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MICROWAVE ABSORBING PROPERTY OF EPOXY RESIN COMPOSITES WITH ADDED Fe₃O₄ NANOPARTICLES

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Abstract- In this paper, we have successfully synthesized nanoparticles of Fe_3O_4 and composites based on epoxy resin with various mass ratios of nanomagnetite were prepared. The structure, morphology and properties of the composites were characterized with X-ray diffraction, scanning electron microscope. Results of measurements of reflection coefficients (R), transmission (T), and absorption (A) of the ER series composites of composites on basis of nano magnetite are presented in this paper.

Key words: Electromagnetic Wave Absorbers, Composite Materials, Fe₃O₄ Nano Powders, Electromagnetic Properties.

I. INTRODUCTION

Electromagnetic absorption materials are the focus of many studies due to increased state regulations for controlling electromagnetic radiation levels, as well as new norms and standards for compatibility and electromagnetic interference produced by these types of devices [1]. These materials are also important tools in electronic warfare because they can be used to exploit potential targets from radar detection.

Related to the electromagnetic properties, the material can be used as an electromagnetic radiation absorption or reflector [3, 4]. When metal is used as a protective material, there are few disadvantages, its weight and its ease of corrosion are the most important because corrosion is the most important.

The matrix of polymer composite contains guiding fillers for protection [5] is attractive because of their processing capabilities, which helps reduce or eliminate seams in the housing that shields. These seams usually encounter metal shields as shields and tend to cause leakage of radiation and reduce the shield effect [6, 7].

The polymer matrix complexes are good-looking because of their low density. The polymer matrix surrounded with micro and nanoparticles which absorb many composites due to its widespread use in electromagnetic wave absorption, ergonomic performance, thermal and electrical conductivities, and various instruments or apparatus.

Electromagnetic materials need to have low weight, thin depth, EM wave absorption capability, wide width and adjustable absorbance frequency, as well as multiple capabilities. Polymer matrix mixtures use process and easy preparation; some kinds of polymers, such as epoxy resins, polyethylene terephthalate and low-cost polyurethane, make composite materials widely used. Accordingly, absorption mechanisms, dielectric wastes, magnetic loss, and conductive loss materials are mainly absorbed into three types of EM materials [8].

It is also used as nanoscale materials, with excellent electrical, magnetic and optical properties due to its specific size, surface, and effect of the quantum tunnel. For example, the density of nanomaterials is less than majority density. It has a certain level of specific surface area, and therefore there are a great number of active atoms on the surface of the material, which is the loss of a large moving dielectric due to polarization of the interface.

The conductivity of the magnetic material in metal form is very high, which makes the actual permeability at high frequencies is reduced due to electrical losses by electromagnetic waves. If the size of unit is under the skin depth, it can lead to a loss of electrical current, which can increase the stability of the absorption waves. Generally, the depth of the skin at microwave frequencies (10 GHz) is about 1 micrometer, so nanoparticles will absorb electromagnetic waves at widespread frequencies [9, 10].

As a matrix, polymer can improve the process, prevent the combination of nanoparticles, modify electrical and magnetic properties of nanoparticles to reduce reflection of the EM wave, and even improve other chemical or physical properties of other materials.

Epoxy resin (ER) is widely used in polymer nanocomposites, as a type of conventional thermoset polymer matrix due to its excellent mechanical properties, chemical and thermal stability, antibacterial properties, low contractility and strong adherence. To be Many nanomaterials, such as single-carbon carbon nanotubes (SWNTs), multi-core carbon nanotubes (MWNTs), carbon nanoparticles, nanofilters, nanoparticles, etc., are derived as emitters in epoxy resins [9, 11].

As a filler magnetite nanoparticle (Fe3O4), not only in the field of magnetic recording media, such as audio and video, and high density digital recording discs, magnetic fluids, data storage, but also in the fields of medical care such as systems medication delivery (DDS), medical programs, including radio frequency hyperthermia, photomagnetic, and magnetic resonance imaging (MRI), medical diagnosis and cancer treatment and microwave devices, optics magnetometers, sensors, high frequency applications, catalytic and magnetic sensor [1-11].

II. EXPERIMENTAL PART AND DISCUSSIONS

In this paper, we have obtained composite materials based on epoxy resin and magnetite nanoparticles and their structure and dielectric properties are investigated.

To synthesis of nanoparticles, necessary amounts of iron salts were taken in the molar ratio of $Fe^{2+}/Fe^{3+}=1/2$. Firstly, 1% aqueous solutions of $FeSO_4$ (99%, South Korea) and $FeCl_3$ (GOST 4147-74) were prepared. They were filtered and mixed together at a temperature of 50-60 °C for 30 minutes with a magnetic stirrer at 500 rpm. Then, at a temperature of ~60 °C, a calculated amount of 25% ammonium hydroxide solution was added to the final mixture, drop by drop to reach value of pH ~10-11. Then, the sediment was left for a one day for aging. After that, a homogeneous precipitate of magnetite was washed with distilled water to get pH=7 for the washing solution. Further, magnetite was stabilized with oleic acid $C_{17}H_{33}COOH$ (TU 6-09-08-2003-88) in hexane n- C_6H_{14} (TU 2631-158-44493179-13).

Ground powders phase analysis was performed using XRD method. Diffractograms were achieved in the variety of 2θ =20-110° with Cu-K α radiation based on Rigaku D/Max diffractometer and at the wavelength of 1.54050 Å.

Microscopic and morphological studies were carried out using an electron microscope (FEGSEM) from the FEI Nova Nano 430 FEG model. The powders were placed with an aluminum sample holder with two-sided adhesive conductive Au strips [12].

The X-ray diffraction patterns of the dried sample are shown in Figure 1. The intensity and the diffraction peaks are consistent with the literature except (111) diffraction peak which was not observed from the result of XRD analysis conducted in D8 Advance Bruker with Cu-K α radiation at a wavelength of 1.540562 Å. This sample shows a very large peak, which represents the incredibly beautiful nature and small crystalline size of particles. Cubic single phase (a=8.322 Å) nano sized Fe₃O₄ powder has been acquired in [13].

Figure 2 shows the SEM microstructures of Fe_3O_4 . The image in Figure 1 indicates that the size of particles is change between 30-40 nm. The particle size is determined by taking the average of the sizes at the peaks D220, D311, D400, D422, D511 and D440 [14].

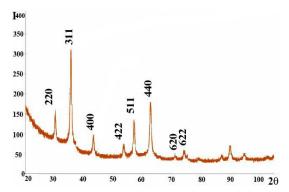


Figure 1. X-ray diffraction pattern of the prepared Fe₃O₄ particles

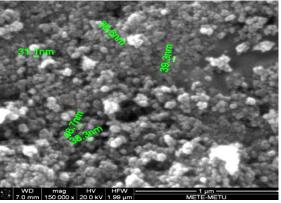


Figure 2. Morphologies of Fe₃O₄

After making sure that obtained results match to the literature composite materials based on nano-magnetite were synthesized. For this, the epoxydian resin ED-20 (GOST 10587-84) was used as a polymeric matrix. For hardening of matrix, methyl-etrahydrophthalic anhydride MTHFA hardener (TU 38.103149-85), Laprol 500 (TU 2226-020-10488057-94) plasticizer and 2,4,6-tris (dimethyl-amino-methyl) phenol UP606/2 (TU 2494-630-11131395-2006) accelerator, were mixed. In order to synthesis samples of an epoxy composite containing nanomagnetite, a mixture of stabilized magnetite in hexane is added to the matrix heated to a temperature of 50-60 °C with constant stirring.

A mixture of an epoxy matrix with nanomagnetite was heated to a temperature of 68-70 °C for evaporation of hexane. After that, semi-liquid of mixture was poured into cylindrical metal molds in which samples were cured in a following stepwise route: 90-100 °C for 1 hour; 110-140 °C for 1 hour; 150-180 °C for 1 hour.

Electromagnetic characteristics of the samples were measured to establish the effect of nanoparticle sizes and volume fractions on the investigated samples. Measurements of electromagnetic properties of composites were carried out in the frequency range 0.1-18 GHz by a coaxial method using a vector analyzer of chains P4M-18/4 manufactured by MICRAN.

The frequency spectra of the refractive index square of the ED $n^2=\varepsilon\mu$ composites are shown in Figure 3. The frequency spectra of the reflection coefficients (R), transmission (T), and absorption (A) of the ER series composites are shown in Figure 4.

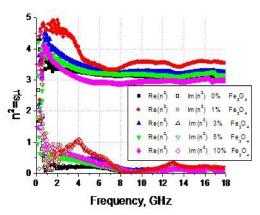


Figure 3. Frequency dependencies of the refractive index square of ER series composites

It can be seen from Figure 3 that the refractive index does not monotonically depend on the concentration of magnetite in the composite. It is interesting that the maximum values of the imaginary part of the refractive index are observed for composites containing only 1% magnetite. It is possible that the samples had a strong inhomogeneity in the distribution of magnetite. The real part of the square of the refractive index decreases with increasing concentration. This may be due to the shift in the frequency of natural ferromagnetic resonance, and the region of lower frequencies with an increase in the concentration of magnetite in the composite.

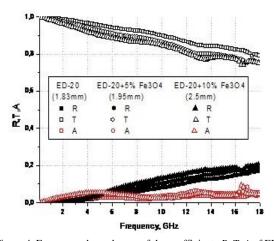


Figure 4. Frequency dependences of the coefficients R, T, A of ER composites

According to the Figure 4 the magnetite composite has a larger absorption coefficient than the empty ED matrix. It should be noted that in order to correctly compare the RTA coefficients, the thickness of the composite samples should be approximately the same. Nevertheless, analysis of the frequency dependences of the refractive index squared of these composites (Figure 3) shows that the increase in the absorption coefficient is due precisely to the presence of magnetite in the material.

III. CONCLUSIONS

To sum up nanoparticles of magnetite Fe₃O₄ (the size of a nanoparticle 30-40 nm) was synthesized by a chemical precipitation method; composites based on epoxy resin with various mass ratios $(1\% Fe_3O_4+99\% EP;$ 3%Fe₃O₄+97%ER; 5%Fe₃O₄+95%ER, 10%Fe₃O₄+90%ER) of nano-magnetite were prepared. It was found that the maximum values of the absorption coefficient (about 10%) were observed for composites with the maximum content of the magnetite. Thus, the materials studied are of interest for electromagnetic applications, including for the development of coatings absorbing microwave radiation.

REFERENCES

- [1] W. Valente, M.H. Amaral, A. Raizer, "EMC Management: How to Compare Electromagnetic Environmental Measurements and Equipment Immunity Levels", Progress in Electromagnetics Research Letters, Vol. 18, pp. 165-177, 2010.
- [2] D.D.L. Chung, "Materials for Electromagnetic Interference Shielding", J. Mater. Eng. Perform., Vol. 9, pp. 350-354, 2000.
- [3] M.N. Afsar, J.R. Birch, R.N. Clarke, G.W. Chantry, "Measurement of the Properties of Materials", Proceedings of the IEEE, Vol. 74, pp. 183-199, 1986.
- [4] E.F. Knott, J.F. Shaffer, M.T. Tuley, "Radar Cross Section", Artech House, 2004.
- [5] X. Huang, Z. Dou, S. Chen, L. Jiang, "Electromagnetic Interfering Shielding of Aluminum Alloy-Cenospheres Composite", Journal of Materials Science, Vol. 42, pp. 2633-2636, 2007.
- [6] G. Wu, X.G. Zhang, Z.Q. Song, B. Liu, "Analysis on Shielding Performance of Metallic Rectangular Cascaded Enclosure with Apertures", Progress in Electromagnetics Research Letters, Vol. 20, pp. 185-195, 2011.
- [7] J.Z. Lei, C.H. Liang, Y. Zhang, "Study on Shielding Effectiveness of Metallic Cavities with Apertures by Combining Parallel FDTD Method with Windowing Technique", Progress in Electromagnetics Research, Vol. 74, pp. 85-112, 2007.
- [8] H. El Ghandoor, H.M. Zidan, M.H. Mostafa Khalil, M. I.M. Ismail, "Synthesis and Some Physical Properties of Magnetite (Fe₃O₄) Nanoparticles", Int. J. Electrochem. Sci., Vol. 7, pp. 5734-5745, 2012.
- [9] H. Jia, W. Li, H. Yu, "Polymeric Nanocomposites for Electromagnetic Wave Absorption", J. Mater Sci., Vol. 44, pp. 3917-3927, 2009.
- [10] T. Zhang, D. Huang, Y. Yang, F. Kang, J. Gu, "Fe₃O₄ Carbon Composite Nanofiber Absorber with Enhanced Microwave Absorption Performance", Materials Science and Engineering B, Vol. 178, pp. 1-9, 2013.
- [11] J. Njuguna, K. Pielichowski, J.R. Alcock, "Epoxy-Based Fibre Reinforced Nanocomposites", Adv. Eng. Mater, Vol. 9, No. 10, p. 835, 2007.
- [12] http://etd.lib.metu.edu.tr.

[13] P. Liu, H. He, G. Wei, X. Liang, F. Qi, F. Tan, W. Tan, J. Zhu, R. Zhu, "Effect of Mn Substitution on the Promoted Formaldehyde Oxidation over Spinel Ferrite: Catalyst Characterization, Performance and Reaction mechanism", Applied Catalysis B Environmental, 2016. [14] T.A. Laxminarayana, S. Kumar Manna, B.G. Fernandes, N. Venkataramani, "Study of AC Magnetic Properties and Core Losses of Fe/Fe₃O₄-Epoxy Resin Soft Magnetic Composite", Physics Procedia, 2015.

BIOGRAPHIES



Arif Mamed Hashimov was born in Shahbuz, Nakhchivan, Azerbaijan on September 28, 1949. He is a Professor of Power Engineering (1993); Chief Editor of Scientific Journal of "Power Engineering Problems" from 2000; Director of Institute of Physics of

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Musa Abdulali Nuriyev was born in Hanifa, Balaken, Azerbaijan on April 10, 1954. He has graduated in the B.S. from Azerbaijan State University (now Baku State University), the PhD in Phys.-Math. The basic scientific fields of his research interests are

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Mazahir Nasraddin Bayramov was born in Baku, Azerbaijan on March 03, 1951. He has graduated in the PhD in physics. The basic scientific fields of his research interests are studying the process of modification of polymer composite materials (fibre

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