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IMPLEMENTATION OF MAGNETRON SPUTTERING METHOD IN OBTAINING A RADIO-ABSORBING THIN FILM BASED ON CoC

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This paper describes the main results of the study of obtaining samples of radio-absorbing CoC films by magnetron sputtering and evaluates the data of experimental and computational studies on the relationship between the structure and properties of CoC films. To eliminate the ferromagnetic effect of cobalt, a composite target was used. The use of the composite target during magnetron sputtering ensures the production of films of a given and necessary composition. Structural-phase state of the obtained films was studied using XRD, SEM and TEM methods. A distinctive feature of the synthesized coating is the absence of a crystalline structure in some areas due to the amorphizing properties of cobalt and its tendency to form metallic glasses. Results obtained by the experiments are in good agreement with computational modeling results. The radio-absorbing properties of the obtained films were confirmed by the results of measuring the reflection loss, standing wave ratio, reflectance, and impedance. Results presented in the article can serve as a baseline for perspective studies in this field.

***Keywords**: structural-phase state, radio-absorbing coatings, magnetron sputtering, films.*

INTRODUCTION

Obtaining materials with specified properties having controlled dielectric and magnetic permeability is a very urgent task [1–5]. The development and increase in the power of microwave radio electronics devices lead to the fact that electromagnetic radiation arising during their operation at frequencies of higher types of harmonics creates significant interference to radio electronic equipment and satellite communications. The impact of electromagnetic radiation (EMR) of various nature on technical and biological objects is a significant factor affecting their functioning. Therefore, the development of new highly efficient broadband radio-absorbing materials is becoming very relevant to solve the problem of reducing interference and electromagnetic compatibility of devices [5]. However, in our opinion, insufficient attention is paid to coatings of the CoC system obtained by magnetron sputtering. In the development of electromagnetic wave absorbers, various materials are used that can absorb electromagnetic radiation in a certain frequency range. The ability of a medium to absorb electromagnetic radiation is determined by its electrical and magnetic properties, which include specific electrical conductivity, dielectric and magnetic permeability. Cobalt has ferromagnetic properties [6] and, in combination with carbon, is promising for creating an inexpensive material with magneto-optical properties [7].

Extensive efforts are being made to create anti-radar coatings for military equipment. This area has become widespread especially abroad, in particular under the Stealth program [3–5]. A significant reduction in mutual interference in the transmitting and receiving paths of devices operating in the range of 300 kHz – 14 GHz is possible due to the use of radio-absorbing magnetic materials of a new generation, providing an expansion of the functional and tactical and technical capabilities of electronic special equipment [3–4].

Modern radio-absorbing materials have low mechanical strength, low temperature resistance and a large reduced specific gravity (10–12 kG/m^2). In contrast, the new generation radio-absorbing materials (RAMs) under study have highly effective absorption of electromagnetic radiation in a wide frequency range with a low specific gravity (<1.5 kG/m^2), high strength and heat resistance, resistance to climatic influences and aggressive environments [8–9]. It is these factors that make it possible to use the materials to reduce the radio visibility of aircraft and light marine vessels, biological protection, as well as solve problems of electromagnetic compatibility in miniature electronic devices. This, in turn, determined the need for further research and the creation of modern technologies for obtaining new generation radio-absorbing materials.

Over the past few years, a few papers have been published describing the results achieved in the study of radio-absorbing materials in the range of 30–60 GHz [5]. Nanostructured materials are known to be used as radio-absorbing materials [10–13].

A radio-absorbing coating containing layers of aramid fabric with film heterostructures deposited on it is characterized by the heterostructure of 2–8 layers of amorphous hydrogenated carbon with 3d metal nanoparticles, while the layers in the heterostructures alternate the concentration of ferromagnetic nanoparticles in the films of neighboring layers is different – low in one (0–30 wt.%), in the second it is high (80–95 wt.%) [14]. The disadvantage of this material is its significant thickness, which is necessary for effective absorption of radio waves.

An electromagnetic absorbing material can be created by merging shielding and thermal insulation properties into a single substance, achieving an optimal balance between density and strength. This material is a granulate composed of a blend of carbon and ferrite powders, with a glass (quartz powder) binder. The material is produced using ceramic manufacturing techniques, including extrusion and casting. Its absorption properties are up to 2 dB across a wide range and can reach 10 dB in a narrow range (up to 5 GHz) [15]. However, this material has some drawbacks, such as low absorption capacity, a high specific weight exceeding 10 kg/m², and a complex production process due to its multicomponent composition, which can result in an uncontrolled final composition.

Another electromagnetic absorbing material is made from a mixture of 0.30–0.45 or 0.55–0.75 mole fractions of strontium titanate and 0.70–0.55 or 0.45–0.25 mole fractions, respectively, with the general formula $BiMO_3$, where M is an element such as chromium, manganese, or iron. This material exhibits high values of the real part of the dielectric constant and significant dielectric losses in the microwave range (ultrahigh frequency) [16]. However, it lacks elasticity, requires considerable thickness for effective absorption, and can only be used in tile form.

Ferrites, magnetodielectrics, and nanostructured polymer-based composite materials can also serve as radio-absorbing materials. $ZnFe_2O_4$ and $NiFe_2O_4$ nanoparticles are of particular interest due to their unique properties, such as high magnetic permeability, high Curie temperature, high electrical resistivity, and low power loss. In the classification of hexagonal ferrites, the resonance frequency (W-type) for $BaM_2Fe_{16}O_{27}$ ferrite (M = Fe, Ni, and Zn) is approximately 36 GHz. Substituting Co in the structure alters the hexagonal configuration, with the resonant frequency for $BaCo_xZn_{2-x}Fe_{16}O_{27}$ estimated to be between 2.5 and 12.0 GHz [17–19].

It is important to note that the effectiveness of protection against microwave radiation depends on many factors, including the frequency of radiation, the power of the radiation source and the thickness and composition of the protective material. Therefore, choosing the optimal material structure for protection requires taking into account specific conditions and requirements.

One of the most effective structures for protection against microwave radiation consists of arrays of microstructures with unique electromagnetic properties not found in natural materials. CoC thin films are particularly promising for these applications. These films can be produced using the magnetron sputtering method, which offers several advantages: a high deposition rate, minimal substrate temperature increase, low film damage, and compatibility with scraper material manufacturing. Films created through this method exhibit high purity, good compactness, uniformity, and excellent adhesion to the substrate. The process is highly reproducible, allowing for consistent film thickness over large areas. Additionally, particle size can be controlled by adjusting the parameters, and various metals, alloys, and oxides can be co-deposited onto the substrate. This method is also conducive to industrial-scale production.

The aim of this study is to create thin CoC-based films using magnetron sputtering technology and to study its structural-phase state and radio-absorbing properties.

MATERIALS AND METHODS

The creation of CoC coatings (CoC_{100-m} – m varied in the range of ~ 30–35 at.%) was carried out by magnetron sputtering in an inert argon medium of graphite (C) and ferromagnetic (Co) targets with a controlled concentration of constituent elements on the Si substrate.

To obtain coatings, a magnetron system with dual magnetrons with a pulse frequency of 40 kHz and a duty cycle of 0.5 was used. A single-crystal silicon substrate was placed at the geometric focus of the magnetrons at 15 cm. Before deposition, the substrate was cleaned with an Ar+ ion source with an accelerating voltage of 3.5 kV for 30 min. The concentration of the constituent elements in the coating is controlled by the power supplied to the targets. The thickness of the layers is controlled by the spraying time. The concentrations of C and Co introduced into the films were changed by changing the power supplied to the target cathodes. The films were obtained by simultaneous sputtering of targets; the film growth rate was on average 10–13 nm/min.

The use of a composite target is due to the fact that such a composite target has a ratio of the surface areas occupied by the composite elements, and during magnetron sputtering ensures the production of films of a given and necessary composition. The target size is chosen as follows: the diameter (C) of the target is 9 mm, the diameter of the disc is 16 mm.

The main limiting factor when implementing the magnetron sputtering of coatings is that cobalt, being a ferromagnet, closes the lines of the magnetic system of the magnetron, transferring it to the diode operating mode, which makes it impossible to use a cobalt disk target. To solve this problem, a composite target was used (Figure 1). As a result of partial closure of the magnetic lines of the magnetron, the discharge did not go into the diode mode, which made it possible to regulate the power density on the target and control the composition of the resulting coating. Graphite target diameter was 9 mm (99.9999% purity with fine grain structure, Phildal Holding Co., China), Co disk diameter was 16 mm (99.99% purity, Phildal Holding Co., China). The diameter of the pressed disks was selected based on the atomization coefficient of this element and its required concentration in the final coating. Pressure before application was $8\cdot10^{-4}$ Pa. Working pressure of Ar was 1 Pa. Additional heating of the substrate was not used; the substrate temperature during the deposition process was <100 °C and measured by using IR-710 infrared thermometer

(Amprobe, USA). The distance from the substrate to the magnetron was 5 cm. The time for applying a separate layer was from 5 to 15 minutes. The Co/C magnetron current was 0.5 A, the Co/C magnetron voltage was 450 V. Thus, samples of CoC film deposited on a silicon substrate with a thickness of 0.35–0.38 μm were obtained.

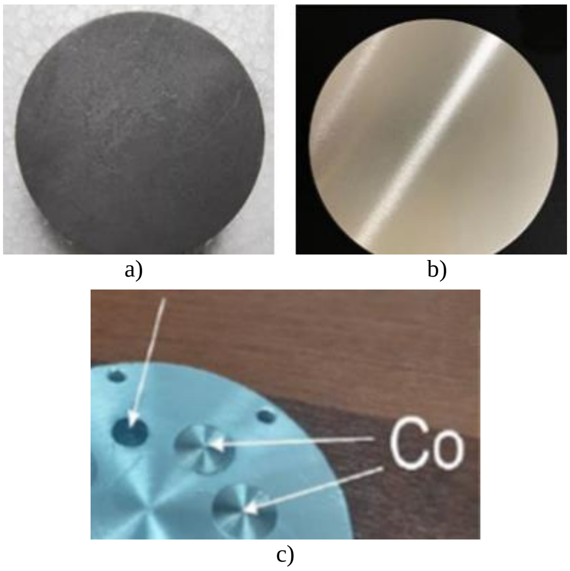


Figure 1. Appearance of: a) graphite target; b) cobalt target; c) composite target

The results of measuring the roughness, microhardness and erosion (abrasive) resistance of the samples are shown in Table.

Table. Mechanical properties of the coating

Parameter	Measurement result
Roughness, Ra, microns	18.4
Roughness, Rz, microns	125.6
Microhardness, HV0.1/10	1023.6
Mass loss / Time	4/20; 5.5/40; 6.2/60; 7/80; 7.3/100; 8.7/120; 9.5/140

X-ray phase analysis was performed on a Shimadzu XRD 6000 device with a Ka Cu anode ($\lambda = 0.154$ nm) by the sliding beam method (shooting angle 15) in the range $2\theta = 20 \div 80$.

The structural state and elemental composition were determined by scanning electron microscopy on a JEOL-2200FS (Japan) (EDX), INCA Energy (Oxford Instruments, UK) scanning electron microscope. Accelerating voltage up to 30 kV; resolution up to 3 nm; instrument magnification up to x300,000.

The microstructure of the films was studied, inter alia, by transmission electron microscopy on a JEOL-2200FS microscope (JEOL, Japan). During the sample preparation, a scratch was applied with a Fine Point Diamond Scriber 54467 (Ted Pella, USA) to the substrate on the reverse side of the sample. The samples are also subjected to additional thinning by ion etching (Gatan Precision Ion Polishing System Model 691).

Studies of the absorbing characteristics of the films were carried out using the Vector network analyzer P9373A equipment in the frequency range 300 kHz – 14 GHz, manufactured by Keysight Technologies (USA). Measured parameters: reflection loss (S11/S22 LogMag); standing wave coefficient (S11/S22 SWR); reflection coefficient (S11/S22 LinMag); impedance (SmithChart). These parameters were measured in the frequency range of 8.2–12.4 GHz.

Modeling of three-dimensional structures and diffractograms of crystals was carried out in the VESTA software package, in accordance with the international database of materials [IRIC], $\lambda = 0.154$ nm. The spatial structure of an ideal crystal is modeled. Further, based on the Wolf-Bragg condition, X-ray spectra of model structures were calculated. The X-ray spectra and interplane distances of the obtained coatings were compared with the spectra obtained by modeling methods and deviations were detected. Also, based on the obtained spectra of coatings, the reverse operation is performed to identify structures by spectra. Thus, it is possible to perform operational control of the received structures without resorting to the use of databases.

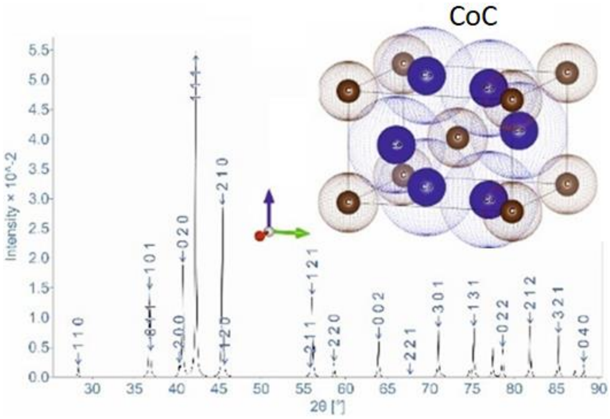
RESULTS AND DISCUSSION

Figure 2 shows the simulated geometry and X-ray spectra of the CoC structure using the Vesta atomic structure software program, which is designed for 3D visualization of structural models and various volumetric data such as electron/nuclear density, and crystal morphology. This program allows you to visualize the morphology of crystals, isosurfaces with several levels, to carry out an advanced bond search algorithm that allows for a more complex search in complex molecules, cellular structures, etc. to carry out calculations of the electron and nuclear density according to the structure parameters, to determine the best planes for selected atoms, etc**.**

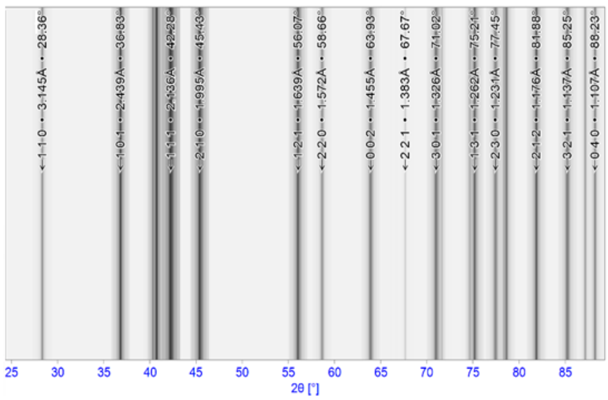
Based on the simulation results, it was found that the structure consists of a crystal with an orthorhombic lattice with an atom in the center, which corresponds to a FCC lattice: $a = 4.469$, $b = 4.426$, $c = 2.9107$, $\alpha = 90$, $\beta = 90$, $\gamma = 90$. Since cobalt is a ferromagnetic material, carbon can affect its magnetic and optical properties. The study of the mutual arrangement of atoms in a crystal after sputtering will allow us to study the magneto-optical properties of magnetron CoC films. This is possible when comparing reference structures and film structures.

Figure 3 shows the structure, electronogram and decoding of the resulting film.

The results of the conducted studies have shown that with the selected mode of thermal exposure in an inert Ar environment at 400 °C, certain modifications occur on the surface of the coating material. The physical and mechanical properties of a surface are determined not only by morphology, elemental composition, but also by phase composition. The main phases were determined using X-ray diffraction analysis.

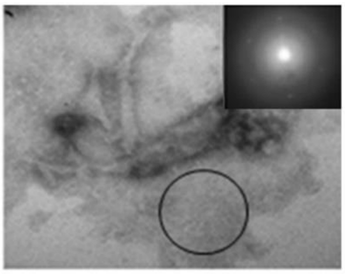


a)



b)

Figure 2. Structure of CoC: a) X-ray spectrum with HKL; b) X-ray spectrum of the CoC structure with d-spacing



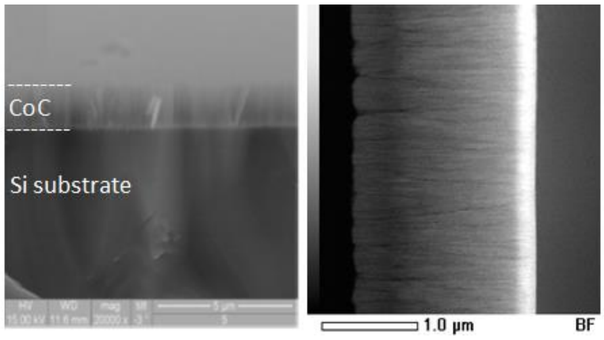
d, nm	(hkl), phase
3.135	(111), FCC
1.934	(200), FCC
1.646	(220), FCC
1.358	(131), FCC

Figure 3. TEM image of the microstructure of the film sample, with magnification x33000

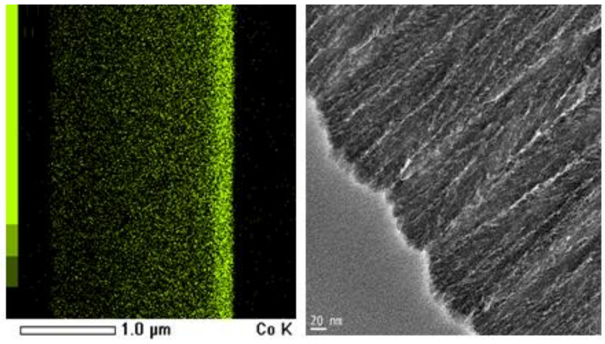
The resulting film structure demonstrates identical HKL planes, but with altered d (nm), which indicates the formation of this structure, but at the same time a slightly deformed crystal lattice is observed. This indicates a change in the parameters a, b, c. It was found that the changes in the lattice lie in the range of acceptable values, which indicates that the resulting structure demonstrates magneto-optical properties.

The results of the microscopy study of the cross section of coatings and the corresponding energy dispersion spectra are shown in Figure 4.

The figure shows that fairly dense coatings have been formed, characterized by a columnar structure, which is common in the metal coating system (Figure 4b, d). The coatings have a highly relief character, formed at the deposition stage of the coating. The thickness of the synthesized CoC coating is equal to an average of 0.3±0.2 microns (Figure 4a, b). Figure 4b clearly demonstrates the formation of a clear interfacial boundary between the coating and the substrate without any diffusion displacement, the interfacial boundary "coating – substrate" is clear. This was achieved by carefully selecting the selected deposition parameters.



a) b)



c) d)

Figure 4. SEM of a cross-section of a coating with an energy dispersion spectrum: a) a general view of the cross section shows the cross section of the substrate and the coating; b) the interfacial boundary between the coating and the substrate; c) the energy dispersion spectrum of Co; d) the columnar structure of the coating

In some places of the surface layer of the substrate, partial deformation occurs, a kind of microchaneling by individual particles of powder components, that is, volumetric saturation of the matrix with the material of embedded particles has been realized. There are areas at the interface where, at the initial stage of coating formation, particles of powder components are embedded in the substrate surface. During the impact, the interaction of dispersed particles with the barrier occurred, where pressures are realized in local areas. In this regard, the interaction of a particle with an obstacle is accompanied by intense deformation in the area directly adjacent to the trajectory of the particle. And as a result, the channel walls are a layer of increased hardness, which introduces an additional strengthening effect.

The study of the local and integral elemental composition of the coating shows that its matrix has a gradient character. Studies have shown that by changing the gradient of the properties of the medium, it is possible to control the structure of the surface wave field, to achieve either its localization in the near-surface zone, or its suppression. According to the data of energy dispersion analysis, the concentration of cobalt in the coating increases with increasing thickness of the sprayed layer, Figure 4c.

Judging by the results of the EDS spectrum of the distribution of the composite coating elements, the distribution of the composite coating elements from point to point practically does not change. We believe that the presence of silicon atoms in the coating is due to its ingress from the detection of the substrate itself, since the distribution of silicon atoms over the depth and width of the coating is homogeneous.

Figure 5 shows transmission microscopy images and the corresponding element mapping. Transmission microscopy results confirm SEM and EDS measurements.

A distinctive feature of the synthesized coating is the absence of a crystalline structure in some areas of CoC coatings, which is most likely due to the amorphizing properties of cobalt and its tendency to form metallic glasses. Obtaining an amorphous state is primarily associated with nonequilibrium processes occurring during the formation of thin films. The state with the absence of long-range order (the absence of correlations between atoms at large distances) is determined while maintaining the short-range order (the presence of such correlations on several (maximum – two or three) coordination spheres. The process of forming the structure of the coatings under study during magnetron deposition is characterized by a number of patterns: The CoC coating has a layered structure with an anisotropic pore distribution; When the coating material is deposited, a certain texture is formed in the form of zones of columnar crystals, while their axes are located normally to the surface.

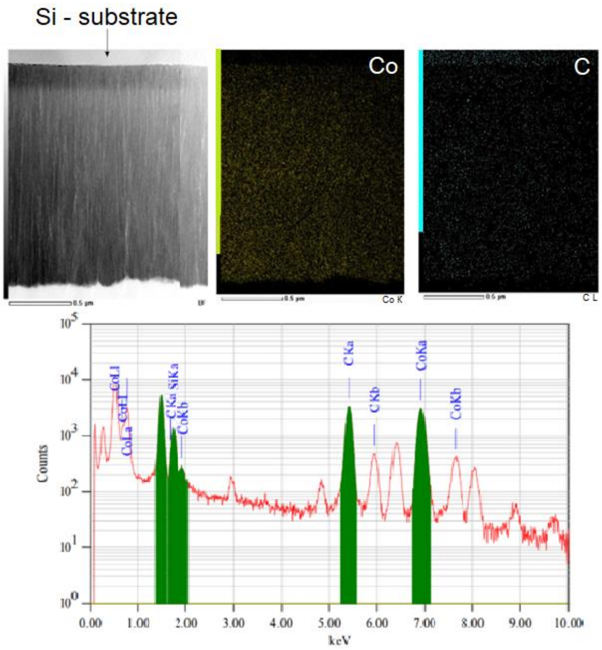


Figure 5. TEM-microscopy of the cross-section of the coating and its element mapping

The results of measuring reflection losses (S11/S22 LogMag), the standing wave coefficient (S11/S22 SWR) and the reflection coefficient (S11/S22 LinMag) from a sample of a film with a thickness of 0.35–0.38 microns are shown in Figure 6.

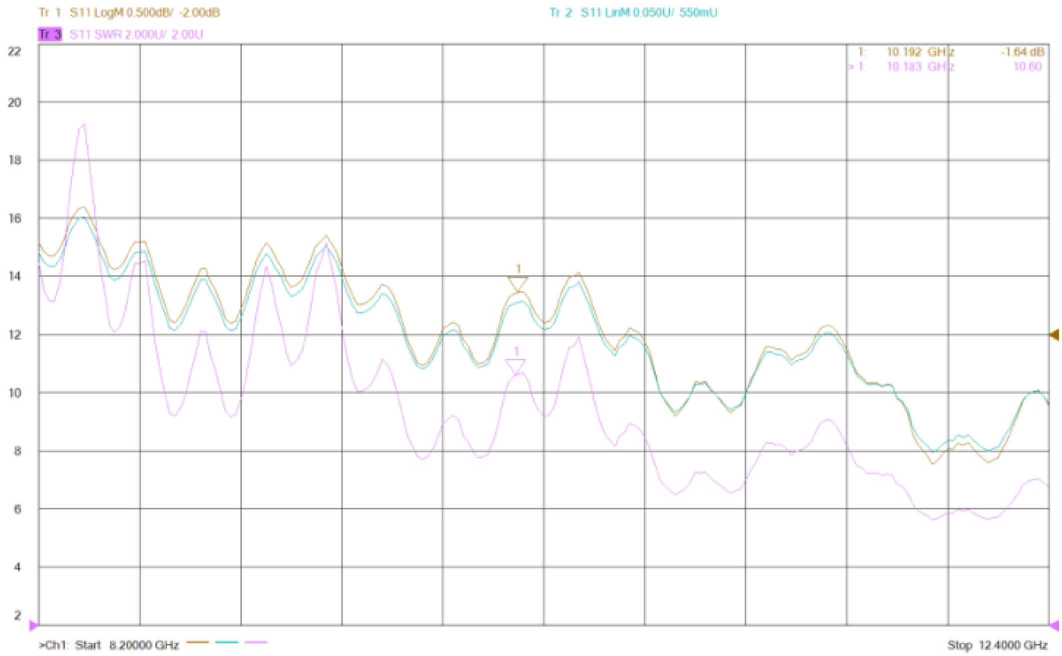


Figure 6. Change in the values of S11/S22 LogMag, S11/S22 SWR and S11/S22 LinMag in the frequency range 8.2–12.4 GHz, for a sample of the CoC film

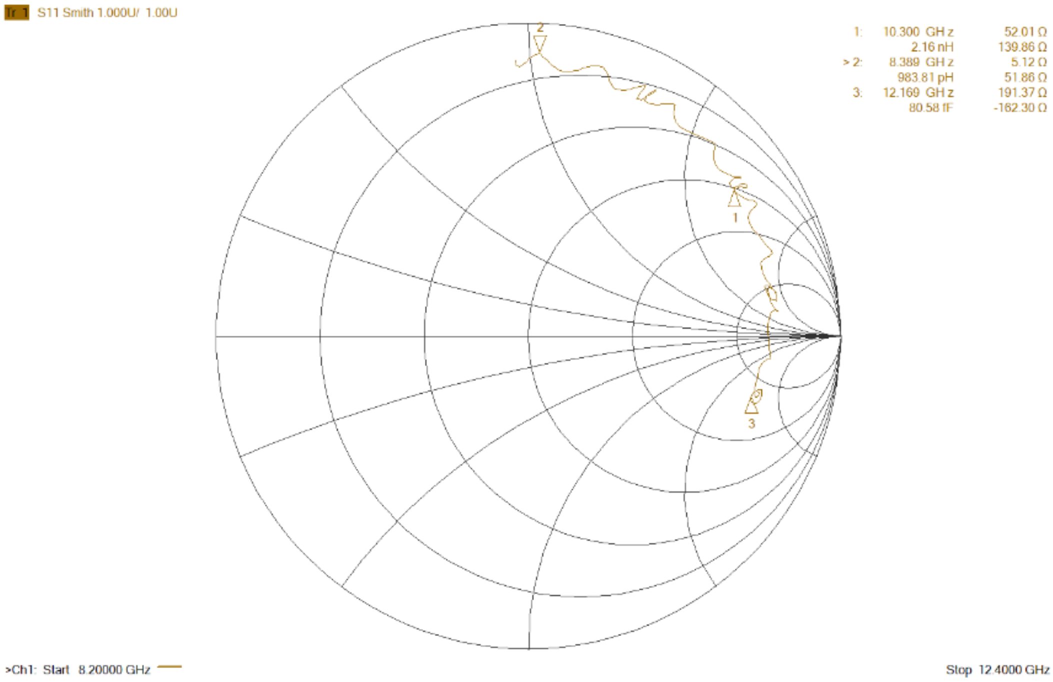


Figure 7. Change in impedance values (SmithChart) in the frequency range 8.2–12.4 GHz, for a sample of the CoC film

In the upper right corner of each graph, numerical values for three markers are shown: 1 – the numerical value at the midpoint of the 8.2–12.4 GHz frequency range; 2 – the maximum value of the measured value; 3 – the minimum value of the measured value.

The results of measuring the impedance values (Smith Chart) from a sample of the CoC film, presented using the Wolpert-Smith diagram, are shown in Figure 7.

In Figure 7, the scale of the pie charts is non-linear. The grid lines correspond to the points of constant active and constant reactive resistance. Points with the same active resistance are arranged in circles. Points with the same reactance form a set of arcs of different curvature. All resistance values are normalized relative to a certain wave impedance of the system $Z_{0,\ -50}$ Ohms. In a pie chart, the grid lines are arranged in such a way that the area of positive active resistances is displayed as a single circle. The central horizontal axis corresponds to zero reactance. The center of the diagram is $Z/Z_0 = 1$, which corresponds to the wave impedance of the system. At the left and right points of intersection of the horizontal axis with the outer circle, the impedance is zero and infinity, respectively. The outer circle corresponds to zero active resistance. The upper and lower halves of the diagram correspond to positive and negative reactivities, respectively. The value of the impedance modulus for the test sample at a frequency of 8.3 GHz (Figure 7) is 5.12 Ohms, at a frequency of 10.3 GHz it increases to 52.0 Ohms and at 12.1 GHz – 191.37 Ohms.

Thus, the resulting CoC film at a frequency of 8.3 GHZ has a reflection coefficient of 900 mU; at a frequency of 10.3 GHz – 805 mU and at a frequency of 12.1 GHz – 718 mU. The study of reflection loss values at a frequency of 8.4 GHz is −0.86 dB, at a frequency of 10.3 GHz −1.86 dB and at a frequency of 12.1 GHz, reflection loss is −2.57 dB. The standing wave coefficient at a frequency of 8.3 GHz is 19, at a frequency of 10.3 GHz is 9.5 and at a frequency of 12.1 GHz is 6.8. The analysis of the general graph of the absorbing characteristics of the material indicates the presence of significant losses of electromagnetic energy in the frequency range 8.2–12.4 GHz. There is a monotonous sinusoidal decrease in the values of the studied parameters with a decrease in amplitude with an increase in the frequency of the electric current. The observed values of reflection losses are typical for volumetric absorbers of electromagnetic energy. This results indicate the possibility of using the studied CoC films as a radio absorbing material, and will serve as the basis for further studies in this direction.

Conclusion

Magnetron sputtering technology was applied to create a thin-film material based on CoC. To eliminate the ferromagnetic effect of cobalt, a composite target was used. As a result of partial closure of the magnetic lines of the magnetron, the discharge did not go into the diode mode, which made it possible to regulate the power density on the target and control the composition of the resulting coating. Structure analysis results showed the correlation with the calculation. A distinctive feature of the synthesized coating is the absence of a crystalline structure in some areas of CoC coatings, which is most likely due to the amorphizing properties of cobalt and its tendency to form metallic glasses. It was found that the obtained material is able to absorb electromagnetic waves due to the structure of the films. Future studies in the field of using the magnetron sputtering method for obtaining CoC material will involve optimization of the synthesis, controlling the parameters, and detailed analysis of the mechanisms of the CoC film formation during the magnetron sputtering. The electronographic analysis established the correspondence of the obtained structures to the calculated structures. The resulting film is a material with similar complex magnetic and dielectric permittivity at a magnitude of magnetic losses several times higher than traditional magnetic materials, but this assumption needs to be proven by further enhanced experiments under controlled conditions and using real samples. These materials can be the basis for the creation of effective broadband electromagnetic wave absorbers.

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CoC НЕГІЗІНДЕГІ РАДИОЖҰТҚЫШ ЖҰҚА ҚАБЫҚШАЛАРДЫ АЛУДА МАГНЕТРОНДЫ БҮРКУ ӘДІСІН ҚОЛДАНУ

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Мақалада магнетронды бүрку арқылы алынған радиожұтқыш CoC қабықшаларының үлгілерін зерттеудің негізгі нәтижелері сипатталған және CoC қабықшаларының құрылымы мен қасиеттері арасындағы байланыстың тәжірибелік және есептеулік зерттеулерінің деректері бағаланады. Кобальттың ферромагниттік әсерін жою үшін композиттік нысана қолданылды. Магнетронды бүрку кезінде композиттік нысананы пайдалану берілген және қажетті құрамдағы қабықшалар алынуын қамтамасыз етеді. Алынған қабықшалардың құрылымдық-фазалық күйі РФТ, СЭМ және ТЭМ арқылы зерттелді. Синтезделген жабынның айрықша ерекшелігі кобальттың аморфизациялық қасиетіне және оның металл шынылар түзуге бейімділігіне байланысты кейбір аймақтарда кристалдық құрылымның болмауы болып табылады. Эксперимент барысында алынған нәтижелер компьютерлік модельдеу нәтижелерімен жақсы сәйкес келеді. Алынған пленкалардың радиожұтқыш қасиеттері шағылу жоғалтуларын, тұрақты толқындардың қатынасын, шағылу коэффициенттерін және кедергілерді өлшеу арқылы расталды. Мақалада келтірілген нәтижелер осы саладағы болашақ зерттеулерге негіз бола алады.

***Түйін сөздер**: құрылымдық-фазалық күй, радио сіңіргіш жабындар, магнетронды бүрку, пленкалар.*

ПРИМЕНЕНИЕ МЕТОДА МАГНЕТРОННОГО НАПЫЛЕНИЯ ПРИ ПОЛУЧЕНИИ РАДИОПОГЛОЩАЮЩЕЙ ТОНКОЙ ПЛЕНКИ НА ОСНОВЕ CoC

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В работе описаны основные результаты исследования образцов радиопоглощающих пленок CoC, полученных методом магнетронного напыления, и дана оценка данных экспериментальных и расчетных исследований взаимосвязи между структурой и свойствами пленок CoC. Для устранения ферромагнитного эффекта кобальта использовалась составная мишень. Использование составной мишени при магнетронном напылении обеспечивает получение пленок заданного и необходимого состава. Структурно-фазовое состояние полученных пленок исследовали методами РФА, СЭМ и ПЭМ. Отличительной особенностью синтезированного покрытия является отсутствие кристаллической структуры на некоторых участках из-за аморфизирующих свойств кобальта и его склонности к образованию металлических стекол. Результаты, полученные в ходе экспериментов, хорошо согласуются с результатами компьютерного моделирования. Радиопоглощающие свойства полученных пленок были подтверждены результатами измерения потерь на отражение, коэффициента стоячей волны, коэффициентов отражения и импеданса. Результаты, представленные в статье, могут послужить основой для перспективных исследований в этой области.

***Ключевые слова**: структурно-фазовое состояние, радиопоглощающие покрытия, магнетронное напыление, пленки.*