

Double-layer Electromagnetic Wave Absorber Based on Carbon Nanotubes Doped with $\text{La}(\text{NO}_3)_3$ and Fe_3O_4 Nanoparticles

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Double-layer structure absorbing materials based on the impedance matching principle and transmission line theory can effectively improve the electromagnetic wave absorbing properties. In this paper, the electromagnetic wave absorbing properties of double-layer absorbers (2 mm thickness), where multiwall carbon nanotube (MWCNT)- $\text{La}(\text{NO}_3)_3$ /polyvinyl chloride (PVC) and MWCNT- Fe_3O_4 /PVC composites had been taken turns as the absorption layer and matching layer, were investigated in 2–18 GHz range. The absorbing properties of single- and double-layer structure and different each-layer thickness with two types of combinations were compared. The results showed that the design of double-layer structure for composites could effectively broaden the absorption frequency area, and increase the absorption intensity. When MWCNT- $\text{La}(\text{NO}_3)_3$ /PVC composite were used as absorption layers with 0.6 mm thickness, the absorption bandwidth (< -15 dB or > 97 %) of double-layer composite was the widest, reaching a maximum of about 3.36 GHz, and the absorption peak value was also the lowest about -46.02 dB at 16.24 GHz.

Keywords: multiwall carbon nanotubes, $\text{La}(\text{NO}_3)_3$, Fe_3O_4 , electromagnetic wave absorbing properties.

1. INTRODUCTION

With mounting electromagnetic pollution, electromagnetic wave absorbing materials have been paid much attention in recent years [1–6]. Many absorbing composites containing carbon fibers [7], carbon nanotubes (CNTs) [8, 9], graphene [10, 11], carbon spheres [12], ferrite [13, 14], carbonyl iron [15], etc. have been most extensively studied. In addition, previous research has found that the absorbing bandwidths of these materials are relatively narrow and weak. Therefore, multilayer absorbing composites with gradient impedance, in which relatively more electromagnetic waves can be absorbed by impedance matching, are fabricated in order to broaden the absorbing bandwidths, enhance the absorbing intensity and improve the absorbing performance. Electromagnetic wave absorbing composites with a suitable absorption performance are usually designed with an appropriate matching layer. It is possible to achieve suitable impedance matching allowing incidents of electromagnetic waves to reach the absorbing layer with the free space [16–18]. Danlée et al. [19] presented that a novel multilayer arrangement of polymer nanocomposites composed of alternating films of dielectric polymer and conducting layers, which consisted of either polycarbonate nanocomposite films with CNTs or a very thin CNTs coating deposited on insulating polymer from a CNT waterborne ink, was able to very effectively absorb electromagnetic wave over a broad frequency range or selectively reflect desired wavelengths. Das et al. [20] found that the double-layer CoZn-ferrite/ TiO_2 (-24.3 dB), where ferrite powders were substituted using Zinc $\text{Me}_{0.5}\text{Zn}_{0.5}\text{Fe}_2\text{O}_4$ (Me = Co, Mn and Ni) had been prepared

by the sol-gel auto-combustion, possessed lower reflection loss than the single-layer NiZn-ferrite (-11.2 dB) of the same total thickness at 12.02 GHz. Ni et al. [21] presented that the magnetic, dielectric and reflection loss value of double-layer nanocomposites consisted of barium titanate (BTO)/CNT 30 wt.% (r) and BTO 30 wt.% (matching layer) thickness were improved compared to single-layer BTO/CNT 30 wt.% nanocomposites. The bandwidth (< -10 dB) covered a wide frequency area from 12.1 to 13.8 GHz when the absorption and matching layer thickness were respectively 1.0 and 0.3 mm, and the minimum reflection loss achieved ~ -63.7 dB (over 99.9999 % absorption) at 13.7 GHz.

In a certain total thickness, each-layer thickness and layer-permutation have exerted great influence on the absorbing properties of double-layer composites. Therefore, we used MWCNTs doped with Fe_3O_4 and $\text{La}(\text{NO}_3)_3$ as an absorbent and insulating resin (PVC) as a matrix to fabricate double-layer composites for high-performance absorbing microwave. The reflection loss of electromagnetic absorbers with various layer permutation and thickness was theoretically estimated by the calculation according to the single- or double-layer absorbers model. Electromagnetic parameters ($\varepsilon_r = \varepsilon' - j\varepsilon''$, $\mu_r = \mu' - j\mu''$) of as-prepared composites were measured by space method in the required frequency range.

2. EXPERIMENTAL

2.1. Materials

MWCNTs were treated with a concentrated acid mixture of H_2SO_4 - HNO_3 (3 : 1 v/v) for 4 h for increasing their activity and purity, and were then ball-milled. MWCNTs doped with Fe_3O_4 or $\text{La}(\text{NO}_3)_3$ with optimal

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microwave absorbing properties were fabricated according to our previous reports: (1) The acid-treated MWCNTs were agitated ultrasonically with 6 wt.% of $\text{La}(\text{NO}_3)_3$ for 1 h, dried out and ground. Then the black MWCNT- $\text{La}(\text{NO}_3)_3$ powder was prepared by a simple method [22]; (2) The acid-treated MWCNTs (100 mg) were ultrasonically dispersed in a mixture solution (250 mL) of $\text{FeCl}_3/\text{FeSO}_4$ (molar ratio = 2:1, FeSO_4 concentration = 0.02 mol/L) for 0.5 h. This solution was heated to 50 °C, while this temperature was maintained for 0.5 h with stirring under N_2 protection, followed by a further heating at 65 °C for 1 h at pH > 12 (adjusted with 6 mol/L NaOH). The solution was added slowly with sodium dodecyl sulfate (SDS, 0.25 g) at 85 °C and cooled until room temperature was achieved. Black precipitates from the solution had been collected by filtration, washed by deionized water to neutral, dried out and milled. Then soft magnetic MWCNT- Fe_3O_4 hybrid materials were obtained via magnetic separation [23].

2.2. Characterization

The samples were prepared by 8 mass % MWCNT- $\text{La}(\text{NO}_3)_3$ or MWCNT- Fe_3O_4 hybrid materials that were homogeneously dispersed in PVC matrix (MWCNT- $\text{La}(\text{NO}_3)_3/\text{PVC}$ or MWCNT- $\text{Fe}_3\text{O}_4/\text{PVC}$), and then made into a rectangular waveguide that has a length of 22.86 mm, a width of 10.16 mm for electromagnetic measurements. The sample complex permittivity and permeability were carried out using a network analyzer (Agilent technologies E8362B: 10 MHz–20 GHz).

2.3. Calculation of reflection loss

According to the transmission theory [24], the formulas of calculation for double-layer absorber were given to determine the reflection loss as follows [25, 26]:

$$R(\text{dB}) = 20 \log_{10} \left| \frac{Z_{in} - Z_0}{Z_{in} + Z_0} \right|; \quad (1)$$

$$Z_{in} = Z_2 \frac{Z_{in1} + Z_2 \tanh(j2\pi f d_2) \sqrt{\mu_{r2} \epsilon_{r2}}}{Z_2 + Z_{in1} \tanh(j2\pi f d_2) \sqrt{\mu_{r2} \epsilon_{r2}}}; \quad (2)$$

$$Z_{in1} = Z_1 \tanh(j2\pi f d_1) \sqrt{\mu_{r1} \epsilon_{r1}}, \quad (3)$$

where Z_{in} is the input impedance at the material interface and free space, Z_{in1} is the interface impedance between absorbing layer and matching layer, Z_1 , Z_2 , Z_0 are the characteristic impedance of absorbing layer, matching layer and a vacuum, respectively, ϵ_r and μ_r are the relative complex permittivity and permeability, respectively, f is the electromagnetic wave frequency in vacuum, d is the layer thickness, and c is the light velocity in vacuum.

3. RESULTS AND DISCUSSION

Fig. 1 shows the complex permittivity ($\epsilon_r = \epsilon' - j\epsilon''$) and permeability ($\mu_r = \mu' - j\mu''$) of MWCNT/PVC, MWCNT- $\text{La}(\text{NO}_3)_3/\text{PVC}$ and MWCNT- $\text{Fe}_3\text{O}_4/\text{PVC}$ composites in 8.2–12.4 GHz. As seen in Fig. 1 a and b, the real part and imaginary part of complex permittivity of MWCNT/PVC

composite have decreased with an increase of frequency, the real part is highest, and the imaginary part is lowest. This can be due to the interface and dipolar polarization and suggests that the dielectric loss of composites increases by adding $\text{La}(\text{NO}_3)_3$ and Fe_3O_4 .

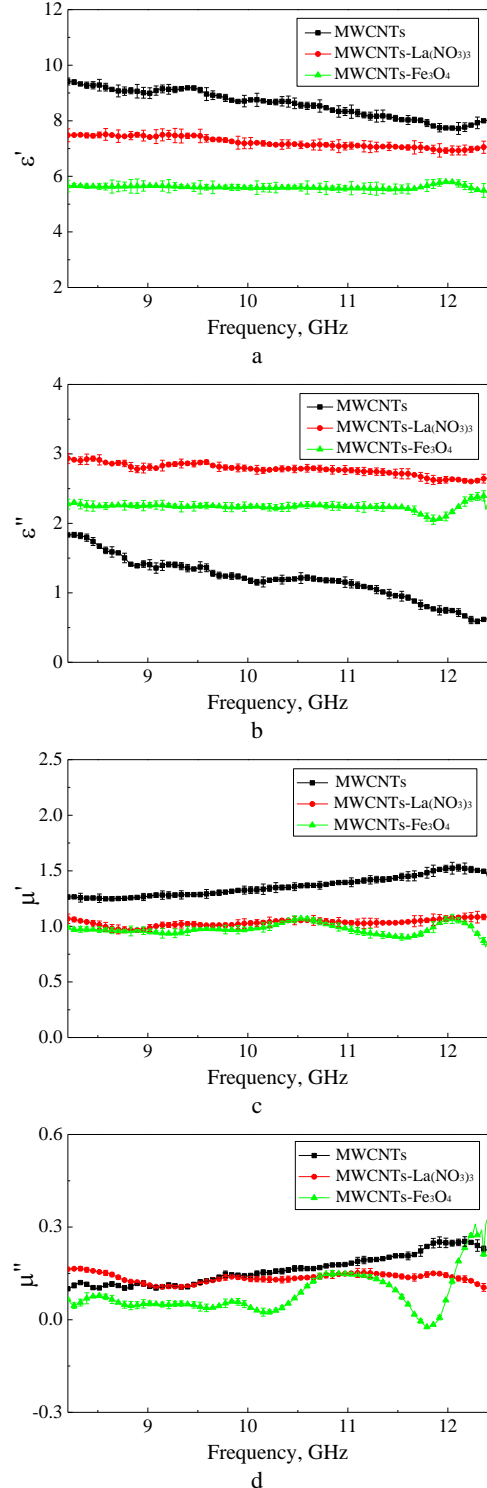


Fig. 1. Complex permittivity (ϵ' , ϵ'') (a, b) and permeability (μ' , μ'') (c, d) spectra for MWCNT, MWCNT- $\text{La}(\text{NO}_3)_3$ and MWCNT- Fe_3O_4 vs frequency

While a small fluctuation is observed in the real part of complex permittivity of MWCNT- $\text{Fe}_3\text{O}_4/\text{PVC}$ composite, which can be ascribed to the dielectric relaxation of the sample interface under the action of the

external electromagnetic wave. A vibration is observed at the corresponding frequency region of the imaginary part. This phenomenon is obviously characteristic for nonlinear dielectric resonance [27]. From Fig. 1 c and d, it can be found that the real part of complex permeability of MWCNT-La(NO₃)₃/PVC composite is lower than that of MWCNT/PVC composite, and the imaginary parts did not appear to be much different. This is mainly because La³⁺ ions could be complex with C=O, -COOH and -OH located on MWCNT surface to enhance magnetic anisotropy and coercivity. The real part and imaginary part of complex permeability of MWCNT-Fe₃O₄/PVC composite are the lowest. This may be due to MWCNTs not only have a unique chiral structure but also a lot of dangling bonds. Electrons can quickly exchange between MWCNTs and Fe₃O₄ nanoparticles to strengthen Fe₃O₄ conductivity and eddy current effect in 8.2–12.4 GHz. Eddy current creates the magnetic field that opposes the original magnetic field, which leads to a decrease in the complex permeability.

Multiple resonance peaks are detected in the imaginary-part high-frequency region. They are mainly caused by natural resonance, domain wall resonance and the eddy current loss arising from intrinsic damping [28].

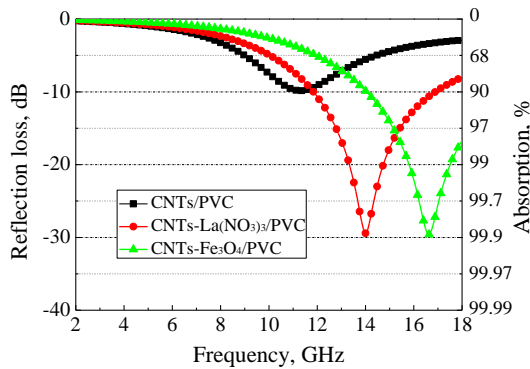


Fig. 2. Reflection losses of single-layer absorbers for MWCNTs, MWCNTs-La(NO₃)₃ and MWCNTs-Fe₃O₄ vs frequency

Fig. 2 indicates the reflection losses of single-layer absorbers for MWCNTs, MWCNTs-La(NO₃)₃ and MWCNTs-Fe₃O₄ with 2 mm thicknesses in 2–18 GHz. Among all of the samples, MWCNT/PVC composite has the highest minimum reflection loss (−9.84 dB, 11.28 GHz) and no absorbing bandwidth (<−15 dB, >97%), while MWCNT-Fe₃O₄/PVC composite, in contrast, has the lowest minimum reflection loss (−29.60 dB, 16.64 GHz) and the widest absorbing bandwidth at about 2.88 GHz (<−15 dB, >97%) from 15.12 to 18 GHz. This is mainly due to the magnetic loss and frequency dispersion on permeability of Fe₃O₄ nanoparticles caused by the natural resonance in the high frequency region. In addition, exchange energy caused by exchange effect can be significantly enhanced owing to the nanoscaled size and high surface anisotropy of Fe₃O₄ nanoparticles. Accordingly, exchange resonance may also contribute to the magnetic loss [29]. For MWCNT-La(NO₃)₃/PVC composite, the dielectric loss is enhanced with adding La(NO₃)₃, so the absorption properties are also strongly improved. As the absorption mechanism of La(NO₃)₃ is not the same as that of magnetic Fe₃O₄, the absorption band is also different. And the electromagnetic

parameter changes lead to the interference condition change, the absorption peak shift to the low frequency region.

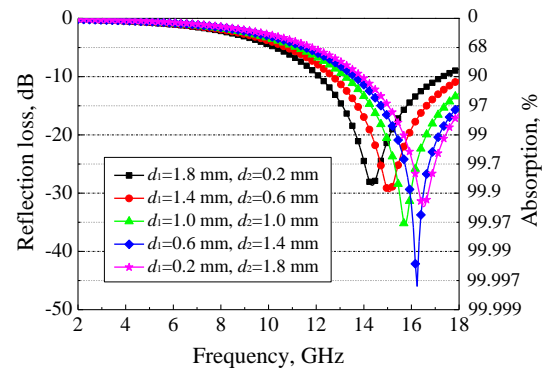


Fig. 3. Reflection losses of double-layer absorbers consisting of absorption layer (d_1) filled with MWCNTs-La(NO₃)₃ and (d_2) matching layer filled with MWCNTs-Fe₃O₄ vs frequency

The reflection losses of double-layer absorbers with MWCNT-La(NO₃)₃/PVC composites as absorption layer and MWCNT-Fe₃O₄/PVC composite as matching layer versus frequency in 2–18 GHz are shown in Fig. 3, and the reflection losses of double-layer absorbers with MWCNT-Fe₃O₄/PVC composites as absorption layer and MWCNT-La(NO₃)₃/PVC composite as matching layer versus frequency in 2–18 GHz are shown in Fig. 4. According to Maxwell equations, electromagnetic wave propagation characteristics in materials are determined by the complex permittivity and permeability. Using the double-layer structure of the composite, electromagnetic wave in space enters into the absorption layer as much as possible by the double-layer matching function and is absorbed by the absorption layer. Electromagnetic wave absorption peak minimum shifts to a higher frequency area with the absorbing layer thickness decreases. When the thickness of absorption layer and matching layer are 0.6 mm and 1.4 mm, respectively, the peak value achieves a minimum of about −46.02 dB at 16.24 GHz, which means that two layers matching is the best. And absorption bandwidth is about 3.36 GHz (<−15 dB or >97%) ranging from 14.64 GHz to 18 GHz. Compared to the single-layer composite as shown in Fig. 2, the double-layer composites are more efficiency for electromagnetic wave absorption in 2–18 GHz. Whereas, when MWCNT-Fe₃O₄/PVC and MWCNT-La(NO₃)₃/PVC composites are respectively used as absorption and matching layer (Fig. 4), and the peak value achieves a minimum about −40.71 dB at 14.32 GHz, with the narrow bandwidth about 2.72 GHz (<−15 dB or >97%). The effective absorption band lies in the relatively low frequency region, and the electromagnetic wave absorption peak minimum shifts to the lower frequency region with the absorbing layer thickness decreases. Two different variation trends are attributed to the different attenuation properties of the dielectric and magnetic materials. Compared to MWCNT-Fe₃O₄/PVC and MWCNT-La(NO₃)₃/PVC composites. This implies that MWCNT-Fe₃O₄/PVC composite would be more suitable for impedance matching with free space, resulting in the overall impedance of double-layer absorber relatively closer to the ideal impedance matching ($Z_{in} = Z_0$)

[30], and when MWCNT-Fe₃O₄/PVC composite is used as a matching layer, more electromagnetic waves can enter the absorption layer, which causes the absorption peak value and bandwidth increases.

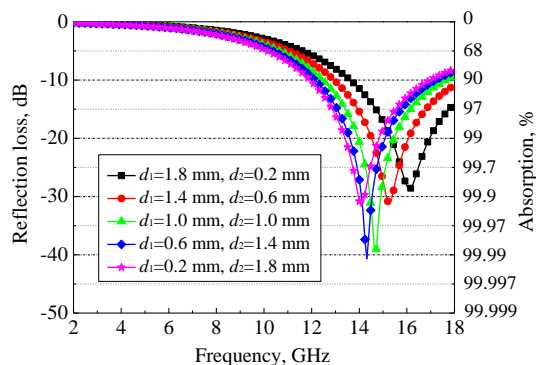


Fig. 4. Reflection losses of double-layer absorbers consisting of absorption layer (d_1) filled with MWCNTs-Fe₃O₄ and matching layer (d_2) filled with MWCNTs-La(NO₃)₃ vs frequency

4. CONCLUSIONS

In summary, the double-layer structure design could have superior absorption property due to the impedance matching of composites. In this investigation, the electromagnetic wave double-layer absorption composites, where MWCNT-La(NO₃)₃/PVC and MWCNT-Fe₃O₄/PVC composite had been taken turns as the absorption layer and matching layer, showed better absorption properties than those of the corresponding single-layer composites when the total thickness was 2 mm. The absorption properties were closely related to the absorber type and thickness of each layer. When MWCNT-La(NO₃)₃/PVC composite was used as absorption layer, strong electromagnetic wave absorption could be obtained in a wide frequency area. Additionally, based on the absorption bandwidth, when the absorption layer thickness was 0.6 mm, the absorption bandwidth (< -15 dB or > 97 %) reached a maximum of about 3.36 GHz (i.e., the reflection loss was the widest). These double-layer absorption composites are promising advanced electromagnetic wave materials.

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