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KOMPOZYTY
COMPOSITES

Vijayan Poornima[1], Sabu Thomas[2], Andrzej Huczko[3]*

[1, 2] Mahatma Gandhi University School of Chemical Sciences, Kottayam, Kerala, India
[3] Warsaw University Department of Chemistry, Pasteur 1, 02-093 Warsaw, Poland
** Corresponding author. E-mail: ahuczko@chem.uw.edu.pl*

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EPOXY RESIN/SiC NANOCOMPOSITES. SYNTHESIS AND CHARACTERIZATION

The sulicon carbide (SiC) nanofibers were produced by self-propagating high-temperature synthesis (SHS). Silicon and polytetrafluoroethylene (TEFLON™) powdered mixture was used as starting reactants. The raw product was chemically processed to isolate and purify SiC nanofibers, several nm in diameter and a length in a micron range. The nanomaterial was used to reinforce epoxy thermosets. Epoxy resin/SiC nanocomposites were prepared by using either ultrasonication or high shear mixing procedures. The dispersion and flexural properties of the nanocomposites prepared by two methods were evaluated and compared. Ultrasonication, in comparison to shear mixing method, yielded superior nanoscale dispersion according to scanning electron microscopy (SEM). As a result of the improvements in nanoscale dispersion, the corresponding improvements in flexural strength and modulus of produced composites were achieved. The better dispersion of SiC nanofibers and properties were obtained with nanocomposite containing 0.25 Phr (parts per hundred epoxy resin) nanomaterial. Thus, even such a low content of 1-D nanomaterial distinctly improves the properties of a composite.

Keywords: epoxy resin, SiC, SHS, nanocomposites, nanofibers, synthesis

NANOKOMPOZYTY: ŻYWICA EPOKSYDOWA/WĘGLIK KRZEMU SYNTEZA I CHARAKTERYSTYKA

Otrzymywano nanowłókna węglika krzemu (SiC) na drodze samorozprzestrzeniającej sie syntezy wysokotemperaturowej. Reagentem była mieszanina proszków krzemu oraz politetrafluoroetenu (Teflon™). Otrzymany produkt poddano obróbce chemicznej w celu izolacji i oczyszczenia nanowłókien SiC, mających średnice rzędu kilkunastu-kilkudziesięciu nanometrów i długość kilku mikronów. Otrzymany nanomateriał zastosowano w celu modyfikacji - wzmocnienia termoutwardzalnej żywicy epoksydowej. Syntezowano nanokompozyty, stosując mieszanie ultradźwiekowe bądź wysoko wydajne mieszanie ścinające. Określono i porównano uzyskany stopień dyspersji oraz giętkości otrzymanych nanokompozytów. Badania mikroskopowe (SEM) wykazały, że mieszanie ultradźwiękowe jest znacznie efektywniejsze, jeśli chodzi o uzyskany stopień dyspersji nanowłókien. W wyniku podwyższenia dyspersji w nanoskali uzyskane nanokompozyty wykazały poprawę właściwości wytrzymałościowych. Najwyższy stopień dyspersji i najlepsze właściwości wykazały nanokompozyty zawierające nanowłókna SiC zmieszane z żywicą epoksydową w stosunku 0,25/100. Tak więc nawet tak niski dodatek jednowymiarowego materiału istotnie polepsza właściwości kompozytu.

Słowa kluczowe: żywica epoksydowa, SiC, SHS, nanokompozyty, nanowłókna, synteza

INTRODUCTION

Incorporation of nanofillers into various types of polymers has aroused great attention in materials science to accomplish multifunctional nanocomposites with enhanced mechanical and thermal properties [1-3]. The new type filler, silicon carbide nanofibers have been attracting considerable attention due to their excellent properties such as high thermal stability, high thermal conductivity, good mechanical properties and chemical inertness [4-6]. Besides, they have been suggested as a good reinforcement material and suitable to be used as the reinforcing component for composites due to their much larger strength over their bulk counterparts and strong interfacial bonding [7]. A recent work on the epoxy based SiC nanofiber composites reported improved wear resistance, hardness and tensile strength much higher than that of pure epoxy resin [8]. In this work, SiC nanofibers were synthesized by self-propagating high-temperature synthesis [9] and composites samples containing SiC nanofibers and epoxy resin were fabricated using two different methods: ultrasonic and high shear mixing methods. The efficacy of the dispersion method on nanocomposite formation has been evaluated using scanning electron microscopy (SEM) while the corresponding improvements in mechanical properties have been determined.

MATERIALS

Silicon powder (Aldrich, 325 mesh, 99% purity) and PTFE (polytetrafluoroethylene) powder (Aldrich, 1 μ) were used as a reducer and an oxidant, respectively. Diglycidyl ether of bisphenol A (Araldite GY 250) with an epoxide equivalent weight of approximately 183 g/eq, provided by Huntsman advanced materials was used. The curing agent and the accelerator were Nadic methyl anhydride (HY906) and Benzyl dimethyl amine (DY062) respectively, also provided by Huntsman Advanced Materials.

EXPERIMENTAL

The SiC nanofibers were synthesized via SHS technique using the stoichiometric mixture of reactants and purified following the procedure outlined elsewhere [10, 11]. Figure 1 presents SEM images of a raw product and purified nanomaterial.

To fabricate composites, SiC nanofibers (0.1 to 0.5 Phr) were added into epoxy resin. The mixtures of epoxy resin and SiC nanofibers were dispersed using two methods:

Method 1: mixed for 10 minutes using high shear mixer (IKA Ultra-Turrax Digital Homogenizer)
Method 2: mixed for 10 minutes using high shear mixture and followed by ultrasonication for 30 minutes using tip sonicator.

After dispersion stoichiometric amount of hardener and accelerator were added into the mixture and mixed well. The mixture was poured into a metallic mould to get a sheet of composites. The cryogenic fracture surface of the nanocomposites was inspected used scanning electron microscopy (JEOL JSM 6390) after platinum coating. SEM images were obtained under conventional secondary electron imaging conditions with an accelerating voltage of 10 kV. Flexural strength and modulus of the samples were measured by the universal testing machine (Tinius Olsen) with a cross-head rate at 1.7 mm/min according to ASTM D790 under a three-point bend configuration.

RESULTS AND DISCUSSION

SEM images of the fracture surface of neat epoxy resin and epoxy resin/SiC nanocomposite prepared by

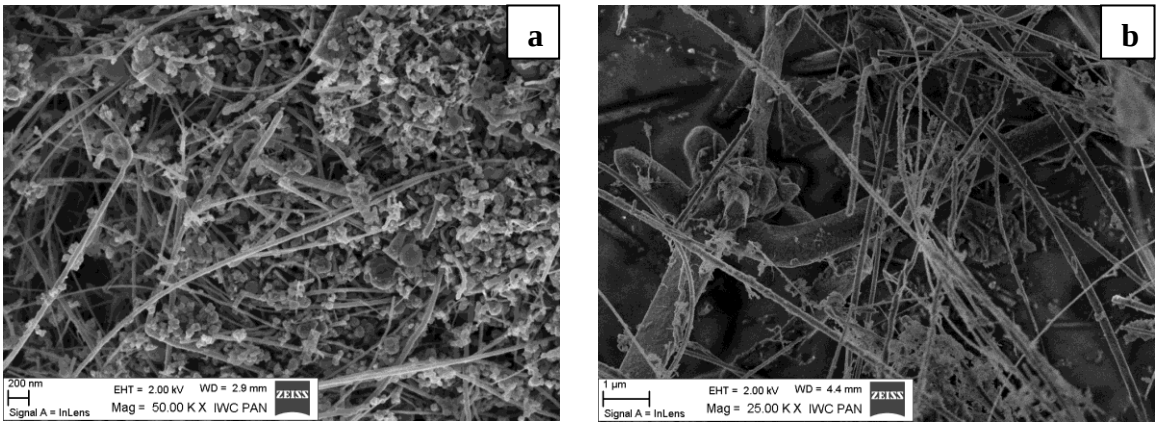

Fig. 1. SEM images of (a) raw product and (b) purified SiC nanofibers

Rys. 1. Zdjęcia mikroskopowe SEM (a) otrzymanego produktu i (b) oczyszczonych nanowłókien SiC

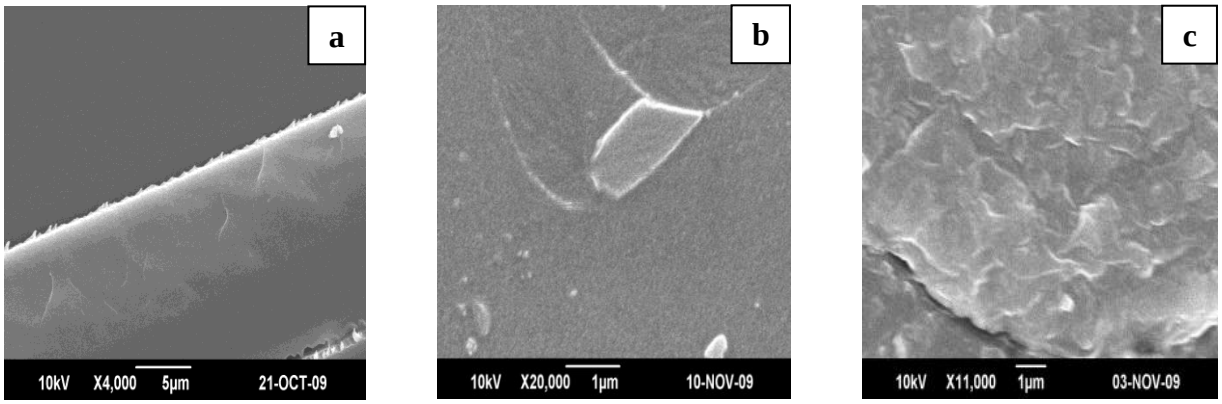

Fig. 2. SEM images of fracture surface of (a) neat epoxy resin; (b) epoxy resin/0.25 Phr SiC nanocomposite prepared by high shear mixing method and (c) epoxy resin/0.25 Phr SiC nanocomposite prepared by ultrasonication method

Rys. 2. Zdjęcia mikroskopowe SEM: a) żywicy epoksydowej bez dodatków; b) nanokompozytu utworzonego (wysoko wydajne mieszanie ścinające) z mieszaniny żywica epoksydowa/0,25 Phr SiC; c) nanokompozytu utworzonego (mieszanie ultradźwiękowe) z mieszaniny żywica epoksydowa/0,25 Phr SiC

both methods are shown in Figure 2. Failure surface of neat epoxy resin shows typical characteristic of brittle fracture. The surface is smooth and crack propagation uninterrupted. As seen in the images, epoxy resin/SiC nanocomposite prepared by ultrasonication have uniform distribution, thus having better compatibility with the matrix epoxy resin, whereas epoxy resin/SiC nanocomposite prepared by high shear mixing method shows dense agglomerates within the matrix epoxy resin.

Figure 3 presents the variation in dispersion with com- position in nanocomposites prepared by ultrasonication. Low magnification SEM images confirm the presence of uniform dispersion in nanocomposite containing 0.25 Phr SiC nanofibers. The dispersion of nanofibers is not uniform in higher loading (0.5 Phr) system.

Table 1 shows flexural modulus, flexural strength and ultimate elongation of epoxy resin/SiC nanocomposites prepared by two different methods. Addition of 0.25 Phr SiC has a high impact on flexural properties of epoxy resin. Epoxy/SiC nanocomposite prepared by high shear mixing shows a 16.2% flexural strength enhancement, while epoxy/SiC nanocomposite prepared by ultrasonication shows a 25% flexural strength enhancement. The enhancement in flexural strength is attributed to the uniform dispersion achieved by ultrasonication method.

Figure 4 shows the variation of flexural modulus and flexural strength with SiC nanofiber content in nanocomposite prepared by ultrasonication method. The modulus of the epoxy resin/SiC nanocomposite increases continuously with increase in SiC content. Better flexural strength has been shown by epoxy resin nanocomposite with 0.25 Phr SiC nanofiber. The strength begins to decrease with 0.5 Phr loading, although the gain in modulus is maintained. The reason for the decrease in

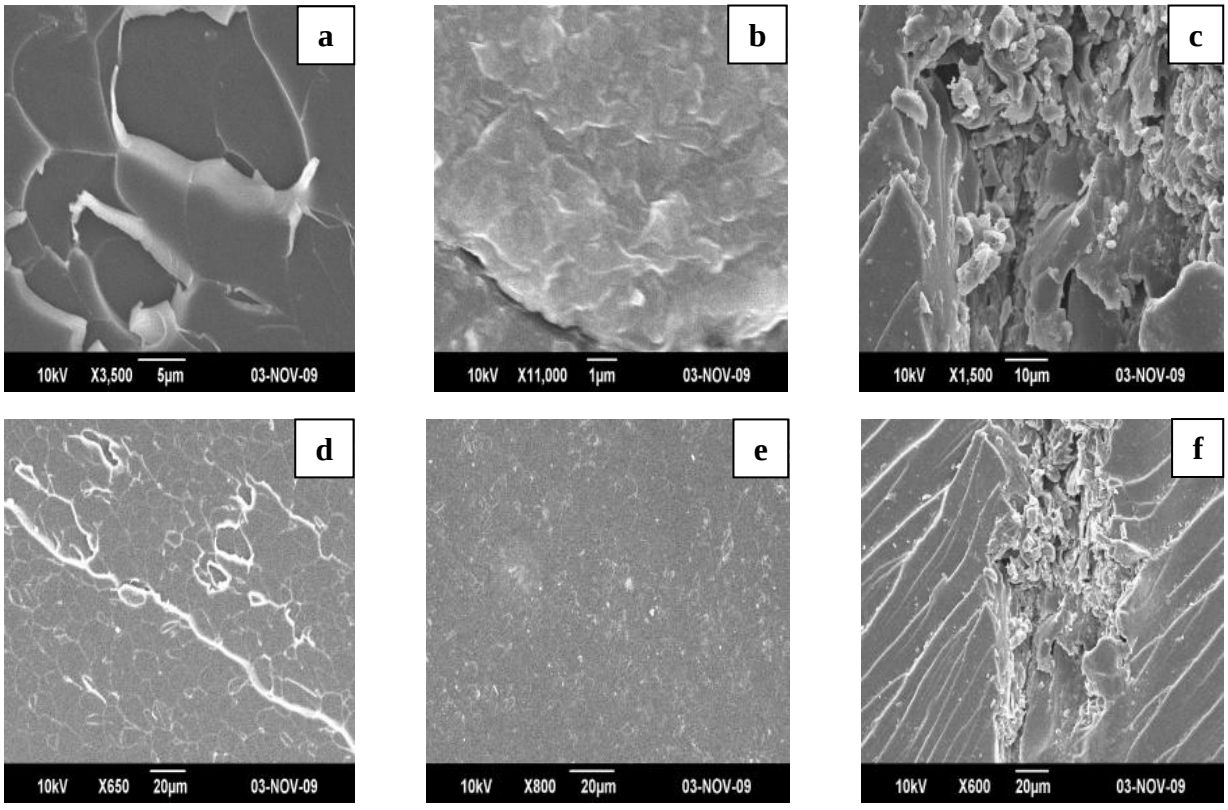

Fig. 3. SEM images of fracture surface of epoxy resin/SiC nanocomposites prepared by ultrasonication method with: a) 0.1 Phr SiC; b) 0.25 Phr SiC; c) 0.5 Phr at high magnifications; d) 0.1 Phr SiC; e) 0.25 Phr SiC and f) 0.5 Phr at low magnifications

Rys. 3. Zdjęcia mikroskopowe SEM przełomu nanokompozytów żywica epoksydowa/SiC otrzymywanych metodą mieszania ultradźwiękowego przy zawartości: a) 0,1 Phr SiC; b) 0,25 Phr SiC; c) 0,5 Phr przy wysokim powiększeniu; d) 0,1 Phr SiC; e) 0,25 Phr SiC; f) 0,5 Phr przy niskim powiększeniu

TABLE 1. **Comparison of flexural modulus, flexural strength and ultimate elongation of epoxy resin/SiC nanocomposite by two different methods**

TABELA 1. **Porównanie modułu giętkości, wytrzymałości na zginanie oraz wydłużenia przy zerwaniu nanokompozytów żywica epoksydowa/SiC otrzymywanych dwoma metodami dyspergowania**

Sample	Flexural modulus GPa	Flexural strength MPa	Ultimate elongation %
Neat epoxy resin	3.18±0.02	86.2±5.07	2.96±0.15
Epoxy resin/0.25 SiC - high shear mixing	3.25±0.04	102.4±3.54	4.42±0.39
Epoxy resin/0.25 SiC - ultrasonication	3.29±0.02	111.4±3.03	4.93±1.08

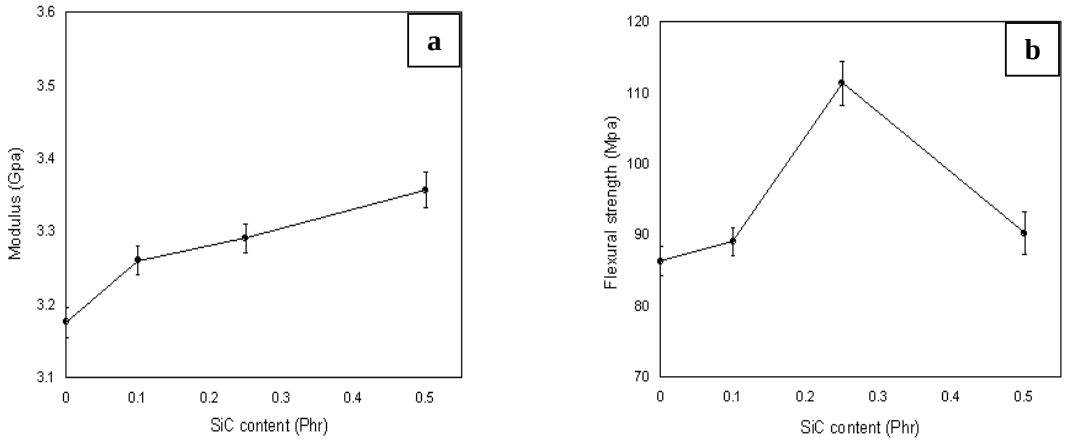

Fig. 4. Variation of flexural modulus (a) (GPa) and flexural strength (b) (MPa) with SiC content in epoxy resin/SiC nanocomposite prepared by ultrasonication procedure

Rys. 4. Zależność modułu giętkości (GPa) i wytrzymałości na zginanie (b) (MPa) kompozytów żywica epoksydowa/SiC, otrzymywanych metodą dyspergowania ultradźwiękowego w zależności od stosunku nanodrutów SiC do żywicy epoksydowej

strength is due to the poor dispersion, which has been confirmed by SEM images.

CONCLUSIONS

The SiC nanofibers were efficiently synthesized via self-propagating high-temperature synthesis. The purified SiC nanofibers have been infused in epoxy resin by different methods to produce nanocomposites. Based on morphological and mechanical results, the following conclusions were reached:

1. Ultrasonication was more effective for the dispersion of SiC nanofibers in epoxy resin than high shear mixing method.
2. Better dispersion and improved flexural strength were found in nanocomposite containing 0.25 Phr nanofibers.
3. The decrease in flexural strength in epoxy resin with 0.5 Phr SiC nanofiber content was attributed to poor dispersions of nanofibers in the composite.

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