Microwave absorption properties of Ag naowires/carbon black composites*

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The composite that can absorb the high-performance electromagnetic (EM) wave is constructed into a sandwiched structure composed of carbon black (CB)/ethylene-vinyl acetate (EVA) and Ag naowires (AgNWs). The AgNWs sandwiched between two CB/EVA layers are used to improve the absorption properties of composite. The effects of EVA-to-CB weight ratio, concentration and diameter of AgNWs with a thickness of 0.4 mm on microwave absorption are investigated. The results indicate that for an EVA-to-CB weight ratio of 1:3, AgNW concentration of 1.0 mg/100 mL, and average diameter of 56 nm, the reflection loss (*RL*) of the composite is below −10 dB in a frequency range of 9.3 Ghz–18.0 GHz, with the minimum values of −40.0 dB and −25.6 dB at 13.5 GHz and 15.3 GHz, respectively. A finite element method (FEM) is used for calculating the *RL* of the composite. The calculated results are in agreement with the experimental data.

Keywords: microwave absorption, sliver nanowires, finite element method, polymer-based composites

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1. Introduction

In order to solve the problems of noise signals and undesirable electromagnetic (EM) wave energy created by wireless communications or electronic devices[1] EM wave absorbing materials are required to be studied. Due to the large electric or magnetic loss, the ferrites and metal-based composites regarded as one of the candidates for EM wave absorbers have particularly aroused the interest of researchers.[2–8] However, the ferrites and metal-based composite absorbing materials have their own disadvantages such as relatively large thickness, high manufacturing cost, high electrical conductivity, and narrow absorption bandwidth, which restrict their widespread applications. It is necessary to seek for thin and lightweight materials with strong microwave absorption properties. More recently, the various types of nanowires were used to fabricate EM wave absorber to meet this requirement. For instance, Liu *et al.*[9] have reported on the microwave properties of ZnO nanowire-polyester composite with a thickness of 1 mm, the results showed that reflection loss (*RL*) of 12.28 dB for 7% nanowire composites can be obtained. Chen *et al.*[10] have fabricated Fe nanowire (70 nm–200 nm in diameter and 20 μm–50 μm in length)/epoxy resin composites for EM wave absorption application, and the resin compacts of 29 vol% Fe nanowires with thickness in a range of 1.3 mm–4.0 mm provided good EM wave absorbing characteristics in a frequency range of 5.6 GHz–18 GHz. Thus, the nanowires are very promising for the fabrication of EM wave absorbers with much thinner thickness in micrometer scale.

Currently, Ag nanowires (AgNWs), which possess unique properties in terms of electronics, thermology, plasmonics, and chemistry,[11–14] have been a notable material in building marcoscale flexible composites because AgNWs possess high conductivity, high aspect ratio, good flexibility, and mature preparation technology. Various flexible conductive materials and devices have been fabricated on the basis of AgNWs.[15–19] On the other hand, it was found that the AgNWs can be used as an absorber with good absorbing properties.[20] In this paper, the AgNWs layer sandwiched between two carbon black (CB)/ethylene–vinyl acetate (EVA) layers is used to improve the absorption properties of composite. The effects of EVA-to-CB weight ratio, concentration and diameter of AgNW layer on microwave absorption are investigated, with AgNW layer being 0.4-mm thick. Free space method with an NRL Arch reflectivity test setup and finite element method (FEM) are used for measuring and calculating the *RL* of the composite, respectively. The calculation results are in agreement with experimental data. Moreover, the absorbing mechanism of the composite is also investigated.

2. Experiments

2.1. Chemicals and materials

Anhydrous ethylene glycol (EG, 99.8%), poly (vinylpyrolidone) (PVP, $M_w \approx 58000$), ethyl alcohol, NaCl, and Tributyl phosphate (TBP, 98%) were all purchased from Hunan Huifeng Reagent Co. Ltd, China. CB powders were acquired from Tianjin Dengke Reagent Co. Ltd, China. EVA was obtained from AkzoNobel. Specialty Chemicals (Shanghai) Co.

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Ltd, China. Silver nitrate ($AgNO_3$, 99+%) was purchased from Sinopharm Chemicals Reagent Co. Ltd, China. $CuCl_2$ was obtained from Tianjin Guangfu Science Co. Ltd, China. All chemicals were of analytical grade and used without further purification.

2.2. Preparation of AgNWs

The used AgNWs were synthesized by polyol technique. In a typical synthesis of AgNWs, 100 mL of EG solution was heated at $\sim$ 160 °C in a three-necked flask (equipped with a condenser, thermocontroller, and magnetic stirring bar). After 1 h, 2 mol/L of $CuCl_2{\cdot}2H_2O$ solution was added into EG solution. After $\sim$ 15 min, 2.5 ml of $AgNO_3$ solution (0.5 mol/L) was added into the hot solution, 6 min later, the 30 ml of PVP solution (0.4 mol/L) was added into hot reaction solution by using a constant flow pump. The reaction mixture was heated at 160 °C for 5 min$\sim$10 min until the supernatant became grey. The growth of nanowires was monitored by sampling small portion of reaction at various reaction times by using an optical microscope (in the dark-field mode). Vigorous stirring was maintained throughout the process. The product could be purified by centrifugation. In our study, the reaction mixture was diluted with ethanol and centrifuged at 4000 rpm for 20 min. The supernatant containing silver nanoparticles could be easily removed by using a pipet. This centrifugation procedure could be repeated several times until the supernatant became colorless. The final products were dispersed in ethanol and stored at room temperature for further use. Figure 1(a) shows the scanning electron microscopy (SEM) images of AgNWs with good size uniformity. Figures 1(b)–1(d) represent different diameters of the single AgNWs used in experiment, in which the average diameters are 32 nm, 48 nm, and 56 nm, respectively.

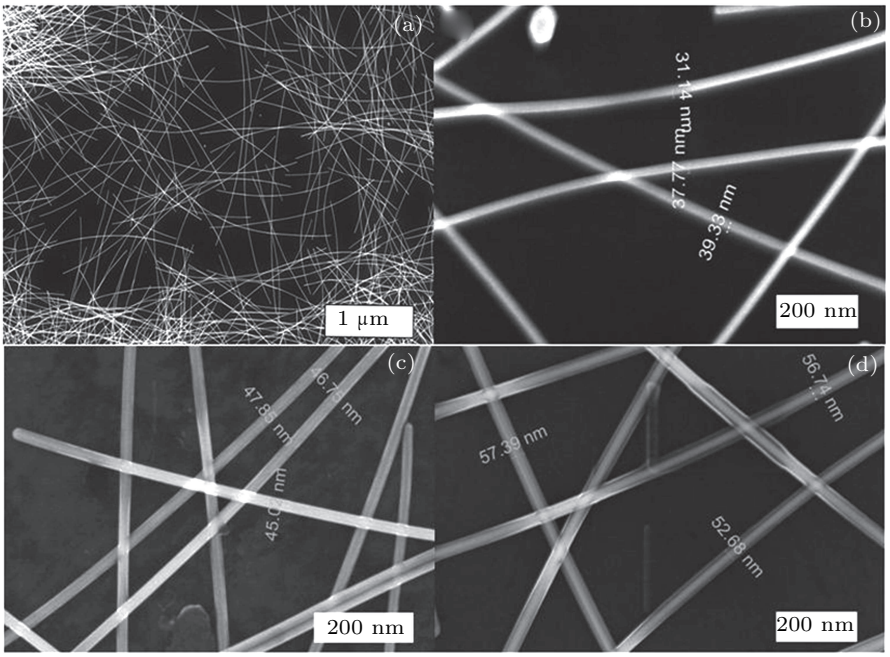


Fig. 1. SEM images of AgNWs with (a) good size uniformity and average diameters of (b) 32 nm, (c) 48 nm, and (d) 56 nm.

2.3. Preparation of the nanocomposite

In order to ensure the accuracy of the measurement of the *RL*, a sandwiched structure has been proposed to ensure the as-fabricated nanocomposites to be positioned in the testing platform and to keep flat when testing.[21–23] The EVA and CB powders,whose EVA-to-CB weight ratio is 1:3, were dissolved in the TBP solvent, the content of mixture powders is 75%. After being stirred for 4 h, the homogeneous suspension was smeared onto an aluminum plate layer by layer by using the wire-wound rod coating method, and then the aluminum plate was immediately placed in the vacuum oven for 2 h at 80 °C to form the matrix films (see Fig. S(1a) in Appendix A). Then the CB/EVA matrix films were cooled at room temperature for further use. Then the as-prepared AgNWs were dissolved in the ethanol and stirred for 20 min to obtain the homogeneous AgNWs solution. Wet AgNWs films (see Fig. S(1b) in Appendix A) with different diameters were prepared on the matrix films and thenplaced in an oven at 60 °C to dry for 5 min before being transferred to a 200-°C oven for 8 min to remove the TBP. Finally, the same matrix films were placed onto the AgNWs film to form a sandwiched structure, and the specimen was placed in the oven for 40 min at 80 °C. The EM absorbing sandwiched structure composites were achieved (see Fig. S2 in Appendix A). The thickness of the specimen was measured to be 0.4 mm by using the vernier caliper. The specimen dimensions were 180 mm $\times$ 180 mm $\times$ 0.4 mm, which was

adhered to a 2-mm thick aluminum substrate. The CB/EVA composite film (0.4-mm thickness) without AgNWs was also prepared for reference.

2.4. Characterizations

The morphologies of the specimens were carried out with field-emission scanning electron microscopy (FESEM, Nova NanoSEM230) operated at an accelerating voltage of 10 kV after sputter-coating specimens with platinum. The *RL* in a 8 GHz–18 GHz frequency range was measured by a free-space method with an NRL Arch reflectivity test setup. The standard size of the sample specimens for the holder was 180 mm×180 mm. The scattering parameters of the CB/EVA matrix films (S_{11}, S_{21}) were tested by using a vector network analyzer (Model. AV3629) with a coaxial line measurement mode over 2 GHz–18 GHz. Standard calibration was initially performed on the test setup in order to remove errors. The real and the imaginary parts of the complex permittivity were determined from the complex scattering parameters by using the Nicolson–Ross model.[24] The samples were washed by using distilled water several times, and dried at 60 °C for 24 h before characterization.

2.5. Numerical calculation

The FEM solution was employed in the numerical study. The simulation model was constructed including all the components of practical structures. The composite structure was embedded within air space, which was cut off in three-dimensional (3D) space by the perfectly match layers (PML)[25–27] to emulate the infinite space. The thickness of the air region between the PML and the composite structure was typically larger than a quarter wavelength at the lowest frequency of interest.

Randomly distributed AgNWs (as shown in Fig. 2(a)) were generated by using the Monte Carlo (MC) method. The primary step generated the location and orientation of the randomly distributed nanowires within a two-dimensional central plane of the sheet. Thereafter, the examination on the overlapping of the nanowires was performed. If no overlapping was found, the nanowires' location and orientation were maintained. If overlapping occurred, a new location and orientation would be generated. This procedure was repeated until the desired number of nanowires was reached. Figure 2(b) shows the schematic diagram of the numerical model. A plane wave with the electric field E parallel to the layer and wave vector k perpendicular to the layer surface illuminates the structure at normal incidence.

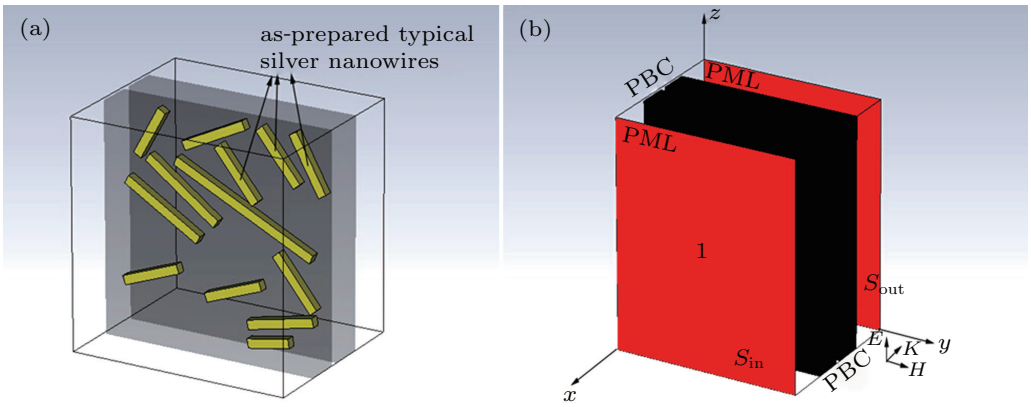


Fig. 2. (color online) Schematic illustrations of (a) the distribution of the AgNWs and (b) the numerical model of planar composite with conductive AgNWs.

3. Results and discussion

Figure 3 shows the variations of the real part ε', the imaginary part ε'' and the loss tangent of permittivity for CB/EVA composites matrix films with frequency at different EVA-to-CB weight ratios. Due to the non-magnetic nature of CB/EVA composite, it is sufficient and appropriate to calculate the complex permittivity. As shown in Fig. 3, it can be seen that the CB/EVA composite matrix films are a kind of strong dielectric lossy material with large ε, of which the real part and the imaginary part both increase with increasing the content of CB. It is noted that the real part and the imaginary part are in the ranges of 197–211 and 200–209, respectively. Especially, as shown in Fig. 3(d), for composite matrix film with the EVA-to-CB weight ratio of 1:4, unreliable ε' and ε'' values at some frequencies are omitted or displayed by dash curves, which is similar to the results reported by Liu *et al.*[28] The used Nicolson–Ross model would produce unreliable results as the sample has a high conductivity. The composite matrix films can attenuate the EM wave by resistance loss of conductivity CB, and the absorbing mechanism of CB can be concluded as follows. (i) Conductive powder, considered as dipole in the process of damped vibration, can attenuate the EM wave. (ii) Multi-reflection can attenuate the EM wave. (iii) The leakage conductance effect between conductive particles can attenuate

the EM wave. At low CB concentrations, for conductive particles in large space, the tunnel effect functions play a major role, which leads to little EM wave attenuation. Conversely, at high CB levels, the interaction and wave loss are enhanced when the electromagnetic wave transmits through the material for the formation of an electric net in CB particles with the narrowing of the space among neighbouring particles. With a combination of the three attenuation functions as given above, the EM wave can be attenuated effectively.

Reflectivity is one of the most important indicators for characterizing the microwave absorbing performances of the composite. It can be written as

$$RL = 10\lg\frac{P_r}{P_i} = 20\lg\left|\frac{E_r}{E_i}\right|, \tag{1}$$

where P_i, E_i and P_r, E_r are the power and electric field of the incident wave and specular reflection, respectively. The *RL* value of −10 dB corresponds to the power reduction by 90 % of the incident wave, and a smaller *RL* value indicates better microwave absorption performance.

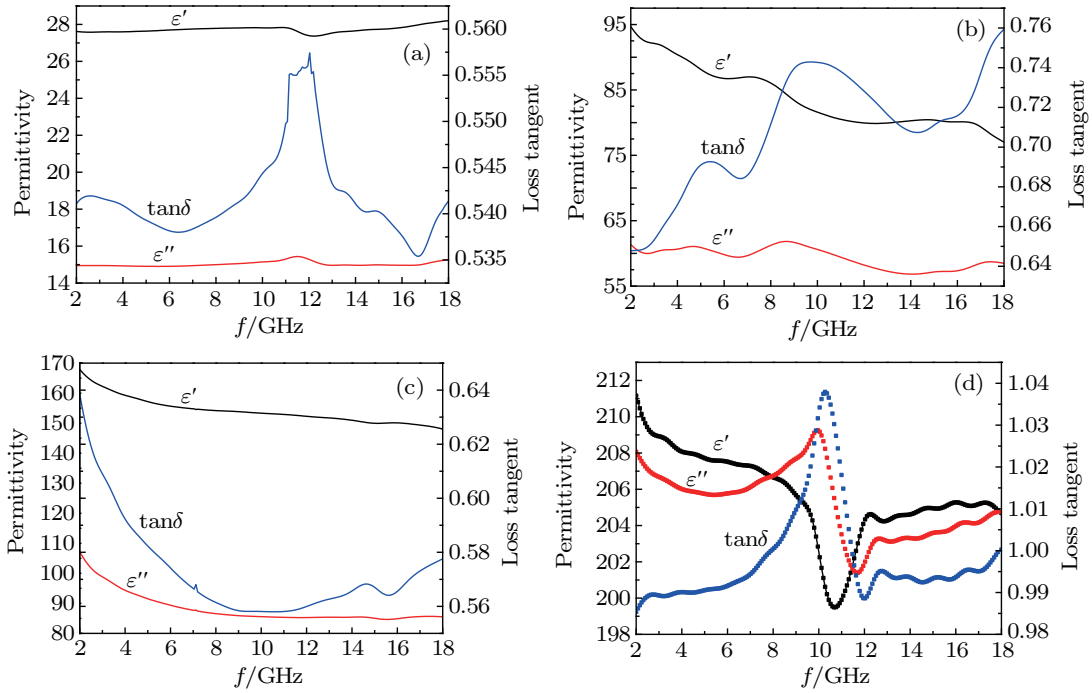


Fig. 3. (color online) Plots of real part ε' and imaginary part ε'' of permittivity for CB/EVA composites matrix versus frequency at different EVA-to-CB weight ratios of (a) 1:1, (b) 1:2, (c) 1:3, (d) 1:4.

Figure 4(a) shows the frequency dependence of the reflection loss of AgNWs/CB/EVA composite absorbers. It can be seen that the pure EVA matrix has almost no absorption, which means that it is transparent to EM wave. For CB/EVA composite matrix film, the *RL* is below −10 dB (90% absorption) in a frequency range of 14.0 GHz–15.7 GHz, with a bandwidth of 1.7 GHz, and the minimum value is −12.8 dB at 14.8 GHz. As shown in Fig. 4(a), it is clear that the microwave absorption is enhanced by embedding AgNWs layer into CB/EVA composites matrix film. The prepared composite samples are named 32c, 48c, and 56c according to the average diameters of AgNWs. For sample 32c, the *RL* value of the corresponding composite is below −10 dB in a frequency range of 11.3 GHz–16.5 GHz, with a bandwidth of 5.2 GHz, and the minimum value is −18.6 dB at 14.0 GHz. For sample 48c, the *RL* value of the corresponding composite is below −10 dB ina fequency range of 11.5 GHz–18.0 GHz, with a bandwidth of 6.5 GHz, and the minimum value is −25.6 dB at 15.9 GHz. For sample 56c, the *RL* value of the corresponding composite is below −10 dB in a range of 9.3 GHz–18.0 GHz, with a bandwidth of 8.7 GHz, and the minimum values are −40.0 dB and −25.6 dB at 13.5 GHz and 15.3 GHz, respectively. It can be observed that the absorption peak frequency of the AgNWs/CB/EVA composite with a thickness of 0.4 mm can be tuned by filling the AgNWs with different diameters into the CB/EVA composites matrix. In these tested samples, 1:3 and 1.0 mg/100 mL are selected as the EVA-to-CB weight ratio and concentration of AgNWs, respectively.

Figure 4(b) shows the *RL* value of the AgNWs/CB/EVA composite with a thickness of 0.4 mm calculated by the FEM. As shown in Figs. 4(a) and 4(b), the calculation results are in agreement with the experimental results. For samples 32c and 48c, the minimum *RL* values calculated by the FEM are −23.1 dB and −35.6 dB, which are lower than the experimental values of −18.6 dB and −25.6 dB, respectively. For sample 56c, the minimum *RL* values predicted by the FEM are −53.1 dB and −36.6 dB, which are lower than the experimental values of −40.0 dB and −25.6 dB, respectively. The

reasons for the differences between the calculated and experimental results are that the finite AgNWs/CB/EVA specimen is supposed to be infinite in the FEM, and the calculated parameters may be different from the experimental procedure and the measurement.

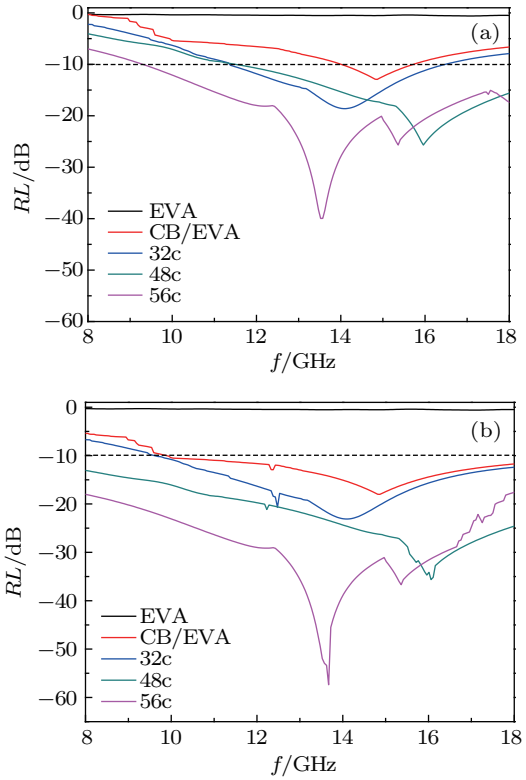


Fig. 4. (color online) Plots of *RL* versus frequency of the CB/EVA composites and AgNWs/CB/EVA absorber, (a) measured by free space method, and (b) calculated by FEM solution.

Figure 5(a) shows the SEM cross-section micrograph of the AgNWs/CB/EVA absorber with an average diameter of 56 nm, EVA-to-CB weight ratio of 1:3, and a thickness of 0.4 mm. The inset in Fig. 5(a) presents the distribution of AgNWs in CB/EVA composite matrix film indicated in a red solid rectangle at high magnification. It can be seen that there exist more defects and suspending bonds (indicated by black arrows in the inset image of Fig. 5(a)) in the AgNWs/CB/EVA absorber. A single AgNW is completely encapsulated in the CB/EVA composites (as shown in the inset image of Fig. 5(a)), which will protect the AgNWs from being oxidized. Stephen *et al.*[29] have theoretically and experimentally demonstrated that for a random distribution of AgNWs (less than 20 nm in diameter), the decreasing of the diameter of nanowires increases the connectivity and conductivity, but the scenario is opposite for nanowires with diameters greater than this size. Therefore we believe that the absorber with the conductivity network composed of AgNWs with diameters of 56 nm can cause more multiple scatterings and interfacial electric polarizations than the samples 32c and 48c, which provides an additional absorbing mechanism. It occurs due to the interaction of microwave radiation with charge multipoles at the interface between AgNWs and CB/EVA, which is similar to that of MWCNT/epoxy resin.[30] The SEM micrograph of the composites absorber is shown in Fig. 5(b). It can be observed that the electrical conducting network is formed in the CB/EVA composite matrix, which is effective for attenuating the EM wave.

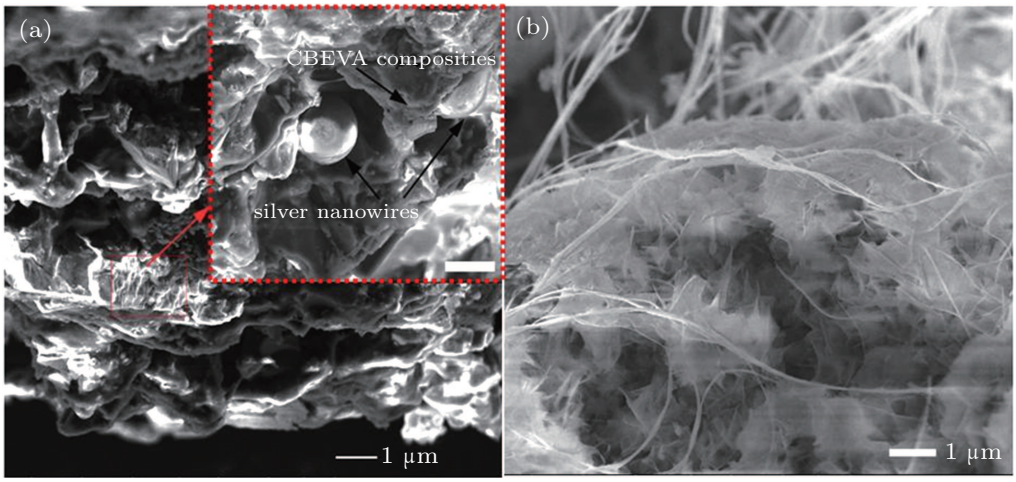


Fig. 5. (color online) SEM micrographs of AgNWs (56 nm in diameter)/CB/EVA composites (a) cross-section image, with the inset image showing the distribution of AgNWs (indicated by black arrows) in CB/EVA composite matrix indicated by a red solid rectangle at high magnification; (b) the micrographs of composite absorber.

With a combination of two CB/EVA layer, incident EM waves entering into the composite are attenuated by reflecting, scattering and absorbing many times such that it is very difficult for waves to penetrate this functional absorber (see Figs. S3 and S4 in Appendix A). However, electric conductivity of AgNWs determines a strong reflection to the incident microwave, which needs a careful match with optimized EVA-to-CB weight ratio. The *RL* curves of the compsoites with different concentrations of AgNWs and EVA-to-CB weight ratio are shown in Fig. 6. As discussed above, for all samples, the

diameter of AgNWs sandwiched between two CB/EVA layers is invariable 56 nm, and the thickness values of all the tested samples are fixed at 0.4 mm. The microwave absorption properties of the prepared AgNWs/CB/EVA composites are summarized in Table 1. As for the absorbing properties of the AgNWs/CB/EVA composite, the concentration of AgNWs and the EVA-to-CB weight ratio both have some effects on the *RL* and can influence the absorbing peak values and bandwidth. It is noted that the microwave absorbing properties will be effectively enhanced with increasing the concentration of AgNWs, with the EVA-to-CB being constant. For instance, as shown in the Table 1, the $RL_{\min}$ of the corresponding composites (as shown in Figs. 6(a)–6(c)) varies from −9 dB to 19 dB and −10 dB to −31.6 dB, respectively. As shown in Fig. 6(d), when the EVA-to-CB weight ratio is 1:4, the observed trend of the *RL* curves of the samples with low concentration of AgNWs (0.4 mg/100 mL and 0.8 mg/100 mL) is similar to that of the other composites (as shown in Figs. 6(a)–6(c)). Hence, the relatively high concentration of AgNWs (1.0 mg/100 mL) should be a factor of this phenomenon. More specially, a relatively high concentration of AgNWs (1.0 mg/100 mL) is sufficient to achieve the best EM wave absorption due to their high aspect ratio.[31] The EM wave absorption is a process, in which the energy of electromagnetic wave is depleted and then transformed into other energy, such as thermal energy, so that the wave cannot be reflected from nor permeated through the material.[32] In the experiment, the observed notable increase in the imaginary permittivity of the CB/EVA polymer matrix (as shown in Fig. 3(d)) will cause too high an electrical conductivity so that the skin depth is very small and nearly most of EM wave would be reflected by the material.[33] This result shows that the parameters in the weight ratio and concentration are chosen reasonably. Comparing our experimental results with the results from Refs. [9] and [10], composites with better excellent absorption performance (minimum *RL* value is −40.0 dB) and thinner thickness (0.4 mm) could also be obtained in our work.

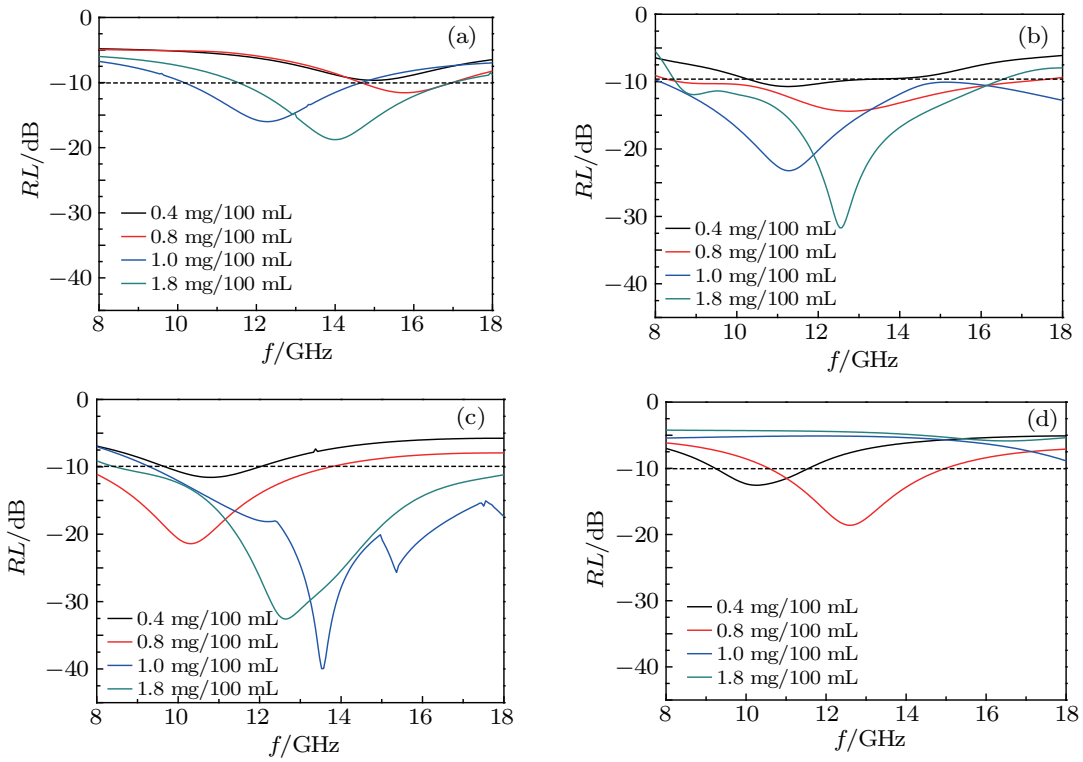


Fig. 6. (color online) Plots of *RL* versus frequency of the AgNWs/CB/EVA composites at different AgNW concentrations in the CB/EVA polymer matrix, with EVA-to-CB weight ratios of (a) 1:1, (b) 1:2, (c) 1:3, and (d) 1:4. For all samples, the diameter of Ag nanowire layer sandwiched between two CB/EVA layers is invariable 56 nm, and the thickness of the absorbing layer is fixed at 0.4 mm.

To the best of our knowledge, the *RL* curves of the composites with multilayer structure have the merits of more interference peaks than those of our structure, which is of benefit to improving the absorbing properties significantly. However, the multilayer structure has some disadvantages, such as longer processing time, inevitably larger thickness, and high-cost preparation method,[34] which is unsuitable for fabricating the absorbing composites on a large scale. As discussed above, the absorbing structure with one AgNW layer in the composite is sufficient to achieve the best electromagnetic (EM) wave absorption. Thereafter, the future work can be done to add more AgNW layers, or splitting the CB/EVA layers by AgNWs to fabricate the multilayer gradient structure composites. Meanwhile, the excellent absorption properties are maintained in those composites with much thinner thickness.

Table 1. Microwave absorption properties of AgNWs/CB/EVA composites. Δf, $RL_{\min}$, $f_{\rm im}$ represent bandwidth of reflection loss less than −10 dB, minimum reflection loss, and frequency of $RL_{\min}$, respectively.

Weight ratio of EVA to CB	AgNWs concentration/(mg/mL)	Δf/GHz	$RL_{\min}$/dB	$f_{\rm im}$/GHz
1:1	0.4/100	0	–9	(15.0)
	0.8/100	2.5 (14.6–16.9)	–11	(15.8)
1:2	1/100	4.5 (10.2–14.7)	–16	(12.2)
	1.8/100	5.4 (11.6–17.0)	–19	(14.0)
1:3	0.4/100	2.0 (10.4–12.4)	–10	(11.1)
	0.8/100	9.3 (8.2–17.5)	–14	(12.6)
	1.0/100	9.8 (8.5–18.3)	–23	(11.3)
	1.8/100	7.9 (8.5–16.4)	–31.6	(12.6)
1:4	0.4/100	2.3 (9.6–11.9)	–12	(10.6)
	0.8/100	5.7 (8.0–13.7)	–22	(10.1)
	1.0/100	9.7 (9.3–18)	–40	(13.5)
	1.8/100	10.4 (8.4–18)	–32	(12.6)
	0.4/100	2.3 (9.2–11.5)	–14	(11.2)
	0.8/100	4.4 (10.5–14.9)	–18	(12.5)
	1.0/100	0	–6	(16.1)
	1.8/100	0	–9	(18.0)

4. Conclusions

In the present work, we show that the flexible Ag nanowires/carbon black composite with a thickness of 0.4 mm exhibits excellent microwave absorption properties. The investigation on the *RL* values suggests that the microwave absorption varies with concentration of AgNWs and EVA-to-CB in composite. The *RL* value is below −10 dB in a frequency range of 9.3 GHz–18.0 GHz, with minimum values of −40.0 dB and −25.6 dB at 13.5 GHz and 15.3 GHz, respectively, for the composite composed of 1.0 mg/100 mL AgNWs and 1:3 EVA-to-CB weight ratio, which is in good agreement with the calculation results. The CB/EVA composite matrix film with strong dielectric loss, and AgNWs layers with perfect connectivity and conductivity are found to be significant factors in microwave absorptions. Moreover, the sandwiched absorbing structure, which integrates the two above-mentioned absorbing mechanisms, would cause more multiple-absorptions such that it is very effective to improve the absorption properties of the composite. The technique and fundamental understanding of absorbing mechanisms provide great opportunities to design and achieve high-performance EM waves absorbing composite with much thinner thickness on a micrometer scale.

Appendix A: Supplementary information

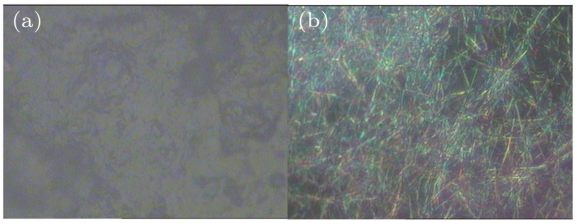


Fig. S1. (color online) Optical micrographs of (a) CB/EVA composite matrix film and (b) Ag nanowires applied by Meyer rods on the matrix film.



Fig. S2. Digital photo of the AgNWs/CB/EVA composite absorber.

EVA, a kind of flexible EM-transparent polymer with excellent high-temperature behavior and good surface smoothness, is used for coating the AgNW film to prevent nanowires from dropping off during scratch and collision and possibly corroded by chemicals. On the other hand, the EVA/AgNWs (1.0 mg/100 mL, 56 nm in diameter)/EVA (named EAE), and EVA/AgNWs (1.0 mg/100 mL, 56 nm in diameter)/CB/EVA (named EACE) are prepared by the same rod-coating technique with the same thickness of 0.4 mm to testify the influence of AgNWs on the absorption properties. The EVA-to-CB weight ratio is fixed at 1:3. As shown in Fig. S3, the *RL* values of EVE and EACE are both less than −10 dB in a frequency range of 8 GHz–18 GHz. It is evident that the performances of these two composites are very weak due to the existence of the connected AgNW layer sandwiched between two polymer ma-

trix films, which would reflect the EM wave effectively. Specially, the *RL* values of the EACE are higher than those of the EAE especially at high frequencies. This may be ascribed to the CB/EVA composite matrix which could absorb the remaining waves propagating through the AgNW layers. Therefore, as shown in Fig. S4, with a combination of two CB/EVA layers, incident EM microwaves entering into the composites are attenuated by reflecting, scattering and absorbing many times, so that it is very difficult for waves to penetrate the functional absorber.

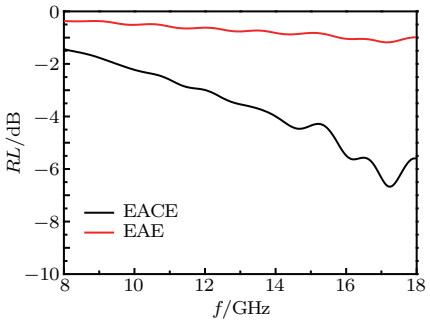


Fig. S3. (color online) Plots of *RL* versus frequency of the EAE and EACE.

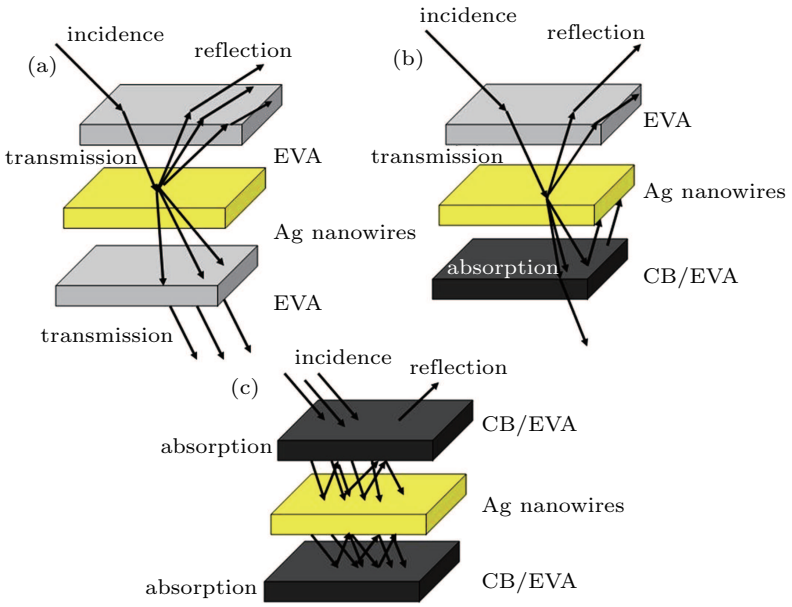


Fig. S4. (color online) Schematics of the general process of EM wave absorption in three kinds of the nanocomposites; (a) EAE; (b) EACE; (c) AgNWs/CB/EVA sandwiched structures composite.

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