



Preparation and characterization of foamed polyurethane/silicone rubber/graphite nanocomposite as radio frequency wave absorbing material: The role of interfacial compatibilization

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ABSTRACT

A novel method for the preparation of radio frequency (RF) wave absorber polyurethane foam (PU) has been developed by impregnation of PU foam in n-hexane solution of room temperature vulcanizing (RTV) silicone rubber (SR) hybridized with graphite nanosheets (GNs) called doping solution. Extent of the GNs dispersion was optimized by the incorporation of a specific type of bifunctional compatibilizer. Insulator to conductive transition threshold as well as electromagnetic wave absorption characteristics of the fabricated nanocomposites was shown to be dependent upon the compatibilizer functionality. All PU/SR/GN nanocomposites generated from bifunctional compatibilizer exhibited higher electrical conductivity with enhanced permittivity implying enhanced formation of conductive networks by GN platelets. Permittivity of the PU/SR/GN nanocomposite based on bifunctional compatibilizer showed to be higher than uncompatibilized counterpart. Electromagnetic reflection loss behavior of the PU/SR/GN nanocomposites exhibited a non-linear correlation with the electrical conductivity. Although all PU/SR/GN prepared nanocomposites exhibited electromagnetic wave reflection loss behavior, but this revealed to be affected by the GN level as well as the size and dispersion state of the graphite nanosheets.

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1. Introduction

Microwave absorbing materials have received increasing attention and interest in electronic and communication industries. The importance of these materials relates to the high demand for the reliability of electronic devices, and also rapid growth of radio frequency (RF) sources and interference of various electronics by radio frequency radiation [1,2]. Microwave absorbing materials are usually supported on a metal surface to prevent the reflection of incident wave. These materials are used as internal lining of the walls of anechoic rooms to prevent the interference of the wave reflected from the room walls with that emitted from the device under test. Moreover, needs for lightweight broad band microwave absorbers which can be used outdoors or in numerous applications where the absorber comes in contact with water or should withstand severe vibrations and/or mechanical impacts arise in airborne, automotive and marine applications [3]. Examples include radar antenna nacelles, antenna or target test mounts in radar

ranges, reducing reflections from buildings on antenna ranges, and producing desired modifications in antenna patterns such as the reduction of side-lobes and back-lobes.

Microwave absorbers have electromagnetic impedance in between the emission environment (usually air) and the incidence surface that they are put over and hence they prevent reflection that is caused by sudden impedance change. As the wave impedance in absorbing lining is a function of the lining thickness and permittivity, the material permittivity and hence, wave impedance in a defined frequency range can be adjusted so that minimum reflection occurs [4]. Materials with foam structure are conventionally used in microwave absorbers because of lightweight, and multi internal reflections due to the presence of large surface area in the material for interaction of electromagnetic field that causes more energy dissipation. However, incorporating of electrically conductive nanofillers has been shown to be effective in both energy dissipation and reflection loss behavior [5,6]. In another words, polymer materials with electrical conductivity are expected to have higher potential to be used for EMI applications.

Electrically conductive polymer nanocomposites have attracted great interest due to the high demand for lightweight absorbing

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materials with acceptable mechanical properties [5–7]. To reduce the weight of the absorbing material, a low insulator to conductive threshold is desired for the incorporated filler. Therefore, both distribution and dispersion state of the nanofiller particles play crucial role in the formation of conductive paths at low filler content [8].

Graphite nanosheets (GNs) have gained great interest because of their high flexibility, large surface area, and lower cost compared with other carbon nanofillers such as nanotubes and nanowires [9,10]. When natural graphite is intercalated by small molecules such as sulfuric acid the spacing between graphite nanolayers is increased and the resulting product is called graphite intercalated compound (GIC). Subjecting the GIC to a sudden thermal shock at a very high temperature (>900 °C), leads to high expansion of the graphite layers in *c*-axis direction, and the product is named expanded graphite (EG). When EG is subjected to ultrasonic powdering in a media with surface tension near to that of graphite, graphite nanosheets (GNs) separate from each other [11,12].

The main challenge for the application of graphite nanosheets is their uniform dispersion in insulating media as matrixes. The strong intrinsic van der Waals attraction between sheets (over 2 eV/nm²) in combination with their high surface area and aspect ratio make the GNs difficult to be stably dispersed in either organic or aqueous solvents. Recently, attempts have been made to prepare stable dispersions of GNs in organic solvents by ultrasonication of EG particles with assistance of a dispersant (BYK-163) and a high molecular weight resin (vinyl chloride-co-vinyl acetate) [13].

The main objectives of the present work are to prepare electrically conductive nanocomposites based on a flexible foamed polyurethane (PU) matrix and conductive graphite nanosheets with emphasis on improved electromagnetic wave (0.5–2.5 GHz) reflection loss characteristic of the nanocomposites, and also sufficient adhesion between GN particles and PU foam structure when it is subjected to any kind of deformation.

2. Materials and methods

2.1. Materials

The graphite intercalated compound, GIC, was purchased from Jiangchem corporation (China) with the trade name of EX095200, and expanded graphite, EG, with a worm-like structure having the sheet edge thickness between 100 and 400 nm was prepared by rapid heating of GIC at 900 °C for 20 s in a furnace. The used room temperature vulcanizing silicone rubber (SR) was commercially available in the form of a viscose liquid with specific gravity of 1.08 g/cm³ and was supplied by Shenzhen Hong Ye Jie Technology Co., China. Normal hexane, water and ethanol were distilled prior to be used.

A bifunctional alkyl ammonium salt with the commercial name of BYK-9076 supplied by BYK chemie (Germany) in the form of a low viscosity liquid and monofunctional cetyltrimethyl ammonium chloride (CTAC) supplied by Tianjin Hero-land S&T corporation (China) were employed as compatibilizer for the preparation of SR/GN doping solutions. The used flexible polyurethane foam was based on diisocyanate and a polyol which was produced by Jalafoam manufacturing company (Tehran, Iran) and had the density of 20 ± 2 kg/m³ and 10 ± 3 pores per inch. The scanning electron micrograph of the used PU foam is presented in Fig. 1.

2.2. Sample preparation methods

In order to evaluate the effect of extent of delamination of graphite layers, two methods were employed to prepare GNs. In the first method, 2.5 g of EG was dispersed in a solvent composed

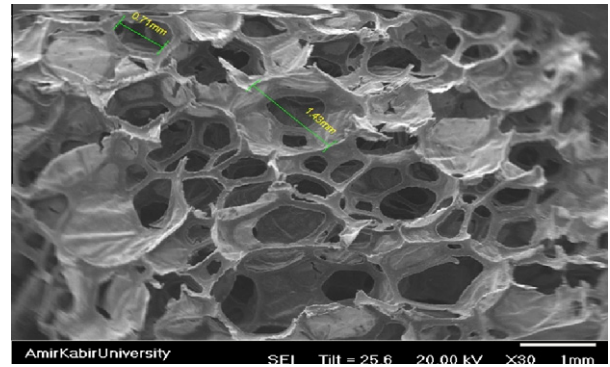


Fig. 1. SEM micrograph of the PU foam which was used in the production of RF absorber nanocomposites.

of 135 ml of ethanol and 45 ml of water and then subjected to powdering process using an ultrasonic sonicator (Hielschar UP400, Germany) for 3 h. After evaporation of the solvent, dried GN was denoted as GNM. In the second method, the prepared EG was first ground in a household-mill for 10 s, and a 10 g sample of the powdered EG was put in a mixture of 135 ml of ethanol and 35 ml of distilled water. This solution was then sonicated for 60 min and then dried via evaporation process. The GN obtained by latter method was denoted as GNS.

Doping solution was prepared by incorporating a calculated amount of dried GN into *n*-hexane and then the solution was subjected to sonication process for 1 min. Surfactant and RTV silicone rubber were then added to the GN solution and the mixture was mechanically stirred for 5 min. Moreover, doping solution contains curing agent in order to crosslink the silicon rubber. The level of curing agent in the composition of all doping solution samples was kept at 20 wt.% of silicone rubber.

The compatibilizer to GN ratio was kept at 0.3 for all prepared doping solution samples as this ratio had been found in our previous work [14] to be optimum for good dispersion of graphite nanoplatelets in RTV silicone rubber. In order to evaluate the electrical conductivity of the synthesized doping solution, after addition of appropriate curing agent into the prepared SR/GN doping solution, it left to be cured at room temperature in the form of sheets with the thickness of 2 mm. To manufacture the PU/SR/GN nanocomposites, samples of PU foam were dipped into the doping solution for a period of time so that the doping solution could uniformly diffuse into the PU foam. The impregnated foams were left at room temperature until the optimized cure of the soaked RTV silicone rubber. All prepared PU/SR/GN nanocomposite samples were coded as presented in Table 1. In this table, B and C indicate BYK and CTAC compatibilizer respectively, and the numbers show the level of GN, where M and S denote the type of the prepared graphite nanosheets. Fig. 2 shows SEM micrograph of typical prepared PU/SR/GN nanocomposite foam.

Table 1

Coding of the PU/SR/GN nanocomposite samples prepared by impregnation of the PU foam (350 cm³) by various SR/GN doping solutions (50 ml *n*-hexane, 10 g SR, and 2.0 g curing agent).

Code	GN type	GN weight (g)	Surfactant type	Surfactant weight (g)
PUB2M	GNM	2.0	BYK-9076	0.60
PUB2S	GNS	2.0	BYK-9076	0.60
PUB1.2M	GNM	1.2	BYK-9076	0.36
PU02S	GNS	2.0	–	–
PUC2.5S	GNS	2.5	CTAC	0.75

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