

# Synthesis, Characterization and Microwave Absorption of Carbon-coated Cu Nanocapsules

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The microstructure and microwave absorption of carbon-coated Cu nanocapsules have been investigated. Carbon-coated Cu nanocapsules have been synthesized by an arc-discharge method. The paraffin-Cu/C nanocapsules composite shows excellent electromagnetic (EM) absorption properties. An optimal reflection loss (RL) value of  $-40.0$  dB is reached at 10.52 GHz for a layer 1.9 mm thickness. RL exceeding  $-20$  dB can be realized in any interval within the 1-18 GHz range by choosing an appropriate thickness of the absorbent layer between 1.1 and 10.0 mm. Theoretical simulation for the microwave absorption using the transmission line theory agrees reasonably well with the experimental results. The EM-wave absorption properties of nanocapsules materials are illustrated by means of an absorption-tube-map. The carbon-coated Cu nanocapsule is an attractive candidate for EM-wave absorption, which significantly enriches the family of EM-wave nanoabsorbents.

**Keywords:** *nanocomposites, carbon, microwave absorbers*

## 1. Introduction

Since the discovery of fullerenes, the carbon-fullerene family of materials have become a focus of much research and exhibited fundamental importance in the advancement of science and engineering<sup>1,2</sup>. In recent decades, much attention has been paid to carbon-coated metal nanoparticles, namely 'nanocapsules'. In recent years, with the development of local electronic devices, microwave communication, and the rising pollution of electromagnetic (EM) interference, materials with EM-wave absorption in a wide frequency range, with strong absorption, low density, and low cost are more and more desirable<sup>3</sup>. The EM-wave absorption properties of magnetic material-based nanocomposites and heterostructured nanocomposites have been intensely investigated, because of the synergetic effect between magnetic and dielectric losses<sup>4,7</sup>. Among the candidates for EM-wave absorbers, magnetic nanocapsules with carbon as shells have attracted intensive interest on account of the following facts: (1) the large saturation magnetization and high Snoek limit, (2) the suppression of the eddy current phenomenon, and (3) being composites with different kinds of EM-absorber materials<sup>8</sup>. However, the EM-wave absorption properties of non-magnetic nanocapsules with carbon as shells have been seldom reported. Carbon-coated Cu nanocapsules were prepared by an arc discharge method in a methane (CH<sub>4</sub>) atmosphere, in which the special microstructures with graphite shells and Cu cores result in a high relative complex permittivity and dielectric loss, exhibiting promising application as a new type of EM

wave shield/absorbent<sup>2</sup>. On the other hand, the graphite shells are outstanding microwave absorption materials due to their dielectric properties, which play an important role in improving the disperse properties of nanocapsules<sup>9,10</sup>.

Actually, Cu nanoparticles possess many good properties such as electric, optical, thermal and lubricating, etc<sup>11-15</sup>. A broad arena in engineering applications is also attributed to these particles and is attracting an increasing number of scientists. Carbon-coated Cu nanocapsules for sensor applications had been produced using a modified flame spray synthesis unit under highly reducing conditions<sup>13</sup>. Zhang et al. reported the synthesis of carbon-coated Cu nanocapsules by means of an arc discharge method in a CH<sub>4</sub> atmosphere, which consists of carbon-coated Cu nanocapsules and onion-like fullerenes<sup>2</sup>. Since then, significant progress has been made in the syntheses of carbon-coated Cu nanocapsules. As well known, the complex permeability, permittivity, the EM impedance match and the microstructure of the absorber determine the EM -absorption properties. Although carbon-coated Cu nanocapsules have been synthesized by various methods, the EM-absorption properties of carbon-coated Cu nanocapsules have been seldom studied. In this study, we have synthesized carbon-coated Cu nanocapsules by an arc discharge technique. The EM properties and microwave absorption properties of carbon-coated Cu nanocapsules have been deeply investigated. Furthermore, an absorption-tube-map has been presented for designing better absorbents with materials of  $\mu_r=1$ . In order to verify the theoretical

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calculation of microwave absorption using the transmission line theory from permittivity and permeability data, the experimental results of microwave absorption have been carried.

## 2. Experimental

Arc discharge is a powerful method for preparing nanoparticles and nanocapsules, which has been described previously elsewhere<sup>16-19</sup>. Briefly, the pure Cu ingot to be evaporated served as the anode on the water cooled copper stage. The upper carbon needle served as the cathode. Ar (16 000 Pa) gas and H<sub>2</sub> (4 000 Pa) and 40 mL liquid ethanol were introduced into the evacuated chamber (1.0×10<sup>-2</sup> Pa) before a potential was applied between the cathode and the anode. The current was maintained at 100 A for 0.5 h. When the arc time reached 0.5 h, the pressure of the chamber can reach 1 atmospheric pressure because of the decomposition of ethanol and the expand of the gas with increasing the temperature. The electric arc can be controlled during operation through adjusting the distance between the two electrodes. After being passivated in air for 8 h, the carbon-coated Cu nanocapsules were collected in the top of the chamber. In order to get the enough nanocapsules for the measurement of reflection loss, the above experiment is repeated several times under the same experiment conditions.

The composition and phase purity of the as-synthesized samples were analyzed by X-ray diffraction (XRD) at 40 kV voltage and 50 mA current with Cu K $\alpha$  radiation ( $\lambda = 1.5418 \text{ \AA}$ ). Transmission electron microscopy (TEM) and high-resolution TEM (HRTEM) images were obtained on a JEOL JEM-2010 transmission electron microscope at an acceleration voltage of 200 kV. The paraffin-Cu nanocapsules composite was prepared by uniformly mixing Cu nanocapsules with paraffin, as described in detail elsewhere<sup>2-10,16-20</sup>, by homogeneously mixing the nanocapsule with paraffin and pressing them into cylinder-shaped compacts. Then the compact was cut into toroidal shape with 7.00 mm outer diameter and 3.04 mm inner diameter. The EM parameters are measured for paraffin-Cu nanocapsules composite containing 40 wt.% Cu nanocapsules, using an Agilent 8722ES network analyzer. The microwave-absorbing characteristics were evaluated by measuring the reflection loss using an HP8757E scalar quantity network analyzer in the 1-18 GHz band range, and the sample sheets (180 mm×180 mm) were mounted onto an aluminum substrate. All the measurements were performed at room temperature. Coaxial method was used to determine the EM parameters of the toroidal samples in a frequency range of 1-18 GHz with a transverse electromagnetic mode. The VNA was calibrated for the full two-port measurement of reflection and transmission at each port. The complex permittivity and complex permeability were calculated from S-parameters tested by the VNA, using the simulation program of Reflection/Transmission Nicolson-Ross model. Air line holder is VNA-8722ES and VNA- HP8757E accessory and Nicolson-Ross model is used on the 85071E Materials Measurement Software. Air coaxial line was used to determine the EM parameters of the toroidal samples in a frequency range of 1-18 GHz. The mismatches between

the cable and sample holder can be eliminated by means of the change of electric delay method in the calibration accessories of network analyzer. The RL coefficient curves were calculated from the relative permeability and permittivity for a given frequency and absorber thickness, according to the following equations:

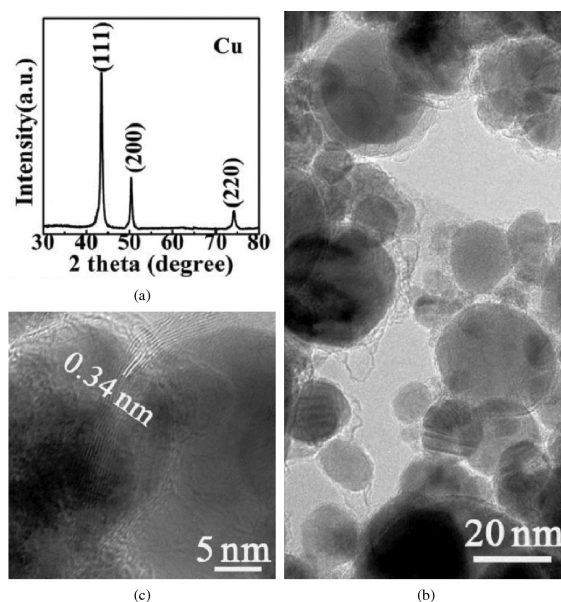
$$Z_{in} = Z_0(\mu_r / \epsilon_r)^{1/2} \tanh[j(2\pi f d / c)(\mu_r \epsilon_r)^{1/2}] \quad (1)$$

$$RL = 20 \lg |(Z_{in} - Z_0) / (Z_{in} + Z_0)| \quad (2)$$

where  $f$ , is the frequency of the EM-wave,  $d$ , the thickness of the absorber,  $c$ , the velocity of light,  $Z_0$ , the impedance of free space and  $Z_{in}$ , the input impedance of the absorber.

## 3. Results and Discussion

Figure 1a shows the XRD pattern of carbon-coated Cu nanocapsules. As shown in Figure 1a, the main peak located at  $2\theta = 43.48^\circ$ ,  $50.44^\circ$  and  $74.13^\circ$ , respectively, corresponds to the (111), (200) and (220) planes of Cu with face centered cubic structure, and the lattice constant of the (111) planes is approximately  $2.08 \text{ \AA}$ . It is then evident that the carbon atoms from the carbon needle hardly affect the lattice constant of Cu nanoparticles to form a Cu-C solid solution. Figure 1b and c show the TEM and HRTEM images of carbon-coated Cu nanocapsules. As can be seen, most of the Cu nanocapsules are spherical in shape, with the mean diameter in the range 10-30 nm. Moreover, from the HRTEM of Figure 1c, it is clearly noted that the whole surface of the nanoparticles is coated by a graphitic multi-layered carbon shell around 1-2 nm in thickness, which is thinner than that of carbon-coated Fe, Ni or Sn nanocapsules<sup>3,10</sup>. The interplanar spacing of 0.34 nm in the coating is approximately close to that of the bulk graphite (002) plane, which is accordance with the previous results<sup>2,3,10</sup>. Moreover, it can be found that



**Figure 1.** (a) XRD pattern, (b) TEM and (c) HRTEM images of carbon-coated Cu nanocapsules.

many lattice defects occur in the carbon shell, including collapses, unevenness, dislocations, etc. The many lattice defects can lead to the high electrical resistivity, which is useful to significantly absorb the microwave energy<sup>21</sup>. In our experiments, during the thermal evaporation process, the Cu nanoparticles were formed at first and then covered by carbon. The formed carbon-coated Cu nanocapsules fall on the surface of the chamber.

To obtain the EM-absorption properties of carbon-coated Cu nanocapsules, RL of the Cu nanocapsules was calculated using the relative complex permeability and permittivity at a given frequency and layer thickness according to the transmission-line theory<sup>3,22</sup>. Figure 2 represents the frequency dependence of the complex relative permittivity ( $\epsilon$ ) and permeability ( $\mu$ ) of the paraffin-Cu nanocapsules composite. As shown in Figure 2a, maximum/minimum values can be found below/above the resonant frequencies in the real part ( $\epsilon'$ ) curve. Accordingly, a peak is observed in the imaginary part ( $\epsilon''$ ) curve near the resonant frequencies. The resonant frequency of  $\epsilon_r$  in the current frequency range is 7.97 GHz, as shown in Figure 2a. Since 1990s, considerable attempts have been done to get an exact solution of the permittivity of heterogeneous mixtures<sup>23</sup>. Tinga et. al<sup>24</sup> have studied multiphase inclusions in the form of confocal ellipsoidal shells dispersed in a host material, which is similar to the present case where carbon-coated Cu nanocapsules are dispersed in a paraffin matrix. Following the effective-medium theory of three-phase inclusions<sup>3,24</sup>, the permittivity of paraffin-Cu nanocapsules composite seems to originate from the special geometric structures of the Cu nanocapsules inclusions, the intrinsic permittivity of each component in the paraffin-nanocapsules composite (Cu, C and paraffin), and the dispersion (volume fraction) of the nanocapsules<sup>3</sup>.

As shown in Figure 2b, the real part ( $\mu'$ ) of  $\mu$ , and the imaginary part ( $\mu''$ ) of  $\mu$ , are almost constant in the 1-18 GHz with values of  $1 \pm 0.03$  and  $0 \pm 0.03$ , respectively. This permeability is expected for a material that has no magnetic properties. The real part of the permeability tends to slightly decrease with increasing frequency. However, a slight

decrease might be expected, in a conductive, non-magnetic material; on increasing frequency, eddy currents start shielding of the material, thus reducing its permeability to 0 (at infinite frequency), as if it were a perfect diamagnet (like a superconductor below its critical temperature)<sup>3</sup>. In any case, the permeability in Figure 2b is that of a non-magnetic system, as expected. It is worthy noted that the negative  $\mu''$  exists in our measured data, as shown in Figure 2b.

To further examine the microwave absorption abilities of the carbon-coated Cu nanocapsules, RL coefficient as a function of absorbing thickness  $d$  and frequency  $f$  were calculated, as shown in Figure 3a, according to Equation 1 and 2. The optimal RL or the dip in RL corresponds to the occurrence of the maximum absorption or the minimal reflection of the microwave power for the particular thickness. An optimal RL value of  $-40.0$  dB, corresponding to 99.99 % absorption, is observed at 10.52 GHz for a layer 1.9 mm thickness. It is worth noting that the absorbent with a thickness of 1.1 mm has RL values exceeding  $-20$  dB at high frequencies in broad frequency ranges, which is thinner than the previous reported results<sup>3-5,8-10,16-20,25-27</sup>. As shown in Figure 3b, for all frequencies within the 1-18 GHz range, RL values exceeding  $-20$  dB can be obtained by selecting an appropriate thickness of the absorbent layer between 1.1 and 10.0 mm. Compared with other carbon-coated nanoparticles or nanowires (e.g., Sn/C<sup>[3]</sup>, Ni/C<sup>[28]</sup>, FeNi/C<sup>[10]</sup>, FeCo/C<sup>[29]</sup>, and FeCoNi/carbon nanotubes<sup>[30]</sup>), the non-magnetic carbon-coated Cu nanocapsules present quite different and excellent EM-wave absorption properties, which may be due to the special microstructure of the Cu nanocapsules, the size of the Cu nanocapsules, and also the weight fraction of paraffin-Cu nanocapsules composite<sup>3</sup>. Generally, there are two mechanisms of energy attenuation in materials: magnetic loss and dielectric loss<sup>3</sup>. However, due to the non-magnetic nature, the present Cu nanocapsules absorb the EM-wave mainly by dielectric losses. And the loss phenomena associated with dielectric mixtures are due to the various polarizations caused by relative displacement of electrons and nuclei, dipolar orientations and interfacial (Maxwell-Wagner) effects at the boundaries between the

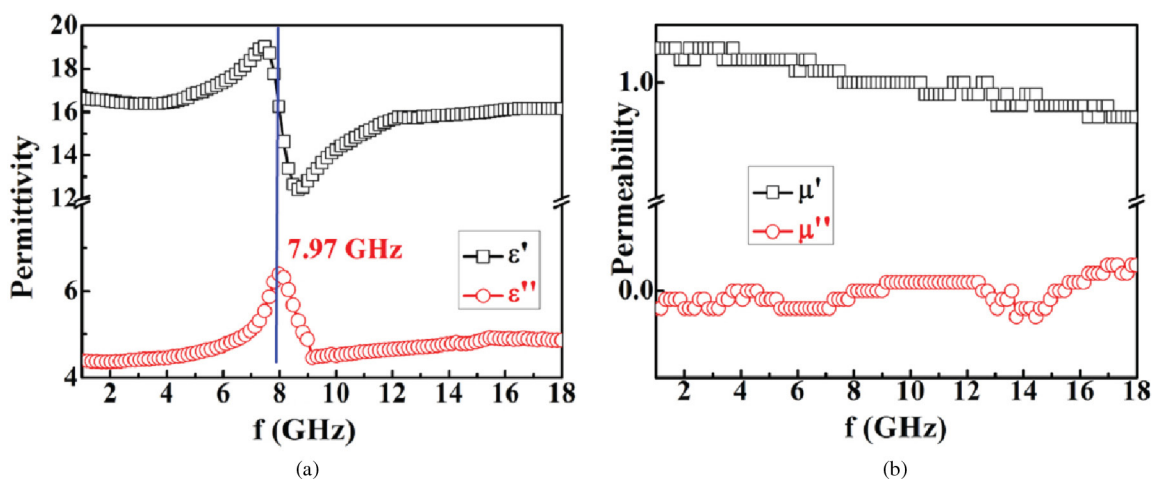
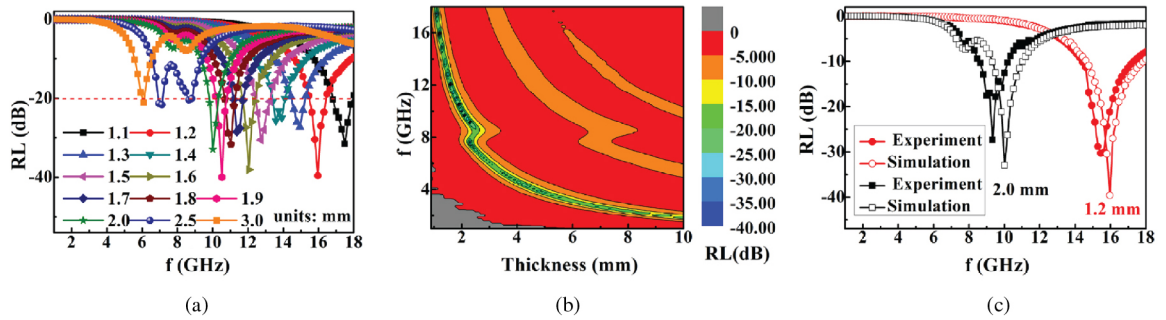
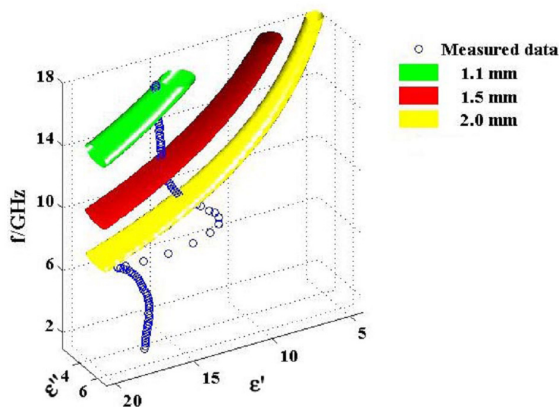


Figure 2. (a) Relative permittivity and (b) relative permeability of paraffin-Cu nanocapsules composite as a function of frequency.



**Figure 3.** (a) Simulated RL coefficient curves of the paraffin-Cu nanocapsules composite as a function of frequency. (b) Two-dimensional representation of RL coefficient derived from the measured and of the paraffin-Cu nanocapsules composite. (c) RL coefficient curves of the composites with the different thickness as the function of frequency for simulation and experiment.



**Figure 4.** Absorption-tubes for materials with  $\mu=1$ . For illustrations, layers with the thickness of 1.1, 2.0 and 3.0 mm are given; open circles are the measured relative complex permittivity of the paraffin-Cu nanocapsules composite.

mixture components<sup>3</sup>. The lossy behavior becomes even more pronounced in the conducting Cu and dielectric carbon interfaces of the Cu nanocapsules<sup>3,31</sup>. In addition, the simulated results show that the RL peak frequency is closely related to the thickness and moves to the lower frequency with the increase of sample thickness, as shown in Figure 3a. It is worthy noted that there is a small peak at around 8 GHz for the layer thickness below 2.0 mm, which is explained by the resonant frequency of  $\epsilon_r$  at 7.97 GHz in Figure 2a. However, there are two clear RL peaks for the sample with the thickness above 2.0 mm, ascribed to the quarter-wavelength cancellation by thickness<sup>21</sup>. For the sample with the thickness above 2.0 mm, the small peak at around 8 GHz disappears, due to the broaden effect by the successive strong RL peaks.

Figure 3c shows the simulation and experimental results for paraffin-Cu nanocapsules composite with the thickness of 1.2 mm and 2.0 mm, respectively. Both the experimental results show that the center frequencies were shifted a little from the theoretical predictions. In the view of the complexity of EM absorption, the theoretical results agree reasonably well with the experimental results in both the curve pattern and absolute values. The slight discrepancy

between the simulated results and experimental ones can be caused by several factors, such as error in fabrication of specimens and measurement of permittivity and reflection loss<sup>32</sup>.

In order to study RL given by Equation 2, the relations between the variables of  $\epsilon_r$ ,  $\mu_r$ ,  $d$  and  $f$  is of great interest to be investigated. Considerable attention has been paid to these kinds of heterogeneous systems, in which nanocapsules with magnetic core and dielectric shell are dispersed in dielectric matrixes (rubber, paraffin or epoxy resin)<sup>3</sup>. However, only a few approximations and numerical methods can be used to resolve Equation 2 to obtain the RL values, because it is too complicated to deal with the six variables in it<sup>3</sup>. In the present study, the situation can be simplified as  $\mu_r = 1$  and  $\mu'' = 0$ , due to the non-magnetic nature of Cu nanocapsules. By simulating Equation 2 with  $\mu_r = 1$ , effective absorption (RL < -20) 'tubes' for the layer thicknesses of 1.1 mm, 1.5 mm and 2.0 mm were obtained, respectively. As shown in Figure 4, the areas where the measured permittivity of Cu nanocapsules goes through the tubes are effective absorption locations. This simulation agrees well with the results in Figure 4, as discussed before. Figure 4 reveals a great potential for non-magnetic materials in EM-wave absorption applications. The absorption tube-map also suggests a general route for finding better absorbents with materials of  $\mu_r = 1$ . For composite with a certain layer thickness, one is only able to get RL values < -20 dB in the whole 1-18 GHz range when the measured permittivity spectra are all located within the tubes of the corresponding thickness<sup>3</sup>.

## 4. Conclusions

Carbon-coated Cu nanocapsules have been synthesized by a modified arc discharge method, in which Cu nanoparticles as cores and carbon as shells. Many lattice defects occur in the carbon shell, including collapses, unevenness, dislocations, etc, which can lead to the high electrical resistivity. By choosing an appropriate thickness of the absorbent layer between 1.1 and 10.0 mm, RL values exceeding -20 dB can be realized in any interval within the 1-18 GHz range. There is a small peak at around 8 GHz for the layer thickness below 2.0 mm, which is explained by the resonant frequency of  $\epsilon_r$  at 7.97 GHz. However, there are two clear RL peaks for the sample with the thickness above

2.0 mm, ascribed to the quarter-wavelength cancellation by thickness. For the sample with the thickness above 2.0 mm, the small peak at around 8 GHz disappears, due to the broaden effect by the successive strong RL peaks. Theoretical calculation of microwave absorption using the transmission line theory from permittivity and permeability data agrees reasonably well with the experimental results. An absorption-tube-map is presented for designing better absorbers with materials of  $\mu_r = 1$ . As a result, the non-magnetic carbon-coated Cu nanocapsules are attractive

candidates for EM-wave absorption, which significantly enriches the family of EM-wave absorbers.

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