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Broadband radio absorbing coating nanostructured by carbon nanotubes

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Abstract: A method for manufacturing broadband radioabsorbing coatings based on a polymer matrix modified with multi-walled carbon nanotube "Taunit-MD" for radio-frequency anechoic chamber application has been developed. It is shown that a high aspect ratio (> 1000) of carbon nanomaterials provides the formation of a developed three-dimensional conductive net in the structure of the polymer matrix at a low additive concentration, which has a positive effect on the electromagnetic energy absorption properties and ensures that the filler does not pour out. Using a 250 mm high pyramid array absorber provides more than –40 dB of reflectance attenuation in the frequency range from 1 GHz, while using a 100 mm high pyramid array absorber provides more than –30 dB of reflectance attenuation in the frequency range from 10 GHz. The full retention of the electromagnetic radiation absorption properties of the developed pyramidal absorbers was demonstrated in the wear resistance test, while the industrial sample with carbon black filler showed a decrease in the absorption of electromagnetic radiation by –20 dB.

Keywords: carbon nanotubes; polymer; composite material; radio absorbing material; electromagnetic radiation.

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Наноструктурированное углеродными нанотрубками широкополосное радиопоглощающее покрытие

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Аннотация: Разработаны методы изготовления и сформированы структурированные многослойными углеродными нанотрубками «Таунит-МД» образцы широкополосных радиопоглощающих покрытий, предназначенных для покрытия внутренних поверхностей и оборудования рабочих мест в высококачественных радиочастотных безэховых камерах. Высокое аспектное отношение (> 1000) применяемых углеродных наноматериалов позволяет сформировать в структуре пенополиуретановой матрицы развитую трехмерную проводящую сеть при низкой концентрации добавки, что положительно сказывается на эффективности поглощения электромагнитной энергии и обеспечивает отсутствие сыпучести наполнителя. При использовании поглотителя с конфигурацией в виде массива пирамид высотой 250 мм обеспечивается ослабление коэффициента отражения более чем на – 40 дБ в диапазоне частот от 1 ГГц, при использовании поглотителя с конфигурацией в виде массива пирамид высотой 100 мм, обеспечивается ослабление коэффициента отражения более чем на –30 дБ в диапазоне частот от 10 ГГц. Испытания на износостойкость продемонстрировали полное сохранение поглощающих характеристик разработанных поглотителей, в то время как для промышленного образца с техническим углеродом наблюдалось ослабление поглощающей способности на –20 дБ.

Ключевые слова: углеродные нанотрубки; полимер; композитный материал; радиопоглощающий материал; электромагнитное излучение.

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1. Introduction

When designing antennas and other transceiver devices, radio engineers should obtain accurate experimental data on radiation patterns, at the same time, these data can be distorted by background electromagnetic radiation. In order to eliminate the influence of background electromagnetic radiation (EMR), experiments are carried out in specialized rooms – the so-called radio frequency (RF) anechoic chambers [1].

The main working elements of such chambers are pyramidal-type radio-absorbing materials (RAMs) based on polyurethane foam matrices impregnated with a special composition of carbon and ferrite particles [2].

The operation mechanism of such materials is to attenuate short-wave EMR due to multiple reflection of the wave in the cavities of the pyramids. In the long-wavelength part of the range, when dimensions of pyramids are much smaller than the wavelength of the incident radiation ($< \lambda/4$), similar RAMs do not differ in their characteristics from single-layer absorbers [1].

The standard technology for manufacturing anechoic chamber absorbers involves using profiled polyurethane foam blanks, which are impregnated with a special composition containing carbon black particles or a mixture of carbon and ferrite particles. In the literature, there are other approaches and methods for the formation of pyramidal RAM aimed at improving functional properties.

For example, to ensure more efficient absorption, the authors of [3] considered the creation of longitudinal corrugations with a depth of 0.25–0.375 wavelengths on the side surface of 45 cm wedge-shaped RAMs. A decrease in the reflection coefficient depending on the depth of corrugations did not affect a significant improvement in performance. The reflection coefficient in the frequency range 100 MHz – 5 GHz was about –30 dB. The authors of [4] also considered an approach with a modification of the pyramid structure. They made pyramids 40 cm high and 20 cm wide with various configurations of absorbing cavities inside the pyramids. Measurements of the reflectivity of the pyramids did not show any improvement in performance, and under some irradiation configurations even led to an increase in reflection. In general, the average level of the reflected signal was at the level of –20 dB in the range of 8–12 GHz.

The authors of [5], using 3D printing technology, formed a hollow absorber with a honeycomb volumetric structure and a pyramidal profile. With a pyramid height of 82 mm and a total product height of 87 mm, the absorber demonstrated the ability to attenuate reflected EMR in the 2–18 GHz range at a level not exceeding –20 dB.

The authors of [6] proposed to combine different types of absorbing products to minimize the reflection coefficient. Pyramidal RAMs, needle-shaped RAMs, and multi-walled RAMs were made. Individually, these materials exhibit EMR absorption at 20, 15 and 10 dB in the range of 1–18 GHz. The combination of multi-walled, pyramidal and needle-shaped coating reduces the reflectance to a minimum of –30 dB, in general, the reflectance shows values at the level of –40 dB.

It was possible to achieve success in the issue of increasing the efficiency of EMR absorption due to the use of a metamaterial substrate with alternating serpentine and helical elements [7]. Additional attenuation of the reflected EMR relative to the pyramidal RAM on a metal substrate was –5÷–10 dB in the frequency range 2–18 GHz.

Such scattering absorbers are used as the main working elements of RF anechoic chambers designed to simulate free space and create a controlled electromagnetic environment in the design and testing of various transceiver electronic equipment [1]. With the development of communication technologies, testing of radio engineering complexes in such chambers is becoming more common due to the fact that it allows to completely eliminate full-scale measurements, which affects cost savings, and the accelerated development of complex equipment.

At the same time, in contrast to free space, in such chambers, the issue of diverting the radiated power can be especially acute. For example, for a massive phased array antenna, the radiation power density due to the extremely narrow beam width and extremely high output power can reach 10 $kW \cdot m^{-2}$ [8]. Therefore, when developing such materials, it is necessary to pay attention to fire safety. As a rule, while working in powerful electromagnetic fields, pyramidal radio absorbers are additionally impregnated with special fire-fighting additives.

However, there are other approaches that provide not only efficient heat removal, but also further facilitate the design. Thus, the authors of [9, 10] fabricated honeycomb absorbers with a wedge-shaped configuration at the top of the product. With a height of 100 mm, these absorbers provide attenuation of the reflected EMR by –40 dB in the range of 18–40 GHz.

The analysis of the above works allows us to conclude that the existing methods for creating pyramidal RAMs are able to demonstrate the maximum attenuation of the reflected signal at the level of –40÷–50 dB, which is certainly an excellent result. At the same time, various modifications of side faces of pyramids lead to more complex production than to an increase in absorption efficiency. And achievement of the required level of attenuation of electromagnetic radiation (–40 dB), as in industrially produced RAMs, is ensured by a large amount of working filler – at least 60 wt. %, which negatively affects mechanical and elastic properties of the product [11, 12].

Therefore, in this paper, in order to improve the performance and absorption efficiency, the use of carbon nanotubes (CNTs) is considered. Due to the creation of developed conductive networks in the structure of polymer matrices [13], these nanomaterials will significantly reduce the concentration of the filler to ensure effective absorbing characteristics and, at the same time, this will preserve elastic mechanical properties.

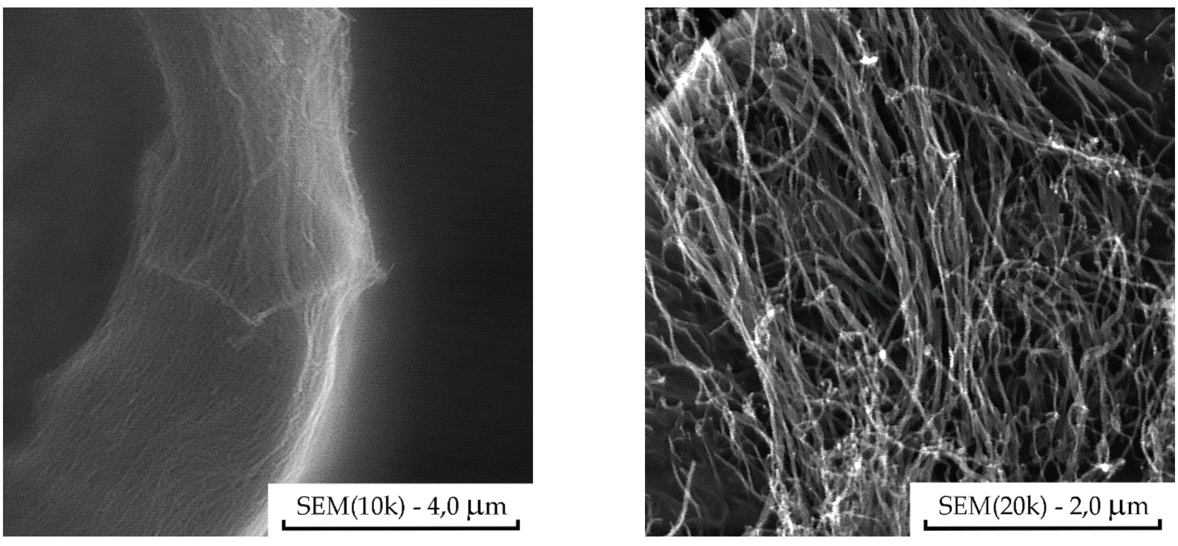
2. Materials and Methods

The object of the study is pyramidal radio absorbing material based on a polyurethane matrix (air volume is 90 %, density is 90 kg·m^{-3}, compression resistance is 5 kPa, pore number is from 80 to 100 ppi, manufacturer is Trest No. 15 Spetsstroy OJSC, Minsk, Belarus) structured with Taunit-MD multilayer carbon nanotubes (NanoTechCenter LLC, Tambov, RF) [14].

Based on the analysis of microphotographs of samples obtained by scanning electron microscopy (SEM) using a Hitachi H-800 electron microscope, it has been found that these structures are characterized by a high length of individual nanotubes (more than 20 μm according to estimates), with relatively small thicknesses, an outer diameter of 10–20 nm, uniform wall sizes and an ordered structure of the array. A pronounced orientation of nanotubes is observed. Sometimes there are formations in the form of bundles with a thickness of 1 to 5 μm (Fig. 1).

A water-acrylic solution of nonionic surfactants “OP-7” (OOO “Sintez-OKA”, Dzerzhinsk, RF) was used as a binder.

The manufacturing process of a pyramidal absorber based on polymer matrices with the addition of carbon nanostructures consisted of the following stages: 1) the formation of test samples of composites to minimize costs in the development of radio absorbers, 2) the study of their electrical characteristics by the transmission line method in the operating frequency range, 3) the selection of the geometric configuration of the absorber, 4) building a model in a software environment based on the data of points 2 and 3 to determine the minimum concentrations of carbon nanostructures required to ensure effective attenuation of the reflected electromagnetic signal, 5) synthesis of a full-scale sample.



(*a*) (*b*)

Fig. 1. SEM images of carbon nanotubes “Taunit-MD”

During the formation of test samples of composites, the problem of creating a homogeneous dispersion of carbon nanotubes is solved. The nature of the dispersion problem for nanosized carbon additives is quite different from other conventional fillers such as spherical particles and carbon fibers because additives such as carbon nanotubes hace a high aspect ratio (> 1000) and an extremely large surface area. As a result, the number of nanosized particles in a given volume at the same mass fractions will exceed the number of microfillers by orders of magnitude [15].

In addition to the size effect when filling materials with a filler, the physical nature of particles also plays an important role in their dispersion in the polymer matrix. CNTs, due to the van der Waals force, can fold into bundles consisting of several hundred individual nanotubes. It has been shown that these bundles and agglomerates lead to a decrease in mechanical and electrical properties of composites compared to theoretical predictions associated with individual CNTs [16]. We also note the fact that carbon atoms on the walls of materials such as CNTs and graphene are chemically stable due to the aromatic nature of the bond. As a result, CNMs are inert and can interact with the polymer matrix mainly due to the forces of electrostatic interaction, which is unable to provide stable bonds through the CNM/polymer interface [17, 18].

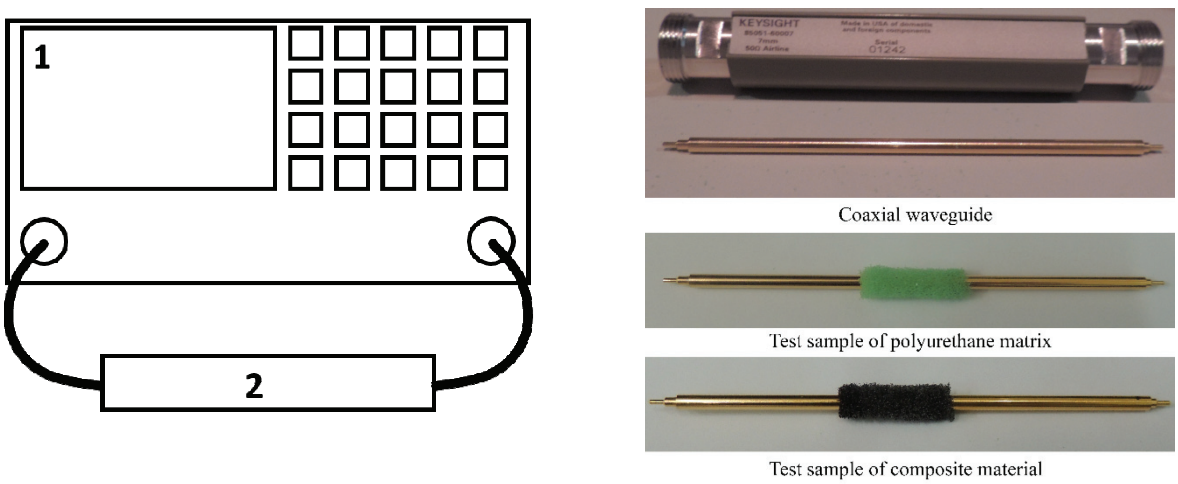
To take into account the above features, the dispersed filler “Taunit-MD” was preliminarily distributed in a 50 mM aqueous solution of anionic surfactants (OP-7) and an acrylic polymer matrix using an ultrasonic homogenizer with a frequency of 24 kHz for 30 minutes with a gradual increase in power up to 80 W. Due to the presence of a benzene ring in the structure and an extended alkyl tail with 8–12 carbon atoms, significant adsorption of surfactant molecules on the graphite surface is ensured due to the “pi-stacking” interaction and repulsion of the adsorbing layer from the surface of surrounding nanoparticles [19].

The use of an acrylic polymer in the liquid phase in combination with a surfactant makes it possible to “glue” MWCNTs to the polyurethane matrix during polymerization.

Upon completion of the procedure for the formation of a water-acrylic dispersion, the procedure for impregnating the polyurethane foam matrix took place. As a result, a number of samples of composite materials were formed with a mass concentration of filler from 1 to 8 wt. %.

The next step was to study the electrical characteristics, complex dielectric and magnetic permeability of the formed composite samples. For these purposes, a hardware-software complex based on the N5290A Keysight Technologies vector network analyzer was applied using the transmission line method in a coaxial waveguide (Fig. 2*a*). The research frequency range was from 1 to 18 GHz. The output data were complex S-parameters describing the ratio of the transmitted and reflected electromagnetic wave to the incident one. On the basis of the registered data, according to the NIST method [20], the parameters of the material were calculated.

As mentioned in the introduction, the operation mechanism of pyramidal RAMs is to attenuate the incident electromagnetic reflection due to multiple reflections in its cavities. Thus, the geometric configuration of the absorber will play a key role in providing absorbing properties in the required frequency range.



(*a*) (*b*)

Fig. 2. Scheme of a measuring system for studying the parameters of materials by the transmission line method (*a*): *1* – network analyzer; *2* – coaxial waveguide with sample (*b*)

The geometrical parameters of the absorber were selected on the basis of calculation by the formulas for the vertical displacement of the beam and the exit of the beam from the absorbing cavity, which made it possible to determine *N* minimum number of re-reflections of a plane wave before it completely left the absorbing cavity [1]:

$$h_n = \frac{2\left(H - \sum_{i-=1}^{n} h_i\right)\mathrm{tg}\frac{\alpha}{2}}{\mathrm{tg}\theta_{n-1} + \mathrm{tg}\frac{\alpha}{2}}, \quad (1)$$

where $\theta_{n-1} = \alpha(n-1) + \theta$, α is the angle of the pyramid base, θ is the incidence angle of the wave, *H* is the height of the pyramid.

$$\sum_{i-=1}^{N+1} h_n < 0. \quad (2)$$

For scattering absorbing materials, configurations consisting of an array of pyramids 75 mm high, a base length of 25 mm and a substrate thickness of 25 mm, operating frequencies from 10–12 GHz were selected, as well as RAMs in the form of an array of pyramids 190 mm high, a base length of 60 mm, substrate thickness 60 mm with operating frequencies from 1 GHz were used. Calculation according to the formulas for the vertical displacement of the beam and the conditions for the beam to leave the absorbing cavity shows that at incidence angles of an electromagnetic wave of 0°, 15°, 30°, 45°, the critical beam undergoes 7, 5, 4, 3 re-reflections in the cavities for 100 mm RAM, and 11, 9, 8, 6 reflections for 250 mm RAM, respectively.

On the basis of the electrophysical data and the selected geometric configurations, a model of absorbers was subsequently constructed [21] and optimal concentrations of carbon nanostructures required to ensure effective attenuation of the reflected electromagnetic signal were determined.

To specify the structure of the absorber, we considered an elementary cell in the form of a pyramid on a substrate made of a similar material with periodic Floquet boundary conditions on four sides [22]. There was an emitting port and a perfectly matched layer (PML) above the pyramid. Under its base there was a perfect electrical conductor (PEC) (Fig. 3).

The space between the PML and the pyramid was an air medium with parameters $\varepsilon_r = 1$ and $\mu_r = 1$. The electrical parameters of the pyramid and substrate material for the corresponding frequency were determined from measurements of a real sample using the transmission line method in a coaxial waveguide (Fig. 4).

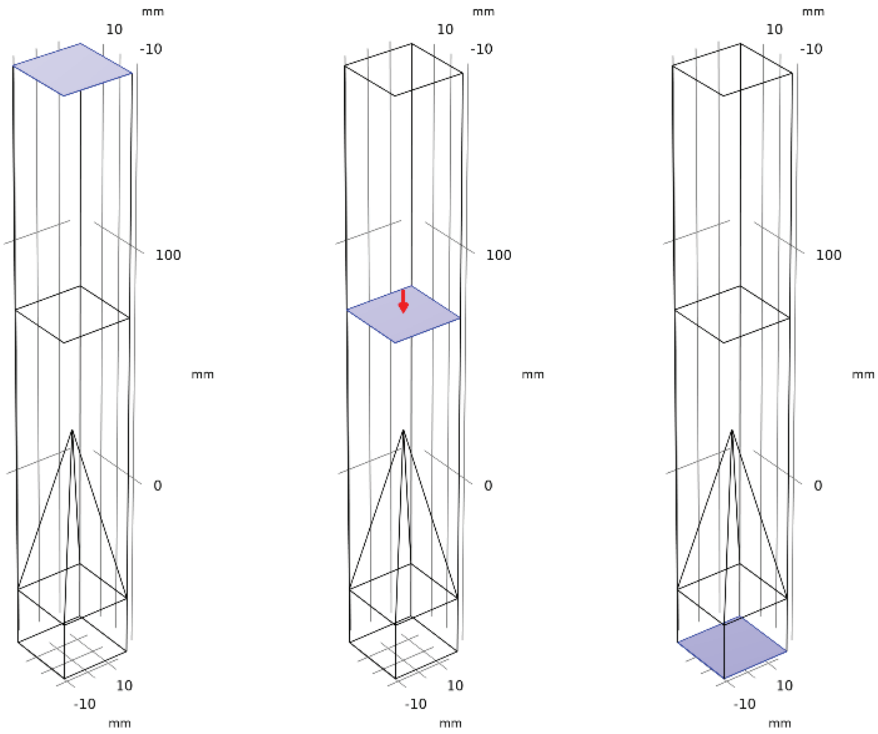


Fig. 3. Location of the matching layer (*a*), emitting port (*b*) and ideal conductor (*c*) in the pyramidal absorber model

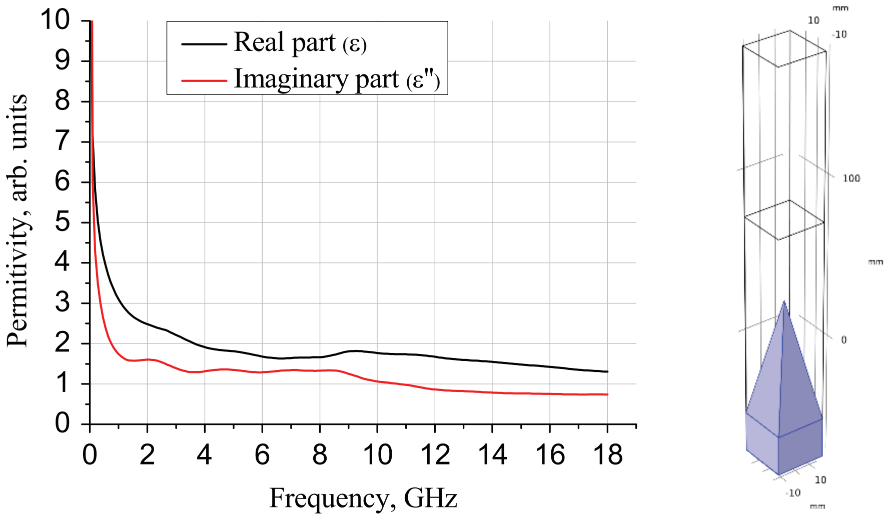


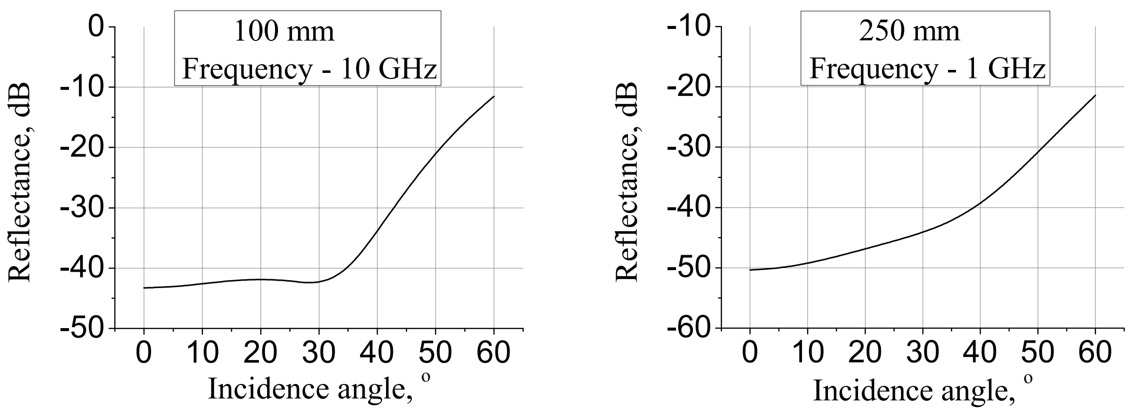
Fig. 4. Frequency dependence of the specified electrophysical parameters of a pyramidal absorber on the example of a composite sample with 2 wt. % CNT"Taunit-MD"

For further studies of the interaction processes of electromagnetic radiation with samples of full-scale pyramidal RAMs, the transmission method in free space using P6-223 and DRH-110 horn antennas in the frequency ranges of 1–15 GHz and 14–50 GHz, respectively, was used.

3. Results and Discussion

During the process of modeling absorbing structures based on polyurethane foam matrices modified with carbon nanostructures, it has been found that only 1–2 wt. % of Taunit-MD MWCNTs is sufficient to produce an effective pyramidal absorber. Figure 5 shows the results of modeling the interaction of EMR with samples of pyramidal RAMs with 2 wt. % CNT "Taunit-MD" with different structural configurations in the form of an angular dependence of the reflection coefficients at operating frequencies. At high frequencies, an increase in EMR attenuation will be observed, since the size of the absorbing cavities and the electrical length of the absorber will also increase with respect to the radiation frequency.

The use of high filler concentrations does not lead to a significant improvement in absorbing characteristics, but at the same time, the complexity of uniform dispersion of high filler concentrations for the manufacture of bulkier products increases. Figure 6 shows the transmission spectra of electromagnetic radiation in the near infrared range (400–1200 nm) through cuvettes with samples of water-acrylic suspensions of carbon nanotubes, recorded using a Lambda 1050 Perkin Elmer spectrophotometer.



(*a*) (*b*)

Fig. 5. Values of reflection coefficients of electromagnetic radiation for pyramidal absorbers with a height of 100 mm (*a*) and 250 mm (*b*) at minimum operating frequencies

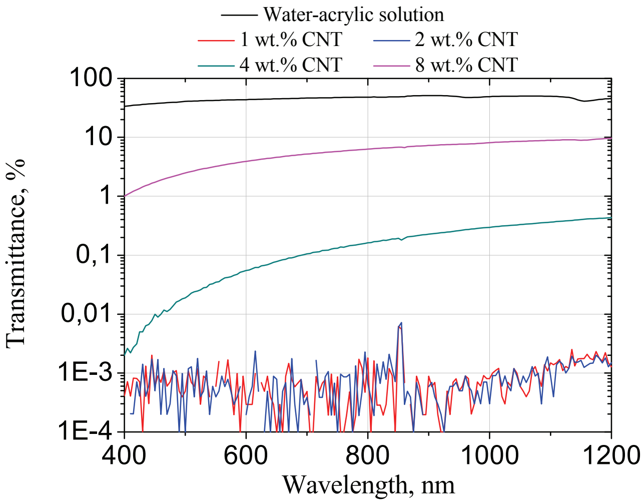


Fig. 6. IR transmission spectra of samples of water-acrylic solutions with different concentrations of carbon nanotubes "Taunit-MD"

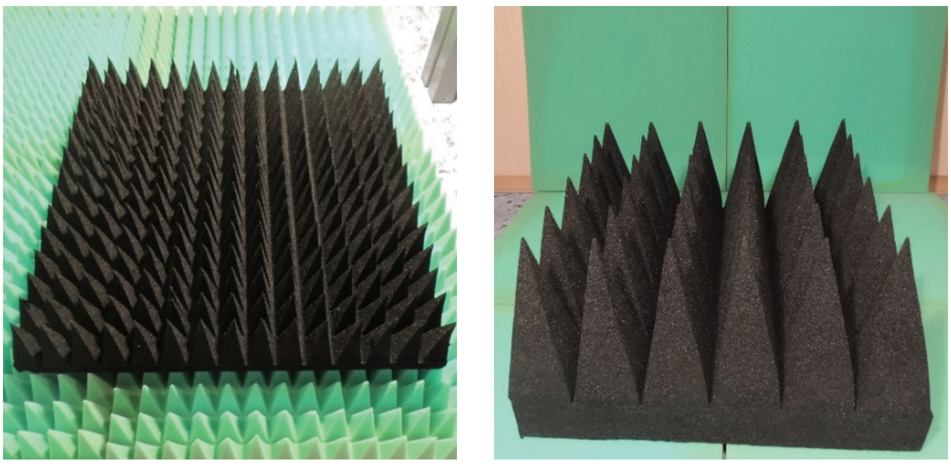
Based on the presented results, it can be noted that for samples with a concentration of 1 and 2 wt. %, almost complete absorption of radiation is observed – the values of transmission coefficients vary within 0.001 %, which may indicate the creation of a homogeneous dispersed medium in the solution. At the same time, despite an increase in the concentration of the absorbing filler, for solutions with 4 wt. % and 8 wt. % carbon nanotubes, a decrease in transmission coefficients is observed. Presumably, this effect is associated with two factors. Firstly, the excess content of carbon nanostructures in the same volume of the solvent leads to an increase in the viscosity of the solution and a decrease in the efficiency of ultrasonic vibrations. Secondly, the concentration of the surfactant relative to the volume of nanotubes decreases. As a result, the repulsive force of carbon nanotubes cannot reach the ability to prevent their agglomeration due to the decrease in the amount of surfactant adsorbed on the surface of nanostructures, so that the dispersion deteriorates, which affects the increase in transmittance [19].

Based on the simulation results obtained, full-scale samples of absorbers based on polyurethane foam and acrylic polymers with dimensions of 500 × 500 × 100 mm and 500 × 500 × 250 mm, modified with multi-walled carbon nanotubes, were formed (Fig. 7).

Figure 8 shows the findings of EMR interaction in the frequency range 1–15 GHz for samples of pyramidal absorbers with the addition of 2 wt. % MWCNT "Taunit-MD". The main measured value was the complex S11 parameter characterizing the ratio of the amplitude of the electromagnetic wave incident on the RAM sample to the amplitude of the signal reflected from the sample. Among other things, the transmitted electromagnetic radiation was completely leveled due to the use of metal shielding on the back of the RAM. Thus, the radiation twice passed through the RAM samples was incident on the receiving antenna.

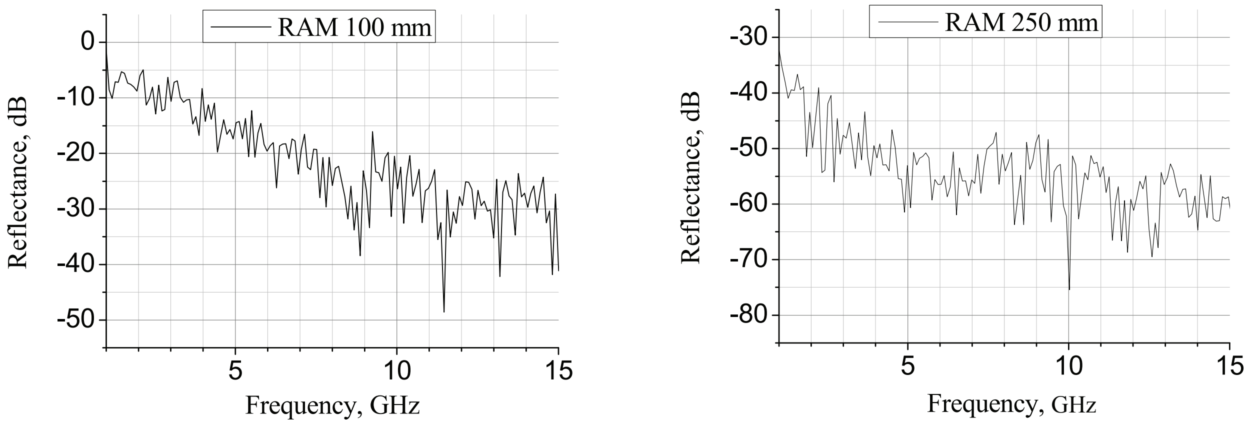
As mentioned earlier, pyramidal RAMs are capable of effectively attenuating incident electromagnetic radiation at wavelengths smaller than the scattering cavities, so it is logical to observe significantly lower reflected radiation attenuation values for 100 mm samples, up to –10 dB at 1 GHz, while at the same time 250 mm sample demonstrates reflected radiation attenuation at the level of –40÷–50 dB over the entire frequency range (Fig. 8).

The use of carbon nanomaterials as a modifier for flexible polyurethane foam is a key feature of the absorber. By creating a developed conductive network in the polymer structure [13], the filler



(*a*) (*b*)

Fig. 7. Radio absorbing composite material based on polyurethane foam modified with multi-walled carbon nanotubes: *a* – 100 mm RAM; *b* – 250 mm RAM



(*a*) (*b*)

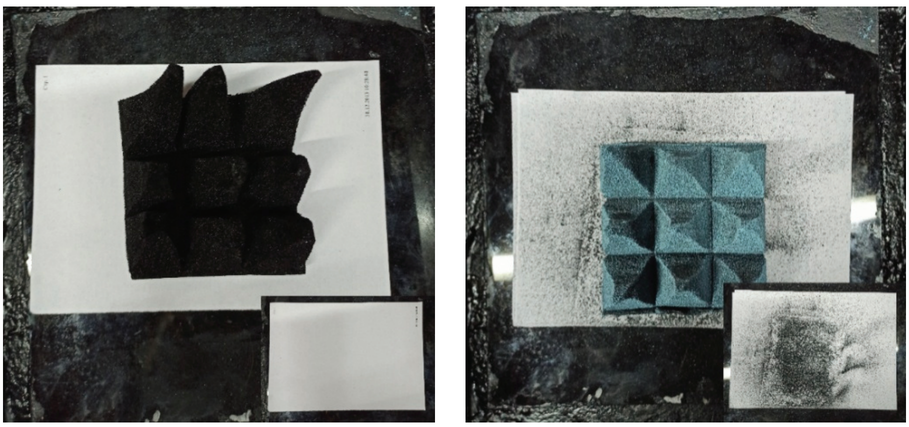
Fig. 8. Frequency dependence of the attenuation of reflection coefficient by samples of radioabsorbing materials on a metal substrate:
a – 100 mm RAM with 2 wt. % CNT "Taunit-MD"; *b* – 250 mm RAM with 2 wt. % CNT "Taunit-MD"

concentration is significantly reduced to ensure effective absorbing characteristics and, at the same time, the absence of filler flowability and the preservation of the elastic mechanical properties of the product are ensured. To confirm this feature, mechanical tests of the wear resistance of a composite sample, as well as a commercially produced RAM image modified with carbon black, were carried out [23]. In this test, segments of RAM samples were subjected to axial compression with a pressure of 10 kPa in a cycle of 200 repetitions. Before and after cyclic mechanical impact, the samples were tested for their ability to shield electromagnetic radiation in the operating frequency range of 14–50 GHz. Figure 9 shows photographs of samples after testing.

The applied load affected the absorber samples in different ways. The sample with the addition of 2 wt. % CNT "Taunit-MD" turned out to be strongly deformed, but after a short time it restored its shape. In turn, a sample of industrial RAM modified with carbon black retained its shape. However, a significant precipitation of the filler occurred, which can be clearly demonstrated in the inset of Fig. 9*b*.

The loss of such a significant amount of filler had a negative impact on the shielding properties of the radioabsorbing material (Fig. 10*a*, *b*). For the RAM sample modified with carbon black, the efficiency of attenuation of the transmitted radiation by –20 dB is observed in the operating frequency range.

In turn, the RAM sample with CNT "Taunit-MD" demonstrated almost complete preservation of EMR shielding properties in the studied frequency range (Fig. 10*c*, *d*).



(*a*) (*b*)

Fig. 9. Images of RAM samples during cyclic axial compression tests:
a – RAM with 2 wt. % CNT "Taunit-MD"; *b* – RAM with black carbon

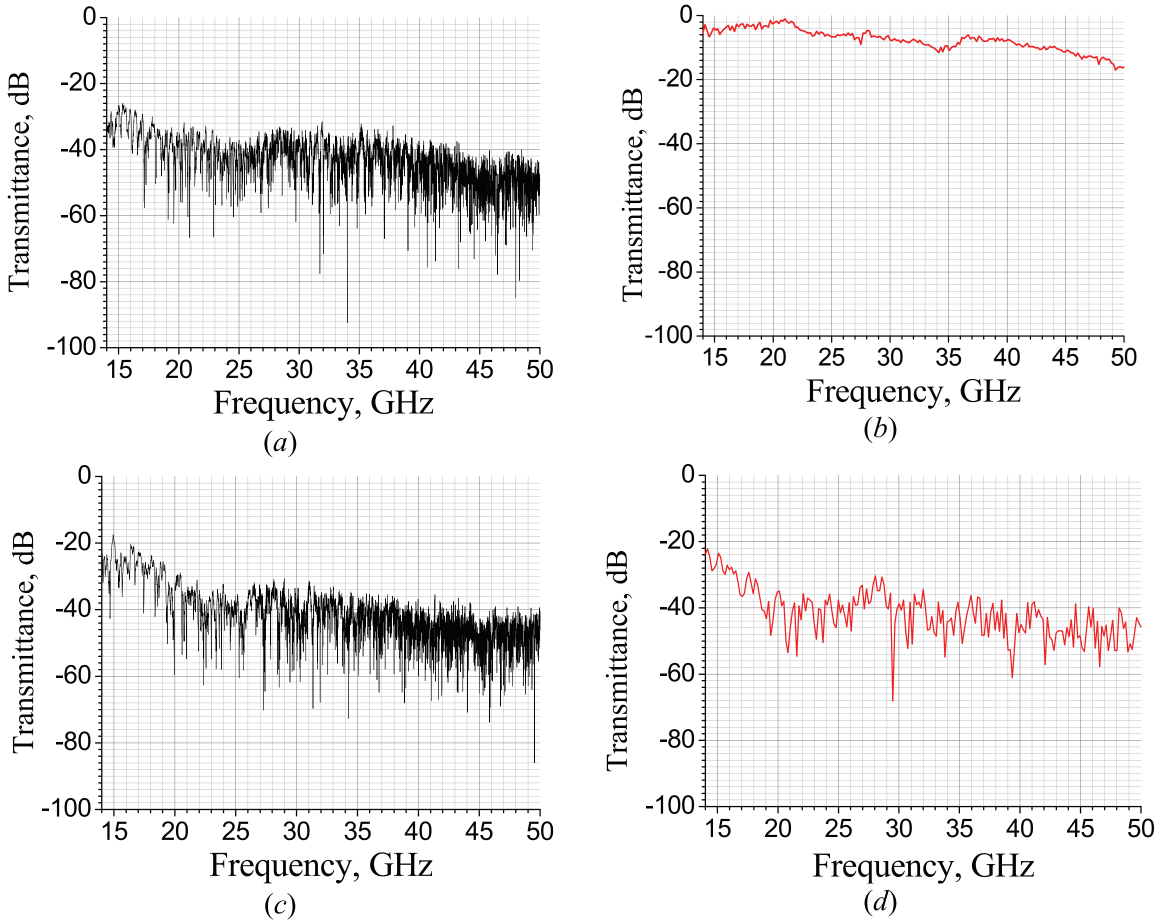


Fig. 10. Frequency dependence of the transmission coefficient of electromagnetic radiation through RAM samples with black carbon (*a*, *b*) and with CNT "Taunit-MD" (*c*, *d*) before (*a*, *c*) and after (*b*, *d*) compression test

4. Conclusion

Based on the evaluation and analysis of the interaction processes of electromagnetic radiation in the microwave range with composite samples of the polymer/CNM system, the prospects and effectiveness of using such materials for solving applied problems of electromagnetic compatibility have been demonstrated. Methods for creating and fabricating effective pyramidal radioabsorbing materials based on a polyurethane matrix with a uniform filling of its volume with multi-walled carbon nanotubes (1–2 wt. %) have been developed. The power of the reflected signal from such materials at normal EMR incidence is less than –40–50 dB in the operating frequency range, which corresponds to the best industrial (commercial) radio absorbing materials containing, in turn, an order of magnitude more filler (~ 30–50 wt. %).

The complete preservation of the absorbing characteristics of RAM structured with carbon nanotubes after wear-resistant tests has been demonstrated. At the same time, for a sample of industrial RAM, a deterioration in the absorbing capacity by –20 dB was observed, which was caused by the precipitation of a significant amount of filler.

5. Acknowledgements

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6. Conflict of interests

The authors declare no conflict of interest.

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