Radio-Absorbing Materials and Technologies for Their Production

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Radio waves cover a fairly wide spectrum of the frequency range (the range of waves used is from ultra-long to millimeter; the range is from centimeters and meters to thousands of kilometers) and are harmonic signals modulated in amplitude and frequency. The main source of powerful electromagnetic radiation is an antenna that radiates a flow of electromagnetic energy in a directionally or non-directionally into the surrounding space.

polymer nanocomposites radio-absorbing materials and coatings

materials with controlled electro-physical characteristics

1. Brief Introduction

Radio-absorbing materials (RAM) are widely used in special-purpose equipment. These materials are designed to ensure the electromagnetic compatibility of radio-electronic equipment and antenna systems [1][2]. The main radio engineering parameters that characterize the RAM are: the values of the real and imaginary parts of the dielectric and magnetic permeability as well as the reflection coefficients (R) and/or transmission coefficients (T) in the frequency ranges of the microwave spectrum.

It is known that an ideal single-layer absorber of electromagnetic waves (EMW) is a material with equal dielectric and magnetic permeability. Herein, the reflections from the front boundary of the material will tend to zero [3].

2. RAM Classification

In real conditions, obtaining RAM with the same values of dielectric and magnetic permeability in the frequency range is an extremely difficult task due to differences in the physical processes that determine these parameters.

RAM can be classified according to various criteria, for example, according to the operating range of effective action: (i) broadband-, when the R values do not exceed a given value in the frequency range $\lambda max/\lambda min \geq 10$; (ii) narrow-range-; (iii) tunable-; and (iv) selective-type RAM.

According to the structure of RAM, they are divided into (i) single-layer constant composition, (ii) gradient-type (multilayer materials with a stepwise change in electrodynamic characteristics from the front boundary to the rear),

(iii) interference-type, (iv) geometrically inhomogeneous (for example, spike-like materials), and (v) combined, i.e., materials with a possible combination of gradient and interference structures.

Most of the existing interference-type RAMs depending on their structure can be divided into the following groups: (i) Salisbury screen based on an outer layer of a thin-layer conductive film located on a dielectric layer screened from the back side, (ii) Dallenbach screen based on absorbing layers placed on a conductive shielding substrate, and (iii) Jaumann screen based on multilayer alternating structures of dielectric and conductive layers [4][5]. As a rule, gradient-type RAMs have more broadband EMR absorption compared to similar single-layer absorbers [6]. A special case of such RAMs are materials with an unfilled input layer with respect to external electromagnetic effects ("soft input"). The values of dielectric permittivity ε of such a layer are as close as possible to ε of the external environment at magnetic permeability $\mu = 1$. The subsequent layer or layers are distinguished by higher values of ε and μ that increase as one approach to the reflective substrate [7]. The thickness of the outer input layer is usually greater than the thicknesses of subsequent layers.

RAMs can be divided into materials of (i) magnetic-type, (ii) non-magnetic-type, and (iii) combined materials depending on the functional filler used. Materials of the magnetic type interact with the H-component of the electromagnetic field and have magnetic losses. Non-magnetic materials interact with the E-component of the electromagnetic field and have only dielectric losses. Combined-type materials contain both magnetic and non-magnetic conductive fillers.

RAMs classification can be carried out according to a number of other features technological, matrix composition and so on. Currently, smart coatings, including active (controlled) radio-absorbing structures, are becoming increasingly important [8]. The principle of operation of such structures is based on the use of external sensors that capture the effective EMR on the object and controlled layers designed to process and restructure the parameters of incoming signals in order to reduce them. In this connection, a new classification of RAM appears: (i) active and (ii) passive. Active RAMs are able to provide values of R of electromagnetic radiation (EMR) from the boundary of the medium and free space, which is close to zero at any polarizations and angles of incidence [9]. Any elements capable of changing the electrodynamic parameters of the material, for example, frequency selective gratings with pin diodes, can be used as active structures for such RAMs and radio-absorbing coatings (RAC). The main preference is given to pin diodes due to their low weight and ease of control by changing the values of the applied external voltage [10].

At present, active RAMs are already used in the fifth-generation Japanese fighter X-2 [11]. Active RAMs and RACs are prone to self-excitation, i.e., generation of their own EMR, which is an unmasking factor. To match the intelligent sensors of active RAMs and RACs, in some cases, it is necessary to use passive radio engineering materials. These materials could be composites with functional fillers. In most cases, obtaining passive RAMs is characterized by ease of manufacture and lower economic costs compared to active structures. In this connection, the works aimed at creating and improving traditional (passive) RAMs do not lose their relevance.

The vast majority of traditional passive RAMs and RACs are composites based on matrices with low dielectric constants and functional fillers. Ceramic [12], polymeric [13], textile [14], mineral materials [15] and so on, can be used as matrices.

3. RAM's Matrixes and Fillers

The choice of matrix and filler is determined by the purpose and operating conditions of the RAM. The main disadvantages of RAMs based on magnetic fillers are high values of bulk density as well as a low operating temperature range. One of the common advantages of such materials is to achieve lower values of *R* of EMR in thinner layers compared to non-magnetic type materials as well as to reduce the contribution to backscattering associated with edge diffraction and surface waves.

In this connection, in most cases, magnetic-type composites are used as coatings. Ferrites and powders of ferromagnetic metals are among the most characteristic magnetic fillers used in the creation of RAMs [16]. To ensure a wider operating temperature range of RPM, as a rule, non-magnetic type fillers are used, which are various electrically conductive functional particles: carbon black, graphite, carbon-containing fibers, powders of diamagnetic metals and their compounds, carbon nanotubes (CNT), graphene and so on [17]. The microwave and electrophysical properties of RAM depend on the composition, thickness, and number of layers as well as on the concentration of functional fillers. By varying these parameters, it is possible to give composite materials (CM) the necessary radio characteristics.

Soot-filled RAMs possess low values of bulk density; however, to achieve low values of *R* in the ranges of the microwave spectrum, thicknesses of more than 10 mm are required [18]. There are a large number of RAMs made using carbon fiber fillers. It is known that such fillers can be used both in materials for anechoic chambers [19] and for structural reinforced plastics [20][21].

4. RAM with Magnetic Fillers

Of greater interest are RAMs with high values of high-frequency dynamic magnetic permeability [22]. Such materials include CMs based on powders of ferromagnetic metals and hexagonal ferrites [23]. There is a known RAM based on nickel-zinc ferrite [24]. It provides efficient EMR absorption in the frequency range from 30 MHz to 1000 MHz. RAMs based on ferromagnetic metals; for example, metals of the iron triad (Fe, Ni, Co) as well as their various alloys and compounds have become widespread [25]. Carbonyl iron powders are of great interest for radio engineering applications and research [26]. For the antennas, developing the RACs based on an epoxy elastomer and carbonyl iron powder is used [27].

The shape and size of the functional filler has a significant impact on the radio-absorbing properties of the RAM [28]. A classic example of varying the electrodynamic properties of RAM based on carbon-containing fibers is a change not only in the concentration of the filler but also in its linear dimensions, for example, by cutting the fiber into lengths from tenths of mm to tens of mm [29].

It is known that powders of ferromagnetic metals and their compounds in the form of flakes and plates significantly increase the values of the dielectric and magnetic permeability of composites based on them compared with materials containing similar spherical ferromagnetic particles [30][31]. This is explained by the higher values of the average polarizability of composites with lamellar inclusions due to the small values of the form factors in the particle planes compared with composites containing spherical inclusions. As a rule, plates and flakes of ferromagnetic powders are produced by high-energy grinding in a closed-circuit bead mill in various organic media, for example, in ethanol and heptane [32]. Commercially available spherical ferromagnetic powders are usually used as the initial raw material for obtaining lamellar particles [33].

The disadvantage of RAM based on lamellar particles at their concentration above 40–50 vol. % is an increase in the values of the dielectric constant (more than a hundred).

The magnetic characteristics of powders of the $La_xNd_{2-x}Fe_{17}$ (x = 0.0, 0.2, 0.4, 0.6) alloy of various morphologies and the electrodynamic properties of corresponding RAMs were studied. The shape changing of the powders was carried out by high-energy grinding. The obtained powders of the investigated flake-shaped alloys provided the *R* of EMR for RAM samples less than −10 dB in a wide frequency range at a thickness of 2 mm. It has been found that the minimum values of the *R* of EMR amounted to −32.5 dB at 9.8 GHz [34].

Among the known magnetic materials for microwave applications, a special place is occupied by ferromagnetic films and CMs (laminates). CMs (laminates) based on ferromagnetic and polymeric films have high magnetic losses in the LF region and have a bulk density not exceeding 2–3 g/cm^3.

As a rule, obtaining a thin layer of a ferromagnetic metal or other material, such as hydrogenated carbon, is achieved using methods of ion-plasma magnetron sputtering on a substrate. The substrates can be polymer films, woven, non-woven materials and so on. The granular films of hydrogenated carbon with nanoparticles of ferromagnetic metals (*Co*, *Ni*) and corresponding RACs were developed [35]. The value of *R* of EMR for obtained RAMs did not exceed −10 dB in the frequency range from 7 to 70 GHz. RAMs were multilayer structures based on aramid fabric coated with films of hydrogenated carbon with different concentrations of ferromagnetic metals from layer to layer and, consequently, varying values of dielectric and magnetic permeability [35]. The films were deposited on the fabric surface by the method of ion-plasma magnetron sputtering. The RAM based on thin films of amorphous hydrogenated carbon with ferromagnetic nanoparticles deposited on a flexible substrate of aramid fabric by ion-plasma magnetron sputtering was developed [36]. According to the results obtained, such RAMs provide a high level of EMR absorption with *R* is in the range of −10 to −30 dB in the microwave frequency range. These materials are characterized by ultra-wideband.

A material that consists of two layers of polymer nanofibers bonded with a radio-transparent material is presented in [37]. A film of hydrogenated carbon with embedded ferromagnetic or ferrimagnetic particles is deposited on each layer of polymer nanofibers by vacuum sputtering. This material provides effective EMW absorption in the frequency range from 5 to 70 GHz at small thicknesses (no more than 2 mm). However, in real conditions, the application of such materials to the surface of a number of objects is an extremely difficult task due to low manufacturability and the need to create additional protective coatings and materials that can improve the physical, mechanical, and tribological properties, for example, erosion resistance. The RAMs described are also characterized by high cost. RAMs based on magnetic fibers have attracted the attention of many researchers. Micro wires can be referred to such magnetic materials [38]. The cores of micro wires are characterized by high values of magnetic permeability. The high value of the dynamic magnetic permeability of fibrous ferromagnetic materials is manifested in the direction along the axis of their fibers; however, the disadvantage is the high dielectric response, which exceeds the magnetic one.

Recently, RAMs based on nanosized fillers and powders with a nanosized crystalline structure have become increasingly important [39][40]. It is known that crystalline ferromagnetic powders have a large domain size, and therefore, the process of rotation of the magnetization vectors is hindered [41]. Such shortcomings are absent in nanocrystalline soft magnetic alloys with a set of *α-Fe* or *α-(Fe,Si)* nanocrystals in the superparamagnetic state and located in the residual amorphous matrix [42].

5. RAM with Nanocarbon Fillers

Nanosized electrically conductive fillers, for example, fullerenes, graphene [43], and CNTs [44], have a wide prospect for creating appropriate RAMs due to their small size and high electrical conductivity. The presence of thin fibrous inclusions in the composition of the CM makes a more significant contribution to the dielectric losses. Inclusions with large linear dimensions limit the technical applications, such RAMs. For example, sheet materials based on carbon fibers of millimeter size are characterized by anisotropy of the permittivity along and across the sheet plane. The clusters can appear in CNT-based RAMs. Its presence as well as their shape and geometric sizes, the value of their capacitances, and conductivities are affected by the frequency dispersion of the permittivity of the material and the expansion of the frequency range. It should be noted that the electrical resistance of contacts between particles and clusters can be much higher than the ohmic resistances inside the particles themselves. In some cases, this effect can make the main contribution to the electrodynamic properties of the material [45][46].

It is known RAMs based on $BaFe_{12}O_{19}$ (BHF) /MWCNTs/PANi nanocomposite [47]. As it was observed due to the presence of PANi, the absorption increased extraordinarily. The absorber exhibited a maximum reflection loss of −24.2 dB at 11.6 GHz.

The manufacturing method and properties of structural RAM based a polyurethane composition with CNTs deposited on walls of honeycomb plastics are described in [48]. By varying the content of CNTs in the compositions, the possibility of tuning the radio technical characteristics of the RAM was shown. The best characteristics on a single-layer composite were obtained at a CNT concentration of 5.6 wt. % (the minimum EC value of *R* was −24 dB at the resonant frequency of the operating frequency range of 2–18 GHz). With an increase in the number of layers of the functionalized honeycomb, a further decrease in the values of *R* was noted.

The methods for regulating the electromagnetic parameters of RAMs based on polyurethane with multiwalled CNTs introduced into its volume are described in [49]. The characteristics of both CNTs and composites were changed by modifying the CNT surface with ferromagnetic metal oxides of various concentrations.

In a number of cases, in order to achieve lower values of the *R* of EMR and expand the operating RPM range, groups of various functional fillers are used. These fillers are introduced as a mixture into the volume of matrices. Both mixtures of magnetic powders and non-magnetic particles in combination with magnetic inclusions can be used as such fillers [50][51][52].

It is known multilayered RAMs with varying values of parts of the magnetic and dielectric permittivity from layer to layer are made on the basis of ferrite and carbonyl iron powders [53][54][55]. Another example of the technical implementation of RAM based on groups of functional fillers is the material described in [56][57]. Here, to ensure the absorption of EMR in the sub-bands of the range from 2 to 60 GHz, powders of hexagonal barium and strontium ferrites were introduced in combination with ultrafine powders of spinel ferrites and iron carbide.

A material based on latexes with functional fillers as combinations of fullerenes with powders of carbonyl iron and ferrites is described in [58]. The resulting composite ensures efficient absorption of the incident EMR energy in the frequency range from 2 to 20 GHz. The aim to broadening the operating frequency range of the RAM is solved through the use of groups and mixtures of various functional fillers. It was shown that at CNTs combining with nanosized magnesium-zinc ferrite powders ($Mn_{1-x}Zn_xFe_2O_4$ (x = 0.0 и 1.0)) the value of the *R* of EMR obtained in the frequency range from 8 to 12 GHz did not exceed −10 dB [59]. The RAMs based on CNTs and magnetite nanoparticles, providing the values of the *R* of EMR of no more than −15 dB in the frequency range from 10.2 to 18 GHz and with a thickness of no more than 3.0 mm, is described in [60].

The RAMs based on nanostructured graphene oxide and magnetite powder with various concentrations are presented in [61]. It was found that the joint introduction of the investigated functional fillers allow to control the values of the dielectric and magnetic permeability in the microwave range. For samples containing a mixture of magnetite powder and graphene oxide (at a concentration of up to 3 wt. %), lower values of the *R* of EMR in the frequency range from 2 to 18 GHz are provided compared with RAM samples based on magnetite powder alone. With a sample thickness of 1.7 mm, the values of the *R* of EMR were no more than −10 dB in the studied frequency region.

It is necessary to note that the choice of matrix material during RAM design is very important due to the need to ensure the required performance depending on the application. It is also necessary to take into account manufacturability and economic indicators.

Currently, there are promising works aimed at creating structural RAMs for various purposes [62][63]. Reinforced composites are known, which are fiberglass based on an epoxy binder containing resistive carbon fibers [64]. Another example of the implementation of a structural RAM is a laminated composite consisting of layers of fiberglass in combination with a lamellar porous structure [65]. Modern methods for modeling and obtaining structural RAMs, including those with a gradient structure, are 3D-printing technologies [3][66]. It should be noted that materials with a large number of air-filled cavities and cells in its volume have lower values of dielectric permittivity compared to similar close-packed matrices. Classical examples are porous-cellular (foam plastics [67] [68], spheroplastics [69]), and porous-fibrous (non-woven fabrics [70][71] and mats [72]) materials.

A lightweight covering RAM based on polyacrylonitrile fibers and nickel with cobalt materials introduced into its volume and applied to its surface at the manufacturing stage is described in [73]. According to the authors' results, the described RPM provides values of the *R* of EMR that do not exceed −20 dB in the operating frequency range.

6. RAM Based on Elastomers

A method for synthesizing a functionalized nickel–carbon porous material where the formation of *Ni* and carbon nanoparticles occurred simultaneously at the material formation stage is presented in [74]. The density of the obtained RAM was 0.1 g/cm^3, the value of the *R* of EMR at a thickness of 2 mm at a frequency of 4.5 GHz was no more than −10 dB, and at a frequency of 13.3 GHz, it was −45 dB.

Increasing the operating temperature range of CM greatly expands the possibilities of their application. Organosilicon materials are among the most heat-resistant (workable up to 300–400 °C) polymer binders and compositions, including those resistant to UV radiation and water. These elastomers are widely used in the creation of RAMs with such fillers as iron nanoparticles, pyrite ash, and carbonil iron particles [75][76][77].

Taking into account the wide range of RAMs and functional fillers of various types, there are a large number of methods for their production and technologies for their manufacture. Questions on the development of promising technologies for the manufacture of RAMs with improved properties still do not lose their relevance.

The following most common manufacturing methods depending on the matrices used in composites can be distinguished. Sintering technologies of raw materials at various temperatures and holding times are usually used to create RAMs based on ceramic matrices [78][79]. For RAMs based on textile and fibrous structures, impregnation methods and needle-punched fabrication methods are used [80][81].

There are a large number ways to produce the RAMs based on polymer matrices. An important role in the choice of processing technology is played the chemical composition, rheology, thermal properties of the binder, the type of curing used, and the composition and concentration of the filling. For RAM based on thermoplastic matrices, the following processing methods are widely used: (i) molding under pressure in a press or autoclave [82], (ii) extrusion methods [83], and (iii) additive technologies [84][85]. Such methods as pouring compositions [86], pressing [87][88] and autoclave molding [89], paint and varnish application methods [90][91], technologies for manufacturing porous structures [92], and impregnation technologies [93][94] are used for production the RAMs based on fiber-reinforced polymer composites. In some cases, to expand the operating radio frequency range and ensure a set of requirements for performance characteristics (physical, mechanical, thermal, resistance to external factors and so on), RAMs based on combinations of different materials manufactured according to their technology are used [95].

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