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ELECTROMAGNETIC INTERFERENCE SHIELDING EFFECTIVENESS OF Cu POWDER/CARBON BLACK/EPOXY RESIN COMPOSITE FILM

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Abstract

A novel composite of carbon black and Cu powder is mixed with epoxy resin in order to produce a composite material suitable for electromagnetic shielding applications. The microstructure of the composite material is investigated by mean of field emission scanning electron microscopy (FE-SEM) and Fourier transform infrared spectroscopy (FTIR). The electromagnetic interference shielding effectiveness (EMI-SE) of composite material is tested over frequency range of 8-12 GHz. According to the experimental results the maximum value of EMI-SE obtained at room temperature is approximately 4 dB and 7 dB for carbon black / epoxy resin sample and Cu powder / carbon black/epoxy resin sample with thinkness 500 µm, respectively. The influence of Cu powder mass and the thick film is also studied. The results indicate that the EMI-SE can reach approximately 25 dB at Cu powder 10 mass %.

Keywords. EMI-SE, carbon, epoxy, copper.

1. INTRODUCTION

Today, the electronic and electrical appliances such as computers, electronic home devices, AC motors have unremittingly developed and become a serious problem for environmental as well as humans. Because of they are capable of emitting electromagnetic waves that can cause diseases including cancer, breast and leukemia and so on. Thus, the studying of materials for blocking undesirable electromagnetic interference from electronic device and communication apparatus is necessary to decrease their impacts on the environment and humans.

Until now, many materials have been investigated for EMI shielding applications such as metals, carbon materials (carbon nanotube (CNTs), carbon fiber, carbon black (CB)) and nanocomposites. Compared to conversional metals, the carbon materials as well as its composites have many advantages for EMI shielding applications such as having light weight, low cost, and high conductivity. In 1999, Xiangcheng et al. [1] reported a carbon matrix composite with continuous carbon fibers for EMI shielding application. The matrix composite was made by forming a mixture of the polymer powder, fillers, subsequent treatment with hot pressing at 310 ^oC for 30 min. They showed that the composite materials with a carbon matrix had more shielding effectiveness than that with an epoxy matrix. The EMI-SE of 124 dB was obtained in the frequency range from 0.3 MHz to 1.5 GHz. In other study, the carbon film based EMI shielding was fabricated by screen-printing method [2]. In this work, multi walled carbon nanotubes (MWCNTs) were made with the metal catalytic decomposition of hydrocarbons process. Then, the MWCNTs were ground by ball milling with organic solvent. The MWCNTs paste was printed through a fine mesh under proper tension onto the plastic substrate and heated at 70 ^oC. The results showed that the MWCNTs film has shield effectiveness of more than 10 dB. They also showed that a thin printed 150 µm, 15 wt.% MWCNTs film exhibits similar shielding performance to a thicker 1.5 mm, 15 wt.% MWCNTs/epoxy composite. In 2010, a novel polyvinyl chloride reinforced graphite copper particles based functional Nano conducting composite was fabricated for EMI shielding application at microwave frequency [3]. The microstructure of Nano composite was examined by scanning electron microscopy (SEM) and FTIR. Hall Effect studies showed that increasing in graphite/copper (GCu) content in the composite would enhance the electrical conductivity of Nano composite film. The EMI shielding experimental results showed that the Nano composites with higher

GCu loading level have been greater EMI-SE which can reach 70 dB in the frequency range of 1-20 GHz. Ho Chang et al. [4] studied electromagnetic shielding of carbon fiber, Ni nanoparticles and MWCNTs in polyurethane. Here, conducting materials such as carbon fiber, nickel nanoparticles, and MWCNTs were added to polyurethance by a polymer blending method. The experimental results showed that the SMI-SE can obtain 28 dB when the inspection frequency of the carbon fiber/Ni nanoparticles is 1000 MHz. When the carbon fiber/MWCNTs film is 0.15 mm thickness, the EMI-SE can reach 33.7 dB. In other study, Nam et al. reported a CNTs incorporated epoxy film for EMI shielding in a frequency band from 1 to 5 GHz [5]. The composite film was fabricated by stacking the CNTs films layer by layer to improve the electric properties of the composite materials. The observed results showed that the reflection loss and absorption loss and EMI-SE of CNT films gradually increased as the thickness of the films increased in a frequency band of 1 and 5 GHz. In 2013, Mohammed H. Al-Saleh et al. [6] reported on EMI-SE of carbon based polymeric materials. In their work, the nanofillers including MWCNTs, carbon nanofibers (CNF), CB nanoparticles and acrylonitrile – butadiene – styrene were prepared by solution mixing. The EMI-shielding mechanisms, the AC electrical conductivity, and the DC electrical conductivity were studied. They found that at nanofiller loading of 5 wt.%, the EMI-SE of CNT-based Nano composite was 2 times higher than that of CNF-based Nano composite and 7 times higher than that of CB-based Nano composite. Recently, Xingmin Liu et al. [7] have studied carbon nanotube reinforced carbon fiber/pyrolytic carbon composites for EMI shielding application. The CNTs were in situ synthesized in C/C composites through catalyzing hydrocarbon gases evaporating out of phenolic resin with nano-sized Ni catalyst. The effect of Ni concentration on the content of CNTs as well as EMI SE of composite was also investigated. The results showed that the EMI - SE increased from 28.3 to 75.2 dB in X-band when content of Ni increased from 0 to 1.25 wt.%. The composite containing 5 wt.% CNTs exposed an EMI-SE higher than 70 dB in the whole X-band. A composite material of CNTs for EMI shielding application was also reported by Renata et al., [8]. The composite film including CNTs, conducting polymer and metal nanoparticles was fabricated. They showed that the coating thickness was 100-200 μm, and the electromagnetic range tested from 200 to 1000 MHz. The studies of electromagnetic shielding found that EMI-SE of material was 15-40 dB. Furthermore, the CNTs were found to be the most effective material compared to others. M. Bayat et al., [9] was studied EMI-SE of hybrid multifunctional Fe_3O_4/CNF composite. Here, they examined the various parameters such as Fe_3O_4 content, carbonization temperature and thickness on total shielding efficiency of different samples. The results showed that the maximum EMI - SE of 67.9 dB is obtained for composite of 5 wt.% Fe_3O_4 with 0.7 mm thickness. Yanju Liu et al. [10] conducted research on the EMI shielding performance of Nano composite with MWCNTs, nanosized Fe_3O_4, and Fe. Here, Nano composite specimens with different fillers were prepared. The microwave absorption of these specimens was determined by vector network analyzer. The results indicated that the Nano composite material has excellent microwave absorption effect in the frequency band (from 13 GHz to 40 GHz). For the Nano composite materials with 10 wt.% MWCNTs, the maximum microwave absorption effect is 56.92 dB. For the Nano composite materials with Nano-Fe_2O_3, Nano-Fe and CNTs, the maximum microwave absorption effect is 100 dB.

Such, it can be seen that the above mentioned research groups are leading ones in the field of study composite materials of carbon materials for EMI applications. The carbon materials have been used including CNTs, carbon filber, and CB material. Additionally, Fe_3O_4, Fe, Ni, Cu powders have been also selected as fillers in the matrix of epoxy. According to our knowledge, the using of Nano composite materials as Cu powder/carbon black/epoxy resin for EMI shielding applications has been not much published. In this paper, we reported a composite film of Cu powder/carbon black/epoxy resin for EMI shielding application in frequency range of 8 to 12 GHz. The influence of Cu powder mass and the thick film was also studied. The results of the present study showed the potential application of these materials for electromagnetic interference shielding effectiveness in the future.

2. EXPERIMENTAL

2.1. Chemical reagents

Cu powder (CAS: 7440-50-8) was supported by Xilong Chemical Co., Ltd, China. Carbon black was purchased from Hangzhou Dayangchem Co., Ltd, China. A commercially available epoxy resin with CAS No: 61788-97-4 was supported by Tianjin Yanhai Chemical Co., Ltd, China. Butyl acetate ($C_6H_{12}O_2$) was obtained by Sigma-Aldrich (CAS:

13864), epoxy curing agent (Aradur 115 from Hunstman company- India). Other chemicals used were of analytical grade.

2.2. Sample preparation

All of composites materials for EMI shielding studies were prepared by solution processing as following: the different amounts of carbon black and Cu powder were suspended in solvent by ultrasonic bath for 5 hour at room temperature. The mixtures (carbon black and Cu powder) suspension was added to epoxy solution and was further ultrasonicated for 5 hours at room temperature. And then, epoxy curing agent was added for 10 min. The mixture was sprayed at room temperature on substrate (20×20 cm^2) and left overnight to evaporate the solvent.

2.3. Characterization

The morphology of composite was characterized by FE-SEM (JSM 7500F). FT-IR spectroscopy was performed on Thermo Nicolet 6700 FT-IR spectrometer. For EMI-SE characterization in the frequency from 8 to 12 GHz, the network analyzer (VNA Master MS2024A) was used. The instrumental setup for shielding effectiveness measurement is shown in figure 1.

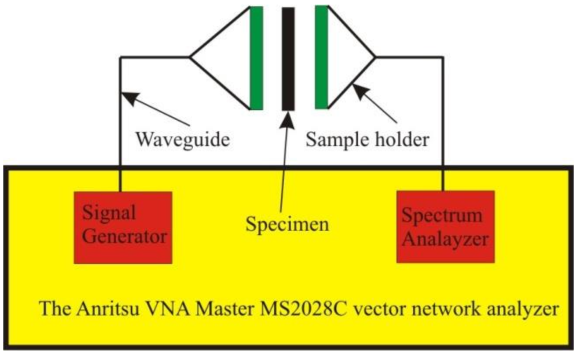

Fig. 1: Instrumental setup for measuring of EMI-SE

3. RESULTS AND DISCUSSION

3.1. Characterizations

3.1.1. FE-SEM & EDX

Figure 2 shows the microstructures of carbon black/epoxy composite film (a), Cu powder/carbon black/epoxy resin composite film (c) and EDX spectra of carbon black/epoxy composite film (b), Cu powder/carbon black/epoxy composite film (d). From the FE-SEM images it can be observed that the fillers such as carbon black and Cu powder are well dispersed in epoxy resin (Fig. 2 a, c). In the figure 2 a, it can be seen that the aggregates and agglomerates of carbon black surrounded by the polyme matrix which have a high propensity to group together and form clusters. With the introduction of Cu powder, the aggregates and agglomerates still were observed. However, the microstructure of the composite material is fairly homogeneous.

Figure 2 (b, d) illustrated EDX spectroscopy of carbon black/epoxy resin composite film (b), and Cu powder/carbon black/epoxy resin composite film (d). It can be seen that the carbon mass is observed at high content (82.2 wt. %), meanwhile the Cu powder is detected at lower content (1.3 wt. %).

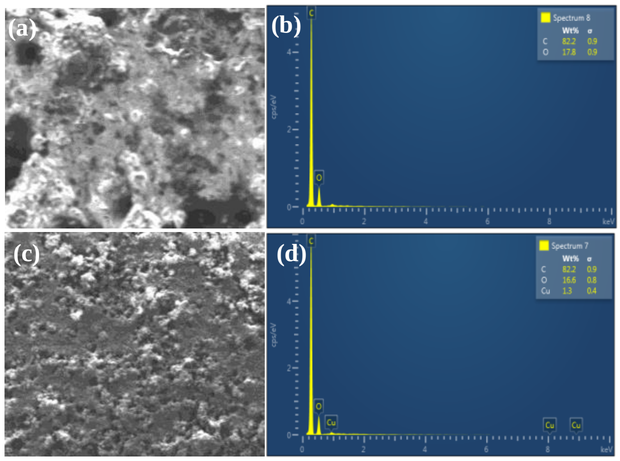

Fig. 2: FE-SEM images (a), (c) and EDX spectra (b), (d) of carbon black/epoxy resin composite film and Cu powder/carbon black/epoxy resin composite film, respectively

3.1.2. FTIR spectroscopy

In this work, the FTIR spectrum was used to monitor of motions occurring between the different chemical bonds of the coating molecules. Figure 3 presents the FTIR spectrum of the Cu powder/carbon black/epoxy resin composite film in the range of 400 to 1600 cm^{-1}. As observed in the FTIR spectroscopy, there is a peak at 1547 cm^{-1}, which corresponds to the C=C bonding of the carbon black. The peak at 1235 cm^{-1} can be assigned to C–H vibration.

The band is observed at 1084 cm^{-1}, which is corresponding to C-C-O bonding and the peak of 520 cm^{-1} can be ascribed to Cu-O bonding.

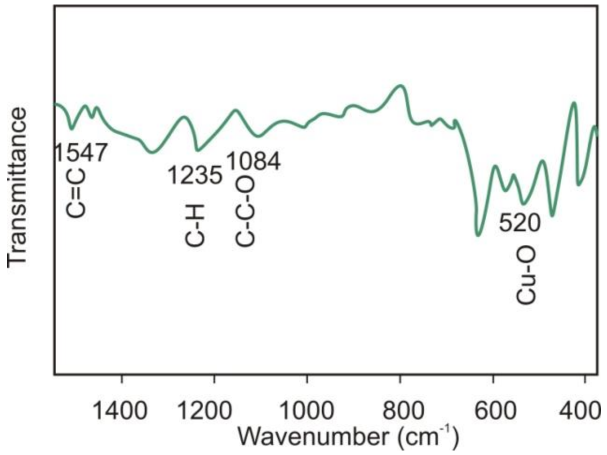

Fig. 3: FTIR spectrum of the Cu powder/CB/epoxy composite film

3.2. Shielding effectiveness of electromagnetic interference

The system used for EMI–SE measurement is schematically illustrated in figure 1. According to D.D.L Chung [11], the primary mechanism of EMI shielding is usually a reflection of the electromagnetic radiation signals incident on surface of composite material. The refection is the result of the interaction of EMI radiation with free electrons on the surface of the shielding. Absorption is usually a secondary mechanism of EMI shielding. Due to that the composite material used for shields has electric or magnetic dipoles. Thus, electromagnetic fields in the radiation had gone through the composite film and then interact with electric/magnetic dipoles of composite material of the shielding. Third mechanism of EMI shielding is multiple reflections, which refer to the reflection of electromagnetic wave at different surfaces of shield. This mechanism requires shield with a large surface area of porous or foam material. Thus, this mechanism can be ignored when shielding effectiveness by absorption is higher 10 dB, or most of the re-reflected wave will be absorbed within the shield [12] or the distance between the reflecting surface is large compared to the skin depth. The mechanism of electromagnetic shielding presents in figure 4.

According to T. A. Skotheim et al. [13], EMI-SE of a material is defined by ratio between the incident powder (P_i) and outgoing powder of an electromagnetic wave (P_t) that can be expressed in a unit of dB by equation below:

$$SE_T\ (dB) = -10 \log (P_t / P_i) \quad (1)$$

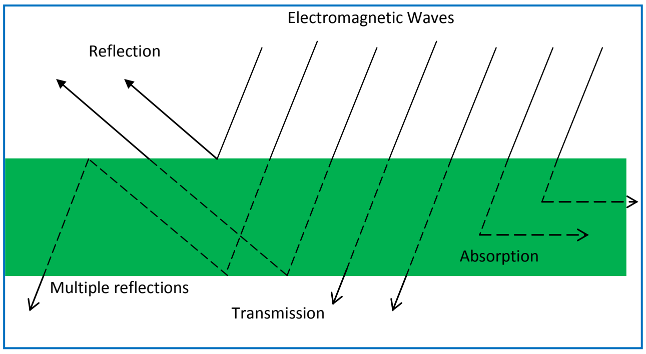

Fig. 4: Mechanism of electromagnetic shielding

When electromagnetic wave is incident on the shielding material, the reflection, absorption as well as transmission takes place. At that time, the total shielding of composite film can be calculated:

$$SE_T = SE_A + SE_R + SE_{MR} \quad (2)$$

where SE_A, SE_R, SE_{MR} are absorption loss, reflection loss and multiple reflection loss, respectively [8]. As above mentioned, SE_{MR} value can be neglected [11, 12]. For this reason, total shielding is calculated as below:

$$SE_T = SE_A + SE_R \quad (3)$$

The transmission (T) of electromagnetic wave is determined by

$$T = P_t / P_i \quad (4)$$

Thus: $$SE_T = -10 \log T \quad (5)$$

The effective absorption is defined as [14]:

$$Aeff = (1 - R - T) / (1 - R) \quad (6)$$

Then, SE due to reflection and absorption can be defined as

$$SE_R = -10\log (1 - R) \quad (7)$$

$$SE_A = -10\log (1 - Aeff) = -10\log (T / (1 - R)) \quad (8)$$

For details of the calculators refer to Xingmin Liu et al. [7], T. A. Skotheim et al., [13], Shailaja Pande et al., [14] and L. Vovchenko L et al. [15].

In this project, the shielding effectiveness of composite films over the frequency ranges of 8-12 GHz is illustrated in figure 5. The signal was plotted for bare substrate (a) epoxy resin coated substrate (b), carbon black/epoxy resin coated substrate (c), and Cu powder/carbon black/epoxy resin coated substrate (d). It can be seen that the EMI-SE of bare substrate sample and epoxy resin sample are approximate zero which was described by line (a) and (b) in figure 5. The EMI-SE is significantly increased that obsevered for carbon black/epoxy resin sample. Meanwhile, the EMI-SE of Cu powder/carbon black/epoxy resin sample is the most increased. Because Cu powder mass added in the composite material, due to Cu powder having high conductivity, thus, it will significantly improve conductivity of composite film. As a result the EMI-

SE is also improved [16]. The maximum value of EMI-SE obtained at room temperature is 4 dB for carbon black/epoxy resin coated substrate sample, and 7 dB for Cu powder/carbon black/epoxy resin coated substrate sample with 500 µm thinkness.

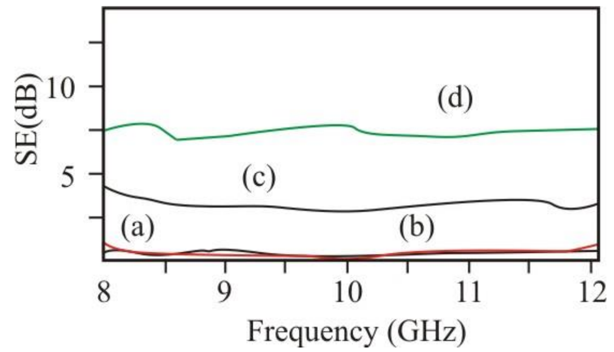

Fig. 5: Electromagnetic interference shielding effectiveness of composite film in range 8-12 GHz, bare sample (a), epoxy resin sample (b), carbon black/epoxy resin sample (c), Cu powder/carbon black/epoxy resin sample (d)

Figure 6 shows the EMI-SE of composite film based on Cu powder/carbon black/epoxy resin with different Cu powder mass over the frequency range of 8–12 GHz. It can be obvious that the composite film is improved with increase of Cu powder mass in the composite film due to the formation of denser conductive networks that enhance the conductivity of composite film [4]. Furthermore, the size of Cu powders is about micrometrics, leading to surface area, the number of dangling bond atoms is all enhanced. These variations make to interface polarization and multiple scattering which are useful for absorption of electromagnetic radiations [17]. Thus, EMI-SE can be also enhanced. In this work, the EMI-SE obtained 25 dB when amount of Cu powder is increased to 10 % mass.

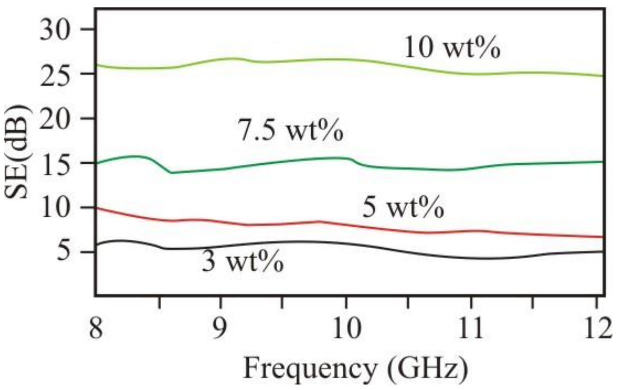

Fig. 6: Electromagnetic interference shielding effectiveness of Cu powder/carbon black/epoxy resin composite film in range 8-12 GHz with different Cu powder mass

In this work, the thickness influence of composite film on EMI-SE was studied. Figure 7 shows the EMI-SE over the frequency range of 8-12 GHz as a function of composite film thickness. As described in figure 7, the EMI-SE is enhanced with increase of the composite film thickness. Due to interaction of conductive network and electromagnetic radiation increase with increasing material thinkness. This leads to better shielding performance of composite film [9].

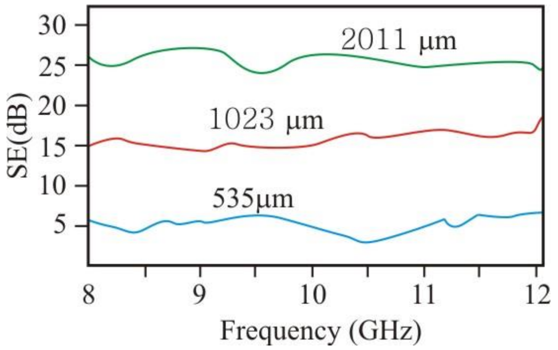

Fig. 7: Electromagnetic interference shielding effectiveness of Cu powder/carbon black/epoxy resin composite film studied in 8-12 GHz with different thinkness film

According to experimental results, the EMI-SE value of composite film increases from 5 dB to approximately 25 dB when the thickness of film is enhanced from 535 µm to 2011 µm.

4. CONCLUSIONS

- The Cu powder/carbon black/epoxy resin mixture prepared composite film has better EMI-SE than that prepared with carbon black/epoxy resin. The maximum value of SE obtained at room temperature is 4 dB for carbon black/epoxy resin and 7 dB for Cu powder/carbon black/epoxy resin sample.
- When using composite film with Cu powder 10 mass %, the EMI-SE obtained 25 dB.
- The SE value of composite film increases from 5 dB to approximately 25 dB when the thickness of film is enhanced from 535-2011 µm.

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