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EDM 2017

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Effect of Ultrasonic Treatment on the X-band Microwave Absorption of Multiwalled Carbon Nanocomposite

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National Research Tomsk State University, Tomsk, Russia

Abstract – **This paper presents results of calculation reflection coefficients multiwalled carbon nanotubes (MWCNTs)-laquer composites at microwave frequencies. Two groups of composite samples with 1.0% weight percentages of MWCNTs were fabricated and tested. The multiwall carbon nanotubes used in the composite were about 18.6 nm and 9.4 nm in diameter. As a result, it is shown that the use different morphology MWCNTs entered in the making of composite material and various ultrasonic treatment times provide the change both absorption region width and absorption peak depth.**

Index Terms – **MWCNT, composite, permittivity, reflection coefficients, ultrasonic treatment.**

I. Introduction

The outstanding properties of multiwalled carbon nanotubes (MWCNTs) represent new opportunities for creating novel multifunctional composite materials. Less weight, excellent thermal, mechanical and electronic properties of MWCNTs can be utilized for various applications, including higher efficiency electromagnetic (EM) wave absorbers for EM interference shielding [1].

Electromagnetic characteristics of CNT-based composites are governed by many factors, such as, polymer nature, geometry of CNTs (length, defect, etc.), concentration and distribution of nanotubes in matrix. Variation in any of these parameters could drastically change properties of the composite materials [2]. Dispersion and allocation of CNTs in polymer matrix are also important for composite properties [3]. It is believed that the amazing electrical properties of CNTs can be utilized to impart electrical conductivity to dielectric host, thereby improving electrostatic charge dissipation and electromagnetic (EM) shielding or absorbing efficiency [4, 5]. In recent years, MWCNTs/polymer composites have been extensively explored for potential applications in EM shielding or absorbing materials [6].

The application of nanotubes as independent materials is troublesome, because they possess volatility and low bulk density. For this reason, nanotubes are used as filler in the manufacture composite [2, 7–8], which can be used to solve problems of electromagnetic compatibility and radio components [9]. The binder material for production of polymer composite should have a good adhesive properties, high fluidity and high polymerization rate [11]. The permittivity depends on the nanotubes concentration in a composite, aspect ratio and the residues of catalytic metals [10–11].

Methods of changes of electrophysical properties of composite materials of external influences on their structure are investigated insufficiently. One way to change the internal structure of composite materials is ultrasonic treatment [12–13]. Ultrasonication can produce thermodynamically stable MWCNTs mixture and after sonication MWCNTs are separated [14].

II. Problem Definition

The present work is devoted to investigation of the impact of ultrasonic treatment and influence of geometry MWCNTs on electrophysical characteristics MWCNT-based composites. Electromagnetic characteristics of composites were studied using a vector network analyzer, Agilent Technologies E8363B, and volume multimode rectangular cavity that cover the frequency range 7 – 12 GHz. We also used Mathcad program for calculation reflection coefficients.

Section III is described calculation absorption characteristics. Section IV is devoted to the measuring equipment and composites manufacturing technique. Section V is devoted to obtained results and substantiated them.

III. Theory

The electromagnetic reflection property of a material is usually characterized in terms of the power reflection of a plane wave reflected from an infinite metallic surface. There is reflectivity or return loss of composites, generally produced for normal incidence, is expressed in decibels [17]

$$R = 20\log\left|\frac{Z_{in} - Z_0}{Z_{in} + Z_0}\right|, \quad (1)$$

where $Z_0 = \sqrt{\mu_0/\varepsilon_0}$ is propagation number of empty space, Z_{in} is free space boundary impedance, we estimate optimal thickness of absorbing layer, located on a perfectly conducting surface. Free space boundary impedance is determined by formula

$$Z_{in} = \sqrt{\frac{\mu_0\mu^*}{\varepsilon_0\varepsilon^*}}\tanh\left(i2\pi df\sqrt{\mu_0\mu^*\varepsilon_0\varepsilon^*}\right) \quad (2)$$

where *f* is electromagnetic field frequency, *d* is material thickness, μ*, ε* are complex permeability and permittivity of material.

Based on (1) and (2), the return losses *RL*, as a function of frequency *f*, are calculated at different thicknesses of samples.

IV. Experimental Results

A. Composites Manufacturing Technique

The fillers of composites were multiwalled carbon nanotubes. MWCNTs were obtained by catalytic gas-phase deposition of ethylene in the presence of FeCo catalyst in Institute of Catalysis SB RAS [15]. The average nanotubes diameters are 18.6 nm and 9.4 nm, purity > 90%. Nanotubes with about 18.6 nm diameter contain remainders of catalyst about 7.0%.

Urethane alkyd lacquer was used for production of experimental samples. In the liquid state its viscosity is known to be small. This allows a filler to be moved easily. For the production of mix for experimental samples 1wt.% of MWCNT was added to 99.0 wt.% of lacquer. The mixture was placed in a glass beaker. The mixture was sonicated by the ultrasonic device “Alena” (Acoustic Processes & Devices lab Biysk Technological Institute, Russia). It consists of ultrasonic generator and ultrasonic vibrating system with installed working tool (the diameter is 10 mm). Working tool was inserted into a glass beaker with mixture. The mixture was sonicated for 1, 2, 3, 4 and 5 minutes at 28 kHz frequency at 50 VA power. Mixtures were molded into a planar plate, which size is 70 mm × 20 mm × 0.5 mm. Process of polymerization was carried out for 48 hours at the room temperature.

B. Measuring equipment

The cavity perturbation technique was used for the evaluation of dielectric properties of the materials in X-band of microwave frequencies. In this technique, a cavity was designed with a very small slot at the centre of the broad/short wall of rectangular waveguide in order to insert the sample material. Agilent’s E8363B vector network analyzer was used to measure of electromagnetic response of the rectangular cavity that covers the frequency range 7 – 12 GHz. When the sample is advanced into the cavity, the intensity of the microwave field in the cavity, the frequency shifts, and the width of the resonance at half-power-maximum are changed according to the electrophysical properties of the sample loading into the cavity. The measuring sample was a long and thin rod with sizes 2×2×70 mm^3. Complex permittivities were calculated using the approximation of the perturbation method [16]. Measurements were made at temperature of 24 ± 1 °C. The measurement error is about 5 percent.

V. Discussion of Results

Fig. 1 shows the complex dielectric permittivity charts of the composite material samples without ultrasonic treatment. It shows that permittivity of the samples is constant over the frequency range. Ultrasonic treatment tends to change of the permittivity value. More details are given in articles [9, 14]. The data from Fig. 1 were taking as a basis for reflection coefficient calculation.

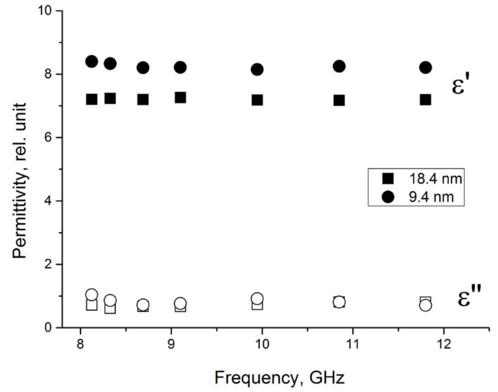

Fig.1. Spectra of complex permittivity of composites (span 8 – 12 GHz)

At present research thicknesses samples were equal 0.2 cm and 0.5 cm. Concentration of MWCNTs in the samples is 1.0 wt.%. Some results are shown in Fig. 2 – Fig. 5. Fig. 2 and Fig. 3 show the reflection curves RL-f for the composites based on MWCNTs with outer diameter 18.6 nm (Set A). Fig. 4 and Fig. 5 show the reflection curves RL-f for the composites based on MWCNTs with outer diameter 9.4 nm (Set B).

All figures show that the reflection coefficient does not change linearly with increasing ultrasonic treatment time. The increase an ultrasonic treatment time leads to displacement of reflection coefficient down and up in frequency range.

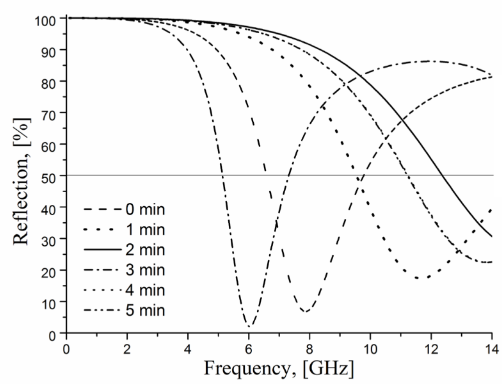

Fig.2. Calculated reflection curves *RL-f* for set A with thickness 0.2 cm

The samples from set A with 4 min and 5 min ultrasonic treatment have equal reflection coefficients. It is reason that there are five reflection coefficient curves in Fig. 2 and Fig. 3.

With regard to samples from set B, we can see all reflection coefficient curves in Fig. 4 and Fig. 5.

The results of investigation of samples with 0.2 cm thicknesses are following. We must to see the level scatter electromagnetic radiation power, when the reflection coefficient is lower 50%. The sample of composite based

TABLE I
EFFECT OF ULTRASONIC TREATMENT ON ABSORPTION REGION OF COMPOSITES

Samples \ Time, min		0	1	2	3	4	5
MWCNTs, nm	Thickness, cm	Absorption region, GHz					
9.4	0.2	> 12.1	> 11.3	> 9.3	> 8.7	> 10.2	> 10.6
	0.5	4.8–7.8	4.5–7.3	3.7–5.9 > 11.4	3.5–5.4 > 10.7	4.1–6.5 > 12.3	4.3–6.8 > 12.9
18.6	0.2	6.6–9.8	> 9.6	> 12.4	5.2–7.3	> 11.2	> 11.2
	0.5	2.6–3.9 8.3–10.7	3.8–6.0 > 11.6	4.9–8.0	2.0–2.9 6.5–8.0 11.2–12.9	4.5–7.2 > 13.4	4.5–7.2 > 13.4

on 9.4 nm MWCNTs without ultrasonic treatment had absorption region at 12 GHz and above, while the sample of composite based on 18.6 nm had absorption region from 6.5 GHz till 10.0 GHz. The absorption region of this composite shifted up and down at frequency range. The ultrasonic treatment is a reason of this move.

According to Fig. 2 – Fig. 5 the Table 1 was compiled. Table 1 contains information about the absorption regions of experimental samples.

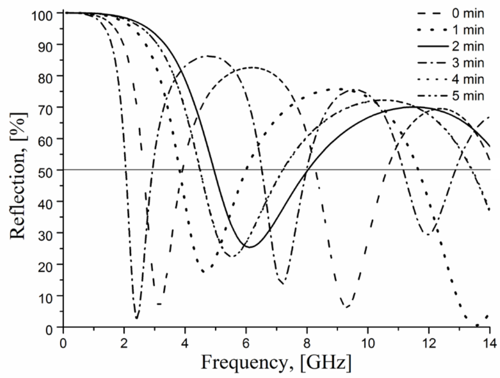

Fig.3. Calculated reflection curves *RL-f* for set A with thickness 0.5 cm

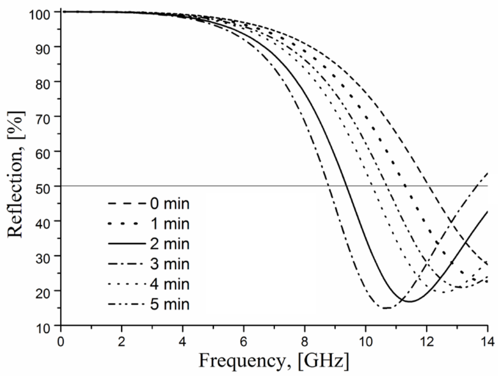

Fig.4. Calculated reflection curves *RL-f* for set B with thickness 0.2 cm

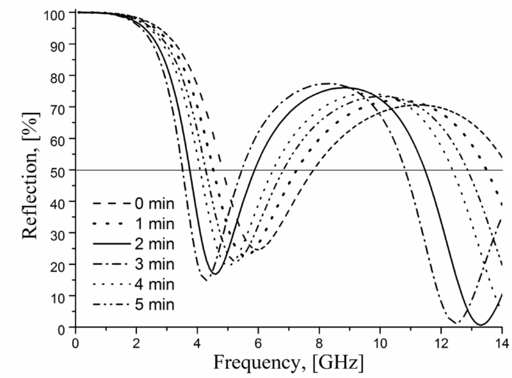

Fig.5. Calculated reflection curves *RL-f* for set B with thickness 0.5 cm

Increasing time of ultrasonic treatment leads to modifies width and frequency boundary of absorption area (see Fig. 2 and Fig. 4). The greatest absorption was observed in both composites after 3 minutes of ultrasonic treatment.

This behavior of reflection coefficients could be explained by the impact of ultrasonic treatment which breaks the agglomerates of MWCNTs and they are evenly distributed over the volume of the composite. MWCNTs have a lot of structural defects [2]. Due to the fact that ultrasonic treatment was carried out by submerged ultrasonic probe, MWCNTs have broken. This led to the modify characteristics of the material. About these conversion electromagnetic characteristics of the composite under the action of the ultrasonic treatment we have already mentioned [9]. It has been shown that there are optimal processing times, which increase both real and imaginary part of permittivity. A study of the reflection coefficient in this article showed similar results.

Thus the results of our investigation are following (Fig. 2 – Fig. 5). We can change the absorption region width of composite sample by changing material thickness and ultrasonic treatment time.

VI. CONCLUSION

Consequently, it is shown that the use different morphology MWCNTs that enter in the making of composite material and various ultrasonic treatment times provide the change both absorption region width and absorption peak depth. On the basis of obtained data it is possible to produce an attenuating material by changing the type of filler or time of the UST.

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