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Electrical Properties of Polycarbonate/Nickel Composites Prepared by a Simple Processing Technique

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Abstract

Polycarbonate-Nickel (PC/Ni) composites fabricated by using a simple solution processing technique followed by hot pressing were studied for the electromagnetic interference shielding application. Optical microscope and scanning electron microscopy showed the uniform dispersion and three dimensional networks of Ni particles in the PC matrix. The dc electrical conductivity of the composites increased approximately fifteen orders of magnitude compared to pure PC. According to power law, the critical exponent and the percolation threshold value are 6.95 and 2.5 vol.%, respectively with regression factor, $R^2 = 0.988$.

Introduction

Due to the fast growth of electronic devices, there is a risk of electrostatic discharge (ESD), electromagnetic interference (EMI) and radio frequency interference (RFI) to the efficiency of electronic devices. The EMI is the electronic disturbance which interrupts, degrade or limit the effective performance of electronic devices. Therefore, protection of devices and circuits against EMI with shielded materials has become an essential issue. For better ESD/EMI/RFI shielding, the shielded materials must possess good electrical conductivity which can be achieved by adding appropriate volume fraction of silver (Ag), copper (Cu), aluminum (Al), nickel (Ni), iron (Fe) etc. to the polymer matrix [1-3]. In conductive filler-polymer composites, the minimum volume fraction of the conductive filler at which drastic increase in electrical conductivity occurs is called percolation threshold which depends upon size, shape and aspect ratio of the fillers, processing method, adhesion between the fillers and the matrix. For metal-polymer composites, it varies between 2 vol% and 57 vol% [4-5]. In addition, the percolation threshold depends upon the packing factor of the filler and polymer crystallinity [6-8]. It is also reported that filler particles are dispersed preferably in the amorphous region of the semi-crystalline polymer.For example, the percolation threshold for the Fe filled high density polyethylene (HDPE) and low density polyethylene (LDPE) composites is 14-15 vol% and 12-20 vol%, respectively [8]. The maximum conductivity reported for poly(vinylidene fluoride)/Ni composites was about 3×10^{-4} S/cm [9]. Nevertheless, the percolation threshold should be as low as possible otherwise mechanical properties of the composites deteriorate [10].

Polymers are widely used for various applications due to their easy processing, low cost, flexibility and chemical resistant property. One of them, i.e., polycarbonate (PC) is an amorphous polymer which has good chemical resistance, good thermal stability and excellent toughness. Moreover, owing to high absorption coefficient and low reflection coefficient, Ni has been widely used as filler for the EMI shielded materials. The Ni has an electrical conductivity of 1.3×10^5 S/cm and relative magnetic permeability of 100 [11-12]. To the best of our knowledge, despite good properties, polycarbonate (PC) based composites containing nickel (Ni) was not studied for the EMI shielding application, yet. In view of this, the objective of this work is to study electrical properties of the PC/Ni composites prepared by a simple solution processing technique followed by hot pressing.

Experimental

Materials

Commercial polycarbonate (PC) was obtained from a local supplier, Pune, India. Tetrahydrofluren (SQ grade) was used as a solvent. The density of PC was 1.2 g/cc. The micro sized Ni powder (Sigma Aldrich Co., USA) was used as filler. According to supplier, the particle size of Ni powder was 5 µm. Laser particle size analyser indicated its particle size between 0.6 µm and 1.2 µm with mean particle size of 0.9 µm. The Ni particles are in aggregated form having dendrites like shape. The size of each dendrite varies from few to several tens of microns [13]. The conductive silver paste was purchased from Electron Microscopy Sciences, USA.

Preparation of PC/Ni Composites

Commercially available PC and Ni powders were pre-dried under a vacuum oven for 3 h at 120 °C and 500 mm Hg pressure vacuum. The appropriate weight of Ni and PC powders were mixed in a THF solvent for 1 h in a beaker using magnetic stirrer and heated the solution to get the viscous solution of PC/Ni/THF. Film was casted on a cleaned glass plate, dried and degassed in a vacuum oven for overnight at 100 °C and 500 mm Hg pressure. Then, dried film pieces were filled in a die and die assembly was placed on a hydraulic press. The sample was heated at a heating rate of 10 °C/min under 45 MPa pressure to a temperature of 260 °C for 25 min. The sample was water cooled to a temperature below 50 °C and ejected. The samples were polished on both flat sides with an abrasive paper to remove excess polymer prior to further

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characterization. PC/Ni composites containing 10 to 70 wt.% Ni particles were fabricated using the above procedure. For comparison, pure PC pellet (as reference) was also made using above mentioned procedure. For a given weight fraction (W_f) of Ni, its volume fraction (V_f) was determined using equation (1) and shown in Table 1.

Table 1: Compositions of the PC/Ni composites

Sample Code	% Ni in PC matrix	
	By wt.	By vol.
C-0	0	0
C-15	15	2.3
C-30	30	5.5
C-45	45	9.9
C-60	60	16.8

Where, C-X; where X indicates wt.% of Ni in the PC matrix.

$$V_f = \frac{W_f}{[W_f + (1-W_f) * {}^{\rho_f}/_{\rho_m}]} \qquad (1)$$

where, ρ_m *and* ρ_f are the densities of PC matrix and Ni powder, respectively.

Characterization

Density

Theoretical density of the composites was calculated by the rule of mixture using the density of 1.2 g/cc for PC and 8.9 g/cc for Ni. The experimental density of the composites was determined by Archimedes's Principle using ethanol medium (specific gravity 0.79 g/cc).

Scanning Electron Microscopy (SEM)

The distribution and morphology of Ni particles in the PC matrix were observed using SEM (JEOL JSM 6360 A). For this, cross section of the samples were polished and lapped. Polished surfaces were coated with a thin layer of platinum using sputter coater (JEOL JEC60) and mounted on metal stub which was grounded with silver paste to minimize charging effects.

Electrical Conductivity

The volume resistivity of compression molded composites was tested using Electrometer (Keithley 6517-B, U.K). Before measurement, samples were coated with silver paste to eliminate contact resistance. This instrument measures volume resistivity of high resistance samples upto 10^{17} Ω. The conductive sample (resistance < 10^6 Ω) was tested with a 7½ digit digital multimeter (Keithley 2001, U.K). The resistivity (ρ) of the nanocomposites was calculated by using equation (2).

$$\rho = (R \times A)/t \quad (\Omega\text{-cm}) \qquad (2)$$

where, R is the resistance (Ω), t the thickness (cm) and A the surface area of the sample (cm^2). The electrical conductivity (σ) (S/cm) of the samples was determined by the reciprocal of the resistivity.

Results and Discussion

Density

Figure 1 shows the theoretical and experimental density of PC/Ni composites. The experimental density of the composites increased with increasing Ni content in the PC matrix. It is due to higher density of Ni (8.9 g/cc) than that of PC. Experimental density of composites is very close to theoretical density indicating porosity free samples.

Optical Microscopy/Scanning Electron Microscopy

Figures 2(a) and (b) show the optical micrographs of polished top surface of 45 wt.% (C-45) and 60 wt.% (C-60) composites. The Ni particles are well dispersed into the PC matrix and form a 3-dimensional network as clearly seen in both composites. Optical micrograph of 60 wt.% composite shows denser Ni content than that of 45 wt.% composites due to decreased interparticle distances. Figures 2(c) and (3d) show the optical micrographs of polished cross section of 45 wt.% and 60 wt.% composites, respectively.

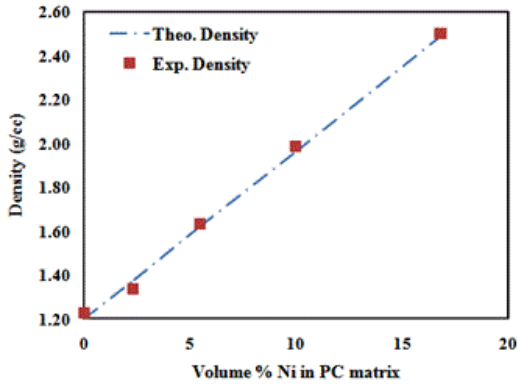

Figure 1: Theoretical and experimental density of the PC/Ni composites.

From all these micrographs, it is clear that Ni particles are uniformly distributed throughout the PC matrix. Figures 3(a-d) show the SEM micrographs of polished composites at 500X and 2000X. SEM images confirm the uniform dispersion of Ni particles in the PC matrix at higher magnifications, which is not clearly seen in the optical images. It also confirms that at higher magnification of 2000X, both cross section show good dispersion of Ni in the PC for 45 wt.% Ni. At higher content (C-60), some aggregates of Ni particles were also observed in both top surface and cross section. This may be due to decreased inter particle distances at higher particle loading as shown in SEM micrograph (Figures 3d and 3f).

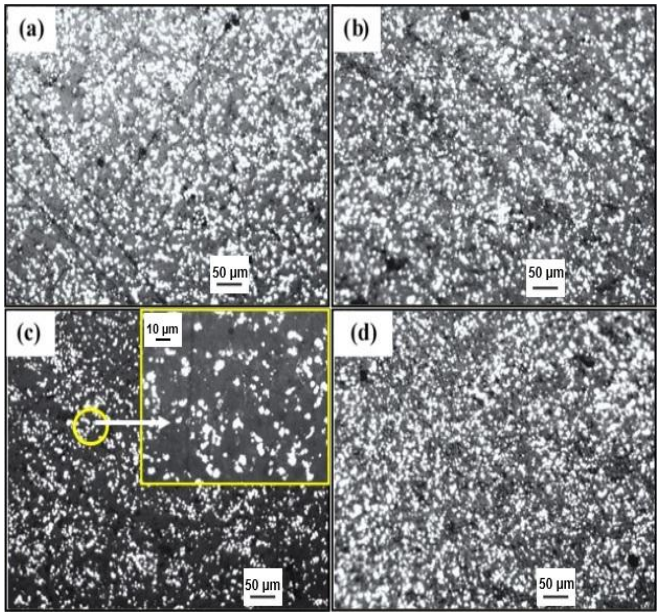

Figure 2: Optical micrographs of polished top (a and b) surface of composites with (a) 45 wt.% Ni and (b) 60 wt.% Ni, and polished cross section (c and d) of composites with (c) 45 wt.% Ni and (d) 60 wt.% Ni at 100 X.

DC Electrical Conductivity

Figure 4 shows the volume electrical conductivity (dc) of PC/Ni composites as a function of vol.% of Ni. The electrical conductivity obtained for the pure PC is 4×10^{-16} S/cm. It increased with increasing Ni content in the PC matrix. There is a sharp increase in the electrical conductivity of the composite between 2.32 vol.% and 5.46 vol.% Ni. The electrical conductivity of 5.46 vol.% composite is 1.62×10^{-6} S/cm. This increased in conductivity is about ten orders of magnitude higher than that of pure PC. The highest electrical conductivity achieved is 0.17 S/cm and 5.7 S/cm for 16.82 vol.% Ni (60 wt%) and 23.93 vol.% (70 wt%) Ni composites, respectively.

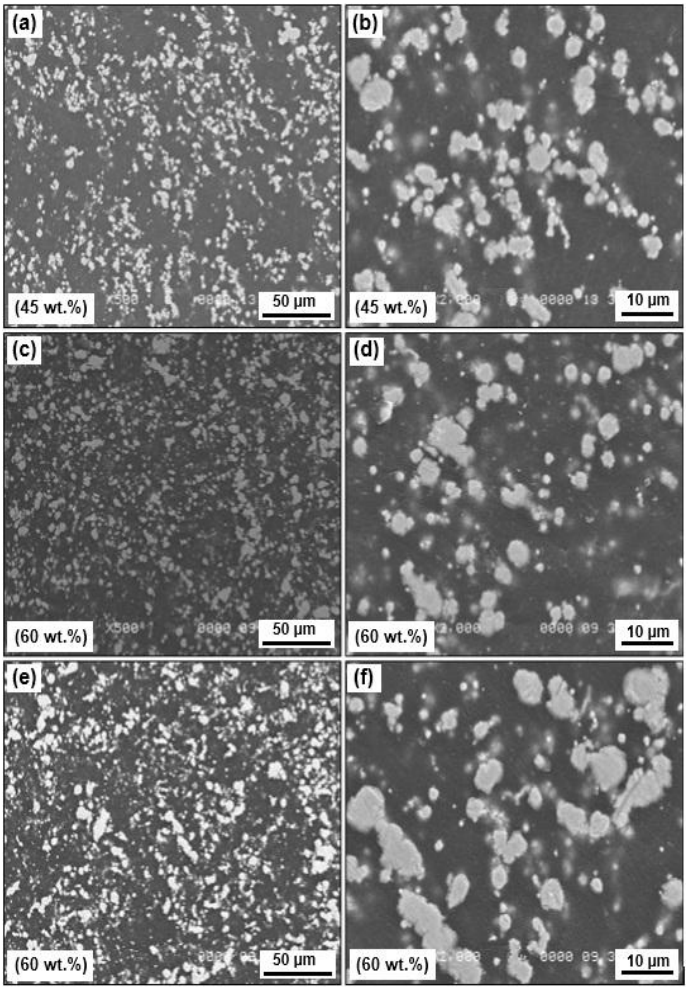

Figure 3: SEM micrographs for polished cross section of composites (a-b) 45 wt.%, (b-c) 60 wt.%, and (e-f) polished top surface of 60 wt.%.

According to classical percolation theory, the conductivity near the percolation range can be estimated by a power law of percolation theory shown in Equation (3).

$$\sigma \propto (\emptyset - \emptyset_c)^t \qquad (3)$$

where, σ is the conductivity of the composites, $\emptyset$ the volume fraction of Ni, $\emptyset_c$ the critical volume fraction of Ni and t is the critical exponent constant, which is related to the system dimensions. The log (σ) verses log ($\emptyset - \emptyset_c$) plot is shown in the inset of Figure 4. To get an estimate for $\emptyset_c$ and the critical exponent t, we fitted the conductivity for $\emptyset > \emptyset_c$. This is done by varying $\emptyset_c$ in the step of 0.01. Critical exponent has been determined from the slop of linear relation between σ and ($\emptyset - \emptyset_c$) on a log-log scale. The best fit line of the conductivity data in the log-log plot for PC/Ni composites give t = 6.954 when $\emptyset_c$ = 2.50 vol.%, and the root mean square error or regression factor is R^2 = 0.988. Mamunya et al. [14] reported a higher critical exponent value (i.e., 8) for blend PE/POM-Fe composite than that of their individual composites (PE/Fe) (t=2.9) and POM/Fe (t=3)). This was attributed because of good dispersion of Fe powder in the PE/POM blend which leads lower percolation threshold, than that of individual matrix.

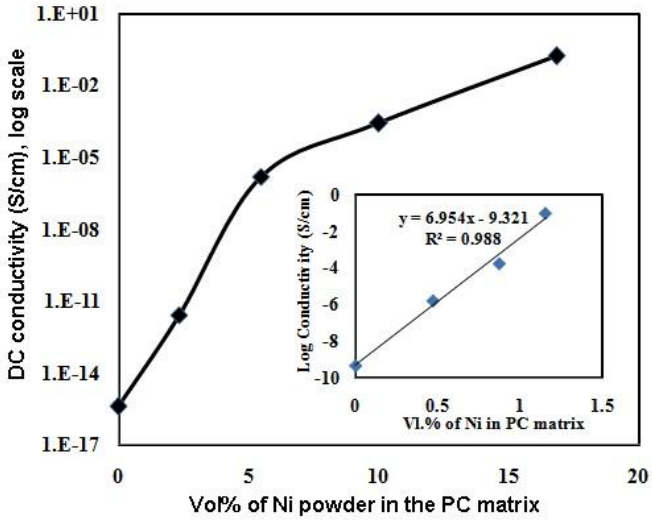

Figure 4: DC electrical conductivity versus vol.% of Ni in the PC matrix. The inset shows the values of conductivity above the percolation threshold (V_f) versus (V_f-V_c).

Conclusions

PC/Ni composites were successfully fabricated using a simple solution processing technique followed by hot pressing. The experimental and theoretical densities of the composites were very close to each other. Optical microscope and SEM showed a uniform dispersion of the Ni particles in the PC matrix. The dc electrical conductivity of the composite with 60 wt.% (16.8 vol.%) and 70 wt% (23.93 vol%) Ni increased to 0.17 S/cm and 5.7 S/cm, respectively. The higher value (i.e., 6.95) of the critical exponent constant was attributed to better dispersion of Ni particles in the matrix.

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