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EMI shielding performance of nanocomposites with MWCNTs, nanosized Fe_3O_4 and Fe

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ABSTRACT

In this paper, nanocomposites based on epoxy matrix with nano-sized multi-walled carbon nanotubes (MWCNTs), Fe_3O_4 and Fe fillers have been developed due to the application in electromagnetic interference (EMI) shielding. The nanocomposites specimens with different fillers, as well as varied filler weight fraction have been prepared. The microwave absorption of these nanocomposites specimens have been verified by vector network analyzer. Furthermore, trilayer-type laminated nanocomposites containing a matching layer with 15 wt% nano-Fe_3O_4, an absorbing layer with 5 wt% CNTs and a reflecting layer with 10 wt% CNTs have been designed and fabricated. Moreover, the permittivity and the permeability for each type of composites are tested as well. Experimental results show that such trilayer-type laminated nanocomposite has excellent micro-wave absorption effect in the frequency band (from 13 GHz to 40 GHz) up to 40 dB, especially in high frequency section. The achieved peak values of the fabricated laminated nanocomposites exceed 100 dB.

1. Introduction

In recent years, the interference shielding of electromagnetic waves continue to attract high interest for commercial and military purposes. Electromagnetic interference (EMI) shielding refers to the reflection and/or absorption of electromagnetic radiation by a material, which thereby acts as a shield against the penetration of the radiation through it [1].

Composites with high electrical conductivity and dielectric constant bring profound development prospect to traditional metal for EMI shielding property. Nanomaterials, especially nanocomposites show excellent EMI shielding performance with larger frequency bandwidth, better compatibility and lower overall mass. Consequently, nanocomposites have been turned out to be the best potential candidate for EMI shielding covering centimeter band and millimeter band [2–4].

Many researches on EMI shielding effectiveness of nanocomposites have been conducted during the past decade. Joo and his colleagues added nano-sized silver into epoxy matrix and obtained the EMI shielding value of −46 dB in the range of 10 MHz–1 GHz. They also carried out the study of the spectra attributes of multilayer absorber composed of materials with different electrical conductivity [5]. Gairola et al. reported a 2.5 mm polyaniline based nanocomposites with nano-ferrite $Mn_{0.2}Ni_{0.4}Zn_{0.4}Fe_2O_4$ which shown high shielding effectiveness up to −50 dB in the frequency band of 8–12 GHz (X-band) [6].

In recent years, the unique electrical, mechanical and magnetic attributes of carbon nanotubes (CNTs) enable it to play an important role in nanoEMI shielding materials preparation [7–9]. Currently, EMI shielding composites prepared by CNTs compounding with organic polymers have become significant field in new generation EMI shielding material research [10–14].

Zhang and his coworkers investigated the EMI shielding effect of a nanocomposite with vapor grown carbon nanofibers (VGCFs) and polyurethane shape memory polymer. Experiments were carried out to evaluate the EMI shielding effect of this type of nanocomposite in three frequency bands as 18–26.5 GHz (K band), 33–50 GHz (Q band) and 50–75 GHz (V band). They found that the EMI shielding effect was dependent of carbon VGCFs content and the specimen thickness. For a specimen made with 6.7 wt% VGCFs and 3 mm thickness, the value of shielding effect exceeded over 30 dB for any of three frequency bands, and the maximum value reached 65 dB [11]. Ma and his research team prepared the composites with maleic anhydride modified MWCNTs (Mah-g-MWCNTs) and poly (methyl methacrylate). The EMI shielding effectiveness (SE) of Mah-g-MWCNTs/PMMA composites increased with the increasing of the Mah-g-MWCNTs content. At the same

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time the wear resistance was enhanced while the loading of the Mah-g-MWCNTs was above the percolation threshold [12]. Parveen et al. from the Indian Institution of Technology investigated the microwave absorption and EMI shielding performance of nanocomposite based on multi-wall carbon nanotubes/polyaniline. The EMI shielding peak value of this composite reached −27.5 to −39.2 dB in the frequency band of 2.4–18.0 GHz [13]. An absorbing nanocomposite made from carbon nanotubes and viton had been developed by Fletcher et al. Its EMI shielding peak value in the 8.2–12.4 GHz (X band) was −42 dB [14].

Multi-layer EMI shielding nanomaterial is typically a laminate containing two or more layers of different materials. The laying up pattern should be carefully determined according to the impedance matching theory or equivalent transmission line theory [15]. The thickness of each layer and the selectance of EMI shielding materials are adjustable in order to achieve the most optimum magnetic parameters matching for wider EMI shielding band and shielding effectiveness [15,16]. Ahn et al. developed radar absorbing structure (RAS), and three-phase composites consisted of {glass fiber}/{epoxy}/{nanosize carbon materials (MWNT and carbon black)} were fabricated. Two types of composites showed outstanding absorbing efficiency over −10 dB between 8–12 GHz frequency range [17]. Kim et al. designed a multi-layered composite, and added MWNT into glass/epoxy plain weave composites to induce dielectric loss. The absorbing bandwidth of −10 dB was 73.8% of whole X band. And the peak value was over −30 dB which means the EM energy absorption of 99.9% [18]. Lee conducted research on preparing nanocomposites with E-glass fabric, epoxy resin, and CNTs. The composite was adhesively bonded to the outside of the sandwich construction in order to absorb EM waves. When the thickness and weight fraction of CNT of nanocomposites were 2.52 mm and 3 wt%, The bandwidth with the EMI SE over 10 dB are 3.3 GHz (that is, the SE between 8.2 and 11.5 GHz are all more than 10 dB) and the maximum absorption rate were 97% [19].

The dielectric property of CNTs, carbon-fibers, carbon blacks composites are also widely studied [20–24], which is regarded as a important part of attenuate the electromagnetic wave [20,23,24]. Meanwhile, the nanosized Fe particle or Fe-doped CNTs composites are fabricated as well, the results demonstrated that the added nanosized Fe particles can strongly enhance the EMI shielding performance of the CNTs composites [24–26].

In this paper, only the microwave absorption properties of nano-composites are focused, carbon nanotubes, nano-sized Fe_3O_4 and Fe powders are selected as the fillers in the matrix of epoxy resin. The reason of choosing these three nanofillers is owing to they are the representative nanoparticles of nonmetal, metallic oxide, and metal respectively. Although there are some researchers already studied the microwave absorption performances of various nano-composites, there is no report of the comparison of the microwave absorption of these three types of nanocomposites with the same mass fraction and composite thickness. The microwave absorption of the CNTs, nano-Fe_3O_4 and nano-Fe filled epoxy nanocomposites have been investigated based on experiments. Owing to the further application of prepared composites are stealth materials for radar waves, so the EMI SE mentioned in this paper are all indicate the absorption part, i.e. the SE_A. (As the total EMI SE (SE_{total}) can be represented by sum of contributions from absorption loss (SE_A), reflection loss (SE_R) and multiple reflections (SE_M), and $SE_{total} = SE_A + SE_R + SE_M$.) Meanwhile, the permittivity and permeability of the three types of nano-composites with various weight fractions are discussed and compared as well. A new kind of laminated structure with three layers of different nanocomposites have been proposed and manufactured in order to obtain better microwave absorption.

2. Material and methods

2.1. Materials

Commercially available multi-walled carbon nanotubes (MWCNTs, for simply, CNTs, same as below) with an average diameter of 50 nm and the length of 10–20 μm are produced by Chengdu Organic Chemicals Co. Ltd. Spherical nano-Fe_3O_4 with an average diameter of 20 nm and nano-Fe with an average diameter of 50 nm are supplied by Nanjing Emperor Co. Ltd. Epoxy resin (AG-80) is produced by Shanghai Institute of Synthetic Resin.

2.2. Preparation of nanocomposite specimens

Preheated MWCNTs are acidificated in an acid of H_2SO_4 and HNO_3 in a ratio of 3:1 for 7 h. Then a certain amount treated MWCNTs together with dispersant CTAB (Cetyltrimethyl Ammonium Bromide) were put into the polymeric mixture epoxy resin and polyamide curing agent. The mixture was agitated by ultrasonic for 40 min. After being mixed uniformly and degassed, the mixture is poured into a plate type curing mould and cured for 6 h at 60 °C in vacuum. Nano-Fe_3O_4 and nano-Fe filled epoxy nanocomposites thin plates are fabricated in the similar way. Different thin plates are made with 5%, 7.5% and 10% weight fractions of MWCNTs. Fig. 1 shows the SEM photos of the 10 wt% CNT/epoxy composite, it can be observed that the CNTs are well dispersed in the epoxy matrix. For nanocomposites filled with Fe_3O_4 and Fe, the fillers weight fractions are 10% and 15%. The thicknesses of fabricated thin plates of all the three types of nanocomposites are 3 mm.

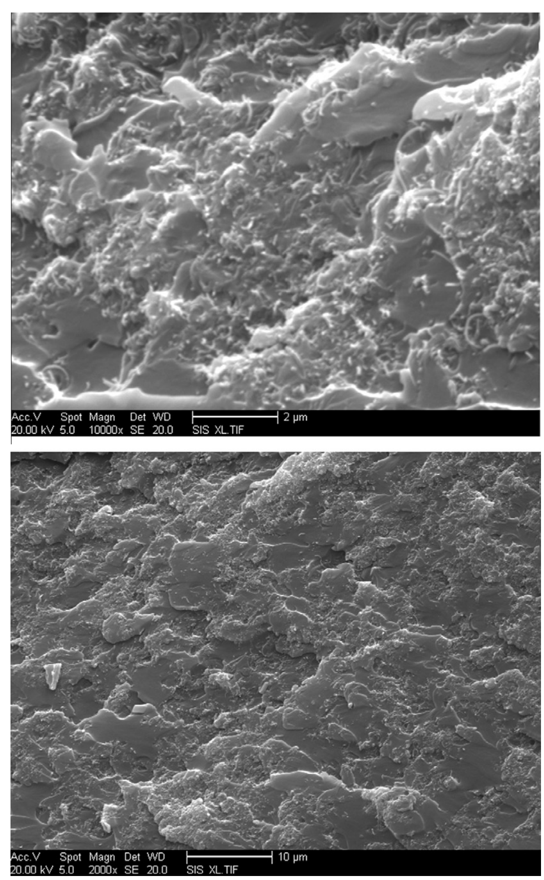

Fig. 1. The SEM photos of 10 wt% CNTs/epoxy composite.

2.3. Testing procesure

In order to measure the microwave absorption of the composite materials, vector Network Analyzer (Agilent 8722ES) and waveguides were adopted. [27]. The frequency range is between 3.22 GHz and 40 GHz. All the composite samples were machined as the same dimension with the corresponding waveguides, which lead to the samples exactly fit the size of waveguides without gaps. For eliminate the influence of the air in the waveguides, the testing procedures are as followed: first, measuring the absorption efficiency of each empty waveguide without the samples, then put the samples with seven different sizes into the corresponding waveguides and test the absorption efficiency of the composites (each type of the composites have seven samples to fit the waveguides of different frequencies), finally subtract the former(the absorption of air) from the latter(the absorption of air and sample), the actual microwave absorption of each composites should be obtained. And the disturbance of the background can be minimized.

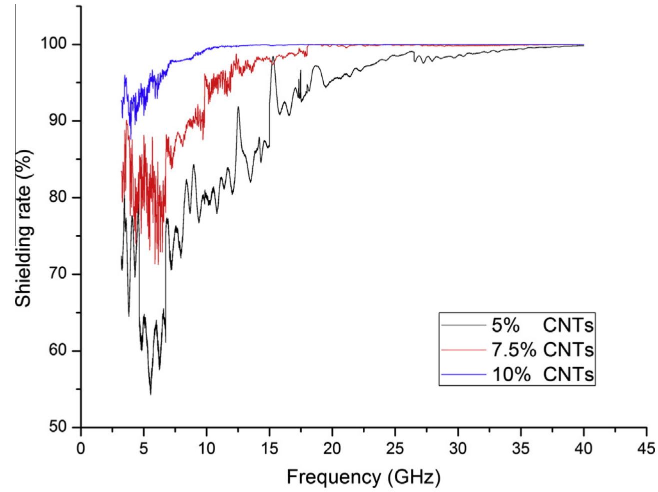

Fig. 3. Shielding (absorbing) rate of CNT/epoxy nanocomposites.

3. Results and discussion

3.1. Microwave absorption of single layer composites

3.1.1. CNTs/epoxy nanocomposite

The microwave absorption of CNTs/epoxy nanocomposites with different weight fractions are shown in Fig. 2. It can be observed that bigger CNTs fractions lead to better absorbing property of the composites. In addition, it is obviously that the property of each composite is getting better with the increasing of the frequency. The Peak absorbing values for the three kind of CNTs/epoxy composites (with the weight fraction of CNTs as 5%, 7.5% and 10%) are 28.08 dB (at 39.989 GHz), 43.103 dB (at 18.82 GHz) and 56.92 dB (at 39.405 GHz) respectively. The negative sign in the figure stands for "absorption", and the absorbing effectiveness of each composite depends on the absolute value [7–9,28].

Shielding rate (here means the percentage of absorption) is generally used to indicate how many percent of incident signal is blocked by the shielding materials (which is another representation of absorbing effectiveness, the unit of the former is dB, and the latter has no unit, it is just the ratio of the absorption part to the initial ones). The transformational relation of the absorbing effectiveness and the shielding rate is as followed: For the absorption is 10 dB, it means 90% of incident signal is blocked, so the shielding rate is 90%. 20 dB means 99% of incident signal is blocked and the shielding rate is 99% as well, etc. Fig. 3 shows the shielding rates of CNTs/epoxy nanocomposites with different weight fractions. All three kinds of specimens have the shielding rate approximate to 100% in the region of relatively higher frequency (near 40 GHz). It demonstrates that CNT is a kind of good fillers of producing microwave absorption composites, which preforms an excellent absorbing property.

3.1.2. Nano-Fe_3O_4/epoxy and nano-Fe/epoxy nanocomposites

The microwave absorption of nano-Fe_3O_4/epoxy and nano-Fe/epoxy composites with different weight fractions are shown in Figs. 4 and 5. It can be observed that the absorbing effectiveness improves slightly with the increasing of the weight fraction. Both the two types of nanocomposites present relatively good absorbing performance in the frequency region of 18–26.5 GHz. Which is similar to the CNTs composites, but the difference is, the microwave absorption of CNTs composites improved with the increasing of frequency, but the microwave absorption of nano-Fe_3O_4 and nano-Fe composites are not gradually changed with the increasing of the frequency, it only presents several "peak values" in the microwave absorption curves. That means in these specific frequency values, the shielding performance are much higher than others.

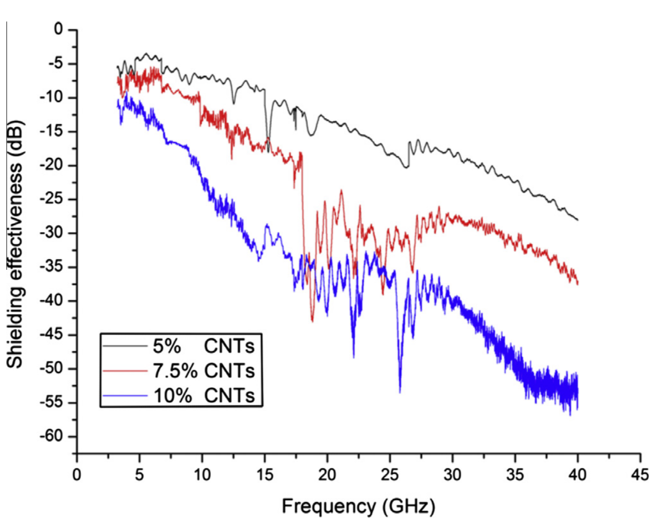

Fig. 2. Microwave absorption of CNT/epoxy nanocomposites.

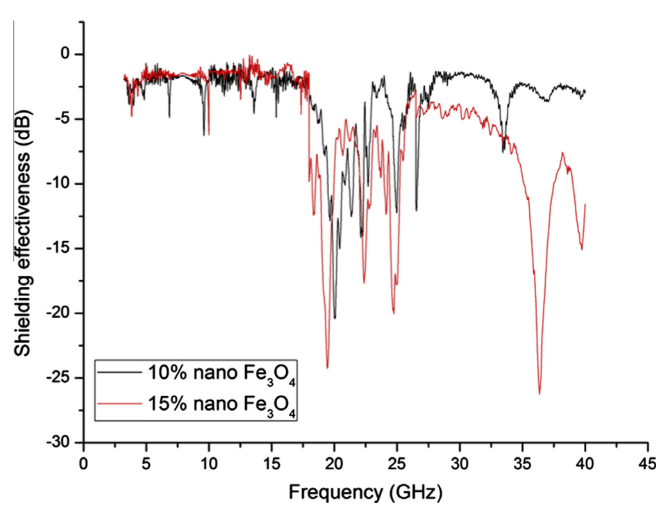

Fig. 4. Microwave absorption of nano-Fe_3O_4/epoxy nanocomposites.

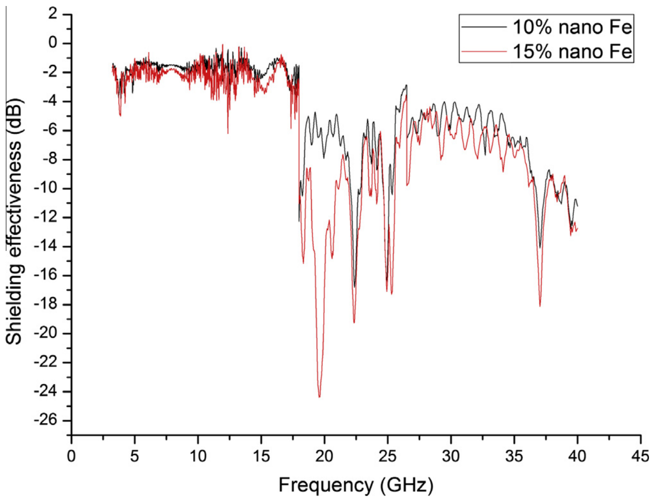

Fig. 5. Microwave absorption of nano-Fe/epoxy nanocomposites.

3.2. Microwave absorption of tri-layer composites

According to the analysis of the microwave absorption of the composites with the three different kinds of nano-fillers and variation weight friction mentioned above, it is necessary to fabricate a kind of multi-layer composites so that a higher microwave absorption should achieved.

Two types of tri-layers microwave absorption nano-composites were fabricated as shown in Fig. 6. Sample 1# was constituted with the impedance matching layer of 15 wt% nano-Fe_3O_4, the absorbing layer of 5 wt% MWCNTs and the reflecting layer of 10 wt% CNTs. Analogously, Sample 2# was constituted with the impedance matching layer of 15 wt% nano-Fe, the absorbing layer and the reflecting layer are same as Sample 1#. This kind of structural design has the functions of simultaneously reducing the reflect loss at the surface of the first layer and increasing the absorption of the waves coming into the composites. The materials adopted as matching layer should have relatively higher impedance which can match with the impedance of the air, and the absorbing layer should has intense absorption of the microwaves, moreover, the reflecting layer should present even higher absorbing property, and strong reflecting property as well, which induce the re-absorbing of the waves incoming in the absorbing layer and lead to the maximum loss in the tri-layer composites. The computational process of each composites are not list in this paper, which could be consulted in reference [29,30]. The microwave absorption for the two types of tri-layer nano-composites are shown in Table 1 and Fig. 7 respectively.

As shown in Fig. 7 and Table 1, the max microwave absorption values of the two types of tri-layer composites both surpass 100 dB, which is far more than the sum of the individual shielding properties of the three single layer nanocomposites. The microwave absorption of sample 1# is superior to sample 2#. In the frequency range of 3.22–40 GHz, microwave absorption of both samples increases with the frequency and the peak values appear around 40 GHz. Table 1 demonstrated that tri-layer nanocomposite have better absorbing performance than single-layer ones, especially in the high frequency range.

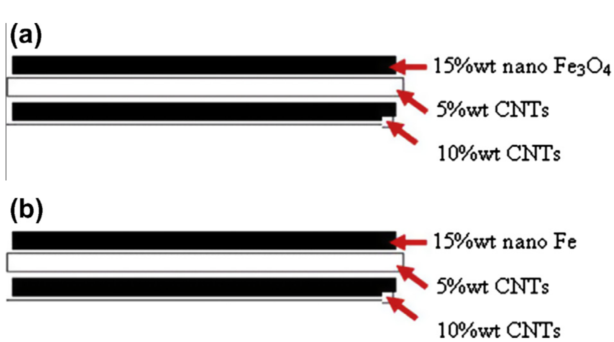

Fig. 6. Tri-layer nanocomposites layer sequence: (a) Sample 1. (b) Sample 2.

Table 1
Tri-layer nanocomposites microwave absorption results analysis.

	Sample 1#	Sample 2#
Peak value	−105.55793 dB	−100.39672 dB
<−20 dB band width	33.01 GHz/89.7%	33.24 GHz/90.36%
<−30 dB band width	28.81 GHz/78.32%	28.92 GHz/78.63%
<−40 dB band width	25.38 GHz/69%	26.46 GHz/71.94%
<−50 dB band width	20.215 GHz/54.96%	22.61 GHz/61.47%
<−60 dB band width	12.69 GHz/34.5%	13.10 GHz/35.6%
<−70 dB band width	8.03 GHz/21.84%	8.70 GHz/23.67%
<−80 dB band width	4.93 GHz/13.4%	5.06 GHz/13.76%
<−90 dB band width	1.08 GHz/2.94%	0.88 GHz/2.39%

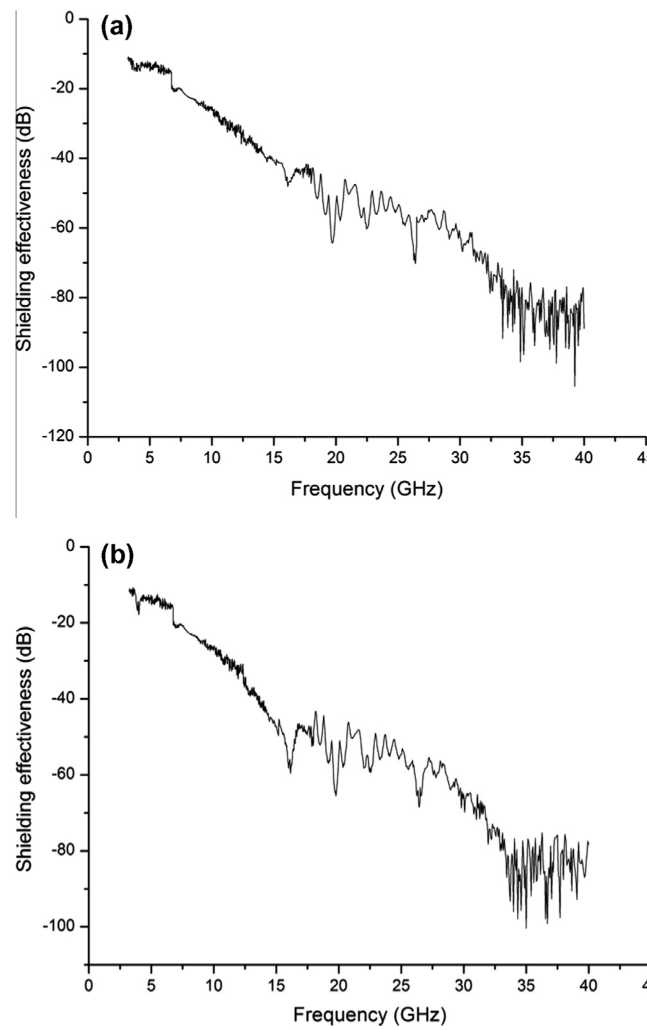

Fig. 7. Microwave absorption of try-layer nanocomposites: (a) 1#, (b) 2#.

3.3. The permittivity and permeability of the composites

Fig. 8 are the real part ε' and the imaginary part ε'' of permittivity for the three types of composites with various mass fractions, it should be noticed that only the properties in 18–40 GHz frequency range are discussed here. From Fig. 8(a) and (b), it can be observed that both the real part and the imaginary part of the CNT/epoxy composites are slightly reduced with the increasing of the frequency, and obviously enhanced with the raise of the mass fraction as well. The enhance level between 7.5 wt% and 10 wt% are higher than which of the 5 wt% and 7.5 wt% which may due to the onset of electric percolation in MWCNTs [24]. Therefore, the experimental results demonstrated that the CNT/epoxy composites perform a big part of dielectric loss in EMI shielding.

Fig. 8(c–f) are the permittivity for nano-Fe_3O_4 and nano-Fe composites with various mass fractions respectively. It can be

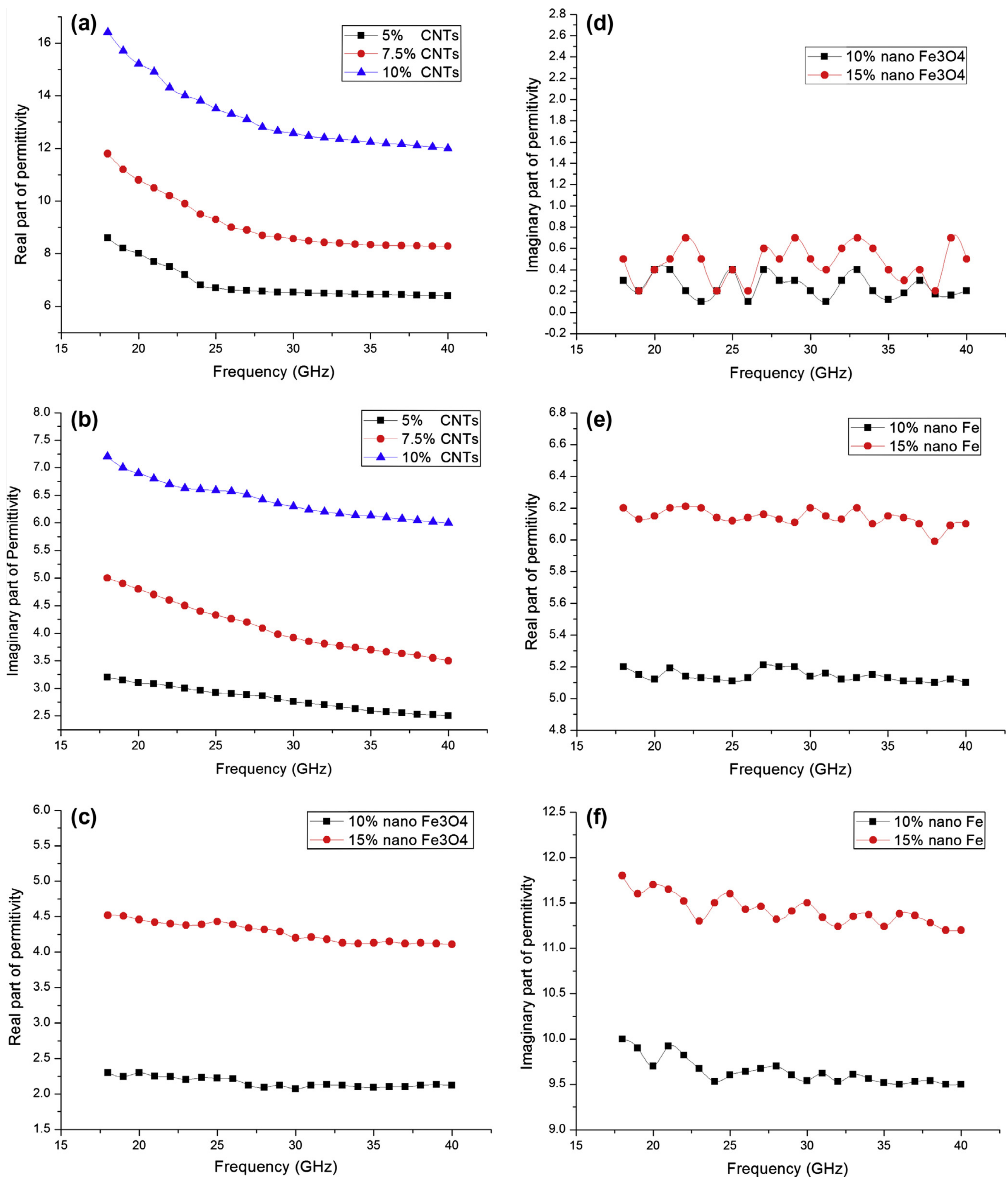

Fig. 8. The permittivity of three types of nanocomposites: (a) Real part of CNT/epoxy composites; (b) Imaginary part of CNT/epoxy composites. (c) Real part of nano-Fe_3O_4/epoxy composites; (d) Imaginary part of nano-Fe_3O_4/epoxy composites; (e) Real part of nano-Fe/epoxy composites; (f) Imaginary part of nano-Fe/epoxy composites.

founded that both the real part and the imaginary part are raised with the mass fraction, which is similar to the property of CNT composites. In addition, the real parts of these two composites are very close, and both much lower than CNT composites, although the mass fractions are relatively higher. Meanwhile, the imaginary part of these two composites are totally different, the imaginary part of Fe_3O_4 composites are close to zero, while the Fe composites are raised to more than 10. As being normally

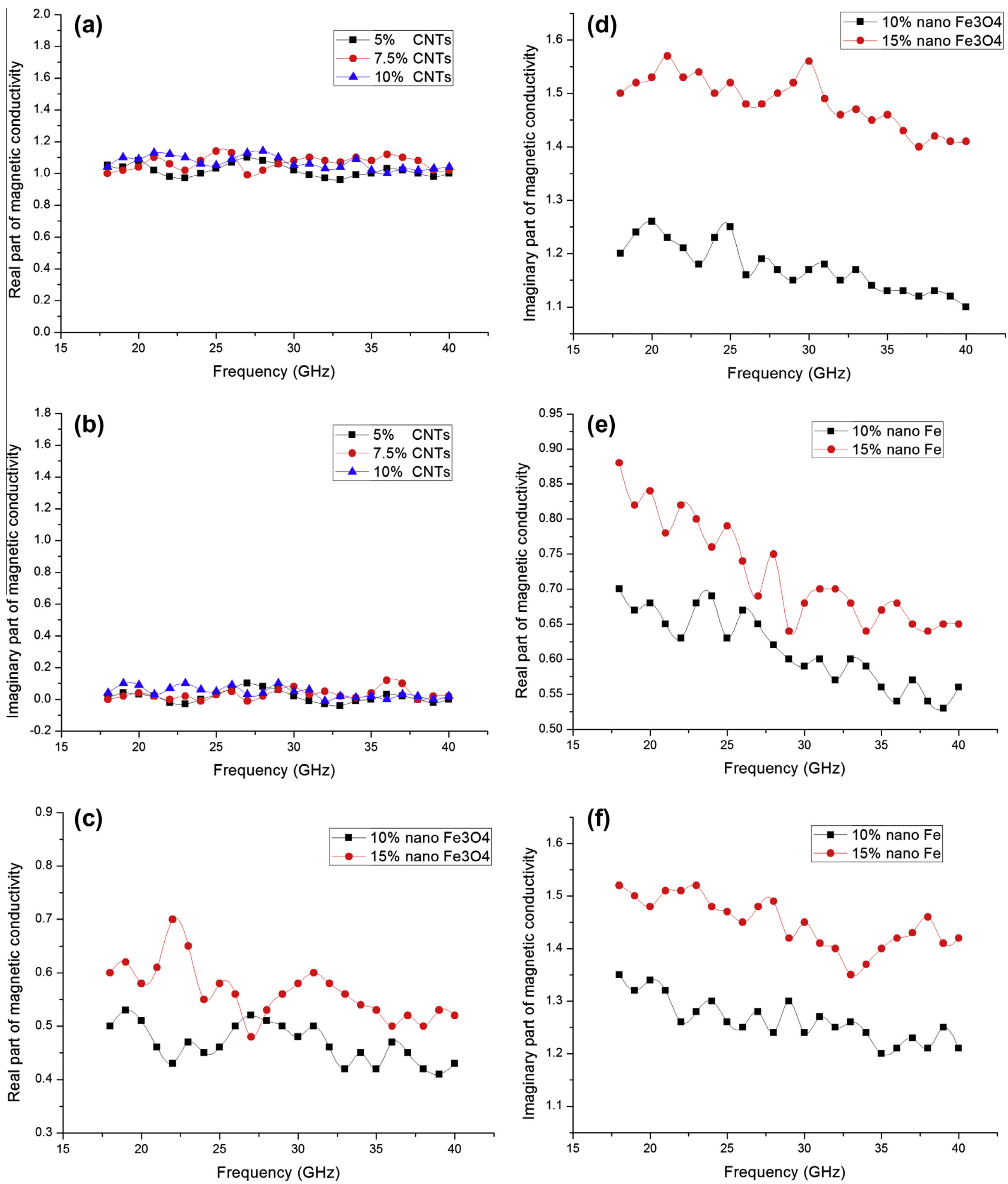

Fig. 9. The permeability of three types of nanocomposites: (a) Real part of CNT/epoxy composites; (b) Imaginary part of CNT/epoxy composites. (c) Real part of nano-Fe_3O_4/epoxy composites; (d) Imaginary part of nano-Fe_3O_4/epoxy composites; (e) Real part of nano-Fe/epoxy composites; (f) Imaginary part of nano-Fe/epoxy composites.

accepted that the real part of the permittivity are due to the presence of polarization effect and the imaginary part are come from the presence of free electrons in materials. So the difference of the imaginary parts indicated the various dielectric properties of nano-Fe_3O_4 and nano-Fe composites.

Fig. 9 is the real part μ' and the imaginary part of permeability μ'' for the three types of composites. It can observed that for the CNT composites (Fig. 9(a and b)), the real part equals 1 and the imaginary part equals zero, so it indicates that CNTs hardly have magnetic loss. Meanwhile, the Fe and Fe_3O_4 composites show better magnetic property, the real parts are both in the range of 0.4–0.9 and the imaginary part in the range 1.2–1.6, and depend on the mass fraction. Which indicate that the nano-Fe and nano-Fe_3O_4 composites both have dielectric loss and magnetic loss in EMI shielding.

Comparing the results with Ref. [24], the authors fabricated the Fe-doped MWCNTs composites and tested the permittivity, permeability as well as the EMI shielding performance in 26–40 GHz, they argued that due to the high aspect ratio of nano-size Fe, the polarized interfaces between Fe and carbon walls would established, which can induce the raise of permittivity of the composites compared with pristine MWCNT composites, and finally enhance the EMI shielding performance as well. Meanwhile, the Fe-doped composites in Ref. [24] still showed a relatively low permeability, the real part and imaginary part are not exceed 1.2 and 0.4 respectively. Which is interpreted as the extremely low concentrations of nano-Fe (0.005–0.008 wt%). In this paper, the mass fraction of Fe have already increased to 10–15%, the 1000 times of the former, but the permeability only have a slightly rise (i.e. 1.6 and 0.9), so it also confirmed that EMI shielding property of the nano-sized Fe are hardly come form the magnetic loss, but dominant over dielectric loss [25,26].

4. Conclusions

The absorbing effectiveness of MWCNTs/epoxy nanocomposite depends strongly on the weight fraction of MWCNTs and the wave frequency. The bigger weight fraction of MWCNTs and the higher frequency both lead to relatively better microwave absorption.

For the nanocomposite with 10 wt% MWCNTs, the maximum microwave absorption is 56.92 dB and the band width of the shielding rate over 99% reaches 34.266 GHz. For the nancomposites filling with nanosized Fe_3O_4 and Fe, the microwave absorption also depend on the weight fraction of the fillers, but the influence of frequency are not so obvious as which of the CNT/epoxy composites, the absorption of the former presents several "peak values" during the frequency while the latter getting bigger gradually with the increasing of the frequency.

The two types of tri-layer composites with nano-Fe_3O_4, nano-Fe and CNTs are all present excellent microwave absorbing performances. The max absorption both exceed 100 dB and in the testing frequency of 3.22–40 GHz, more than 90% band width has the microwave absorption over 20 dB, which illustrate that the tri-layer nano-composites have powerful and stable microwave absorbing property in testing frequency.

The dielectric permittivity and the magnetic permeability of nano-Fe_3O_4, nano-Fe and CNTs composites increase with the raise of the mass fraction, and EMI shielding property of the three types of composites are all dominant over dielectric loss, rather than magnetic loss.

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