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Development and Application of Thin Wide-Band Screening Composite Materials

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The paper is dedicated to the development of effective composite coatings with the use of carbon fillers of different morphology, their research and application in the broadband frequency range.

Electromagnetic loss studies were performed according to international standards ASTM D4935, IEEE-STD-299 and the US Department of Defence standard MIL-STD 461F.

The impact of hybrid carbon nanomaterial "graphene / nanotubes" on the electrophysical properties of the composite material has been analysed. As a result, the research laboratory technologies of production of composite coating on water and non-water (alcohol) basis are developed based on the carbon fillers of various morphology and also magnetite. The shielding properties of most of the created composites are estimated in the frequency range from 50 MHz to 30 GHz.

The state enterprise “All-Ukrainian centre for standardization, metrology, certification and consumers’ rights protection” (here and after “Ukrmetrteststandart”) conducted comparative tests of the developed coating (in the form of paint) with a protective coating # 842 MG Chemicals (Burlington, Ontario, Canada) based on silver microparticles. It is concluded that the developed protective coating is not inferior to the Canadian reference sample in the entire studied frequency range, while having a much lower cost and simplified application technology.

The research has been carried out to study the application of composite paint to cover the components of some radiation monitoring devices of the ECOTEST brand, in particular a multi-purpose dosimeter-radiometer MKC-UM type.

Developed composites on a non-aqueous basis have already found wide practical application to solve the problem of electronic compatibility by applying a layer of 150-200 microns on the inner surface of thermal imagers and optical sights ARCHER brand.

Developed water-based composites can be used for interior decoration, in the formation of electromagnetic screens, thin gradient coatings to protect people from electromagnetic radiation in the microwave range.

Keywords: hybrid carbon nanomaterial, polymer matrix, electromagnetic shielding, broadband shielding composite.

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Introduction

The rapid development of electronic devices and gadgets in wireless communication, and their constant use in everyday life inevitably creates a strong electromagnetic radiation (EMR) in the environment [1]. Due to the intensive growth of the spread of electronics, broadcasting, and telecommunications, it is impossible to avoid the influence of various types of EMR [2]. To date, people are surrounded by EMR arising from a variety of electronic devices (Fig. 1).

EMR not only affects the operation of electronic devices (the so-called "compatibility" problem), but also significantly harms the environment [3-6] and affects human health [7], causing under certain conditions such serious diseases as brain tumours [8] and leukaemia [9, 10]. At the same time, the issue of information security is very important, which requires not only shielding, but also a comprehensive technical solution.

Today, the main way to reduce the negative impact of EMR is to develop means of protection for electronic devices, humans and the environment, namely the

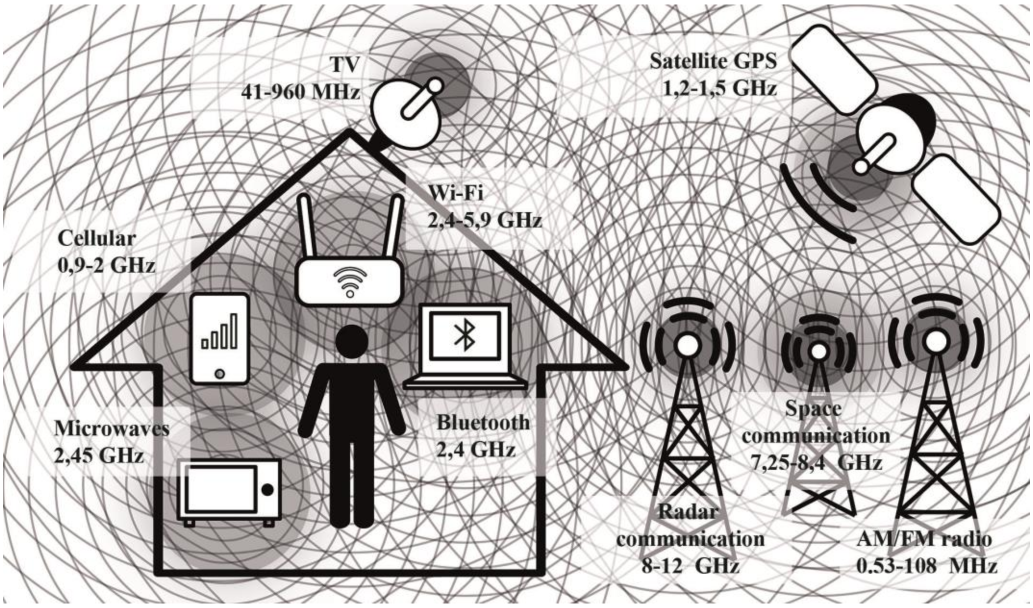

Fig.1 The main sources of electromagnetic radiation in human everyday lives.

creation of various types of shielding [11] and absorbing [12, 13] materials. Such materials have a wide range of applications, including commercial and scientific electronics, antenna systems, space exploration and medical devices [14], a wide range of military programs [15-18]. As of today, metals remain the best materials for electromagnetic shielding, but they have a number of significant disadvantages: high cost, high density, most of them have poor corrosion resistance, complexity of installation of structures, very low EMF absorption [19, 20]. That is why polymer composites filled with carbon materials can be even more promising in use.

The combination in composites of EMR-transparent polymers and carbon fillers of different structure and morphology allows to create materials that partially or completely cover the shortcomings of metals [21]. Such composite materials have low density, high conductivity, good manufacturability, and lower cost in comparison to protective materials filled with metals [22-24]. Sheet materials, electronic equipment housings, and paint coatings can be made of polymer composite materials. The latter are of particular interest due to their prospects: they are easy to work with in complex profiles, it is possible to provide a complete seamless coating that reduces the risk of energy leakage, it is possible to form a reliable thin electromagnetic screen.

Typically, electromagnetic shielding in electronic equipment often uses paint coatings with metallic fillers of nickel, copper, and silver. Such composite materials are close to thin metal screens in terms of shielding level and provide shielding efficiency up to -70… -80 dB. However, they have a few significant disadvantages associated with metallic fillers: the use of toxic organic solvents, oxidation of the metallic filler, rapid settling of the filler, fast curing time, as well as a significant contribution to the final weight of the device and its cost. Therefore, paint coatings filled with metal fillers are difficult to use to protect large (anechoic chambers, living quarters, large electrical panels) and complex-profile objects (eg, thermal imager housings, radiation reconnaissance dosimeters, etc.). As an alternative to metal fillers, carbon materials are widely used.

The Department of Electrochemical Power Engineering and Chemistry of KNUTD traditionally conducts research on various carbon graphite materials, electrically conductive polymers, carbon nanomaterials, hybrid polymer composites as active materials, mainly for electrochemical power sources (http://www.en.knutd.com.ua/university/faculties/fcht/hteheh/). With the beginning of military operations in the East of Ukraine (2014), the department began the studies on the protection of electronic equipment and humans from the effects of EMR, given the importance of the problem and the existing experience of investigations with carbon, polymer and composite materials. Scientists from different countries have shown some efficiency in the use of carbon materials such as graphite [25], carbon black [26, 27], carbon nanotubes [28], carbon fibres [29], graphene [30] and their mixtures in carbon-polymer composites for electromagnetic shielding.

In particular, in [31] the combination of multilayer carbon nanotubes (MCNTs) of 4 wt.% with microfibers of silicon dioxide provides a shielding efficiency of -40 dB at a sample thickness of 2 mm in the frequency range 8 – 12 GHz. Scientific groups are working on the latest lightweight shielding materials, among with foamed polymer matrices with a graphene content of only 0.8 wt.% provide shielding efficiency up to -23 dB at a thickness of 1.6 mm [32]. A combination of nanostructures "carbon nanotubes / multilayer graphene" gives good results, which allow to obtain -48 dB at a thickness of 1.6 mm and a density of 8.9 mg/cm^3 [33]. The electrical properties of single-layer carbon nanotubes (CNTs) depend significantly on their geometric structure, on diameter and chirality. It should be noted that the specified shielding efficiency depends strongly on the measurement technique (which does not always meet existing standards, as standardized equipment is very expensive), as well as on the frequency range and layer thickness (which are not always given). Therefore, the quantitative data presented in the literature should be treated with caution.

The problem with the use of CNTs is the complexity of their dispersion in an aqueous medium. There are many known methods for creating dispersions of CNTs, these include methods of surface functionalization, which are described in detail in [34, 35], ultrasonic dispersion, including the use of surfactants [36] and other methods [37]. In the production of paint coatings, this introduces additional operations and energy consumption, and surfactants adversely affect the process (increase foaming) and impair performance (reduce the amount of adhesion to the substrate and increase the hydrophilicity of film materials). Therefore, the most technological among carbon materials are graphite, carbon black, carbon fibre and ready dispersions of nanosized carbon materials without the use of surfactants.

Summarizing the above, it can be noted that the vast majority of scientific work is aimed at creating and researching shielding materials that give good shielding performance, but at relatively large thicknesses (2 mm or more). There is almost no work on the development of thin film materials (up to 1 mm), especially on the basis of aqueous emulsions of polymers, which would provide shielding of -30 dB or more. That is why the main purpose of the paper was to develop multi-carbon composite materials such as water-based and non-water-based paints, which would provide shielding efficiency not worse than -30 dB in the wide frequency range -50 MHz - 30 GHz with a layer thickness of not more than 500 μm for further its application in various technical solutions to the problem of electromagnetic shielding (EME).

I. Materials and methods

As the main materials colloidal graphite with an average particle size of 5 μm, flake accumulator graphite GAK-2 with an average particle size of 25 - 30 μm (manufactured by Zavallivskii Graphite, Ukraine) were used; lamellar graphite with an average particle size of 70 - 100 μm; carbon fibres with particle sizes: diameter d = 7 μm, length l = 50 - 100 μm; highly dispersed carbon black of the N330 brand, graphitized carbon black of the Pureblack brand (USA); styrene-acrylic emulsion, dispersant for carbon fillers in aqueous systems and defoamer based on modified silicone; polyvinyl butyral; composite carbon nanomaterial type "grapheme / nanotubes" SQG-CNT-90 brand (China).

The following equipment was used to make samples of polymer composites: laboratory vibrating screen with multilevel type sieves, with a frequency of 1400 vibrations per minute; laboratory speed mixer up to 5000 rpm with a bowl with a volume of 2 litres and a cutter with a diameter of 50 mm; frame applicator for coatings of a given thickness of 0 - 2 mm, a step of 1 μm, and a working width of 100 mm. The technology of sample production includes the following stages: 1) classification (sieving) of colloidal graphite preparation and plate graphite using a vibrating screen, for screening of graphite particles larger than 40 μm and 100 μm, respectively; classification of carbon fibres for screening long fibres and balls larger than 100 μm, which may impair the homogeneity of the composite; 2) preparation of soot dispersion: the required amount of water, dispersant and defoamer is loaded into the high-speed mixer - the mixture is stirred for 5 min, after which the required amount of highly dispersed granular soot is gradually introduced into the resulting solution; 3) mixing the finished soot dispersion with other carbon fillers and functional additives for 30-60 minutes to form a homogeneous state. The viscosity of the paint was measured and controlled by a viscometer B3-4 GOST 9070-75, the flow time through the calibrated capillary 4 mm should not exceed 100 s. Films of composite materials were formed on an insulating basis from glass by means of a manual frame applicator at room temperature, dried under normal conditions for 30 min, and at a temperature of 55 °C for 60 min. The obtained films were characterized by a uniform and integral surface and a thickness of approximately 30 - 60 μm.

The adhesion of the samples to the substrate was measured by the method of applying lattice incisions ASTM D3359.

The specific surface electrical resistance was measured by a standard method of measuring resistance or conductivity at direct current of conductive materials in accordance with ASTM D4496.

Standardized methods and equipment in accordance with ASTM D4935 and IEEE-STD-299 standards were used to measure the shielding efficiency of thin samples (up to 2 mm). The essence of the method according to the ASTM D4935 standard is to use compact TEM-cells (Transversal ElectroMagnetic Cell). ASTM D4935 standardizes a fairly narrow frequency range: from 30 MHz to 1.5 GHz. This is due to the fact that in TEM-cells, which have the design described in the standard, with increasing frequency, various parasitic processes are observed, which make it impossible to accurately measure the shielding efficiency. In our paper we used advanced TEM-cells of KEYCOM Corp. (Japan).

KEYCOM Corp. expanded the frequency range of measurements to 18 GHz. To study the frequency range above 18 GHz, studies were performed in accordance with the IEEE-STD-299 standard. The shielding efficiency of the samples according to the IEEE-STD-299 standard is calculated by the difference in signal level between the antennas. The receiving antenna is located in the middle of the shielded room opposite the hole. Shielding efficiency was determined by measuring the signal with an open hole. and when it is closed by the test sample.

The measurement according to the US Department of Defence MIL-STD 461F did not apply to individual materials, but to the devices as a whole, the inner surface of which was covered with one or another coating. These measurements were performed on the appropriate equipment at the State Enterprise "Ukrmetrteststandard", Kyiv.

Table 1

The composition and some properties of the paint samples based on polyvinyl butyral

Component/Parameter	Measurement unit	Sample Number					
		1	2	3	4	5	6
Graphite from “Zavalivskii Graphite”, UA	wt.%	80	-	60	60	50	50
PUREBLACK from “Superior Graphite Co.”, USA	wt.%	-	80	20	20	17	17
Magnetite from KNUTD, UA	wt.%	-	-	-	-	17	17
PVB and solvent	wt.%	20	20	20	20	16	16
Layer thickness	microns	60	60	60	125	60	125
Sheet resistance	Ohms per square	15333	6667	400	128	133	24

II. Results and discussions

At the very beginning of a research, the task was to improve the first generation of KNUTD paint, developed on the basis of colloidal graphite, graphitized carbon black and polyvinyl butyral (PVB) [38]. Scientific studies have shown a clear synergistic effect when using a mixture of carbon fillers with different structure and morphology (in this case - graphite and carbon black), which leads to a significant increase in shielding efficiency (SE) than would make a simple arithmetic sum of SE and each a separate component. The nature of this synergism can be explained by the fact that the absorption and reflection of EMR is carried out mainly at the interface of the particles, the surface area of which increases when using certain combinations of fillers. Therefore, the use in the composite of certain combinations of dissimilar materials with different structure and morphology of particles, especially combinations of micro- and nanomaterials, should provide a sufficient value for practical purposes SE = -30 dB (equivalent to attenuating the EMR intensity by 1,000 times).

As an electrically conductive micro-material in this embodiment served as colloidal graphite, as another nanostructured material - graphitized carbon black, in which microscopy clearly fixes graphene nano-sized areas.

Some improvement of shielding properties of the first generation of paint (up to -30 dB and more) and adhesion (up to 4-5 points for glass, ceramics, metals, polar plastics) was achieved due to the following factors:

1) the introduction of an impurity of magnetite, which partially compensates for the magnetic component of the electromagnetic wave;

2) replacement of highly dispersed colloidal graphite with larger, but at the same time much more conductive graphite GAK-2; this significantly affects the electrical component of the electromagnetic wave;

3) the use of ultrasonic dispersion of a mixture of components in an alcohol solution, which contributes to the disintegration of agglomerated particles, creating a reliable electrical contact and more uniform mixing of the components.

It should be noted that despite the introduction into the mixture of such a poorly conductive component as magnetite, due to the optimal content of components and method of mixing it is possible to obtain samples with much lower resistance (№ 5, 6) than without magnetite at the same thickness (№ 3, 4) - Table 1.

The paint developed and improved in this way allowed to solve successfully the problem of electromagnetic compatibility of thermal imagers with other electronic equipment (in particular - a radio station) and to reduce the "visibility" in the radio frequency range. The coating is easily applied in a thin layer (150 μm) on the inner surface of the body parts of thermal imagers of complex profile, provides reliable adhesion and efficiency of EMR shielding. Currently, more than 3,500 thermal imagers and optical sights of the ARCHER brand have been manufactured using this development for the needs of the Armed Forces, the National Guard and the Border Troops of Ukraine.

In addition, successful tests on the use of composite paint to cover the components of some radiation monitoring devices of the ECOTEST brand, in particular, a multi-purpose dosimeter-radiometer type MKC-UM in the amount of more than 40 sets.

The state enterprise “Ukrmetrteststandart” (Kyiv) compared this paint with the nearest foreign analogue – protective coating # 842 MG Chemicals (Burlington, Ontario, Canada) based on silver microparticles. As a result of this comparison, it was concluded that: 1) the developed protective coating is not inferior to the Canadian reference sample based on silver particles in the entire studied frequency range, while having a significantly lower cost and simplified application technology; 2) KNUTD protective paint provides effective internal shielding of electromagnetic radiation in accordance with the requirements of the standard MIL - STD 461F of the US Department of Defence in the investigated during the comparison frequency range from 30 MHz to 1 GHz.

An important characteristic feature of this type of paint is the use of PVB as a polymeric binder. This provides a fairly high adhesion of the paint to a wide range of body polymeric materials, as well as complete moisture resistance of the paint, as PVB is well soluble in ethyl and isopropyl alcohols, but not in water. Such properties are very important when using paint for electronic equipment, protective screens, ships, submarines, etc.

It should be noted that due to the full moisture resistance and adhesion to metals, the paint exhibits

Table 2

Composition and properties of basic carbon composites based on water-soluble polymer matrix

Component/Parameter	Measurement unit	Sample Number			
		7	8	9	10
Colloidal graphite	wt.%	13	-	26	-
Flake graphite	wt.%	-	17	-	-
Carbon black	wt.%	7	9	-	-
Carbon fiber	wt.%	20	26	-	26
SQG-CNT-90	wt.%	-	-	9	9
Polymer emulsion and functional additives	wt.%	15	15	15	15
Water	wt.%	45	33	50	50
Surface resistance	Ohms per square	6.3	7	6.3	16.2
Adhesion to glass	points (from 0 to 5 points)	5	3	5	3

excellent anti-corrosion properties in a wide temperature range, at least from -30 to +120 ˚C. Therefore it can find wide practical application in construction and in a life, in particular as an anticorrosive covering of pipelines of hot and cold water supply.

It is clear that for the shielding of internal special and domestic premises for information protection, protection of people from EMR, it is desirable to have a water-soluble paint on a suitable polymer, in particular on the basis of styrene-acrylic emulsion. Therefore, the main further efforts were devoted to the development of water-soluble paint.

The ratio of fillers was selected according to the structure and morphology of the materials. Carbon Black and grapheme / nanotubes are characterized by low bulk density and large specific surface area, so the most rational, in terms of efficiency of electrophysical parameters and rheology, will be the dry residue content of not more than 48 %, and in the case of nanosized fillers this figure can be reduced to 35 - 30 % (Table 2).

By experimental modelling of composites, the optimal ratio and combination of carbon materials of three types was found: carbon fibres, colloidal graphite and soot in a ratio of approximately 3: 2: 1 mass parts (composites 7 - 8, Table 2), respectively. In such a composite, carbon fibres form a conductive framework and act as a reinforcing component: they increase the strength and hardness of the composite film.

Graphite and carbon black form branched aggregate structures around the fibres, increasing the number of phase boundaries, increasing the cohesion and adhesion of the composite material. The main interaction of electromagnetic waves with nonmagnetic materials occurs at the interface and consists of repeated reflection from the surface of particles in the middle and on the surface of the composite and the interaction of the electromagnetic field with the molecular and electronic structure of such material. with material.

The results of measurements of shielding properties (in absolute value) of such a composite material are shown in Fig. 2 for the composite № 7.

Figure 2 clearly shows that the composite № 7 in the frequency range of 1.5 - 12 GHz shows stable characteristics and provides shielding efficiency of at least -23 dB, with a sample thickness of 100 μm (2 layers). In the frequency range of 12 - 15 GHz (Ku band) there is a rapid decrease in the magnitude of electromagnetic losses and the restoration of a linear nature in the range of 15 - 30 GHz. This effect requires further study, because it can be caused by both the features of the device and the interaction of electromagnetic waves with the components of the composite in this frequency range.

In papers [30, 39, 40] high efficiency of use of graphene for shielding is noted. However, obtaining thin-layer coatings in the form of paint using graphene or nanotubes is very difficult. Therefore, we decided to try to create a paint using a hybrid composite carbon nanomaterial SQG-CNT-90.

The material is interesting for use in shielding composites due to its structure. It is synthesized from graphene and carbon nanotubes in a certain ratio and combines the properties of both materials - high electrical conductivity of graphene and the absorbing properties of MCNT.

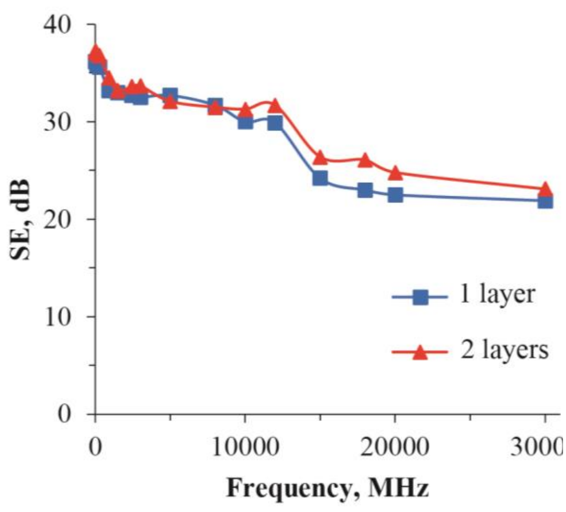

Fig.2. Dependence of shielding efficiency of composite № 7 on the number of applied layers.

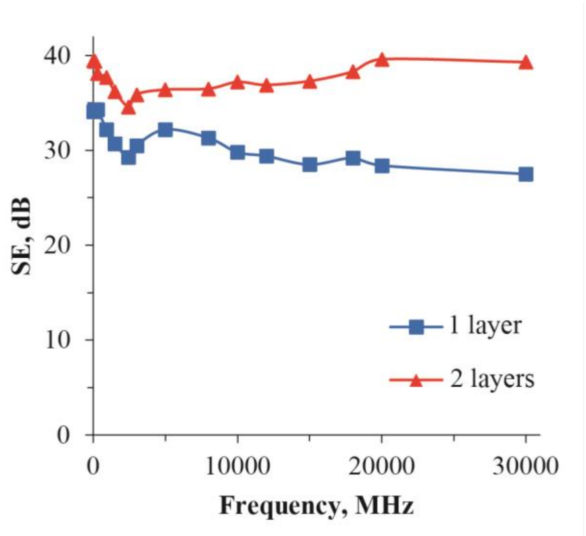

Fig.3. The dependence of the shielding efficiency of the composite №9 on the number of applied layers.

It should be noted that the use of this composite simplifies the technology of manufacturing the composite material, because it does not require prior preparation of the carbon black dispersion and classification of fibers, as it completely replaces them. This, in turn, saves material costs and energy costs in the manufacture of composite material № 9. The composite material №9 was also characterized by a lower viscosity and a lower degree of filling, which is also an important economic indicator.

The use of graphene / CNT nanocomposite together with other carbon materials allows to reduce the total content of carbon materials and significantly improve the electro-physical properties of film materials (Table 2, sample № 9, Fig. 3). Interestingly, the combination of graphene / CNT nanocomposite with carbon fibers did not give the expected results, but the combination of grapheme / CNT nanocomposite with colloidal graphite provides a shielding efficiency of -35… -40 dB in the entire frequency range from 30 MHz to 30 GHz at a thickness of 60 µm (2 layers).

Conclusions

The developed coating (paint) based on the non-aqueous basis (alcohol), ensures the effective shielding at the level of -30 dB and more in the frequency range at least from 30 MHz to 30 GHz; excellent adhesion to the wide range of materials (polar plastics, metals, ceramics, glass, wood, etc.); full moisture and anticorrosive resistance and founds a practical application in electronics, constructions and in a common life.

Composite materials in the form of paint based on aqueous emulsion of polymers have been developed, which can provide high shielding efficiency at the level of -33 ...- 23 dB in the frequency range from 50 MHz to 30 GHz.

During the research, the optimal ratio and combination of carbon materials of three types was found: carbon fibers, colloidal graphite and carbon black in a ratio of approximately 3: 2: 1 mass parts, respectively.

A broadband shielding composite material in the form of a water-based polymer emulsion paint using a composite carbon nanomaterial has been developed that can provide high shielding efficiencies of -35… -40 dB in the frequency range from 50 MHz to at least 30 GHz.

The use of a composite carbon nanomaterial in the paint coating allows to reduce some economic indicators, in particular the degree of filling, the number of required operations in the manufacturing technology, the manufacturing time of the final composite.

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Розробка і застосування тонких широкополосних екрануючих композиційних матеріалів

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Робота присвячена розробці ефективних композиційних матеріалів з використанням вуглецевих наповнювачів різної морфології, їх дослідженню та застосуванню в широкосмуговому діапазоні частот.

Дослідження електромагнітних втрат проводили за міжнародними стандартами ASTM D4935, IEEE-STD-299 та стандартом Міноборони США MIL- STD 461F.

Проаналізовано вплив гібридного вуглецевого наноматеріалу «графен/нанотрубки» на електрофізичні властивості композиційного матеріалу. В результаті досліджень розроблені лабораторні технології виготовлення композиційних покриттів у формі фарби на водній і неводній (спиртової) основі з використанням вуглецевих наповнювачів різної морфології, а також магнетиту. Екрануючі властивості більшості створених композитів оцінені в діапазоні частот від 50 МГц до 30 ГГц.

Державним підприємством «Всеукраїнський центр стандартизації, метрології, сертифікації і захисту прав споживачів» (тут і далі «Укрметртестстандарт») проведені порівняльні випробування розробленого покриття (фарби) з захисним покриттям #842 MG Chemicals (Burlington, Ontario, Canada) на основі мікрочастинок срібла. Зроблений висновок, що розроблене захисне покриття не поступається канадському еталонному зразку в усьому дослідженому діапазоні частот, маючи при цьому значно нижчу собівартість і спрощену технологію нанесення.

Проведені дослідження по застосуванню композитної фарби для покриття складових деталей деяких приладів радіаційного контролю бренду Ecotest, зокрема багатоцільового дозиметру-радіометру типу МКС-УМ.

Розроблені композити *на неводній основі* вже знайшли широке практичне застосування для вирішення проблеми електронної сумісності шляхом нанесення шару 150 – 200 мкм на внутрішню поверхню тепловізорів і оптичних прицілів бренду ARCHER.

Розроблені композити *на водній основі* можна використовувати для внутрішньої обробки приміщень, при формуванні електромагнітних екранів, тонких градієнтних покриттів для захисту людини від електромагнітного випромінювання в НВЧ-діапазоні.

Ключові слова: гібридний вуглецевий наноматеріал, полімерна матриця, електромагнітне екранування, широкосмуговий екрануючий композит.