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Enhanced electromagnetic absorption properties of carbon nanotubes and zinc oxide whisker microwave absorber

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ABSTRACT

CNTs/ZnOw composites were fabricated by a simple mechanical mixing method. The effects of the different CNTs and ZnOw mass fractions on the electromagnetic parameters and wave absorbing properties of the composites were studied. The complex permittivity and attenuation constant increase with increasing mass fractions of CNTs and ZnOw. However, the microwave impedance decreases with the increasing concentrations of CNTs and ZnOw. The experimental results show that 4 wt% CNTs mixed with 10 wt% ZnOw has the optimum microwave absorption ability with a thickness of 2.0 mm. The minimum reflection loss is −37.03 dB at 12.24 GHz and the bandwidth corresponding to the reflection loss below −10 dB is more than 4.04 GHz. Compared with pure CNTs or ZnOw, it cannot only greatly reduce the mass fractions of microwave absorbing materials, but also enhance the microwave absorption properties. The results indicate that the prepared CNTs/ZnOw composites have excellent absorbing properties with thin thickness and lightweight.

1. Introduction

In recent decades, the microwave absorbing materials have attracted considerable interest in both commercial and military purposes. The microwave absorbing materials can be used to minimize the electromagnetic reflection from the metal plate such as aircrafts, ships, tanks and the walls of an echoic chambers and electronic equipments [1–4]. Extensive studies have been carried out to find suitable microwave absorbers [5–7]. Ferrites [8–12] and carbonyl iron [13–15] have been utilized as absorbing materials in various forms due to their large magnetic loss and large resistivity. However, unavoidable disadvantage of overweight of these magnetic absorbers has greatly limited the applications. Much effort has been devoted to the development of better radar absorbing materials with lightweight, thin thickness and wide band absorption [16–18].

Compared with magnetic absorbers, the dielectric absorbers are relatively of low density for their low mixing ratios, which are more suitable for radar absorbing materials with light weight and thin thickness. Since the discovery in 1991 [19], carbon nanotubes (CNTs) have been widely studied as microwave absorbing materials for higher aspect ratio and electrical conductivity. The concentration of CNTs is extremely important for the application as microwave absorbing material. A certain amount of CNTs is required for the attenuation of the microwave, however, a low loading is expected to avoid or minimize the degradation of other performance aspects, such as the mechanical properties [20]. Zinc oxide whisker (ZnOw) is comprised of a central part and four needle crystal projections extending from said part in plural different axial directions [21], has shown great potential as radar absorbing material [21–25]. On the basis of these characteristics, novel electromagnetic wave absorption properties are expected from the combination of ZnOw and CNTs. Li et al. [26] revealed the CNTs/T–ZnO/EP composite exhibited excellent microwave absorption properties compared with CNTs/EP and T–ZnO/EP composites. A minimum reflection loss of −23.00 dB were obtained when the content of CNTs and T–ZnO are 12 wt% and 8 wt%, respectively. However, further studies are still necessary to optimize the mass fraction of CNTs and ZnOw for a better microwave absorbing ability. Furthermore, a high concentration of CNTs in the composites is expected to be avoided, which will result in degradation of other performance.

In the present work, CNTs/ZnOw composites were fabricated by a simple mechanical mixing method. The effects of the different concentrations of CNTs and ZnOw on the electromagnetic parameters and microwave absorbing properties of the composites were studied. The optimization of CNTs and ZnOw concentrations

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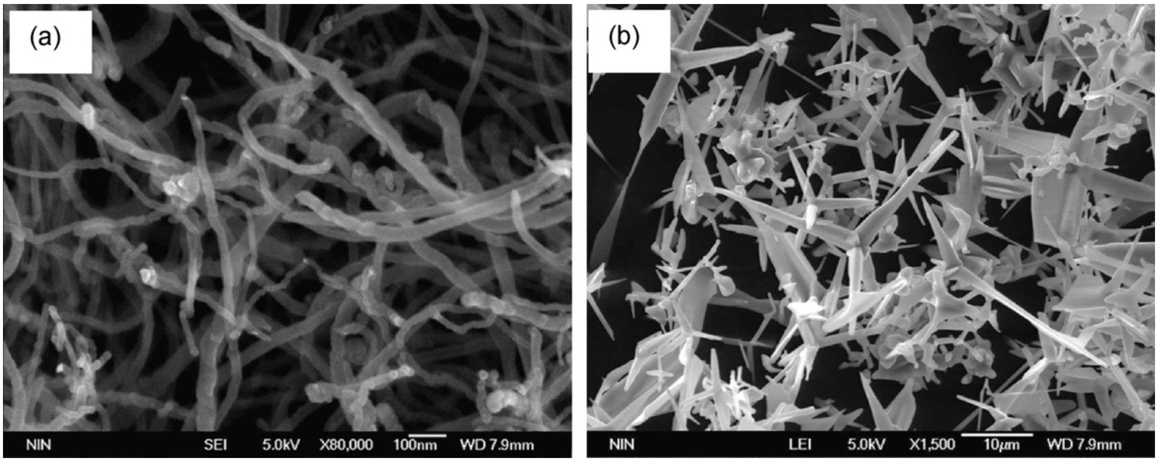

Fig. 1. Scanning electron microscopy (SEM) of CNTs (a) and ZnOw (b).

was realized. The results show that optimized composite possesses excellent microwave absorbing property.

2. Experimental

Multi-walled carbon nanotubes were synthesized by chemical vapor deposition with diameters ranging from 5 to 15 nm and lengths ranging from 10 to 20 μm. The surface morphology of CNTs was examined by scanning electron microscopy (JSM-6700), which is shown in Fig. 1(a). ZnOw were synthesized with equilibrant reaction method. Details of the preparation method were described elsewhere [27]. The length and basal diameter of the needles of the whiskers were 15–120 μm and 1.8–6.6 μm, respectively (Fig. 1(b)).

The CNTs/ZnOw complex absorbers were obtained by homogeneously mixing the CNTs and ZnOw with various weight fractions instead of the complicated chemical methods devices. Therefore, this method is in close relation to industry processes. For measurements of the electromagnetic parameters, CNTs/ZnOw complex absorbers were mixed with paraffin and poured into coaxial clapper in a dimension of outer diameter of 7.0 mm, inner diameter of 3.0 mm, respectively. The proportions of each component are shown in Table 1. The complex permittivity $\varepsilon_r(f)$ and permeability $\mu_r(f)$ of CNTs/ZnOw composites were measured by the T/R coaxial line method in the frequency range of 2–18 GHz using a network analyze (Agilent technologies E8362B:10 MHz–20 GHz). The microwave absorbing performances were evaluated by the following equation [28]:

$$R = 20\log\left|\frac{Z_{in}-Z_0}{Z_{in}+Z_0}\right| \tag{1}$$

where R denotes the reflection loss in dB unit. Z_0 is the characteristic impedance of free space. Z_{in} is the input characteristic impedance at the absorber/free space interface, which can be expressed as

$$Z_{in} = Z_0\sqrt{\frac{\mu_r}{\varepsilon_r}}\tan h\left(j\left(\frac{2\pi fd}{c}\right)\sqrt{\mu_r\varepsilon_r}\right) \tag{2}$$

where c is the velocity of light, f is the frequency of electromagnetic wave, d is the thickness of an absorber. In this paper, all d values are in mm unit. $\varepsilon_r = \varepsilon' - j\varepsilon''$ is complex permittivity, $\mu_r = \mu' - j\mu''$ is complex permeability.

Table 1
Proportion of the components of the samples.

Samples	CNTs (wt%)	ZnOw (wt%)
1	4%	0%
2	6%	0%
3	8%	0%
4	10%	0%
5	0%	15%
6	2%	15%
7	4%	15%
8	6%	15%
9	8%	15%
10	4%	5%
11	4%	10%
12	4%	20%
13	4%	30%

3. Results and discussion

3.1. Electromagnetic and absorbing properties of CNTs

Fig. 2 shows the real and imaginary parts of complex permittivity of CNTs/paraffin with four concentrations (4, 6, 8 and 10 wt%) in the frequency range of 2–18 GHz. The real and imaginary parts of complex permittivity of the CNTs samples show a similar variety trend. With increasing frequency, the values of real part of complex permittivity decrease slightly. It can be noticed that the higher is the CNTs weight percent addition, the greater are both the real and imaginary permittivity in the frequency range of 2–12 GHz. Increase of the real part of the complex permittivity can be mainly ascribed to dielectric relaxation and space charge polarization effect, whereas an increase of the imaginary part of the complex permittivity can be attributed to the enhanced electrical conductivity of the material [29].

In order to further characterize the absorbing performances of the CNTs with different mass fractions, the reflection loss of CNTs absorbents were calculated based on Eqs. (1) and (2). Fig. 3 shows variation of the reflection loss versus frequency of CNTs absorbents with a constant thickness (2.0 mm) in the frequency range of 2–18 GHz. With increasing of CNTs contents, the microwave absorption peak firstly increases then decreases. The reflection loss reaches a minimum of −18.06 dB with 6 wt% CNTs, and the minimum reflection loss decreased to −13.9 dB and −5.4 dB with the increasing contents of the CNTs (8 and 10 wt%), respectively. Additionally, when it is related to same thickness the values of reflection loss shift to lower frequency with increasing contents of CNTs.

3.2. Effect of CNTs concentration on electromagnetic and absorbing properties of CNTs/ZnOw composites

The CNTs/ZnOw complex absorbers were obtained by homogeneously mixing the CNTs and ZnOw with various weight fractions (sample 5#, 6#, 7#, 8# and 9#). The weight content of ZnOw was 15 wt%, and the weight content of CNTs ranged from 0 wt% to 8 wt%. We hope that the combination of ZnOw and CNTs will improve the microwave absorption properties of CNTs/ZnOw composites. Moreover, the effect of CNTs concentrations on electromagnetic and absorbing properties of CNTs/ZnOw composites was also greatly expected.

Fig. 4 shows the real and imaginary parts of permittivity of CNTs/ZnOw composites. It can be seen from Fig. 4(a) that the real part of complex permittivity of ZnOw remains nearly constant (2.7).

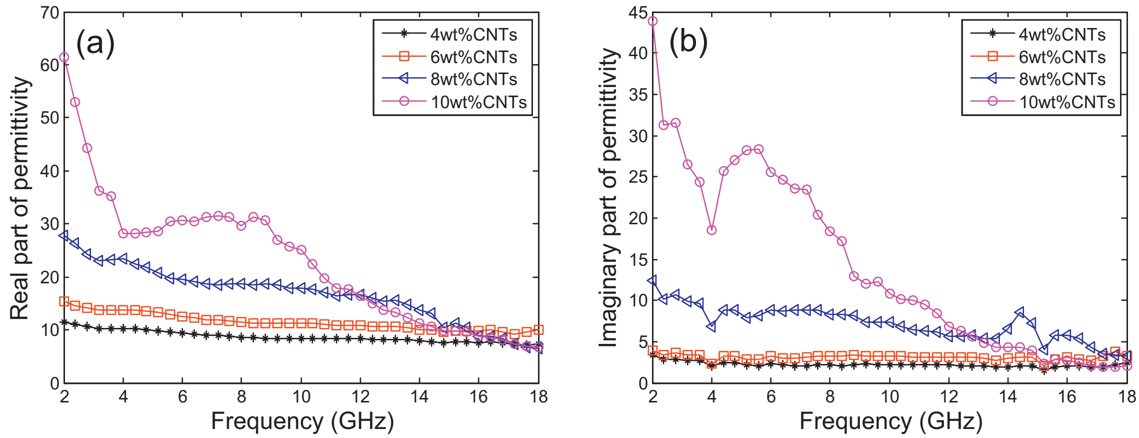

Fig. 2. The real (a) and imaginary (b) parts of CNTs/paraffin.

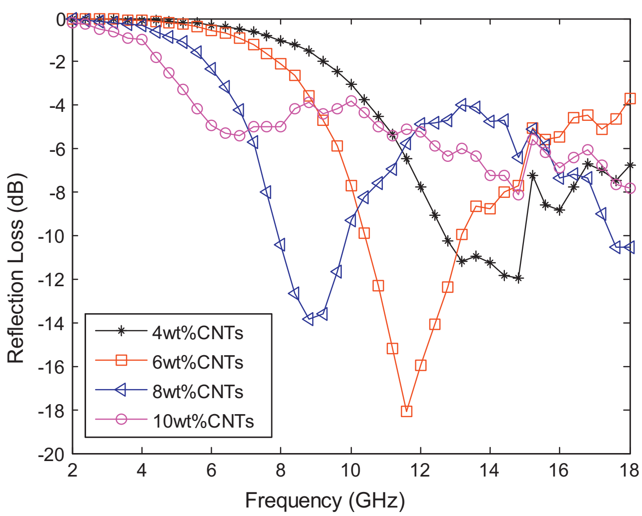

Fig. 3. Reflection loss of CNTs with 2.0 mm thickness.

However, the real part of complex permittivity of CNTs/ZnOw composites shows a great increase with the increasing concentration of CNTs and exhibits dependence with frequency. With increasing frequency, the real part of complex permittivity of CNTs/ZnOw composites with 8 wt% CNTs decline from 55.1 to 24.2. The imaginary parts of permittivity of CNTs/ZnOw composites show a similar tendency compared with the real part of complex permittivity, as shown in Fig. 4(b). It is found that both the real and imaginary parts of complex permittivity of CNTs/ZnOw composites are higher than that of ZnOw at the full test frequencies. The complex permittivity of CNTs/ZnOw is sensitive to the content of CNTs. This result can be explained by the effective medium theory [30], the dielectric constant increase with elevated concentration of CNTs.

A good absorbing material must exhibit the ability of attenuating the microwave propagated within it. According to the theory of electromagnetic wave, the attenuation constant α is given by [30]:

$$\alpha = \frac{2\pi f}{\sqrt{2}c}\sqrt{\mu''\varepsilon'' - \mu'\varepsilon' + \sqrt{(\mu'^2 + \mu''^2)(\varepsilon'^2 + \varepsilon''^2)}} \quad (3)$$

Fig. 5 presents the attenuation constant of CNTs/ZnOw composites. The attenuation constant of CNTs/ZnOw composites is higher than that of ZnOw and increases with the mass fraction of CNTs. The attenuation constant of 15 wt% ZnOw increases from 75.8 Np/m to 603.9 Np/m with increasing frequency. When the CNTs addition increases to 8 wt%, the attenuation constant increases from 353.8 Np/m to 1926.9 Np/m. The addition of CNTs has effectively improved the microwave attenuation ability of CNTs/ZnOw composites. Research by Cao et al. [31] has proved that two important conditions should be satisfied for a good absorbing material. The first is the “matched characteristics impedance”, in which the intrinsic impedance of the material is made equal to the impedance of the free space. Second, the incident electromagnetic wave must enter and get rapidly attenuated through the material layer. So, the microwave characteristic impedance of CNTs/ZnOw composites is presented below.

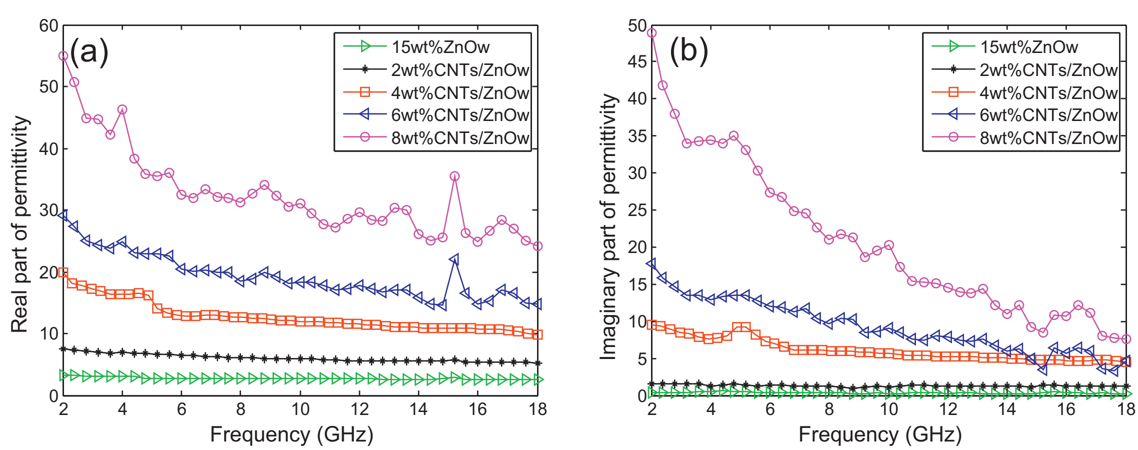

Fig. 4. The real (a) and imaginary (b) parts of CNTs/ZnOw composites with 15 wt% ZnOw and different contents of CNTs (0, 2, 4, 6 and 8 wt%).

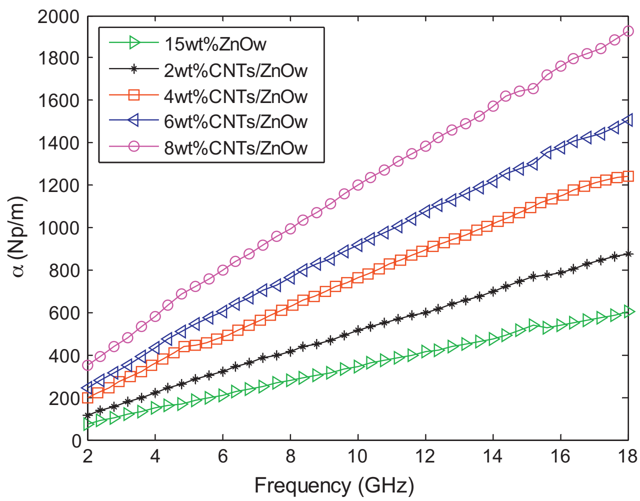

Fig. 5. The attenuation constant of CNTs/ZnOw composites.

The wave impedance η is expressed by the equation [32]:

$$\eta = Z_0\sqrt{\frac{\mu_r}{\varepsilon' - j\varepsilon''}} \quad (4)$$

The microwave impedance of the composites evaluated across the tested frequency band is shown in Fig. 6. With elevated concentration of CNTs, the microwave impedance of CNTs/ZnOw composite declines gradually compared with that of 15 wt%ZnOw. CNTs/ZnOw composite with 8 wt% of CNTs shows the lowest wave impedance value. Addition of CNTs could effectively improve the microwave attenuation ability of CNTs/ZnOw composites. However, the microwave impedance was also reduced much smaller than that of free space wave impedance (about 377 Ohm). The duplex effects will finally influence the absorbing performance of the CNTs/ZnOw composites. So we calculated the reflection loss of CNTs/ZnOw composites at 2.0 mm thickness as shown in Fig. 7. It can be seen that 4 wt% CNTs/ZnOw has the optimum microwave absorption. The minimum reflection loss is −18.91 dB at 10.96 GHz. As a result, we concluded that the concentration of CNTs had great effect on the microwave absorbing performance of CNTs/ZnOw composites. Moreover, we also observed higher values of microwave absorption at a lower loading fraction of CNTs (4 wt% CNTs) compared with pure CNTs (6 wt% CNTs). A better electromagnetic matching could be established due to the combination of CNTs and ZnOw with proper contents. For further optimization of the weight contents of CNTs/ZnOw composites, the effect of ZnOw concentration on electromagnetic and absorbing properties of CNTs/ZnOw composites was discussed in the following section.

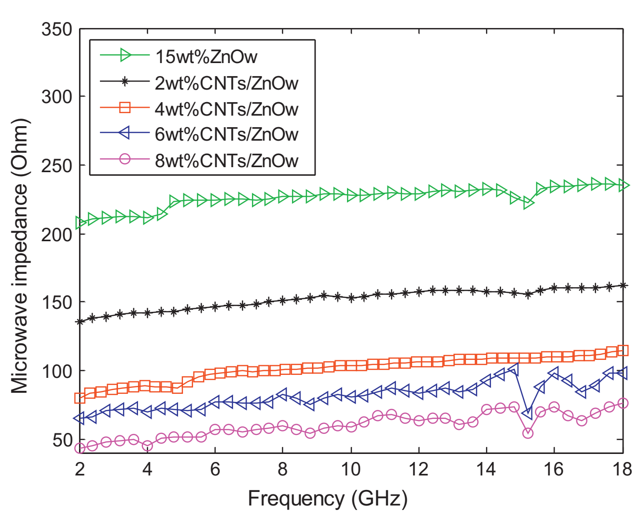

Fig. 6. The microwave impedance of CNTs/ZnOw composites.

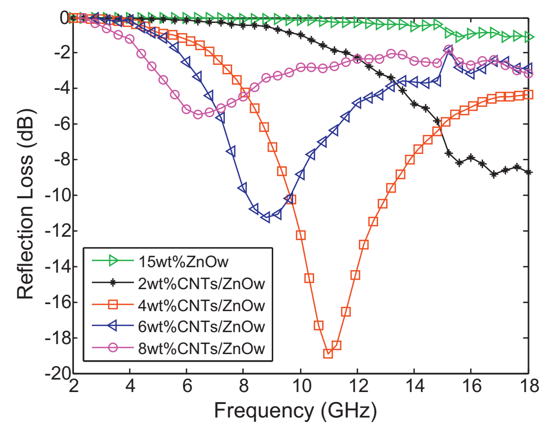

Fig. 7. Reflection loss of CNTs/ZnOw composites with 2.0 mm thickness.

3.3. Effect of ZnOw concentration on electromagnetic and absorbing properties of CNTs/ZnOw composites

The complex permittivity of CNTs/ZnOw composites with 4 wt% CNTs and different content of ZnOw (5, 10, 15, 20 and 30 wt%) were shown in Fig. 8. It can be seen that the complex permittivity of CNTs/ZnOw is sensitive to the concentration of ZnOw. Both the real and imaginary parts of complex permittivity increase with the increasing concentrations of ZnOw and exhibits dependence with frequency. With increasing frequency, the values of complex permittivity decrease slightly. Based on Eqs. (3) and (4) the attenuation constant and microwave impedance were also calculated as shown in Figs. 9 and 10. The attenuation constants of CNTs/ZnOw composites increase with ZnOw weight percentage. The additions of ZnOw have effectively improved the microwave attenuation ability of CNTs/ZnOw composites. When the ZnOw addition increases from 5 wt% to 30 wt%, the average attenuation constant of CNTs/ZnOw composites increases from 589.5 Np/m to 917.7 Np/m. On the contrary, the average wave impedance value of CNTs/ZnOw composites decreases from 130.7 Ohm to 82.8 Ohm.

The reflection loss of CNTs/ZnOw composites with 2.0 mm thickness is shown in Fig. 11. In the low concentration (the concentration of ZnOw < 10 wt%), the reflection loss decreases with the increasing weight fraction of ZnOw. The composite of 4 wt% CNTs mixed with 10 wt% ZnOw has the optimum microwave absorption. The minimum reflection loss is −37.03 dB at 12.24 GHz. The bandwidth corresponding to the reflection loss below −10 dB is more than 4.04 GHz (10.72 GHz–14.76 GHz). The maximum bandwidth under −5 dB is 8.56 GHz (9.44 GHz–18 GHz). The absorption peaks shift to low frequency with elevated concentration of ZnOw, which results from the increasing number of ZnOw, more energy absorption, and increasing equivalent wave paths [33]. Fig. 12 shows variation of the reflection loss versus frequency of the composite with 4 wt% CNTs and 10 wt% ZnOw at different thickness in the 2–18 GHz range. It can be seen that the peak for the reflection loss shifts to a low frequency with increase of the thickness. The CNTs/ZnOw possesses excellent microwave absorbing ability at the thickness of 1.7–3.5 mm. We also observed higher values of reflectivity at a lower loading fraction of CNTs (i.e., −37.03 dB with 4 wt% of CNTs) when the cooperative effect of CNTs and ZnOw occurred compared with pure CNTs, e.g., −18.06 dB with 6 wt% CNTs (Fig. 3). Additionally, our values are comparable to cage-like ZnO/SiO_2 (−10.68 dB, 20 wt% ZnO/SiO_2) [22], tetrapod-shaped

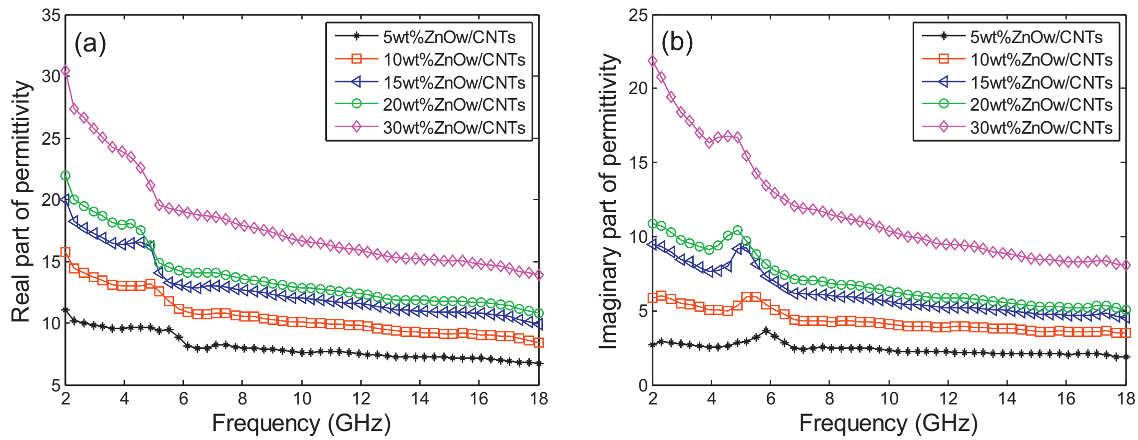

Fig. 8. The real (a) and imaginary (b) parts of ZnOw/CNTs composites with 4 wt% CNTs and different contents of ZnOw (5, 10, 15, 20 and 30 wt%).

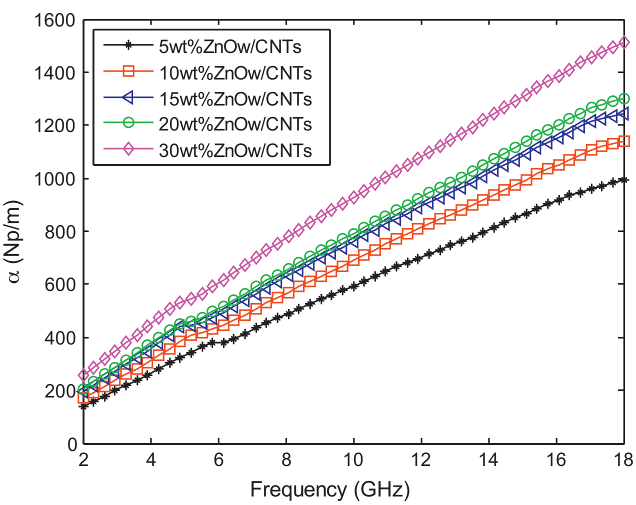

Fig. 9. The attenuation constant of ZnOw/CNTs composites.

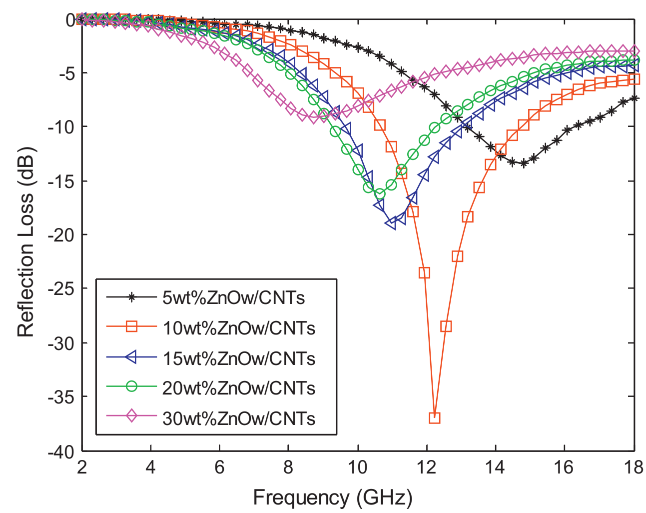

Fig. 11. Reflection loss of ZnOw/CNTs composites with 2.0 mm thickness.

ZnO/CNTs composites (−23.00 dB, 12 wt% CNTs mixed with 8 wt%, T–ZnO) [26] and CNTs filled with Ag nanowires [34], Ni nanowires [35], Er_2O_3 [36], Sm_2O_3 [37] and Fe [38].

The results show that 4 wt% CNTs mixed with 10 wt% ZnOw has the optimum microwave absorption ability with a thickness of 2.0 mm. Compared with pure CNTs or ZnOw, it cannot only greatly reduce the mass fractions of microwave absorbing materials, but also enhance the microwave absorption effect. This enhanced microwave absorption property of CNTs/ZnOw composite is result

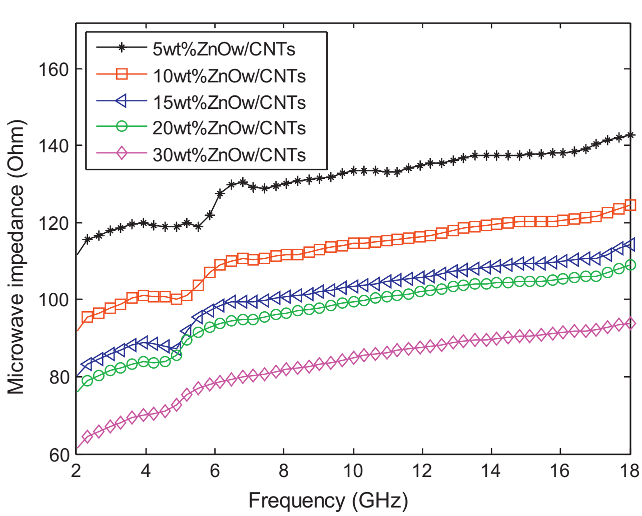

Fig. 10. The microwave impedance of ZnOw/CNTs composites.

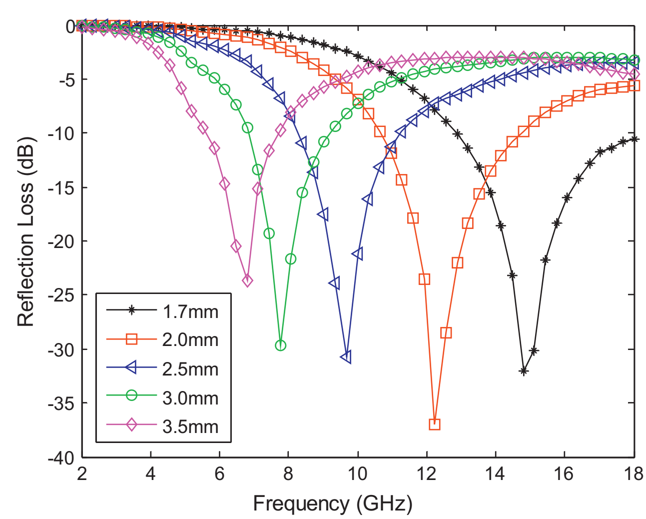

Fig. 12. Reflection loss of 4 wt% CNTs with 10 wt% ZnOw at different thickness in the 2–18 GHz range.

from the cooperative effect of CNTs and ZnOw. Mixing of CNTs and ZnOw is an efficient way to modulate the electromagnetic parameters of the composite and thus improve the microwave absorbing performance. The addition of ZnOw to CNTs might increase interfaces and it should be noticed that the multi-interfaces between ZnOw and CNTs can be benefit for the microwave absorption due to the interactions of microwave radiation with charge multipoles at the interfaces [39].

4. Conclusions

The electromagnetic and microwave absorbing properties depend on the contents of CNTs and ZnOw were investigated. The complex permittivity and attenuation constant increase with increasing contents of CNTs and ZnOw, while the microwave impedance decreases with the elevated contents of CNTs and ZnOw. The cooperative effect of CNTs and ZnOw contribute significantly to the microwave absorption properties of CNTs/ZnOw composites. Our results show that 4 wt% CNTs mixed with 10 wt% ZnOw has the optimum microwave absorption ability with a thickness of 2.0 mm. The minimum reflection loss is −37.03 dB and the bandwidth corresponding to the reflection loss below −10 dB is more than 4.04 GHz. The results indicate that the prepared CNTs/ZnOw composite has great promise in the potential application to lightweight and strong absorption microwave absorber.

Acknowledgments

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