Radio-shielding metamaterials transparent in the visible spectrum: approaches to creation

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Abstract. The approaches to creation of the materials providing simultaneously high indicators of transparency in the visible spectrum and shielding in a wide radio frequency band are considered in this paper. The analysis and comparison of the main designs of such materials, as well as approaches to their creation, including multilayer and conductive mesh structures, is carried out. The results of our own theoretical studies of the disordered mesh structure are presented, which allow one to obtain a light transmission coefficient from 90 to 98 % in combination with an electromagnetic interference shielding efficiency from 50 to 65 dB. The best results practically achieved to date (shielding efficiency equal 45 dB in the range from 10 kHz to 20 GHz with a light transparency of more than 80 %) were obtained on mesh structures by photolithography, which is a significant limiting factor of this approach. The created multilayer structures show, in general, lower characteristics. However, the technology for their production is better scaled, and the optimization of the thicknesses and chemical composition of multilayer structures can significantly increase them. In this regard, technological aspects may come to the fore when taking into account the possibility of subsequent scaling of the technology and economic indicators when choosing an approach for the implementation of the materials with the required characteristics.

1. Introduction

The rapid development of information technology in the modern world leads to the widespread use of various electronic devices, which increasingly makes the problem of their electromagnetic compatibility more relevant. The operation of many electronic systems and micro-electromechanical devices requires their isolation from exposure to extraneous electromagnetic waves and noise, or vice versa, the need arises to protect against the radiation of the devices themselves, which today is becoming a serious problem due to its adverse effects on human health [1, 2].

To date, the most common shielding method is the creation of protective metal cases. However, under modern conditions there is an increasing demand for materials that should not only have high shielding properties, but also have a different combination of other required operational properties, such as flexibility, transparency in different spectral ranges, weight and size characteristics, air permeability, etc. This makes it impossible to use traditional metallic materials and requires the development of new approaches and materials to protect electronic devices from electromagnetic waves.

This work focuses on the creation of electrically conductive materials that are transparent in the visible spectrum range, which, in addition to the task of electromagnetic radiation shielding, are widely used as transparent electrodes in portable electronic devices. At the same time, solutions used in industry allow to provide either high transparency (more than 90 %) at low electromagnetic interference (EMI)
shielding efficiency (SE) values (up to 20 dB), or high SE with a significant reduction in light transparency. In this regard, it is necessary to search such structures and technologies that allow to achieve high transparency and EMI shielding efficiency simultaneously.

Currently, two main approaches are used to obtain such structures: continuous multilayer structures and conductive mesh structures, as well as their combination. Each approach has its advantages and disadvantages, allowing to achieve various combinations of transparency and shielding efficiency in conjunction with other operational characteristics. This article is devoted to a review of promising design solutions and analysis of approaches to creating materials with high transparency and EMI shielding efficiency.

2. Multilayer structures
Such structures can be realized on the basis of doped wide-gap semiconductor materials from metal oxides and nitrides (ZnO, TiO₂, In₂O₃, SnO₂, Zn₃N₂, TiN, etc.), thin metal layers, and conductive organic compounds. The most commonly used transparent conductive layers are Indium tin oxide (ITO coatings) systems, which currently have the best ratio of electrical conductivity and light transparency in the visible range (at least 70%, with a sheet resistance of 15 - 20 Ω/sq, which corresponds to SE of about 22 dB), good adhesive and satisfactory corrosion properties. Moreover, their optical properties can be improved (light transparency of more than 90% at a sheet resistance of 20 Ω/sq) by applying antireflection layers. Figure 1 shows the results of measurements of such coatings obtained by the authors of the current work.

Figure 1. EMI SE (a) and transparency (b) of ITO-coating.

One of the disadvantages of ITO coatings is the high temperature of their growth and annealing, which complicates the deposition of such films on flexible polymer substrates and leads to their somewhat worse characteristics compared to ITO layers on glasses. In view of the recent intensive development of the technology of flexible LCD touch screens, an urgent area of research is improving the technical characteristics of flexible conductive layers.

In [12], the authors compare the characteristics of an ITO coating (95% In₂O₃ + 5% SnO₂) with the structure of ZrO₂ (90 nm) – Ni (8 nm) – ZrO₂ (90 nm) – Cu (22) – Ni (5 nm) – ZrO₂ (35 nm) based on
thin metal films, which are made in the form of thin alternating layers of metal with high electrical conductivity and magnetic permeability, separated from antireflective oxide layers, in order to increase light transparency. Both coatings were applied to glass substrates. The ITO coating after annealing in vacuum at a temperature of 350–400 °C with a transparency of 95 % has a sheet resistance of 10 Ω/sq (SE 15 - 20 dB in the range of 6 - 17 GHz and 26 – 27 dB in the range of 0.7 – 5.0 GHz). A structure based on thin metal films with a transparency of about 60 % has a sheet resistance of 2 – 3 Ω/sq (SE 26 – 33 dB); however, its deposition technology does not require high-temperature annealing, which allows it to be applied to non-heat-resistant flexible polymeric materials. Similar characteristics were obtained in [7] on the structure (ZrO$_2$, HfO$_2$, TiO$_2$) + Ni + (ZrO$_2$, HfO$_2$, TiO$_2$) + Au + Ni + (ZrO$_2$, HfO$_2$, TiO$_2$) deposited on glass. The shielding efficiency of such samples was not less than 25 dB in the range of 100 kHz – 10 GHz, the light transparency at a wavelength of 580 nm was not less than 55 %, and the sheet resistance of the screen was 2.5 – 3.0 Ω/sq.

In [13], a structure with a total thickness of 312 nm from seven alternating layers of silver and zinc oxide (Ag (11 nm) / ZnO (81 nm) / Ag (22 nm) / ZnO (81 nm) / Ag (22 nm) / ZnO (81 nm) / Ag (11 nm)) was deposited on a polycarbonate substrate, which had an SE in the range from 30 kHz to 1 GHz of more than 40 dB with a transparency of about 60 %. In [14], a dielectric-metal-dielectric shielding structure based on an ultrathin (8 nm) copper alloyed silver film (the electromagnetic Ag shielding, EMAGS) was proposed, with a transparency of 96 % and a SE of about 26 dB in a wide frequency band 32 GHz, covering all the X, Ku, Ka, and K bands. Coating of two such structures made it possible to achieve SE more than 30 dB in the range of 8 – 40 GHz with a transparency of 93 %, and their separation by a layer corresponding to a quarter of the wavelength was up to 50 dB. It is worth noting that the proposed EMAGS structure can be applied on a large surface area using vacuum deposition with roll-to-roll system.

In order to further improving the characteristics of transparent conductive multilayer coatings, a number of structures based on a combination of thin metal layers with wide-gap oxide semiconductors are proposed. In [15], optical materials consisting of alternating ITO and Ag layers were developed and produced. There shielding efficiency at layer thicknesses of 40 nm and 20 nm, respectively, exceeded 40 dB in the range from 0.1 GHz to 18.0 GHz, and even reached 70 dB at a frequency of 0.1 GHz. Experimental study of the coatings’ transparency with different ratios of layer thicknesses showed that the highest transparency about 90 % in the visible range was achieved for ITO and Ag thicknesses of 60 nm and 20 nm, respectively. The transparent layers based on conductive organic compounds have not yet allowed to achieve high functional characteristics. In [4], a thin film of polyaniline (PANI) synthesized by the method of self-stabilized dispersion polymerization (SSDP) [5-6] at a sheet resistance of 500 Ω/sq, which corresponds to SE 2.7 dB, showed a transparency of 55 - 60 % at wavelengths of 500 - 600 nm. Thus, the ITO coatings that are widely used today do not allow to obtain a high SE without a significant decrease in light transparency, which, as a rule, does not exceed 25 dB with a transparency of 90 %, while on flexible substrates they have shown a significant decrease. Higher values of these parameters can be obtained using multilayer structures based on thin metal layers (Ag and Ni, less often Au) in combination with antireflective oxide dielectric or semiconductor materials (ZrO$_2$, ZnO, HfO$_2$, TiO$_2$, ITO, etc.).

3. Mesh structures
A constructive feature of such structures is the creation of the conductive mesh on or inside the surface of a transparent substrate with certain parameters, such as: orderliness of conductors; cell geometry; geometry of conductors forming conductive tracks; electrical conductivity of the tracks.

Disordered conductive mesh are most often formed on the basis of silver nanofibers (AgNWs) randomly oriented on the substrate surface [16-21], while ordered grids are obtained by microelectronics methods [22-36]: photolithography or direct laser etching, etching through masks, and etc. At the same time, some authors have attempts to create ordered grids using methods of self-organization [37-42]. Different combinations of transparency, shielding, or sheet resistance can be achieved by adjusting the parameters of the mesh structures [22].
3.1. Disordered meshes
A common method of forming such meshes is application of a suspension containing metal nanoparticles (nanofibers) to a transparent substrate [16-21]. The metal particles form a disordered conductive network on the surface (Figure 2a) after removing the dispersing medium during the drying process.

In [16] a polyimide coating with AgNWs was obtained (average diameter 30 nm, length 30 μm), which made it possible to obtain transparency of about 90 % in the range 450 – 750 nm and SE about 25 dB in the frequency range 100 – 1500 GHz. In order to increase the electrical conductivity of the layer, galvanic deposition of copper was carried out, which made it possible to reduce the sheet resistance from 500 to 450 Ω/sq with a decrease in transparency to 80 % during the deposition of Cu for 6 min. An increase in the deposition time of up to 8 min reduced the resistance to 28 Ω/sq, which provided a shielding efficiency of 55 dB with a decrease in transparency up to 58 %.

In [17] a shielding film based on a disordered network of AgNWs, calcium alginate and polyurethane were presented. This structure has a high transparency (T = 92 %) and a shielding efficiency (SE = 20.7 dB). With an increase in the surface density of silver nanofibers from 58 mg/m² to 174 mg/m², the shielding coefficient can be increased to 31.3 dB while maintaining the transparency of 81 %. In addition, these transparent films have high mechanical stability under difficult operating conditions with 98 % and 96 % shielding preserved even after 30 minutes of ultrasonic treatment and 5000 bending cycles (radius 1.5 mm), respectively.

In order to improve the ratio of transparency and shielding (electrical conductivity of the layer), a number of studies have proposed various methods for ordering the location of Me nanoparticles on the surface of a substrate [37] or for creating grooves of a certain geometry with their subsequent filling with metal. In [43-45], such grooves are obtained using self-organization mechanisms associated with the destruction (cracking) of the sacrificial layer. The parameters of the resulting grooves depend on the chemical composition (for example, on the mass ratio between methyl methacrylate (MMA) and butyl acrylate (BA) in the emulsion) and the thickness of the applied sacrificial layer.

In [43], a method for producing a Ag mesh on an quartz substrate with a line width of 0.5 μm and a grid pitch of 30 – 80 μm by using the destruction of a sacrificial layer of 3 μm thickness, deposition into the obtained grooves Ag with a thickness of 200 nm, and subsequent etching of the sacrificial layer was described. In comparison with the ordered Ag grid (line width 5 μm, grid pitch 250 μm, layer thickness 200 nm), which gives a transparency of 95 % in the range 300 – 800 nm and shielding efficiency from 17 to 20 dB in the range 12 – 18 GHz, the obtained sample (the mass ratio between MMA and BA in the emulsion is equal to 2) has very similar transparency characteristics; however, the SE is only about 10 dB. By increasing the mass ratio between MMA and BA in the emulsion to 3, it is possible to increase...
SE to 28 dB with a transparency of 92%. An important feature of this technology is the possibility of applying conductive coatings on surfaces of complex shape and large area, including domed glass.

From our point of view, another promising approach that can be used to form conductive meshes of a given geometry is based on the technology of self-organizing “breathing figures” [37-39], which allows one to produce porous polymer films with a controlled pore size (Figure 1b). The concept of “breathing figures” describes the condensation of many drops of moist air upon contact of water vapor with a cold surface. At the same time, not all the processes and results of this method obtained in practice have been explained and modeled theoretically. Therefore, we will look towards the development of this method in our further works.

An analysis of the works on the creation of radio-shielding (conducting) transparent materials based on disordered meshes showed that some ordering of the conductors improves their transparency and shielding efficiency; however, the characteristics of such materials are still far from materials based on ordered grids obtained by lithography methods. At the same time, the development of technologies and methods of self-organization has great potential for improving the performance of the resulting structures with a slight increase in the cost of the technological process and the possibility of its scaling.

3.2. Ordered Grids

The most common methods for ordered grids producing are based on photolithography processes [22-30], which allow one to obtain a given geometry in a controlled manner (Figure 2c), while achieving maximum values of the shielding and light transparency of such materials. In [27], a Ta / Al / Ta multilayer grid (the width of conductive tracks is 15 μm and the grid pitch is 400 μm) was created by combining the magnetron sputtering process and photolithography. With an Al layer thickness of 150 nm this grid has a transparency of 86% in the range 500 – 2000 nm, sheet resistance 10.2 Ω/sq and SE 46 dB at 6 GHz. It is worth noting that the deposition of an ITO layer on such a structure with a slight decrease in light transparency (from 84% for ITO on glass to 80% for ITO over Ta / Al / Ta for 500 nm) allows one to achieve sheet resistance of the ITO + Ta / Al / Ta structure 7.8 Ω/sq which improves the shielding efficiency of the created coating with a slight loss of transparency.

In [28], by UV lithography followed by galvanic deposition of Cu with annealing in air, an oxidized copper grid of 2 μm thickness was obtained, which shows a transparency of about 80% in the range 400 - 800 nm and SE from 30 to 20 dB in the range 12 - 18 GHz. Similarly, metal grids with a grid pitch of 50 μm from Au and Cu were produced on a polymer substrate in [26]. These structures showed a transparency of 72% and 87% (at a wavelength of 550 nm) with a sheet resistance of 0.2 Ω/sq and 0.7 Ω/sq for Au and Cu, respectively, which is significantly lower than the resistance, for example, of ITO films with the same transparency.

It is worth noting that today most microelectronic factories and laboratories are equipped with equipment designed for substrates with a diameter of 300 mm, and the limiting factor is precisely the photolithography plants, which makes it difficult to obtain such conductive grids on large samples. In order to solve this problem, the process of forming the polymer “stamp” by the lithographic method was described in [46], which is used then to form a network of grooves of a given topology over a large area using the nanoimprinting method. In order to metallize the obtained topology, a paste based on silver nanoparticles was introduced into the formed system of grooves, followed by galvanic interrogation of Ni. The resulting materials with a light transparency of 80 – 85% (without antireflection layers) have SE 45 dB at a frequency of 8 GHz.

The lithography process can be used, not only to create a protective mask with subsequent etching of the structure through it, but also to create a conductive structure that is networked in the process of photochemical or photothermal synthesis from solutions of photosensitive heterometallic complexes. Such an approach was proposed in [29], where exposure to an emulsion layer of silver halide grains dispersed in gelatin on a PET substrate makes it possible to create a network of such grains with a line width of about 15 μm and a grid pitch of 300 μm. At the same time, the authors can achieve a distance of SE 40 dB in the range 1 – 800 MHz with a decrease in light transparency by 5% compared with the original substrate.
As an alternative to the lithography process, laser etching can be used. A similar approach, for example, was applied in [31], where the authors create a two-layer Au-Ni grid with a line width of 10.5 μm and a grid pitch of 200 μm by laser etching using a femtosecond laser. With a quartz substrate thickness of 16 mm on a two-layer sample, a 76 % transparency was obtained in the range of 400 – 900 nm at SE 60 – 70 dB in the range 1 – 5 GHz, while a single-layer sample showed 84 % transparency at SE 45 dB.

In a number of works [32, 33], 3D printing was used to create grid metal structures. In [32], using this method, grid and line structures with different parameters were obtained. For a silver grid with a line width of 15 μm and a grid pitch of 1000 μm, a transparency of about 93 % at 550 nm was achieved with a sheet resistance of 0.2 Ω/square. In the work, it is shown that line structures make it possible to achieve higher transparency with an increase in the layer resistance compared to grids.

A structure with high transparency and shielding indices can be constructed on the basis of clusters of metal rings that form a mesh similar to a conducting substrate [34-36]. In [34], such a structure, created by multi-step UV lithography followed by plasma-chemical etching of an Al layer 300 nm thick deposited on a quartz substrate (Figure 3), has a transparency of 95 % in the range 400 - 2400 nm at SE 26 dB at 12 GHz.

Figure 3. The structure of metal rings in the form of a floral pattern [34].

It is worth noting that the creation on the surface of an ordered grid of metal conductors often leads to the appearance of Moiré waves, which sharply reduce the contrast of the image. For this effect, we proposed the use of an artificially disordered mesh [47, 48]. Moreover, periodic (with toroidal topology) Voronoi diagrams of the set of points randomly distributed inside the unit cell with a uniform probability density were taken as the basis. In order to increase the optical homogeneity of the meshes, the Voronoi diagrams were additionally subjected to Lloyd relaxation [49]. The design of the proposed mesh is shown in Figure 4.

Based on the theoretical studies, the authors of this work proposed options for the geometric parameters of the mesh, which allow one to obtain a light transmission coefficient from 90 to 98 % in combination with a shielding coefficient in the radio wavelength range from 50 to 65 dB (Figure 5), which is typical for existing opaque materials. The technology of producing such meshes is currently under development. Thus, the analysis of practical results on the creation of materials based on a grid of ordered conductors showed the possibility of achieving high shielding efficiencies of up to 45 or more dB in the range from 10 kHz to 20 GHz with a light transparency of more than 80 %. Theoretical calculations show great potential for improving these indicators; however, the limiting factor of this approach is the complexity of the process and the issue of its scalability. At the same time, it is worth noting the recent work on obtaining such grids by the methods of additive technologies, which, with their further development, can solve these limitations.
Figure 4. (a) Transparent mesh formed by opaque conductor wires (b) single unit cell of the simplified model used in this study for numerical simulations (c) SEM image of the obtained structure

Figure 5. Theoretically calculated values of shielding (SE), transparency (T), and field of view (FOV) coefficients for various mesh sizes (l)

4. Hybrid structures
The methods for producing such materials combine two or more approaches to the production of electrically conductive transparent coatings, discussed earlier, in order to use the advantages of each method. Such structures are often a multilayer material containing a metal mesh in combination with other layers that also have electrically conductive properties [50-54]. In [50, 51], the processes of fabricating hybrid structures based on either randomly oriented AgNWs on polymer substrates in combination with a layer of reduced graphene oxide (RGO) [50] or a high-conductivity layer of two-dimensional graphene nanoplates (GNS) [51], respectively (structures A / RGO / AgNWs and GNS / AgNWs, where A is a protective layer of acrylic polymer). The shielding coefficient SE of the A / RGO
/ AgNWs structure was more than 24 dB, which is approximately two times higher than that of an ITO film on glass (about 10 dB) with a similar relative transparency (84 - 85 %).

The addition of a graphene oxide layer to a layer of randomly oriented AgNWs nanofibers [18] gives a slight increase in SE (the GNS / AgNWs film shows SE up to 26 dB in the Ku and K frequency ranges with a transparency higher than 78 %). It is worth noting that the examined films have good flexibility and high structural stability under various operating conditions. In [52, 53], hybrid ones were built on the basis of an Al metal grid with square cells formed on a quartz substrate by photolithography and graphene layers. Moreover, in [53] a different combination of metal and graphene layers is considered. With an optical transparency of 94 %, a sheet resistance of 20.7 Ω/sq was obtained in [52], and the maximum EMI SE value was 21 dB at 12 GHz, which is comparable with the best samples of ITO coatings on glass substrates [15].

The GMTD hybrid structure [53] (Figure 1) with two graphene layers and two metal grids with a period of 160 μm provides an SE exceeding 48 dB in the Ku band and SE exceeding 32 dB in the Ka band with a visible transparency of ~ 85 % at 700 nm. In [54], a hybrid structure was obtained by hot pressing two PET films, on one of which an ordered silver grid was formed by photolithography (with a period of 1, 2 or 3 mm), and the other was coated with a network of randomly arranged AgNWs with the addition of RGO, addition of which leads to an increase in the electrical conductivity of the layer. Such a structure with an Ag network with a step of 1 mm has the highest SE value of 43 dB at a frequency of 1.8 GHz with a transmission coefficient of 74 %, while the SE of the AgNWs / RGO layer does not exceed 10 dB.

Thus, hybrid structures can slightly improve the shielding and transparency of the resulting layers compared to, for example, metal meshes or ITO coatings, while providing other operational characteristics of the formed layer, such as structure flexibility, mechanical stability, and resistance to environmental factors environment, etc. At the same time, this approach leads to some complication in the technological process of obtaining such structures, therefore, the feasibility of its application will depend on the particular problem being solved.

5. Discussion

Based on the results of the analysis of scientific and technical literature, a consolidated graph has been constructed (Figure 6), which displays the practically obtained shielding efficiency (SE) and light transparency (T) for the various types of structures considered above. When constructing it, the frequency dependences of SE and T, methods and measurement technics, which can lead to significant errors, have not been taken into account. However, the obtained data still allows to identify promising approaches for obtaining materials with a certain combination of transparency and radio shielding efficiency.

If the task is to obtain materials with SE less than 30 dB and maintain light transparency at 80 %, all the considered approaches (multilayer coatings, including ITO, ordered and disordered mesh structures) can be used to obtain specified characteristics. However, the use of ITO coatings is currently limited to application to inflexible substrates (such as glass), since their manufacture with SE up to 25 dB and a transparency of about 90 % requires the operation of high-temperature annealing. An increase in the SE requirements to 40 dB or light transparency up to 90 % or more leads to the need to complicate the structure and the technological process of obtaining such a layer, including the increase of the layers number in a multilayer structure or adding continuous conductive layers based on wide-gap semiconductors or graphene to disordered conducting networks, thus obtaining hybrid structures. In this case, replacing disordered fibers with an ordered conducting grid in such a hybrid structure with graphene oxide layers can increase SE from 26 to 48 dB with the same light transparency (up to ~ 85 %).

Broadly speaking, the highest characteristics (SE more than 45 dB at a light transparency of about 85 %) were obtained experimentally on ordered mesh structures. Theoretical calculations show that, by optimizing the parameters of the conductive grid, it is possible to achieve 60 dB or more on such materials with a transparency of about 90 %, but the imperfection of the resulting structures can significantly reduce the calculated parameters.
It is worth noting that the practical results obtained on multilayer continuous structures turned out to be generally lower compared to metal meshes. Such coatings, as a rule, lead to a decrease in transparency to 60% or less when reaching SE up to 40 dB. In a number of studies, higher values SE were obtained, but in narrow frequency bands. At the same time, our theoretical calculations, which are beyond the scope of this article, show that the optimization of the thicknesses and chemical composition of heterostructure layers can significantly increase the functional characteristics of such coatings, bringing them closer to metal meshes. However, this issue requires further serious study, since in addition to structural parameters (layers thickness, chemical composition, electrical conductivity), technological aspects of manufacturing developed multilayer structures will affect the obtaining characteristics.

6. Conclusion
The results analysis allows to conclude that both approaches (fabrication of multilayer structures and mesh conductive structures) make it possible to obtain materials with high values of shielding efficiency in the radio range and transparency in the visible wavelength range. Currently, among practical results, the best characteristics have been achieved on ordered mesh structures obtained by lithographic methods. However, the need to use microelectronics technologies in their creation is a significant limiting factor of this approach. Moreover, it can be expected that the development of self-organization methods and additive technologies will lead to a significant simplification of the technological process, lower cost and the possibility of scaling it to large-area samples.

At the same time, the task of technology scaling in the case of multilayer structures can be solved much easier, since the technologies of applying thin-film coatings on roll materials are widely used in...
industry already now. There is a great potential for improving multilayer structures by optimizing their design and technological solutions, despite experimentally they have lower shielding efficiency and light transparency compared to metal grids. In this regard, the technological aspects can come to the fore, when an approach for the implementation of the considered materials with given characteristics are chosen, taking into account the possibility of subsequent scaling of the technology and economic indicators when choosing an approach for the implementation of the materials with the required characteristics.

Acknowledgments
This work was supported by the Ministry of Science and High Education of the Russian Federation (state task No. 0705-2020-0032).

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