

Microwave Properties of Carbon Magnetic Shell Structures, Based on Glass Microspheres, With a Coating Consisting of Ferromagnetic Compounds and Nanocarbon

O.S. Yakovenko*, L.Yu. Matzui, L.L. Vovchenko, O.V. Turkov, V.V. Olyinyk, O.V. Zhuravkov

Taras Shevchenko National University of Kyiv, 64/13, Volodymyrska Str., 01601 Kyiv, Ukraine

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In the present work, regularities of changes in the frequency dependence of microwave shielding properties of fabricated 2D and 3D metastructures with periodic lattices based on glass microspheres coated with GNPs, GNPs-NiFe, MoS₂ and a mixture of MoS₂ + 20 % GNP were established. The influence of the size of carbon-based globular structures, the type of globule coating and the type of polymer matrix on the value and balance between the indices of electromagnetic radiation reflection, absorption and transmission in the frequency range of 25-37 GHz is considered. Glass balls with a diameter of 600 μm and 1.8 mm were used as a glass core. It was established that the increase in the size of glass microspheres leads to an increase in the shielding efficiency, and a large amount of GNP or GNPs-NiFe in the 3D structure contributes to the effective attenuation of electromagnetic radiation by increasing the reflection and absorption of microwaves. The presence of NiFe magnetic component on the glass microspheres leads to an increase in the absorption coefficient *A*, while the reflection coefficient *R* decreases compared to composites based on glass microspheres covered only with GNPs.

Keywords: Microwave Properties, Polymer Nanocomposites, Shell Structures, Ferromagnetic, Nanocarbon, Reflection Loss, Absorption, NiFe, MoS₂.

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1. INTRODUCTION

Electromagnetic pollution from electromagnetic interference (EMI) of waves in the microwave range is harmful to commercial devices, biological systems, high-end information technology, and defense security technologies. Therefore, the development of high performance EMI shielding materials has attracted wide attention [1, 2]. One of the approaches in the creation of composite materials (CMs) that effectively absorb microwave radiation is the use of different types of nanoparticles as fillers, the specific properties of which ensure an increase in absorption efficiency. The combination of nanocarbon 1D, 2D fillers with high dielectric (BaTiO₃, TiO₂) [3-5] and magnetic particles (Fe, Fe₃O₄) [6, 7] significantly expands the possibilities for changing the electrodynamic parameters of these CMs both due to the combined action of the materials themselves and due to the influence on the level of interphase interaction, synergistic effect, formation of an electrically conductive cluster in the CMs.

One of the modern directions of obtaining materials with high absorption characteristics, in contrast to the approach discussed and studied by us earlier, based on the formation of CMs with multicomponent fillers, is the idea of using spherical particles covered with a layer of another material as fillers [8, 9]. Such a specific structure allows the use of different types of materials as a "core" and "shell", thereby providing the possibility of varying their properties. These structures can not only make the incident microwave radiation generate multiple reflections, refractions and scattering to attenuate the electromagnetic energy through a prolonged propagation path, but also increase the dielectric loss due to space charge polarization, interfacial polarization and synergistic effect.

Currently, various types of shell structures with high absorption properties, for example, Fe₃O₄/CoFe₂O₄, Fe₃O₄/ZnO, SnO/FeNi, etc., including hollow, porous shells, are intensively researched. As a new type of lightweight material, hollow glass microspheres (HGMs) have shown a good potential for functional applications with a high compressive strength, high corrosion resistance, low thermal conductivity, low thermal shrinkage coefficient and other advantages [10-13]. Coating of HGMs by metallic or magnetic materials give a good opportunity to use them for electromagnetic shielding with the ultralight characteristic [14]. However, using the metallic materials in composites usually have limitations in terms of application in the field requiring lightweight.

In this paper, we report the results of the study of the microwave shielding properties of the composites based on glass microspheres coated by graphite nanoplatelets (GNPs) and GNPs decorated with NiFe and MoS₂ nanoparticles. The influence of the size of the carbon-based globular structures, the type of coating of the globules and the type of the polymer matrix, on the value and balance between the EMR reflection, absorption and transmission have been established.

2. MATERIALS AND METHODS

2.1 Brief Characteristics of the Materials

Glass microspheres (GM) with a diameter of 600 μm and 1.8 mm and a density of 2.3–2.8 g/cm³ were used, the main components being SiO₂ (50–60 %) and Al₂O₃ (27–35 %).

As a coating on the surface of GM, we used graphite nanoplatelets (GNPs), GNPs decorated with NiFe and MoS₂ nanoparticles.

*alena-ya@ukr.net

GNPs were obtained by thermo-exfoliation of oxidized natural graphite with following ultrasonication (in Baku 9050 device with a maximum output power of 50 W and an ultrasound frequency of 40 kHz) in acetone medium for 3 hrs [15]. Thermally exfoliated graphite (TEG) was obtained by rapid heating (thermal shock) of intercalated graphite compounds, which leads to "swelling" of graphite particles due to evaporation and decomposition of the intercalant. The wall thickness of the individual flake of the TEG can be estimated as 20-40 nm, the pore size ranges from 5-10 μm . The TEG particles are long, about 3-5 μm , and have diameters of 50-100 μm . The peculiarity of TEG particles morphology is the worm-like shape and the layered structure. In order to obtain GNPs, ultrasonication was performed until complete destruction of the TEG worm structure is achieved, after which the dispersed GNPs were dried at room temperature till the complete evaporation of the acetone. GNPs are disk-shaped plates with the lateral dimensions of 1–10 μm and the thickness of 15–45 nm with the maximum of thickness distribution at 30 nm [16].

Method of salt impregnation was used for fabrication of FeNi-decorated GNPs [17]. Metal powders of the reagent grade iron (Fe, 99.95 %) and nickel (Ni, 99.95 %) were supplied from Aldrich. Concentrated Merck's nitric acid (65 vol. % HNO_3) was used for preparation of metals nitrate solution. Two gases were used for reduction of metals oxides: hydrogen (H_2) taken from the Flame Ionization Detector (FID) gas station (hydrogen generator) and helium (He) taken from balloons, supplied from Linde Gas Ukraine, 99.995 vol. %. Before feeding, H_2 was dehydrated in a dry scrubber system.

The typical preparation procedure for FeNi-decorated GNPs with 60 wt. % of FeNi alloy (nominal content of 80 wt. % of Ni and 20 wt. % of Fe - $\text{Fe}_{20}\text{Ni}_{80}$) is as follows.

First, a mixture of 1 g of Fe and 4 g of Ni pure metal powders was dissolved in 50 ml of 65 vol. % HNO_3 and refluxed using a sand bath for 30 min. When both metals were completely dissolved, the nitrate solution was cooled down to room temperature. The appropriate mass of GNPs was impregnated by the nitrate solution (at 60 wt. % of metal component per GNPs). Then, the obtained wet substance was dried in a sand bath. A porcelain dish was used for the evaporation of the rests of the solvent at 100 $^\circ\text{C}$ for 4 hrs and the following drying in a muffle at 350 $^\circ\text{C}$ for 4 hrs to yield a mixture of Fe_2O_3 and NiO powders. The reduction of the obtained oxides to Ni-Fe metal mixture was performed by the gas stream. As-received, $\text{Fe}_{20}\text{Ni}_{80}$ -decorated GNPs were characterized using SEM and X-ray instruments. The results of SEM investigations (at different magnification) of the morphology of $\text{Fe}_{20}\text{Ni}_{80}$ -decorated GNPs powder, preliminary dispersed in ethanol, are presented at SEM images in Fig. 1.

Fig. 1 a, b shows general view of GNPs- $\text{Fe}_{20}\text{Ni}_{80}$ powder at low magnification. As it is seen in Fig. 1 c, d, the metal component of powder is in the form of granules and is fairly evenly distributed over the surface of the GNPs plates. $\text{Fe}_{20}\text{Ni}_{80}$ nanoparticles are mostly rounded and they are approximately of 20-40 nm in diameter; the nanoparticles agglomerations with sizes of 100-200 nm are found though. Also, there are some areas of metallic phase accumulation (lighter areas of the image) as agglomerates of different shapes: spheres, spiculae, and others (Fig. 1 d). SEM images of nanodispersed $\text{Fe}_{20}\text{Ni}_{80}$ -decorated GNPs at higher magnification (Fig. 1 c, d) show that the metal component is distributed not only on the side surfaces of GNPs but also on its edges.

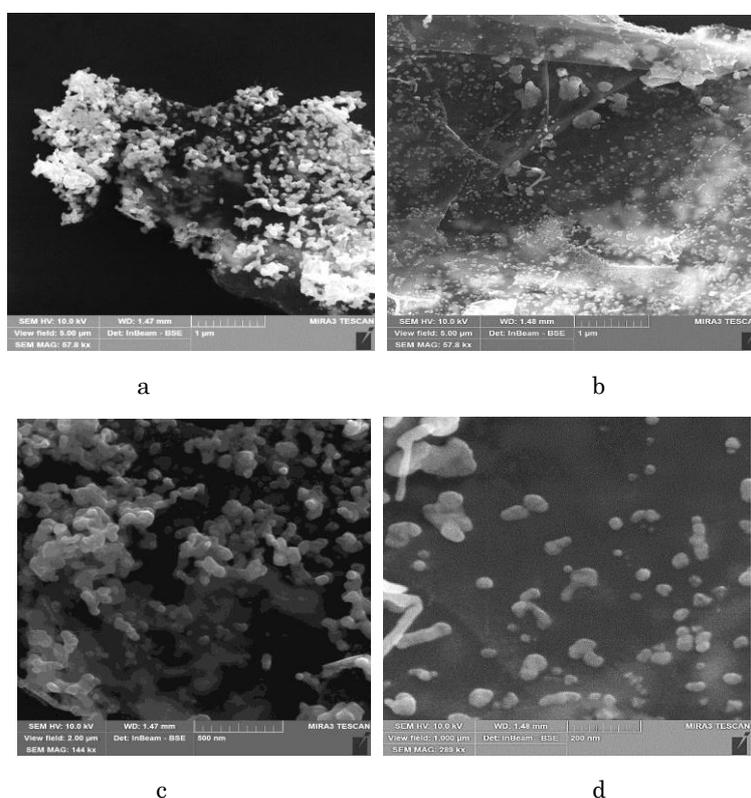


Fig. 1 – SEM images of GNPs decorated by $\text{Fe}_{20}\text{Ni}_{80}$ at different magnifications: a)-57800x, b)-57800x, c)-144000x, d)-280000x

MoS₂ powder (the lateral dimensions of which were 4-70 nm; No. 808652 Sigma-Aldrich, 99.995 %) was used as a dielectric shell. Carbonyl iron powder was purchased from Ukrsplyv Inc. Individual Fe particles have an irregular lamellar shape, and the average lateral size of the particles is about 3-7 μm, while the thickness varies from 0.6 to 2 μm.

Larit 285 epoxy resin or Larit 285 epoxy resin with 60 wt. % of iron nanoparticles was used as a polymer matrix in the fabrication of composite samples.

2.2 Methods of Obtaining of Glass Microspheres-Based Composites with a Carbon Magnetic Coating and Studying of their Morphology by Optical Microscopy

To obtain 2D-3D composites with periodic gratings which are based on glass microspheres (GM) coated with GNPs, GNPs decorated with FeNi (GNP-FeNi), MoS₂ and a mixture of MoS₂ + 20 % GNPs, a mixture of microspheres and coating nanoparticles were homogenized by thorough mixing using an IKA ULTRA TURRAX TubeDrive homogenizer in test tubes with a stirrer. The rotation speed was 3000 rpm. To determine the optimal mixing time to create a carbon layer on the surface of the polymer

particles, mixing was carried out for 10, 20, 30, 40, 60 min., and then the glass balls were examined under an optical microscope. It was determined that 40 min. is sufficient mixing time, during which the nanocarbon coating powder is evenly distributed around the glass spheres. Fig. 2 shows optical microscopic images of GM coated with GNP after 20 min (left) and 40 min (right) of mixing. The thickness of the coating during 60 min of mixing is ~1 μm.



Fig. 2 – Optical microscopic images of GNP-coated GM after 20 min (left) and 40 min (right) of mixing

To obtain composites with a 2D structure, GM coated with GNPs, GNPs-NiFe, MoS₂ and MoS₂ + 20 % GNP were arranged in 1 layer, forming a periodic 2D structure. To prepare 3D structures, GM coated with GNP, GNP-NiFe, MoS₂ and MoS₂ + 20 % GNP were stacked in 3 layers as shown in Fig. 3.

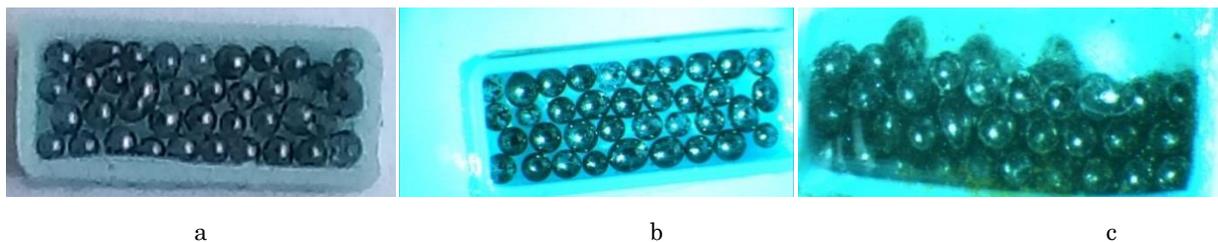


Fig. 3 – Composites with 2D structure of GM (top view) (a), (b); composites with 3D structure of GM – 3 layers (side view) (c)

The list of fabricated composite samples with 2D and 3D structure is given in Table 1.

Table 1 – The list of fabricated composite samples with 2D and 3D structure

Number of sample	The composition of the coating of microspheres	Filling material	Number of layers	Size of glass microspheres
N1	20 %GNPs	Wax	1	0.6
N2	20 %(GNPs/60%(Ni ₈₀ Fe ₂₀))	Wax	1	0.6
N3	20 %GNPs	L285	3	0.6
N4	20 %(GNPs/60%(Ni ₈₀ Fe ₂₀))	L285	3	0.6
N5	20 %MoS ₂	L285	3	0.6
N6	20 %(MoS ₂ 20 %GNPs)	L285	3	0.6
N7	20 %GNPs	L285 + Fe 30 %	3	0.6
N8	20 %MoS ₂	L285 + Fe 30 %	3	0.6
N21	20 %GNPs	L285	1	1.8
N22	20%(GNPs/60%(Ni ₈₀ Fe ₂₀))	L285	1	1.8
N23	20 %GNPs	L285	3	1.8
N24	20 %(GNPs/60%(Ni ₈₀ Fe ₂₀))	L285	3	1.8
N25	20 %GNPs	L285 + Fe 30 %	3	1.8

2.3 Research Methods of the Structure and Microwave Properties of Composites

The structural and morphological features of filler particles and of filled epoxy composites were investigated using optical microscopy ("Mikmed-1" with an ETRK PCM-510 attachment).

The electromagnetic shielding properties of the

composites were tested in the frequency range of 26-37 GHz at room temperature using P2-65 scalar microwave network analyzer. The rectangular waveguides have the following cross sections: 7.2 × 3.4 mm². Samples for research were cut from the billets of investigated composites using a milling machine with numerical control positioning resolution

of which is 0.025 mm. The shape of the investigated samples provided complete filling of cross sections of the above mentioned waveguides while the samples thicknesses were 1 mm.

The physical parameters for evaluating of electromagnetic shielding performance are: the total SE (SE_T), reflection SE (SE_R), and absorption SE (SE_A).

These parameters can be calculated using microwave scattering parameters (S_{11} and S_{21}) by equations (1)-(6). The absorption (A), transmission (T), and reflection (R) coefficients for an incident EM wave on a layer of EMI shielding material, as well as the corresponding SE_A , SE_R , and SE_T , are [18-21]:

$$R = S_{11}^2, \quad (1)$$

$$SE_R = 10\log(1 - R), \quad (2)$$

$$T = S_{21}^2, \quad (3)$$

$$SE_T = 10\log(T), \quad (4)$$

$$A = 1 - R - T, \quad (5)$$

$$SE_A = 10\log\frac{T}{1-R}, \quad (6)$$

where EMR transmission (T) and reflection (R) indices are determined as $R = |E_R/E_I|^2$, $T = |E_T/E_I|^2$, where E_I , E_R , E_T are the electric field strengths of the incident, reflected, and transmitted waves, respectively.

3. RESULTS AND DISCUSSION

The study of the microwave shielding properties of the fabricated 2D and 3D metastructures was carried out in order to establish the influence of: 1) the size of the carbon-based globular structures, 2) the type of coating of the globules and 3) the type of the polymer matrix, on the value and balance between the EMR reflection, absorption and transmission indices.

Data on the shielding efficiency and the ratio of reflection, absorption and transmission of EMR of composites with 2D/3D structures of GM depending on the type of covering of the shell structures are presented in Fig. 4. To analyze the influence of the type of coating and the size of the GM, the effectiveness of SE_T shielding (Fig. 4 a, b) and the ratio between the EMR reflection, absorption and transmission indices (Fig. 4 c, d) of different types of composites with 2D (Fig. 4 c) and 3D (Fig. 4 d) structure of GM were investigated in the frequency range of 25-37 GHz using the GM with a diameter of 600 μm and 1.8 mm. Glass-based globules with different shells, namely GNPs and GNPs-NiFe were investigated in the frequency range of 25-37 GHz.

As can be seen from Fig. 4, shielding efficiency, SE_T , for GNPs-coated microspheres with 2D structure, as well as for the 3D structure, is not high and practically does not depend on the glass size, which can be explained by the very thin GNP coating on the glass balls. In addition, the coating of glass microspheres with GNPs-NiFe leads to more effective attenuation of EMR compared to the material consisting of GM-GNPs. In this case, the effectiveness of SE_T shielding increases significantly; 2-4 times for 3D structures in relation to composites with 2D

structure. Increasing the size of the GM also results in increased shielding efficiency. Such a large difference in SE_T is associated with a larger thickness of 3D structure ($\sim 2.7 / (5.4)$ mm for GM with the size of 0.6 mm and 1.8 mm correspondingly) and a thicker conductive coating on the surface of the polymer globules. Thus, the large amount of GNPs or GNPs-FeNi in this 3D structure contributes to the effective attenuation of EMR by increasing the reflection and absorption of microwaves.

It should also be noted that the presence of the NiFe magnetic component on GM leads to an increase in the absorption coefficient A , while the reflection coefficient R decreases compared to composites based on GNPs-coated GM. For 2D structures, the A/R ratio is much larger for all the studied structures and reaches a maximum in the region of 29-30 GHz for (GNP-FeNi)/L285 composites (Fig. 4.3 e, f). Such an increase in the absorption coefficient A may be associated with additional magnetic losses, as well as electrical losses and EMR scattering at the glass/GNPs/NiFe interfaces.

For further analysis, the effect of the type of coating, the size of the glass balls and the character of their location in the sample on the absorption properties of the developed materials, have been studied. The results of investigation of the frequency dependence of RL , reflection losses, are shown in Fig. 5. It is known [22] that when an electromagnetic wave vertically falls on a sample, the reflection loss RL depends on the material parameters ε' , ε'' , μ' , μ'' , sample thickness d and EMR frequency f . A good microwave absorber requires two basic conditions: when the microwave is incident on the surface of the absorber, the direct reflection of the microwaves must be minimal; at the same time, it is possible that microwaves propagate in the absorber. This requires the input impedance Z_{in} to be equal to the free space wave Z_0 (377 Ohms). In addition, according to a previous study [23], for a single-layer absorber, the RL minimum occurs due to the suppression of two microwaves at the front surface of the absorber. Two waves: the reflection wave, which is reflected at the front interface of the absorber, and the outgoing wave, which is reflected by the metal reflector.

The minimum RL directly depends on the intensity of two waves. At the point of minimum reflection, the input impedance Z_{in} is assumed to be equal to the free-space wave impedance Z_0 , which means that the imaginary input impedance Z_{in-im} approaches zero ohms, while the corresponding real input impedance Z_{in-re} approaches 377 Ohms [24].

As can be seen from Fig. 5, for composites GM/GNPs/L285 and GM/(GNPs-NiFe)/L285 based on microspheres with a diameter of 0.6 mm, RL is more than -10 dB in the entire studied frequency range, i.e. more than 90 % of the introduced EMR is absorbed, which is the target value for electromagnetic absorbers from an industrial point of view. A clearly defined RL minimum for the composite GM/(GNPs-NiFe) (sample 2-1 layer of coating) is observed at a frequency of 29.1 GHz and it is -22.4 dB. An increase in the thickness of such a composite (comparing structures with one and three coating layers) leads to the fact that the minimum in the studied frequency interval shifts to the region of lower frequencies, and its depth decreases. This trend is clearly visible with an increase in the diameter of the used

microspheres for other samples as well (Fig. 5 b). Thus, for a 1-layer composite GM/GNPs (sample 1), $RL_{min} = -16.7$ dB and is observed in the frequency range of 33-34 GHz, with an increase in the diameter of the

microspheres used, i.e., with an increase in the thickness of sample, $RL_{min} = -20.0$ dB and is observed at a frequency of 27 GHz.

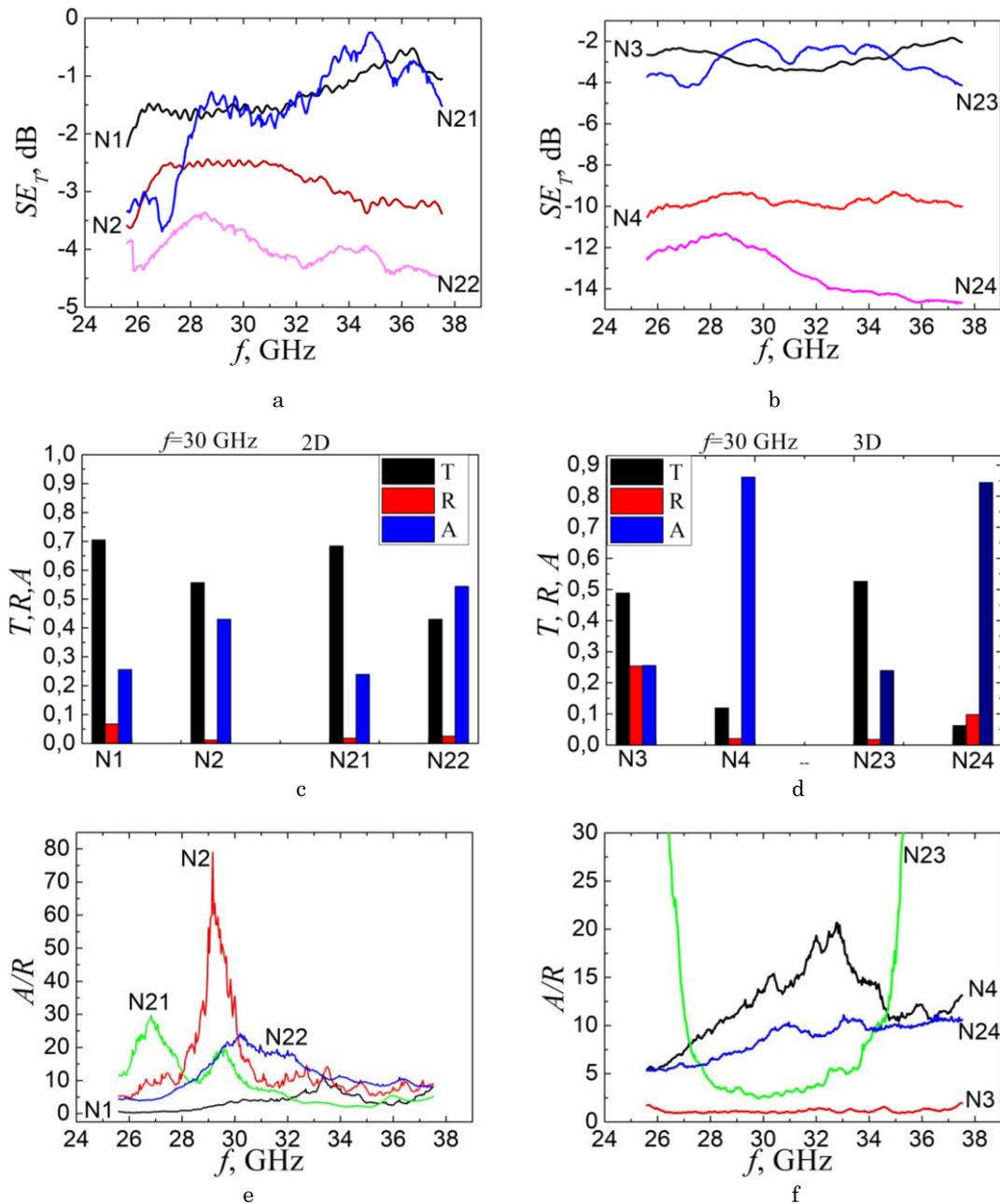


Fig. 4 – Frequency dependences of shielding efficiency SE_T (a, b), the ratio between the coefficients of EMR transmission, absorption and reflection (c, d), A/R ratio (e, f) for composites with 2D (a, c, e) and 3D (b, d, f) structure based on microspheres of different sizes (0.6)/1.8 mm covered with GNPs or GNPs/NiFe. Samples were numbered as in Table 1

The influence of the matrix type on the shielding properties was studied for 3D composites based on GM/GNPs (samples 3, 7, 23, 25), GM/MoS₂ (sample 5, 8) and MoS₂+GNP (samle 6) located in different polymer matrices: epoxy resin L285 and epoxy resin L285+30 % Fe. Fig. 6 shows the ratio between the EMR reflection, absorption and transmission indices for above mentioned 3-layer structures with polymer matrices: L285 and epoxy resin L285+30 % Fe.

As can be seen, the addition of dispersed Fe particles

to the epoxy resin leads to an increase in EMR attenuation and the maximum of SE_T shielding efficiency is observed for 3D composite GNPs/L285+Fe when GM with a diameter of 1.8 mm are used. Fig. 7 shows frequency dependences of A/R for 3-layer structures with GM/GNPs/L285 and GM/MoS₂/L285 fillers with polymer matrices: 30 % Fe in epoxy resin.

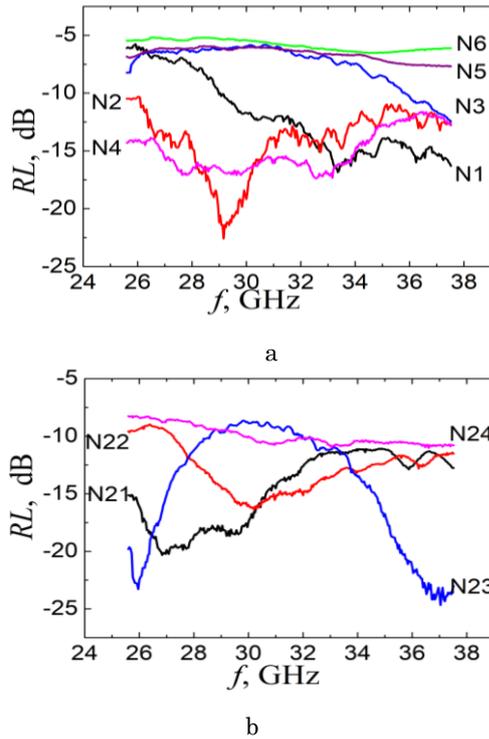


Fig. 5 – Frequency dependences of RL, reflection loss, for the investigated composites. Samples were numbered as in Table 1

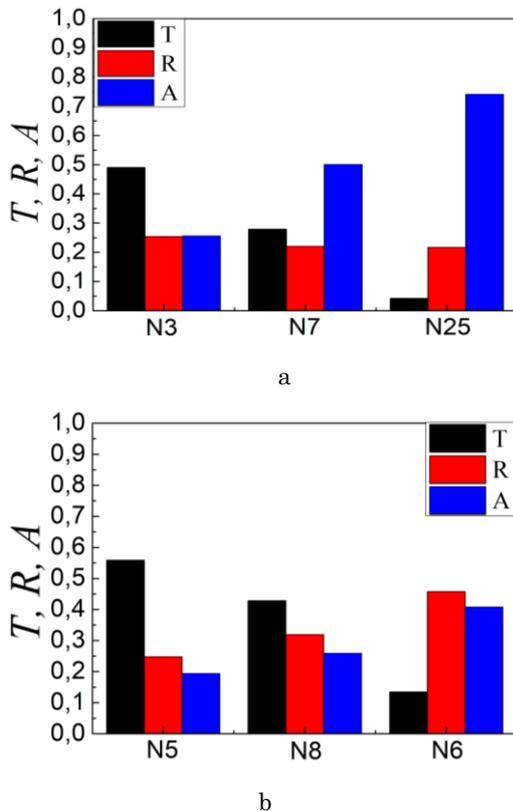


Fig. 6 – The ratio between the EMR reflection, absorption and transmittance indices for 3-layer structures with GNP (a) and MoS₂ (b) fillers with polymer matrices: epoxy resin and 30 % Fe in epoxy resin. Samples were numbered as in Table 1

As for the ratio between the EMR reflection and absorption indices, R decreases and A increases, and such a change in the A/R ratio reaches 3.4 times for the 3D composite GM/GNPs/L285+Fe for microsphere with a diameter of 1.8 mm. As can be seen from Fig. 7, with an increase in the EMR frequency, the A/R ratio increases significantly and a maximum is observed on the frequency dependence, the position of which and the value of A/R in it depend on the size of the GM, that is, on the thickness of the composite sample.

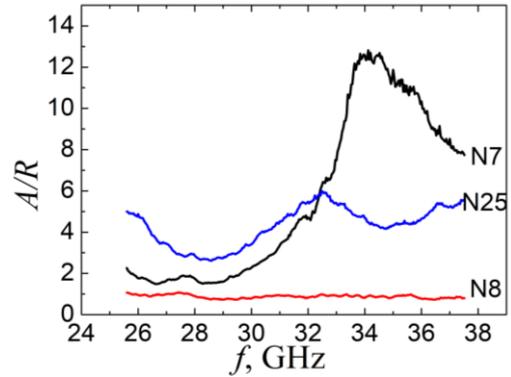


Fig. 7 – Frequency dependences of A/R for 3-layer structures with GM/GNP and GM/MoS₂ fillers with polymer matrices: 30 % Fe in epoxy resin. Samples were numbered as in Table 1

As in the case of GM/GNPs/L285 and GM/(GNPs-FeNi)/L285 2D structures, the increase in the absorption index A can be associated with additional magnetic losses (for Fe), dielectric losses (GNP) and EMR scattering at interfaces: Fe-epoxy resin, GNP-epoxy resin, GNP-Fe.

4. CONCLUSIONS

Investigation of the microwave characteristics of 2D and 3D structures based on glass microspheres of various sizes covered with a layer of GNPs and GNPs-NiFe, MoS₂ showed that RL, reflection loss, in the entire studied interval (25-37 GHz) of frequencies is more than -10 dB, and for GNPs-NiFe-coated structures based on microspheres with a diameter of 0.6 mm, a well-defined RL minimum is observed at a frequency of 29.1 GHz and it is -22.4 dB, which means that more than 98 % of the introduced EMR is absorbed, which is the target value for electromagnetic absorbers from an industrial point of view.

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Мікрохвильові властивості карбонових магнітних оболонкових структур, на основі скляних мікросфер, з покриттям, що складається з феромагнітних сполук та нановуглецю

О.С. Яковенко, Л.Ю. Мацуй, Л.Л. Вовченко, О.В. Турков, В.В. Олійник, О.В. Журавков

*Фізичний факультет Київського національного університету імені Тараса Шевченка,
вул. Володимирська 64/13, 01601 Київ, Україна*

У роботі встановлено закономірності зміни частотних залежностей мікрохвильових екрануючих властивостей виготовлених 2D і 3D метаструктур з періодичними ґратками на основі скляних мікросфер, покритих ГНП, ГНП-NiFe, MoS₂ та суміші MoS₂ + 20 % ГНП. Розглядається вплив розміру глобулярних структур на основі вуглецю, типу покриття глобул і типу полімерної матриці на значення та баланс між показниками відбиття, поглинання та пропускання електромагнітного випромінювання в діапазоні частот 25-37 ГГц. В якості скляного сердечника використовувалися скляні кульки діаметром 600 мкм і 1.8 мм. Встановлено, що збільшення розміру скляних мікросфер призводить до підвищення ефективності екранування, а також велика кількість ГНП або ГНП-NiFe у 3D структурі сприяє ефективному ослабленню електромагнітного випромінювання за рахунок збільшення відбиття та поглинання мікрохвиль. Наявність магнітного компонента NiFe на скляних мікросферах призводить до збільшення коефіцієнту поглинання A , тоді як коефіцієнт відбиття R зменшується порівняно з композиціями на основі скляних мікросфер, покритих лише ГНП.

Ключові слова: Мікрохвильові властивості, Полімерні наноконізати, Оболонкові структури, Феромагнітний, Нановуглець, Втрати на відбиття, Поглинання, NiFe, MoS₂.