoxy resin matrix.

The process of dispersion was not optimized, yet. Scanning Electron Microscope pictures of nanocomposites samples showed a slight dispersion of nanofibers into the epoxy matrix, as it possible to see in the figure 6.

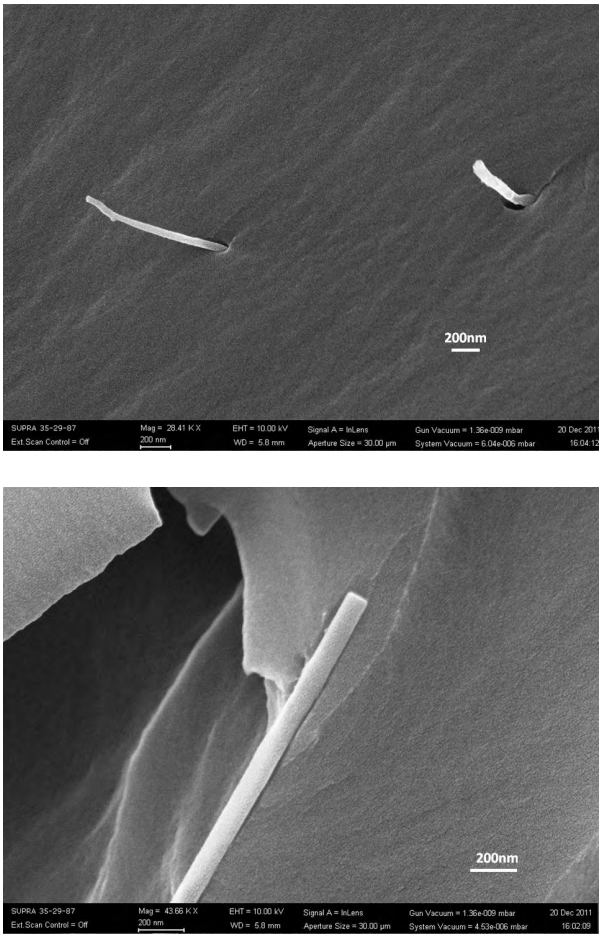


Fig. 6. Images of SiC nanofibers which come out form epoxy matrix (Epikote828) loaded at 0.5%wt; the scale bar length is 200nm.

4. CONCLUSIONS

A variety of 1-D nanostructures (1-D nanocarbons and different 1-D SiC nanosamples) have been successfully produced by SHS technique, including bundles of SiC nanofibres. The latter were never used before as composite filler (in contrast to SiC particles) and because of their high structural organization they seem promising for further detailed investigations.
The mechanical properties of the obtained nanocomposites turned out to be improved by the presence of the nanofiller, with respect to the properties of the pure matrix. Possible applications are being currently considered.
Positive ultrasonication influence on material properties has been confirmed. As the Young modulus of purified SiC reach the level of 450 GPa, there is still room for a significant improvement in which finding out the type of resin providing an optimal interaction with the filler, as well as setting up an efficient dispersion method are ingredients of crucial importance.

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