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Effect of iron-deposited graphene oxides on the electromagnetic wave absorbing property of polymer composite films with Fe-based hollow magnetic fibers for near-field applications

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Iron (Fe)-deposited graphene oxide (GO) particles with different ratios of GO to Fe were fabricated and analyzed using TEM, SEM, and VSM to optimize the fabrication conditions with respect to proper magnetization and improved dispersion. The Fe-deposited GOs with a GO:Fe mass ratio of 0.5:1 were used as secondary filler to assist the primary hollow magnetic fibers for the improvement of near-field electromagnetic absorption property measured by a microstrip line method. We prepared composite films with 30 wt% primary hollow fibers and 2 wt% of several kinds of secondary fillers: Fe-deposited GO, GO, and Fe nanoparticles. The power losses of the composite films showed that the incorporation of a small amount of Fe-deposited GO into the film alongside the hollow fibers could lead to a dramatic increase in the power loss compared to the other secondary fillers. According to the experimental results, it was concluded that this increase was due to the fact that Fe-deposited GOs, which were aligned in the in-plane direction and placed among the hollow fibers throughout the film, played a role in connecting the hollow fibers and spreading the electric and magnetic fields originating from the signal.
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1. Introduction

Since the use of electronic devices such as laptops and mobile phones has recently increased at a rapid rate, the main operational frequencies have moved to the GHz range owing to the saturation in the MHz frequency band on which most devices have operated until now. At the same time, as these electronic devices get smaller and thinner, the electronic components must be integrated on a large scale and the dimensions of the electric circuits have become comparable to the wave length of the electrical signals [1]. These similarities can give birth to impedance mismatches at the electrical junctions resulting in generation of noise and unnecessary electromagnetic (EM) radiations. In addition, the radiations also can be induced by the magnetic field of the signal line; for instance, on a microstrip patch antenna or on printed circuit boards [2]. These kinds of the electromagnetic interference (EMI) can have a detrimental influence on the reliability and EM compatibility of electronic systems.

Much attention has been paid to avoiding EMI between the electrical components and signals. In particular, it is essential that the signal integrity be maintained and concurrently the noise should be minimized [3]. In order to suppress or absorb the noise, loss generation by magnetic resonance is typically utilized. Many researchers have reported noise suppression results using amorphous magnetic layers or polymer composite films. They are incorporated in the vicinity of noise sources from which the EM radiation at near-field cannot be assumed as plane waves. Magnetic layers of amorphous and nano-granular type materials such as $Ni_{0.2}Zn_xFe_{2.8-x}O_4$, CoNbZr, MnZn-ferrite and CoZrO can increase the EM absorbing properties which correspond to the value of power loss (P_{loss}) divided by an input power (P_{in}) [4–7]. However, these layers have some problems for widespread use as absorption layers with regards to the cost of the fabrication process and raw

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materials.

Polymer composite films including magnetic fillers have been considered a promising EM wave absorber in near-field applications and have already been realized in commercially available products due to their excellent processability and cost-effectiveness. These noise absorption films usually contain conventional semi-hard magnetic particles or flakes of metal alloys that have both low coercive force and high magnetization [8]. Some organic reagents or polymeric materials were used as binding the fillers. However, there are still some drawbacks of this approach. Since the densities of the magnetic fillers are very high (5–8 g/cm^3), the absorbers with the fillers may be heavy [9–12]. Moreover, it is difficult to fabricate lightweight absorbers because they should contain at least 85 wt% of fillers into the polymer matrix to obtain practically effective noise suppression. Therefore, it is of great practical importance to develop a low-density magnetic filler without significantly impairing the magnetization. Many studies have been attempted with respect to fabrication of lightweight magnetic fillers [13–20]. However, high loading amount of the fillers are required for suitable performance because most of the fillers have low magnetic properties. In addition, these fillers have difficulty in increasing their weight fraction in the matrix owing to the low bulk density resulting from their very small size and high surface area.

Here, in order to overcome such limitations, we propose an iron (Fe)-deposited graphene oxide (GO) which is especially used as auxiliary magnetic fillers. There are many reported researches about composites with graphenes and magnetic graphenes as main magnetic filler exhibiting excellent EM wave absorbing properties. Kong et al. [21] synthesized reduced graphene oxide (RGO) nanosheets combined with surface-modified Fe_2O_3 nanoparticle clusters and the paraffin-based composites with 50wt% of the filler exhibited a low EM reflection coefficient (RC). They emphasized that the low RC is attributed to the interfacial polarization of the cluster and the compromised electrical conductivity of the RGO. Zong et al. [22] synthesized RGO/$CoFe_2O_4$ and the highest reflection loss of the EM wave absorber with 50wt% loading was −47.9 dB at 12.4 GHz. Rugby-shaped $CoFe_2O_4$/RGO [23] and hollow sphere type $CoFe_2O_4$/RGO [24] exhibiting both magnetic loss and dielectric loss were synthesized by a vapor diffusion method. According to the reported results, the composite with 60wt% of the filler had a high reflection loss. Zheng et al. [25] reported an EM wave absorbing material containing 60wt% of Fe_3O_4/graphene nanosheets. Al-Ghamdi et al. [26] prepared high electrical conductive foliated graphite nanosheets and the conducting networks of 40wt% loading in the epoxy matrix played a role in showing 35–55 dB of EMI shielding effectiveness. However, despites the benefits of the graphene-related materials, more than 40wt% of the filler is still necessary. Moreover, most of the results are only related with far-field EM wave absorptions or EMI shielding applications [21–34].

In this work, we investigated the effect of Fe-deposited GO as a secondary filler on the near-field EM absorbing performance of films containing primary magnetic fillers for near-field applications. If the Fe-deposited GOs can be well distributed and especially placed among isolated primary magnetic fillers, it is expected that the filler system even at low concentration can form the effective magnetic linkages in the polymer matrix. Furthermore, it is expected that the Fe-deposited GOs will be able to orient in the in-plane direction inside the polymer film because of their 2D structure originating from the GO. This orientation can enhance the absorbing properties of the polymer composite films compared to films with randomly distributed Fe particles. As the primary filler, hollow magnetic fibers fabricated according to our previous work [20] were used.

2. Experimental

2.1. Synthesis of primary magnetic fillers

As the primary magnetic filler, we used hollow magnetic fibers fabricated according to the experimental protocol outlined in our previous work [20]. The fibers in this work consist of an inner nickel layer and an outer iron-cobalt alloy layer. All experimental conditions for the current fabrication were identical except for the fiber length and elemental composition of the outer layer. The polymer substrate fibers were cut into pieces about 180 μm in length and then hydrolyzed in a solution of 8.5 g/L NaOH maintained at 120 °C for 50 min. The average diameter of the remaining fibrils was about 2.5 μm. Iron (Fe) and cobalt (Co) were simultaneously deposited on the surface of the Ni-coated fibers resulting in an Fe–Co alloy layer. The plating solution included two metal salts: $FeSO_4 \cdot 7H_2O$ and $CoSO_4 \cdot 7H_2O$. $NaBH_4$ and NaOH were used as a reducing agent and pH-controlling agent, respectively. The added weight ratio of the Fe salt and Co salt was 50:50. To remove the polymer substrates fibers, a heat treatment was conducted at 700 °C for 1 h under an argon atmosphere, resulting in the hollow structure.

2.2. Synthesis of Fe-deposited GOs

Graphene oxide (GO) was prepared by modified Hummers method [35,36]. For the synthesis of Fe-deposited GOs, we modified the fabrication method reported by Guo et al. [37] in order to increase the magnetic properties of the particles. 100 mg of the prepared GO was added to 100 mL of deionized (DI) water and treated for 2 h by a sonicator. $FeCl_3$ was dissolved in 50 mL of DI water and the solution was poured into the aqueous GO solution and stirred for 12 h at room temperature. The mass ratios of GO to Fe were varied through different concentrations of $FeCl_3$ in the Fe source solution as shown in Table 1. Next, the aqueous $NaBH_4$ solution was slowly added to the mixture to reduce the Fe^{3+} ions and GO and the reaction was maintained for 30 min. After the reduction, the solution was washed with an excess amount of water by centrifugation to remove unreacted reagents and homo Fe particles. The remnant was dried overnight at room temperature in a vacuum oven. The samples were thermally treated at 500 °C under an argon atmosphere for 2 h. For comparison, GOs were reduced by the same procedure without the addition of $FeCl_3$ and Fe nanoparticles with size of ~200 nm were also synthesized (Table S1, Fig. S1 and Fig. S2 in the Supplementary material).

2.3. Fabrication of composite films with the fillers

The hollow magnetic fibers and the Fe-deposited GO particles were used as primary and secondary fillers, respectively. Composite films with the primary and secondary fillers were fabricated by a doctor blade method. First, a mixture of a binder resin (Thermoplastic urethane, Elastollan, BASF), a blended solvent of dimethylformamide/acetone (1:1), and the secondary filler were

Table 1
Compositions of the solutions for the synthesis of GO/Fe.

	Solution			Mass ratio
	GO solution	Fe source	Reduction	
DI water	100 ml	50 ml	50 ml	–
GO	100 mg	–	–	–
$FeCl_3$	–	116 mg		GO: Fe = 2.5: 1
		580 mg		GO: Fe = 0.5: 1
		2900 mg		GO: Fe = 0.1: 1
$NaBH_4$	–	–	54 mg	–

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