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New Antistatic Charge and Electromagnetic Shielding Effectiveness from Conductive Epoxy Resin/Plasticized Carbon Black Composites

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Recently, there is an increasing interest in electromagnetic interference shielding due to the rapid increase in electromagnetic pollution and the wide use of commercial and military products. Conducting polymer composites were prepared in the presence of epoxy resin and plasticized carbon black (CB). The structural characteristics of the composites were examined by means of scanning electron microscopy, cross linking density, and interparticle distance among conductive particles. The mechanical properties such as Young's modulus, elongation at break, and hardness of the composites were investigated as a function of CB content. The results indicated that CB could improve the composite microstructure. The higher mechanical behavior than green epoxy can be attributed to the interaction between CB particles and epoxy resin. The conductivity, mobility carriers, and number of charges of the composites at room temperature were found to be dependent on CB content. The applicability of the composites to antistatic charge dissipation was examined. Dependence of the microwave properties of the epoxy/ CB composites on the volume fraction of CB particles and frequency were studied. Moreover the permittivity as a function of frequency of the composites was studied. The electromagnetic wave shielding of epoxy/CB composites is dominant by both reflection and absorption. Composites can find applications in antistatic charge dissipation and in suppression of electromagnetic interference and stealth technology. POLYM. COM-POS., 28:000-000, 2006. © 2006 Society of Plastics Engineers

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INTRODUCTION

Conducting polymer composites (CPCs) are ubiquitous in technological applications and constitute an ongoing topic of tremendous commercial interest [1-3]. Electrically conductive polymer composite is obtained when conductive particles such as metal powder, carbon black (CB), and carbon fiber etc. are implanted to an insulating polymer matrix [4–8]. The level of electrical conductivity in these heterogeneous materials depends primarily on the concentration and geometry of the filler [9-11]. The CB produces large cohesive strength (between the individual fine particles), which leads to aggregates of particles, having diameters of not less than several microns [12]. In the field of conducting polymeric composites, there is one main objective: minimizing the filler concentration, due to the fact that high concentration of the conductive filler could lead to deterioration of the mechanical properties of the composites [13], an increase of the melt viscosity [14], a decrease of the impact resistance [15], a contamination in clean room environment, and an increase of the final product cost [16]. Electromagnetic interference (EMI) shielding problems are increasing in electronic and military communication owing to sensitive electronic devices and densely packed systems. Hence the research on the EMI shielding has received more and more attention in recent years [17-21]. In fact, the stealth technique can be categorized into two methods. The first method is dealing with the shape optimization of the body so that the incident electromagnetic wave can be scattered, yielding a minimum reflection loss. The second method considers the use of electromagnetic wave absorption or structures [22-24]. For EMI shielding materials, typical metals such as copper or aluminum, which have high conductivity and dielectric constant, have been used. While



typical metals have good mechanical and shielding properties, they have disadvantages such as heavy weight, easy corrosion, and poor processibility for shielding material [15, 25–27]. CPCs have inspired much interest because of their light weight, hard corrosion, good processibility, and easy control of the conductivity [28–31]. With the aforementioned consecration in mind, in this study, the plasticized carbon black was implanted into an epoxy resin matrix, aiming to obtain conducting composites with high electrical conductivities and with low filler content. The relationships between the microstructure mechanical and electrical properties of the composites were studied. The permittivity of the composites as a function of frequency was examined. The applicability of the composites for static charge dissipation and EMI shielding was tested.

EXPERIMENTAL

Materials

The matrix used in the present study is epoxy resin polymer based on an Epikot resin 828 and an aromatic hardener 128 made by Kayo, Tokyo, Japan. The standard curing mixture contains 75 wt% epoxy resin and 25 wt% hardener according to the manufacturing data sheets; both components are delivered in the liquid state. The conductive filler is a high abrasion furnace black (CB) with a primary particle diameter of about 3 μ m. A glycerol is used as a plasticizer and is mixed with the epoxy resin in 8% weight quantity to reduce the viscosity of the resin and facilitate easier mixing of high volume fraction of particles in the resin.

Composite Preparation

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Samples containing 5, 10, 15, 20, 25, and 30 parts of CB per hundred parts of resin were prepared according to the recipe shown in Table 1, and composites are donated as CB5, CB10, CB15, CB20, CB25, and CB30, respectively. The epoxy resin, hardener, glycerol, and CB were centrifuge-mixed for 1 min at 8,000 rpm and then further sonication for an additional 30 min at room temperature. The composite particles were transferred to a preheated compression Teflon mold and then hot-pressed at 130° C under 200 kN/m² for 1 h. The final product was obtained in the form of sheets with a dimension of $2 \times 1 \times 0.3 \text{ mm}^3$.

TABLE 1. Ingredients of th	e investigated	epoxy composites.
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Ingredients (wt%)	CB5	CB10	CB15	CB20	CB25	CB30
Epoxy resin	75	75	75	75	75	75
Hardener	25	25	25	25	25	25
Plasticized	8	8	8	8	8	8
CB	5	10	15	20	25	30

Characterization

Scanning electron microscope (SEM) analysis of the samples was carried out using a A JEOL-JSM 5800 at an accelerating voltage of 5 kV. The surfaces of the specimens were coated with silver to obtain a conductive layer. The specific electrical conductivity for the composites was performed by measuring the current through the sheet sample under a steady constant voltage using a digital Keithley 642 electrometer. The two sides of the samples were bended with Cu rod during curing process to reduce the contact resistance. The real and imaginary permittivities were measured using RLC Bridge (3535 Z-Hitester, Hioki, Japan). Stress-strain data were determined on a Shimatzo universal testing machine, using C-type dumbbell specimens. The test was done at 20°C and at a crosshead speed of 300 mm min⁻¹. The hardness of the specimen was also measured, according to ASTM D 2240-81 using Shore A type durometer. The cross linking density (n) was calculated according to the theory of rubber elasticity by using the following equation [2, 26].

$$E_{\rm s} = nRT \left(E_{\rm r} - \frac{1}{E_{\rm r}^2} \right) \tag{1}$$

where E_s is the tensile stress, R is the universal gas constant, T is the temperature in Kelvin, and E_r is the extension ratio.

The distance among conductive filler particles (g) is given by [3]:

$$g = D\left[\left(\frac{p}{V_{\rm CB}}\right)^{\frac{1}{3}} - 1\right] \tag{2}$$

where D is the diameter of filler particles, P is the packing density coefficient of fillers and is about 0.64 for statistically packed monodispersed spherical particles of any size [10].

The static energy (SE) of epoxy as a function of CB content was measured using static charge meter (mode AX-221-Japan) and is given by [27]:

$$SE = \frac{1}{2}CV_i \tag{3}$$

where C is the capacitance of the base plate and V_i the voltage indicated on the static charge meter.

For measuring the discharge properties the tested samples were charged at room temperature for 15 s in a 10 kV electric filed (corona discharge). The decrease of the electrical surface potential of the specimens over time was displayed and recorded. Electromagnetic wave shield-ing properties were determined by the Hewlett–Packard waveguide line containing spectroanalyzer, power meter, coefficient of reflection meter, and coefficient of attenuation meter. The measurements were carried out in the frequency ranges 1–10 GHz and the thickness of the sample is about 1.0 mm.



(a)



FIG. 1. Scanning electron micrographs of two epoxy/CB composites: (a) epoxy containing 5 wt% CB. (b) epoxy containing 30 wt% CB.

RESULTS AND DISCUSSION

Network Structure and Mechanical Properties of Composites

The dispersion state of the CB in composites was investigated by scanning electron microscopy (SEM). Scanning electron micrographs of two epoxy/CB composites containing 5 and 30 wt% CB are shown in Fig. 1a

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and 1b, respectively. A comparison of the surface morphology of two samples shows a marked change in the surface properties and texture of the microstructure of the composites. Figure 1a shows a random distribution of CB particles in the epoxy matrix and CB particles are separated to each other, indicating weak epoxy/CB interfacial bonding. In Fig. 1b the sample CB30 exhibits a spherical morphology, and the particles are held together by short range surface forces. The forces may be electrostatic and van der Waal's attraction between particles or liquid capillary forces due to the presence of the plasticizer within the granules [1, 2]. Furthermore, the CB particles are segregated in the interfacial zones of epoxy particles, and they formed conductive pathways throughout the composites. Hence, small amounts of CB particles were enough to establish the conductive networks within resin matrix, resulting in lower percolation threshold value as confirmed latter by conductivity measurements (see Fig. 4).

For further understanding of the effect of CB particles on the network structure, we examined in more detail the crosslinking density (n) and the interparticle distance among conductive particles (g). The dependencies of nand g of epoxy/CB composites versus volume portion of the CB are plotted in Fig. 2. The n was found to increase while g decreases with increase in content of CB in the epoxy composites. This is associated with the increase of the intermolecular forces among epoxy chains and CB particles. Furthermore, because of the lower viscosity of the resin during processing, the degree of intermixing between CB and resin particles was strong, resulting in increase of crosslinking density [11, 32].

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Young's modulus (Y_m) , hardness (H_A) , and elongation at break (EB) of epoxy composites versus volume fraction of CB are shown in Fig. 3. It is clear that Young's modu-



FIG. 2. The dependencies of n and g of epoxy/CB composites versus volume portion of the CB.

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FIG. 3. Experimental and theoretical values of Young's modulus and elongation at break of epoxy composites versus CB content. Hardness of the epoxy composites versus CB content.

lus increases with increase in CB content in the composites. This fact is associated with the higher interfacial adhesion and crosslinking density among filler and matrix with increase in CB loadings. Similarly, Shore A hardness of epoxy composites (see Fig. 3) increases with increase of the CB content, due to the fact that the CB included into the epoxy matrix behaves like physical crosslinking points and hence restrict the movement of epoxy chains [16]. The improvement in the Y_m and H_A may be caused by the strong interactions between epoxy matrix and CB, which leads to a good dispersion of CB in the composites. These well dispersed CB may have an effect of physical crosslinking points, thus increase both Y_m and H_A . The Guth–Smallwood equation is widely used to predict the increase of Y_m in polymer-filler composites [19, 21]:

$$Y_{\rm m} = Y_{\rm me} \left(1 + 2.5 V_{\rm CB} + 14.1 V_{\rm CB}^2 \right) \tag{4}$$

where $Y_{\rm me}$ is the modulus of an epoxy matrix.

The calculated values of $Y_{\rm m}$ for epoxy/CB composites were compared with experimental values as shown in Fig. 3. The experimental values of $Y_{\rm m}$ are much higher than the theoretical ones. This means that strong surface interactions are developed between CB particles and epoxy matrix in composites as confirmed before. The EB values of the samples are shown in Fig. 3. When the CB content was increased, the EB values decreased, as expected. There are two possible reasons evoked on decrease in EB with increase in CB content. First, the inclusion of CB in the epoxy matrix acts as a reinforcing agent, leading to the formation of sites of stress concentration into composites. The other possible reason is due to the crosslinking networks (chemical bonding) between flexible epoxy matrix and stiff CB or the epoxy inter-molecular chain. The experimental data of EB are compared with Nielson model given by the following equation [23, 24]:

$$\frac{\text{EB}_{\text{m}}}{\text{EB}_{\text{e}}} = 1 - V_{\text{CB}}^{\frac{1}{3}}$$
(5)

where EB_e is the elongation at break of a green epoxy matrix.

Good agreement between the model and experimental data is observed in Fig. 3. This fact can be caused by lower agglomeration of the CB as well as by sufficient wetting of particles due to the presence of plasticizer [1]. Finally, it should be emphasized that the plasticized CB improves the interfacial bond with epoxy matrix and the corresponding molecular structure and bulk mechanical properties.

Effect of CB on the Static Conductivity

The volume electrical conductivity of the epoxy resin/ CB composites as a function of the volume fraction of the CB at room temperature is depicted in Fig. 4. The volume conductivity increases with the increase in the concentration of CB in the composite. At low CB loadings, a large change in volume conductivity can be



FIG. 4. Dependence of room temperature conductivity μ and N with respect to CB content of epoxy/CB composites.

observed because the gap distance among CB particles are small enough. Gap distance decreased as the amount of CB was increased in composites as observed in Fig. 2. As expected, a decrease in gap distance as reflected by increase in crosslinking density is observed as the amount of CB is increased in the samples, which in turn shows an increase in the composites conductivity of the corresponding samples. Furthermore, increasing the CB content makes CB particles more crowded and linked, leading to the rapid increase of bulk conductivity. This indicates that the conducting particles come into contact with each other or closed up enough to allow the carriers mobility hop by tunneling, thus forming continuous conducting mesh [4]. A feature of interest in this figure is the critical concentration i.e. the minimum CB volume fraction that yields a measurable conductivity. When the volume fraction of CB reaches 9 wt%, the conductivity of the composite is higher than that of green epoxy resin by 8 orders of magnitude. The value of critical concentration is quite low as compared to theoretically predicted value (about 17 wt%) for the CB embedded in insulating polymer matrix [15]. The lower percolation threshold value can be attributed to higher degrees of intermixing [17]. The intermixing increases the concentration of CB in the interfacial regions or builds up the direct contact between CB particles, resulting in a lower percolation threshold. To support the aforementioned facts, the mobility carriers (μ) were calculated from the following formula [9]:

$$\mu = \left(\frac{v_0 e g^2}{KT}\right) \tag{6}$$

where v_0 is the optical phonon frequency (generally $v_0 \approx 10^{-13}$ s), *e* is the electron charge, and *K* is the Boltzmann constant.

The density of charge carriers (N) is given by [27]:

$$N = \frac{\sigma}{e\mu} \tag{7}$$

The variation of μ and N with respect to CB content of epoxy/CB composites are plotted in Fig. 4. It can be seen that both μ and N increases with increase in CB content into composites. This is attributed to a high degree of bridging among CB particles and intermixing with increasing CB loading level in composites.

Antistatic Charge

Static electricity on a surface is generated due to the build-up of charges. This could happen either by triboelectric effect-rubbing, sliding, separating of nonconductive materials, or by an electrostatic field, created when one charged body induces charge on a nearly second body [26]. Antistatic composites are widely used in media drives and in storage devices, since they need rapid dissipation of surface charges generated during their operation. The variation in SE with respect to CB content for epoxy





FIG. 5. (a) The variation in SE and decay time constant with respect to CB content for epoxy composites. (b) Decay voltage versus time of epoxy/CB composites.



FIG. 6. (a) EMI and skin depth as a function of CB content. (b) The theoretical and experimental results of EMI of epoxy/CB composites versus frequency.

the range of $1.62 \times 10^7 - 1.1 \times 10^4$ Ohm cm, and here the generated electric current flows in the composites through CB particles, thus becoming well dissipated. With higher

CB content (i.e. $CB \ge 15 \text{ wt\%}$), the CB particles come fully in contact with one another but may act rather as electric conductor, and as a result, the electric energy is not fully dissipated. For further verification of the aforementioned results, the static decay time of the epoxy/CB composites was determined by noting the time taken from the dissipation of static charge on going down from 10 kV to 0 V. The variation of the decay voltage with time for epoxy/CB composites is shown in Fig. 5b. The decay voltage–time curve can be described by the following exponential decay equation.

$$(V - V_{\rm m}) = (V_{\rm m} - V_0)(1 - \exp(-t/\tau))$$
 (8)

where $V_{\rm m}$ is the maximum voltage and τ is a decay time constant that depends on CB content and is calculated at $t = \tau$.

The calculated values of τ as a function of CB content is illustrated in Fig. 5a. It is seen that the value of τ decreases as CB content increases. It is clear that at low CB content the decay time constant is faster and less than 10 sec. With increase in CB content the decay time constant is higher than 10 sec. The results indicate that conducting epoxy/CB composites can be used effectively in antistatic protection devices at low CB loading level.

Electromagnetic Wave Shielding Effectiveness (EMI)

EMI is another property of importance in many applications [25]. Electronic devices operating normally in their intended environment, without conducting or radiating excessive amounts of electromagnetic energy, or not being susceptible to such energy from internal or external sources, are in the state of electromagnetic compatibility. EMI is radiated or conducted energy that adversely affects a circuit's performance, and thus disrupts a device's electromagnetic compatibility. Many types of electronic circuits radiate or are susceptible to EMI and must be shielded susceptible to EMI. Electromagnetic shielding means that the energy of electromagnetic radiation is attenuated by reflection or absorption of an electromagnetic shielding material. CB/epoxy composites are a kind of EMI shielding materials with good prospect of application because of its electrical conductivity and dielectric constant. The EMI is defined in terms of the ratio of the electric field of incoming and outgoing waves by the following expression [2, 18]:

$$EMI = 20 \ \log_{10}\left(\frac{E_i}{E_o}\right) \tag{9}$$

where E_i and E_o are the electric field of incoming and outgoing waves, respectively.

Reflection, transmission, absorption, and multiple reflection are observed when the plane wave is incident on shielding material. The total EMI efficiency is written as [28]:



FIG. 7. (a) The real of permittivity as a function of frequency of the epoxy/CB composites. (b) The imaginary parts of permittivity as a function of frequency of the epoxy/CB composites.

$$\mathbf{EMI} = A + R + M \tag{10}$$

where A, R, and M are the shielding efficiency due to absorption, reflection, and multiple reflection, respectively.

Absorption loss for electric field in a sample of thickness (d) is given by [30]:

$$A = 20 \, \log\left(e^{-\frac{d}{\delta}}\right) \tag{11}$$

where

$$\delta = \text{skin depth} = \sqrt{\frac{2}{w\mu\sigma}}$$
 (12)

 σ is the electrical conductivity, μ is the permeability of the shield material, and *w* is the angular frequency.

The reflection loss for a plane wave of frequency (f) is obtained as

$$R = 31.5 + 10 \, \log\left(\frac{\sigma}{f\mu}\right) \tag{13}$$

The correction term for multi-reflection within the shield depends upon the skin depth as shown in the following expression:

$$M = 20 \, \log\left(1 - e^{-\frac{d}{\delta}}\right) \tag{14}$$

The measured values of the shielding effectiveness and skin depth for the different loading level of CB in the epoxy matrix are shown in Fig. 6a. Perusal of Fig. 6a indicates that 5, 10, 15, and 20 wt% loading of CB show a shielding effectiveness of 1.5, 4.1, 9.1, and 29.5 dB, whereas a 25 wt% loading of the CB in epoxy gives a shielding effectiveness of 37.3 dB. A 30 wt% loading of the CB in host epoxy matrix gives a shielding effectiveness of 44.0 dB. The results indicate that it increases the percentage of CB in the host epoxy matrix, resulting in an increase of the shielding effectiveness value. This can explained as follows: an increase in the number of conducting paths increases the electrical conductivity by reducing its volume resistivity. The increase in the number of CB particles also allows the creation of particleparticle contacts, resulting in increase in the electromagnetic wave shielding. This means that conductive network paths are formed upon increasing the CB content in the epoxy matrix as confirmed before by conductivity results (see Fig. 4).

The theoretical and experimental data of the EMI of epoxy/CB composites versus frequency are presented in Fig. 6b. It is noted that the EMI shielding decreases with increase in frequency. This is attributed to the increase in the number of conducting paths. It is interesting to mention that rippling the CB particles increases the amount of absorption within the composite. Further, there is an increase in the amount of CB surface scattering, which increases the number of internal reflections within the composite and hence increases the energy of absorption within the composite. This supports that the proposed composites are promising candidates for stealth technology.

To verify the aforementioned results the real and imaginary permittivity of the composites as a function of frequency were studied and presented in Fig. 7a and 7b, respectively. It is clear that the real and imaginary permittivity increase with increasing CB content in composites, due to the fact that the CB particles get closer to each other, resulting in an increase of permanent electric dipoles

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within the composites [4, 15]. Also the real and imaginary permittivity decrease with increase in frequency. A low CB content reduces the contribution from orientation polarization, thus leading to the lowest values of real and imaginary permittivity in the composites. With increasing CB loading level, this constitute a heterogeneous system and interfacial polarization, which is another important polarization process and associated relaxation also will give rise to loss mechanism [1, 12]. This is due to the dipolar and interfacial polarization increase with increase in CB content in the composites. Therefore, the dipolar and interfacial polarization are the dominant polarization and the associated relaxation phenomena constitute the loss mechanism. The aforementioned results indicate that low CB loading in the epoxy composites can be used for electrostatic charge dissipation, whereas higher loading of the CB can be used for the shielding of EMI at microwave frequency.

CONCLUSIONS

The results obtained in the investigation of CB/resin composites allow drawing the following conclusions.

- 1. The morphology indicates that the composites treated with plasticized CB exhibit much better bonding between CB and resin matrix. In addition, the inclusion of plasticizer with CB in epoxy matrix improves the quality of the network structure and increases the cross linking density of the composites.
- 2. Plasticizer CB surfaces improved the mechanical properties of the composite samples because plasticizer provided better adhesion of the CB to the resin matrix, leading to efficient load transfer.
- 3. Room temperature conductivity, mobility carriers, and number of charges increases by increase in plasticized CB volume fraction in the composites.
- Interfacial adhesion among CB and resin enhances the polarization into composite and increases the permittivity properties.
- The epoxy/CB composites are certainly promising candidates as antistatic charge dissipation at low CB content and electromagnetic wave shielding with high CB loading in stealth technology and microwave frequency materials.

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