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EMI Shielding using Composite Materials with Plasma Layers

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1. Introduction

Electromagnetic compatibility (EMC), including the engineering of materials used for shielding,, is currently one of the most extensively developing field of applications of composite materials (Bula et al., 2006; Jaroszewski & Ziaja, 2010; Koprowska et al., 2004, 2008; Sarto et al. 2003, 2005; Wei et al., 2006; Ziaja et al., 2008, 2009, 2010). The development of lightweight, mechanically resistant, shielding materials is possible by using plasma technology. Due to rapid increase in the number of sources generating the electromagnetic (EM) fields, e.g. radio broadcasting, television, radio communication, cellular networks, continuously extending range of applied frequencies, and increasing power generated by PEM sources, the shielding design is getting more and more challenging. These challenges stem from the fact that complex EM power engineering systems are built of miniaturized electronic circuits. The progressing miniaturization reduces the resistance of the electronic circuits to electromagnetic exposure. Therefore, the choice of suitable materials for the shields and their appropriate arrangement has an essential meaning.

2. Criteria of selection of materials and fabrication technologies for shielding materials

Materials used in the technique of electromagnetic field shielding must meet following conditions:

- have a suitably high coefficient of the shielding effectiveness SE,
- be resistant to mechanical impact and easy to handle (rigidity, elasticity, gravity, the way of installation, sealing),
- be resistant to harmful influence of external environment (oxidation, corrosion),
- durable,
- homogenous,
- easy to form the shield,
- low costs of production.

Shields made as metal sheets or foil, and metal mesh are characterised by a good EM field shielding effectiveness coefficient. However they are characterised by low resistance to environmental impact. Their fundamental disadvantage is weight. They are primarily used in low frequency electromagnetic field shielding.

The alternative to metal shields is the use of composite shields, that have lately found a wide application in the EM field shielding technology (Wojkiewicz et al., 2005; Holloway et al., 2005; Ziaja et al., 2008a, 2008b), and they are interchangeable equivalents for metals. Main advantages these materials are: good mechanical and chemical properties, lower weight, higher rigidity and strength, resistance to corrosion, lower costs of treatment and easier processing. Based on their structure composite materials are divided into two groups:

- laminar systems,
- inclusions in substrate (admixture materials in a form of fibres, flakes or particles).

The first group is made in the special fabric form (fabrics with entwined conductive fibres e.g. Zn, C, Cu, and Ag): nonwoven fabrics with conductive layers deposited or laminates with pressed in conductive layers. Conductive layers may be put on by using the following methods: silk-screen printing (Wang et al., 2009); vacuum evaporation or magnetron sputtering (Ziaja, 2007; Ziaja et al., 2008). They are characterised by easy deposition on different kind of surfaces and the shielding efficiency coefficient above 50 dB. However not every material is suitable for conductive coating. It is determined by surface properties of the substrate material.

The second group covers conductive glues or dyes.

While designing composite shielding material, it is necessary to take into account, that efficiency of shielding depends on the following factors:

- volume fraction of inclusions,
- electrical and magnetic properties,
- shape and size of inclusions, and the way of their orientation,
- EM field frequency,
- number and sequence of layers.

Choice and optimisation of appropriate composite structure is made on the basis of assumed mathematical models (Sarto et al., 2004; Szhulz et al., 1988; Pospieszna, 2006). These models must assume effective application of different mechanisms of shielding:

- effectiveness of the reflection mechanism on interfacial surfaces, which among other things depends on their size. In case of composite materials this effect is obtained by implementing extenders with expanded specific surfaces,
- losses caused by multiple reflections are negligibly small, when a distance between successive reflection surfaces (interfaces) are big in comparison to the depth of penetration,
- because of the skin effect, for effective use of whole cross section area of the elementary unit of extender, its size should be comparable or less than a depth of penetration,
- effective absorption of radiation ensures material with a high permittivity and / or with a high magnetic permeability.

The distribution of inner field is mainly determined by the orientation of fibres and the polarisation of the incident field. In order to achieve a relatively high and constant value of the shielding effectiveness SE, it is necessary to use anisotropic materials with certain number of layers displaying different orientation of fibres.

The surface of composite materials, which reflects the EM wave must be electrically conductive, although its high conductivity is not required. It means, that in a case of composite material, containing conductive extender, a conductive connection between extender units is not required, even though such connection improves shielding. Good shielding effectiveness values are obtained in polymer composites containing carbon powder (Kim et al., 2004; Wu et al., 2003; Huang et al., 2007) or silver powder (Hong et al., 2001).

Coating of materials by thin layers (metallic, ceramic and polymer) meets wide use in many branches of technique, both in laboratory scale and in industry. The winning of materials with unique properties, a possibility of miniaturization, improvement of the lifespan and reliability, and a high energetic efficiency of electronic and optoelectronic devices are main driving force of thin-layer technique development to produce EM composite shileding. The most often used methods of layers winning are: the chemical deposition from the gas phase (CVD) and the physical deposition from the gas phase (PVD).

PVD technologies are based on deposition of a conductive layer on the substrate from the gas phase using physical phenomena, such as: metal evaporation (vacuum sputtering), sputtering in vacuum, gases and metallic vapour ionisation. For most of them the common feature is a process of deposition of a layer from stream of gas containing ionised molecules. Any differences between PVD methods are due to the way of winning of ionised metal vapours.

Nowadays, the most often used method of the obtaining different kinds of layers is magnetron sputtering (glow discharge, plasmatic). This method is based on bombarding of surface of the sputtering electrode (the target) with high energetic ions of working gases. Ions knock out atoms or molecules of material from the target, which next are deposited on substrates. This way metallic layers, dielectric semiconductor layers and high-melting layers can be deposited. Sputtering devices using an electrical and a magnetic field are called magnetron or plasmatic guns. In electrical and magnetic fields, ions from working gases obtain very high kinetic energy, and to the layers can be deposited at a very high speed,, which for metals is a few μm/min and for dielectric layers is in the order of 10-100 nm/min. Such high speeds of putting on layers are not available in other methods. The magnetron structure differentiates from other atomiser structures by a dominating presence of secondary electron ionisation (surface ionisation effect). Thanks to that, current density flowing through the target is at least one order of magnitude greater than in other structures.

Additionally during a process of sputtering, plasma cleans and activates surface of substrates. Therefore with magnetron methods one can do metallization of materials such as: PTFE (polytetrafluoroethylene) or PP (polypropylene), which can not be transformed by metallization with other methods, because of their surface properties.

Universality of magnetron sputtering devices gives opportunity of using them in both production lines on an industrial scale and also in small scientific laboratories. The placement of the target with regard to substrates being coated is almost optional and the deposition area is virtually unlimited. Magnetrons of rectangle shape with targets up to 4000 mm long are used to cover wide glass sheets with layers with different optical properties. It even ensures coating of a layer with the retention of stoichiometry of winning compounds and the thickness repeatability of single multilayer structures. The measure of usefulness of magnetron systems, is among other things the ability to realise precise requirements for optical coatings (e.g. $Ti0_2$, $Si0_2$, Si_3N_4, $Sn0_2$, ZnO) for needs of automotive and spacecraft industry.

In the project for winning electrical field shielding layers, on textile carriers, magnetron gun WMK-100 was used. The deposition experiment was prepared by the Laboratory of Vacuum and Plasma Technology of Wrocław University of Technology. This magnetron gun is characterised by a power densities of (~ 300 Wcm^{-2}) striking targets, which are not obtainable in standard devices. Additional advantages of the magnetron gun are:

- the easiness of target exchange,
- the possibility of target sputtering of thickness from 1 to 6 mm,
- the possibility of spraying of dielectric target.

Magnetron guns are powered by direct (DC) and high frequency alternating (AC) current sources. DC currents are primarily used for metallic targets. Increasing the supply current frequency allows to sputter dielectric materials. Sources with radio frequency (RF) are also used; however, they didn't meet broader application in industry.

In recent years impulse current sources have been developed for magnetron gun power supply. In standard systems, the power emitted on a target is controlled by the magnetron supplying current value, but in impulse systems the power is controlled by the length of impulse time. On Fig. 1 types of power supply modulation of magnetron sputtering devices are shown. During the impulse duration t1 sputtered material is accumulated in the plasma area. During the intervals between impulses (t2-t1), the deposition of atoms on the substrates surface takes place. Applying uni-polar pulses to metallic targets (Fig.1 b) and bipolar ones to dielectric ones (Fig.1 a) is a common practice. The advantage of this type of solution is a possibility of making stable reaction processes. This stability stems from elimination of non controlled arc discharges. Since in a single impulse, there is always the maximum sputtering speed, the impulse modulation lets to increase the ratio of sputtered material to impurities.

Describing the power supplied we use two electrical quantities: the real power P and the circulate power Pc. The first defines directly the effectiveness of the layers coating, the second is a certain property of the power supply system. It reflects changes of plasma parameters (change of impedance) connected with a change of concentration and type of charged particles. It is especially seen during sputtering in oxygen.

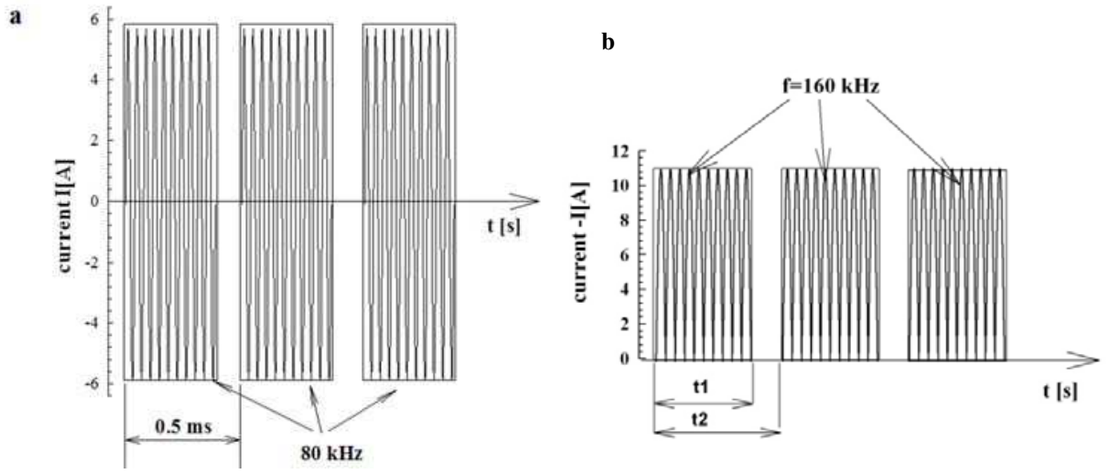


Fig. 1. Types of impulse modulation: a - alternating current (bipolar), b - variable unipolar current

Recent current source solutions let not only to change the length of impulse time t1, but also the current bursts repeating frequency 1/t2.

This solution is useful during executing reactive processes, because the ratio of both times t1/t2 is a characteristic for coming into existence of each chemical compound. By changing this ratio, one can change chemical composition of emerging compounds. In reactive

processes (winning of oxides or nitrides) the dwelling time of atomised material in the plasma area plays the key role in a winning of layers with the pre-set chemical composition. If times t1 are too short, monoxide layers obtained, may not have the required stoichiometric composition. Even when sputtering metallic titanium in pure oxygen, one can obtain metallic layers. Therefore the proportion t1/t2 is a very important technological parameter.
Processes of sputtering titanium (99.9 %) and zinc (99.9%) targets were realised by use of magnetron launcher WMK-100 with the magnetic system of diameter f= 75 mm. The argon pressure (99.999 %) used as a working gas was changed from $7*10^{-3}$ to $3*10^{-2}$ Tr. It was established, that pressure of $1*10^{-2}$ Tr was optimal, because the etching area of the target surface is then the widest. It ensures 85 % utilisation of the target area. This technological parameter is very important from the industry point of view, because it ensures the most advantageous use of target material. In this case, it may be increased using the magnetic system of a wider diameter. For this magnetron the optimal diameter of magnetic system is 100 mm. The distance between the target and nonwoven fabrics has been set to 100 mm.
The magnetron gun was energized by impulse current source type DPS (DORA POWER SYSTEM) which works in AC-M and DC-M modes controlling power up to P= 12 kW. This source can control group frequency in the range of fg=50 Hz – 5 kHz.



Fig. 2. The zinc target installed on the WMK-100 magnetron

3. Barrier composites on textile substrates

When designing of shielding materials in a composite structure polymer-conductive layer, it is necessary to take into account not only electrical parameters (resistivity ρ, permittivity ε), but also the thickness of layers and the morphology of their surface. The layer thickness may be controlled by both: the power emitted on atomised electrode and by the time of atomisation.
The appropriate choice of these parameters guarantees obtaining of uniform layers with the expanded specific surface and numerous conductive bridges. The volume resistivity ρ_v plays the significant role in the shielding mechanism, however it is not possible to measure this quantity in these type of composites.. Good solution seems to be a measurement of the surface resistivity ρ_s and its influence on a value of the shielding coefficient SE.

The ρ_s value of metallic layers is determined by the surface morphology of used nonwoven fabric and by the number of conductive bridges.
Titanium layers were deposited on polypropylene nonwoven fabric of the base weight of 60 g/m² and 150 g/m² , also on nonwoven fabric with consistence of viscose 30%-polyester 70 % and the base weight of 70g/m² (VISC30+PES70). To establish the structure of obtained metallic layers, the surface of cross section areas of samples were examined at magnification 300x. On Fig. 3 nonwoven fabric surfaces before and after the metallization process are shown.
Nonwoven fabric VIS30%+PES70% is characterised by the non-uniform expanded surface with clear seen fibres. Also for propylene, the influence of the basis weight on the morphology of nonwoven fabric clearly appears. PP samples of the base weight of 60 g/m² still have apparent single fibres. PP of the base weight of 180 g/cm² has the most uniform surface.
The power P emitted by the gun was varied from 0,5 do 3 kW. Increasing of power above 3 kW causes heating of nonwoven fabric, which may even lead to melting . Heating of nonwoven fabric results in desorption of gases from its volume or chemical dissolution causing arising of reactive gases in the magnetron chamber. These gases react chemically with atomised titanium and form its compounds, which are characterised by other electrical properties than metallic titanium. Time of depositing was varied from 0,5 to 20 min.
The investigation of the surface morphology was conducted with the use of scanning microscope Quanta 200 in the low vacuum mode (without covering of preparation surface with a gold layer), The sample surface was in its natural state. The acceleration voltage 15,0 kV of an electron bunch and the SSD detector were used.

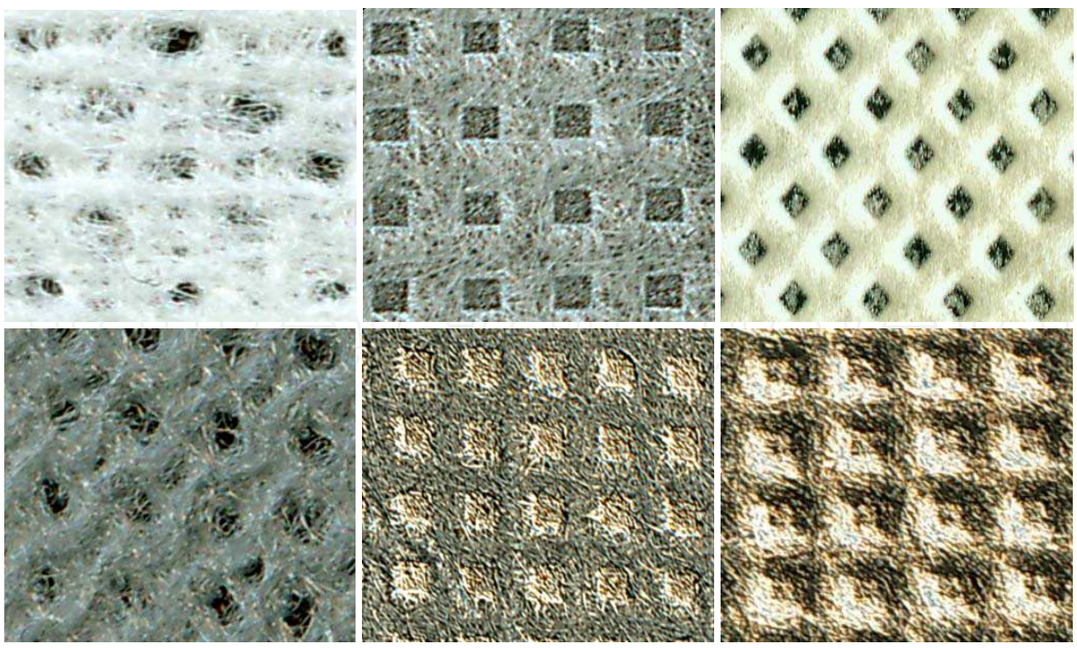


Fig. 3. Textiles' surfaces: a- before deposition of the Ti layer, b- after deposition of the Ti layer.

The microscope examination (Fig. 4) has shown, that deposited layers are not uniform, what is probably caused by penetration of metal into areas between fibres.
In covering metallic layers, there are few cracks and splits, and their adhesion to substrate is very good. For PP nonwoven fabric, titanium layers also form themselves into areas between interleaves. Increasing the base weight of the fabrics causes forming of more solid layers and uniform metallic layers. The covering process of layers Ti begins with forming of nuclei of crystallization centres (for depositing time less than 10s) on passing holes of nonwoven fabric (Fig. 3). X-ray radiography examination shown, that Ti layer is formed by crystallites of dimensions from 8 to 13 nm. Increase in the depositing time causes the spreading of layers so that a continuous structure can be obtained.
Interesting part is also building over areas between passing holes of nonwoven fabric and deposition of Ti layer on fibres, taking place inside nonwoven fabric (Fig. 4). Such extension is characterised by a big specific surface, which increases the reflection and the dispersion of the electromagnetic wave effect.

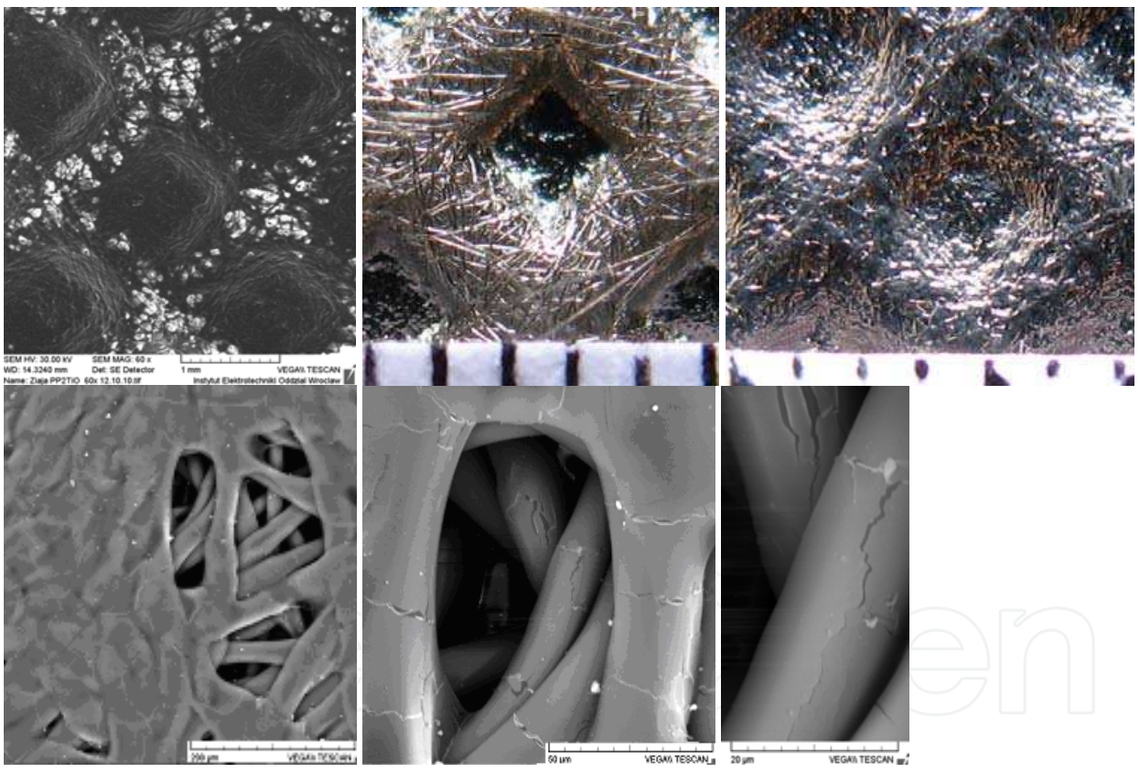
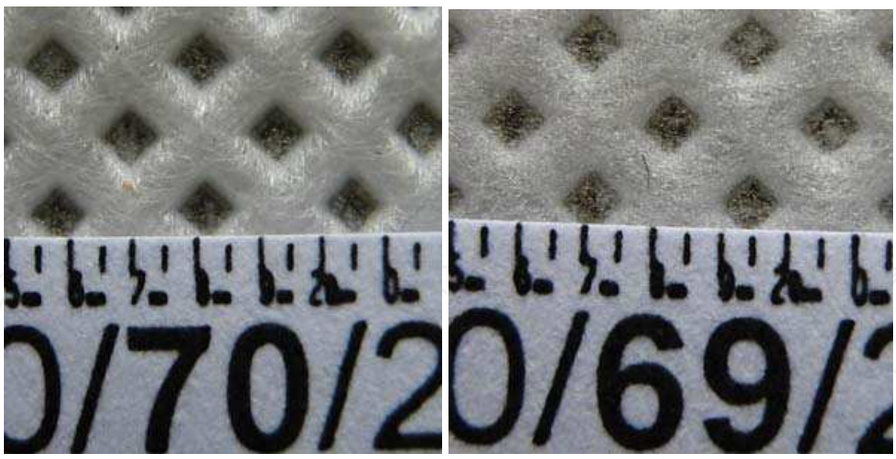


Fig. 4. Microscopic image of the Ti surface at different magnifications.

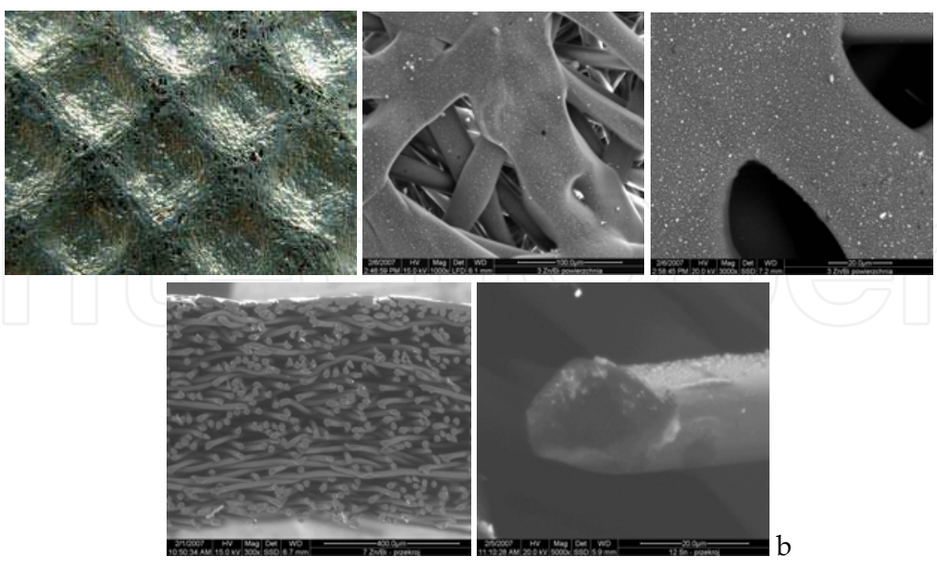
Values of surface resistivity ρ_s of titanium layers depend on morphology of the nonwoven fabric surface. The precise microscopic analysis has shown obvious differences in the surface morphology of either side of the nonwoven fabric PP150 (Fig. 5). The one side of the surface is almost smooth, consistent and without any expanded porous structure; whereas the second surface is scabrous and porous, with visible micro-fibres forming a spongy microstructure. Such a surface makes it impossible to form a nucleus of crystallization centres, which form conductive metallic bridges.



a b

Fig. 5. The surface morphology of each side of polypropylene textile of the base weight of g=180gcm^{-2} , a- smooth surface, b- scabrous surface.

The process of winning of zinc layers and zinc-bismuth layers on PP onwoven fabric was made by sputtering metallic targets with the weight consistence of 0.9Zn-0.1Bi and 0.96Zn-0.04Bi, and metallic zinc with the purity of 99.9 %. Metallic layers were deposited on polypropylene nonwoven fabric of the different base weight, alumina ceramics, CORNIG glass covered with conductive and transparent layer of ITO (the solid solution SnO_2 and In_2O_3).
To identify the structure of the obtained metallic layer the surface of the samples cross section was examined at magnifications: 300x, 1000x (Fig. 6). In covered layers Zn there are few cracks and splits, and their adhesion to substrate is very good and similar to titanium layers. In magnified figures below (Fig.6), one can observe well formed, conducting zinc bridges.



a b

Fig. 6. The microscopic pictures of (a) the surface and (b) the cross-section of Zn layer.

During the deposition of Zn and Zn-Bi target, in contrast to titanium ones, one is not allowed to use high values of power. Exceeding the value of power over 2 kW (for magnetic

system use), causes uncontrolled arcing discharges on defects and impurities of the target surface, which leads to overheating. Further increasing of power heats the target in such a way, that process of melting follows it, and then it causes evaporation of the target material. This effect leads to damage of the target and eventually to water leak from cooling system into vacuum chamber.

Zinc and zinc-bismuth layers, just like titanium, are half-amorphous. The X-ray analysis (Fig. 7) has shown, that the average dimension of crystallites is of the order of 10 nm. Lines characteristic for Zn are characterised by a small intensity and a big half-width.

In Fig. 8 the dependence of group frequency of a magnetron gun power supply and types of nonwoven fabric, also their base weight on a surface resistivity ρ_s of titanium layers is presented. It was determined, that the value of the surface resistivity depends on the substrate base weight. PP150 nonwoven fabric is characterised by the lowest value ρ_s of the order of 10^3 Ω. It is a big value and according to the expanded surface of nonwoven fabric, also numerous of resistance bridges, in spite of that the magnification picture of cross section suggests continuity of the layer. For nonwoven fabric PP60 the surface resistivity is one level higher. The highest value ρ_s was obtained for VISC30+PES70, which is above 10^7Ω. This is the result of lack of the metallic continuity of layers.

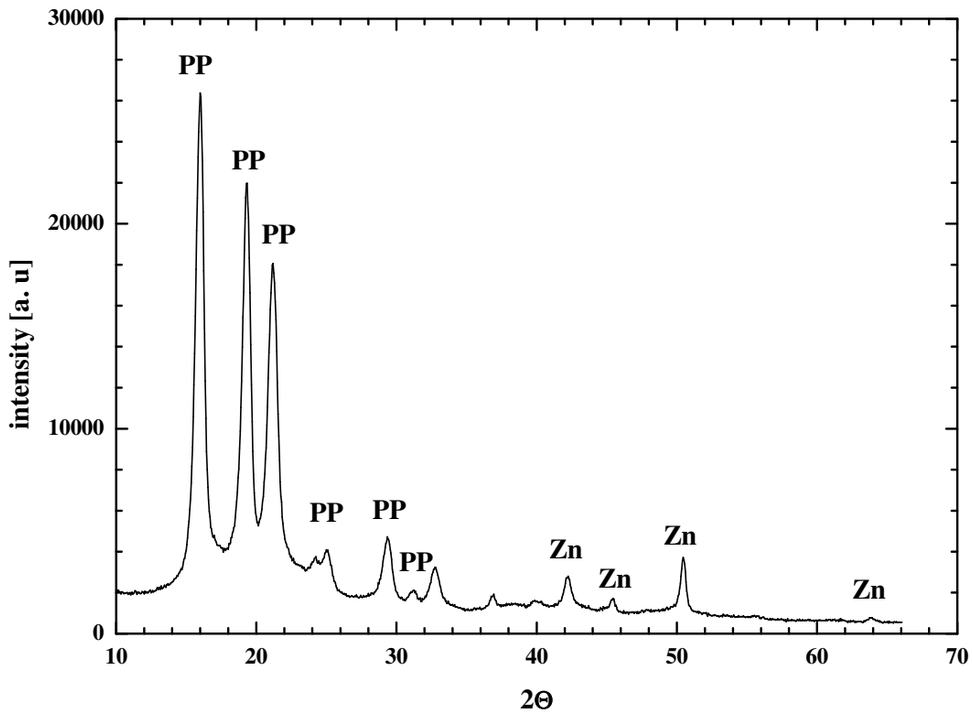


Fig. 7. X-ray picture of PP textile with covering Zn layer for radiation wavelength Co.

It was determined, that with increase in group frequency, value of ρ_s was increased slightly. It is the result of the longer time of sputtering t1 and the longer dwelling time of the substrate in the area of plasma.

Probably a chemical dissolution of substrates occurs as a result of the effect of the higher temperature. Substances are released, which react with titanium layer changing its electrical properties.

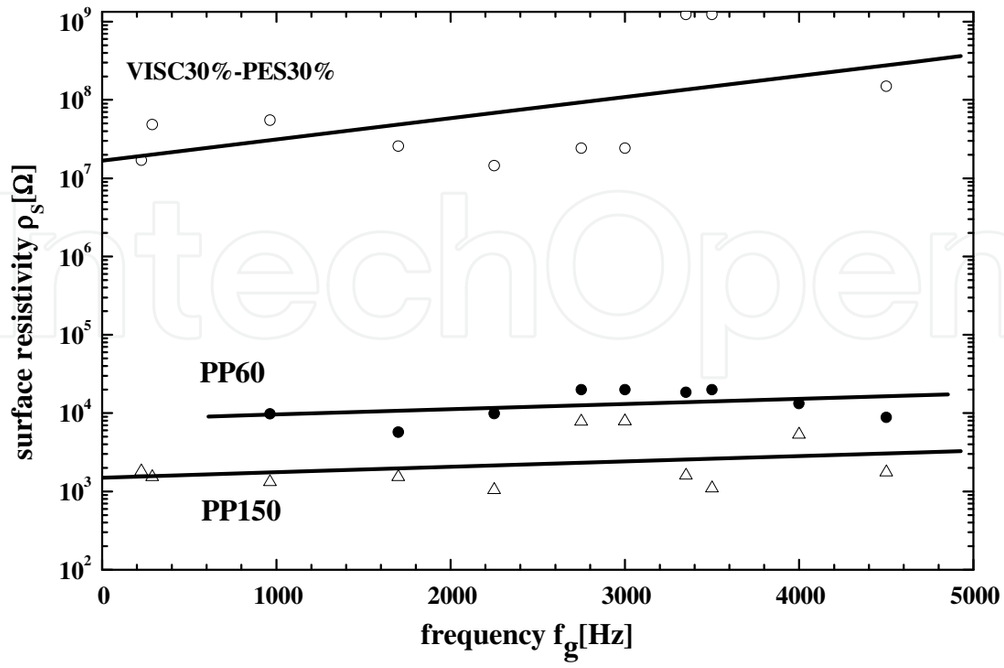


Fig. 8. Influence of the group frequency fg on the surface resistivity ρ_s of titanium layers, P_{Ar} =2*10^{-2}, power emitted on the target P=1 kW, the depositing time t=10 min.

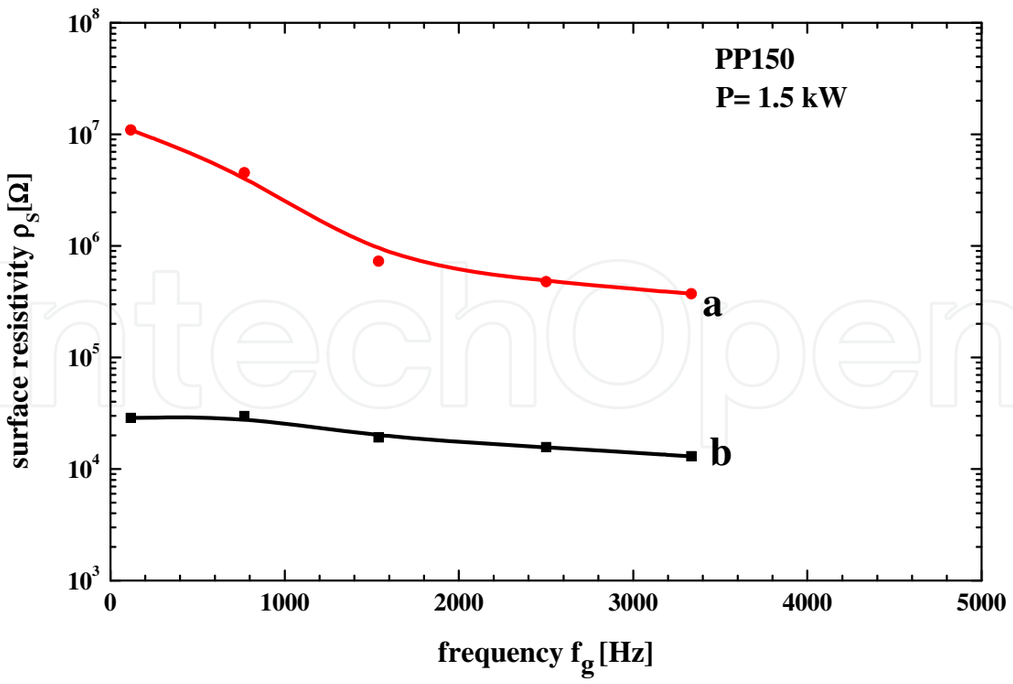


Fig. 9. Influence of the group frequency fg on a surface resitivity ρ_s of titanium layers, P_{Ar}=2*10^{-2}, the depositing time t=3 min. in case of unipolar DC-M supply

The surface microstructure has the decisive impact on the value of the surface resistivity ρ_s . The porous side of PP nonwoven fabric with an extended structure is characterised by a very high surface resistivity of the order of 10^7 Ω (Fig. 9) and it is two levels higher than on the smooth side ($\rho_s = 3*10^4$ Ω). With increasing of group frequency of the power supply, ρ_s gets lower into value $3*10^5$ Ω, but even then it is one level higher than on a smooth side.
The value of ρ_s depends also on the time of depositing metallic layers. Increasing the time, the covering layer not only increases its thickness, but it gets more solid and uniform. After obtaining the specified thickness, value ρ_s does not change.

4. Shielding properties of PP/Ti and PP/Zn composites

Measurements of shielding attenuation were realised on the test setup prepared in the Institute of Telecommunications, Teleinformatics and Acoustics of Wrocław University of Technology in accordance with the method of ASTM D4935-99. The test diagram is presented on Fig. 10. The test settup consists of network analyser - model HP 8711A of Hewlett-Packard firm and measuring adapter, which is a section of an air concentric line of characteristic impedance 50 Ohm. Measuring uncertainty is approximately 2 dB. Measurements of attenuation were made using standard sample. Shielding attenuation is calculated as a difference between transmittances or between attenuation of a standard and an examined sample.

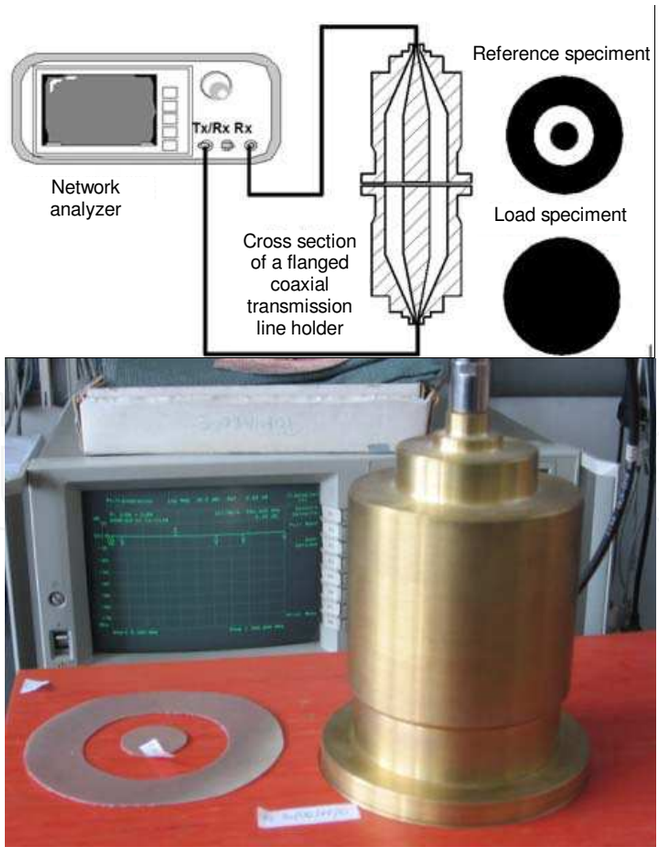


Fig. 10. The diagram of the test setup of shielding attenuation

Examinations performed have shown an influence of types of used substrates on a value of shielding attenuation of metallic layers. On Fig. 11, increasing of shielding attenuation Ti layers of 10 dB for substrates of smooth surface (CORNIG glass) in comparison to the substrate of extended specific surface (nonwoven fabric PP) is observed. This observation points to a possibility of obtaining higher values of SE for nonwoven fabrics.
The highest values of shielding attenuation coefficient (SE>50 dB) are obtained for composites PP/Zn (Fig. 12). The value of SE may increase by doping zinc layers with bismuth. Increasing bismuth concentration from 4 to 10 % of atomic, causes increase in SE by approximately 20 dB (Fig. 13 and 14). Further increase in concentration of Bi does not cause any increment of SE. Observed changes of SE value as a function of power released on the target, are caused by increase in Zn layer thickness.

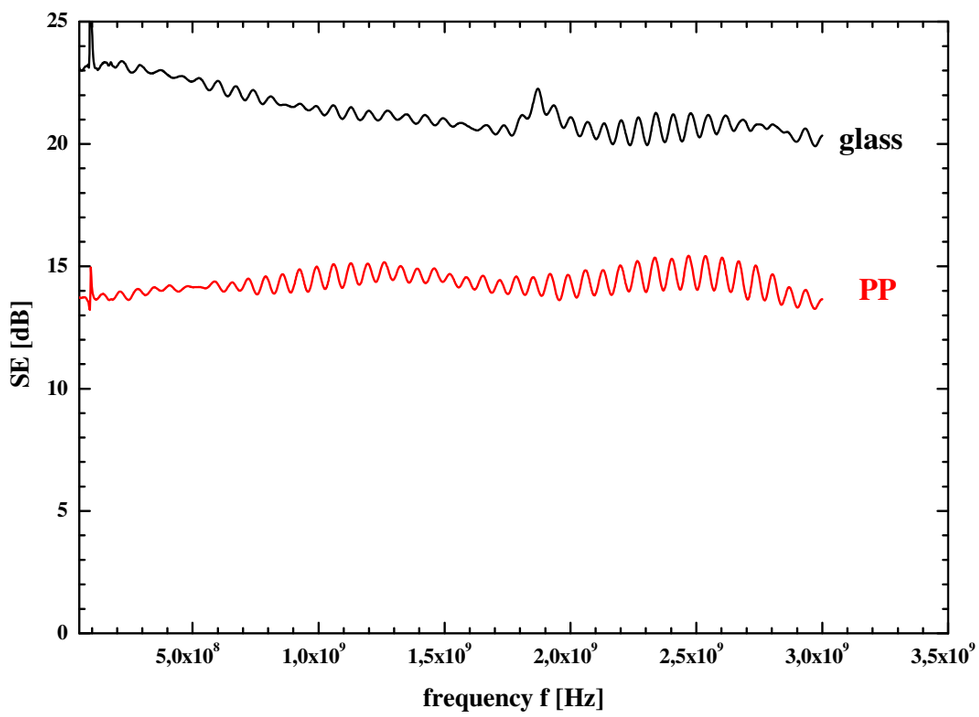


Fig. 11. The coefficient of shielding efficiency SE versus frequency f for PP/Ti and glass/ Ti composites.

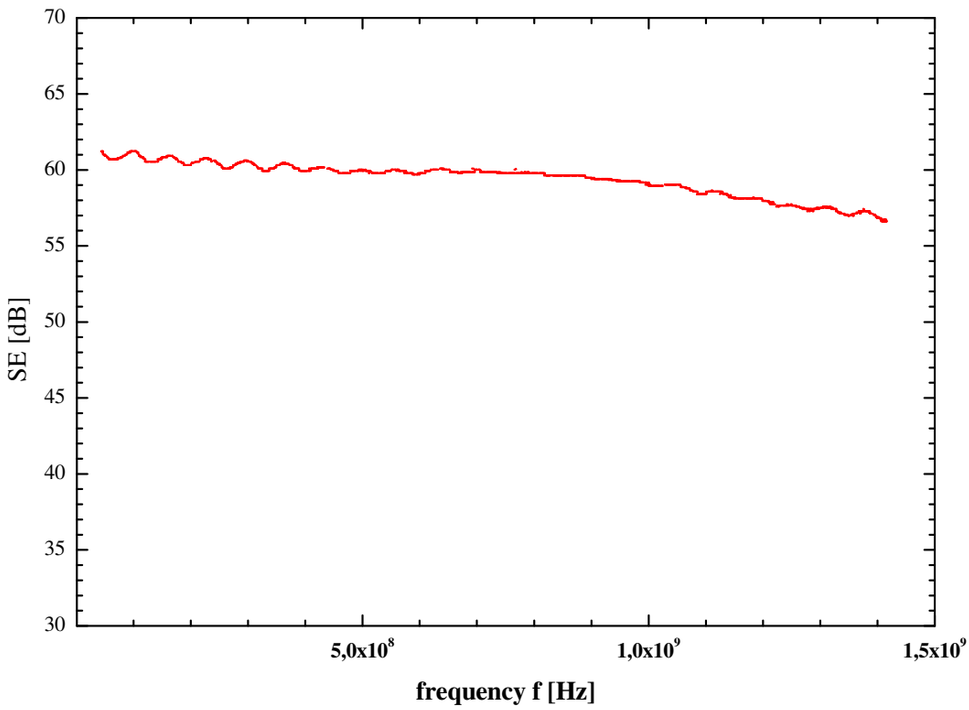


Fig. 12. The coefficient of shielding efficiency SE versus frequency f for PP/Zn composites.

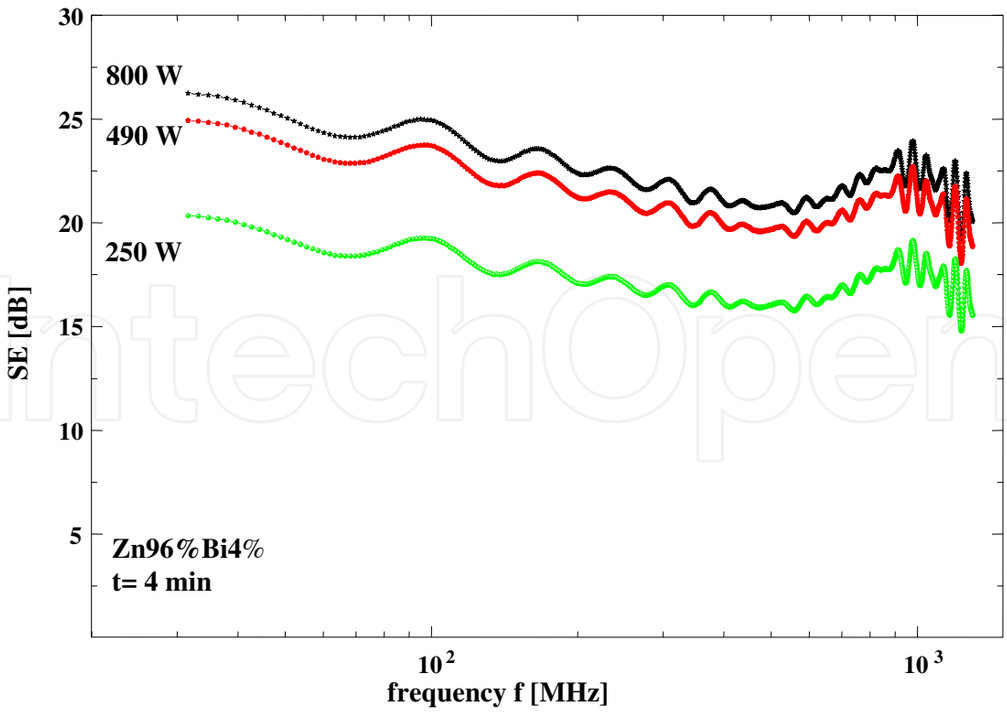


Fig. 13. The coefficient of shielding efficiency SE versus frequency f, for different powers released on the Zn96%Bi4% target in case of unipolar DC-M supply.

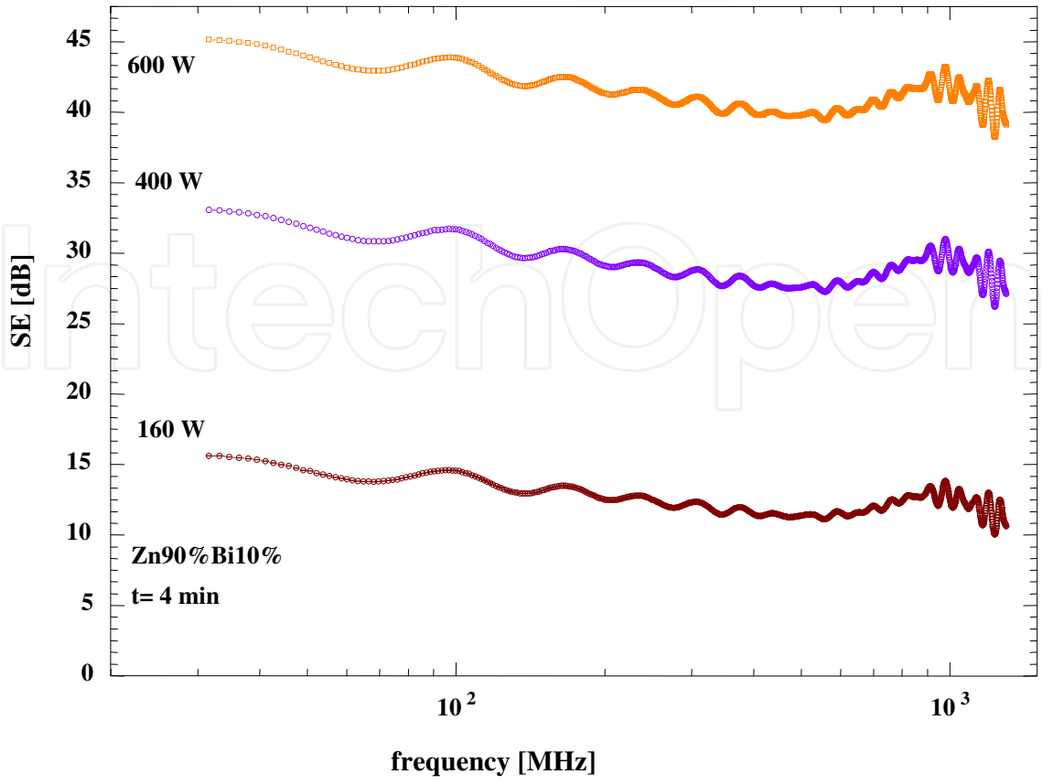


Fig. 14. The coefficient of shielding efficiency SE versus frequency f, for different powers released on the Zn96%Bi4%target in case of bipolar DC-M supply.

Despite of the initial assumption, that the surface resistivity may be a good tool for testing composites PP/Me (Me- metal) for evaluation of their shielding properties, it has been found, that deciding role is played by the thickness of layers. In case of composites PP/Zn and PP/Ti, for the same ρ_s, different values of shielding coefficient were obtained. On Fig. 15 , over twofold increase in SE value, for the same values of surface resitivity, was observed. In this case, Zn layer is characterised by higher thickness and uniformity than Ti layer. Higher uniformity and continuity of Zn layers causes increase in the reflecting effect of electromagnetic wave from this surface.

5. Assessment of barrier shielding materials with plasma layers deposited on the fabrics and parameters of technological process with the impedance spectroscopy method

Shielding effectiveness of composite material polymer-metal is not only determined by its surface resistivity. There are other physical phenomena occurring both in each composite layer and on interphases between them that play very important roles.
Composite materials working in alternating electrical fields are characterised by a presence, beside conductivity, also polarisation phenomena, that is they get a certain electrical moment m. The total dielectric polarisation is a superposition of three polarisation mechanisms:

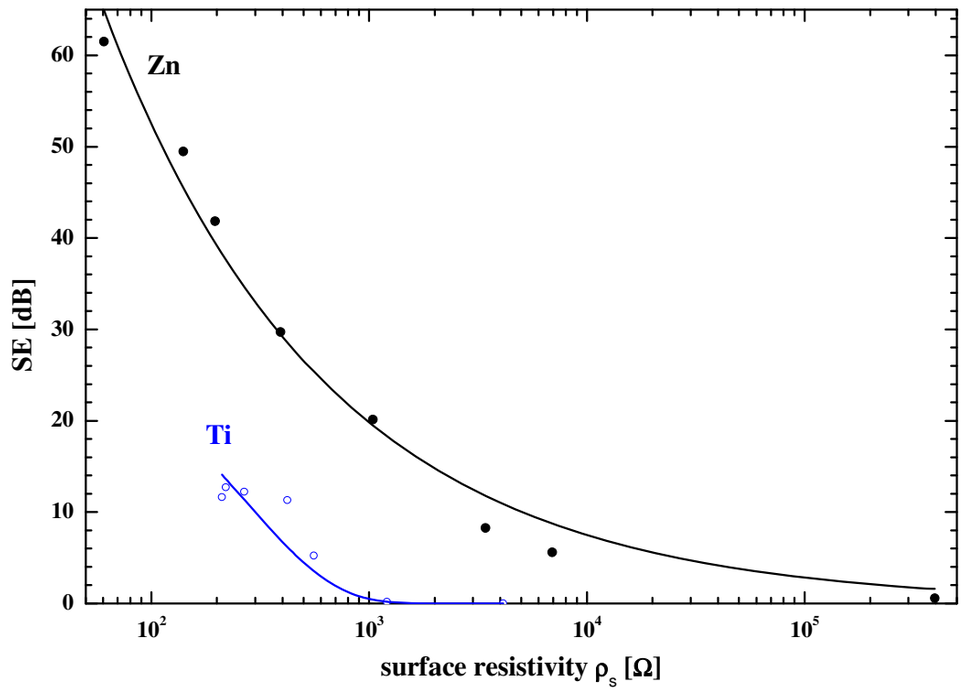


Fig. 15 . The coefficient of shielding efficiency SE versus frequency f, for different powers released on the Zn96%Bi4%target in case of unipolar DC-M supply.

- distortion polarisation, which occurs both in dipole dielectrics and in non dipole dielectrics, and being a sum of:
  - electron polarisation resulting from a deformation of electron shell, and
  - atomic polarisation resulting from a displacement of atoms from their original positions,
- orientation (dipole) polarisation, which occurs only in dielectrics with permanent dipole moments resulting from an alignment of dipole axes along direction of outer electric field lines,
- ion polarisation, which occurs in dielectrics with ion bonds, resulting from displacement of ions in relation to each other from the place of balance.

Generally the polarisation phenomenon is that an arising on a dielectric surface, inserted into electric field, induced electric charges, which are bonded charges ρ_p. We can treat them as charges of dipoles chain ends, directed by the outer electric field. If we take into consideration the surface charge density for a capacitor not filled with dielectric ρ_0, then the total charge density for a capacitor with dielectric is a sum of the bounded charge ρ_p and the free charge ρ_0. Inserting of dielectric into electric field (flat capacitor configuration) causes increasing of the capacitance from value C_0 to value C. The ratio of the capacitor capacitance increasing to its capacitance C_0 is defined as the electric susceptibility of the dielectric χ, but the proportion of the capacitor capacitance with a dielectric to its capacitance without a dielectric, as the electrical permeability ε. Thus we can define the density of the total capacitor charge as:

$$\rho = \rho_0 + \rho_p = \rho_0 + \chi\rho_0 = \rho_0(\chi + 1) \tag{1}$$

A measure of this charge is the dielectric displacement vector (the vector of an electrical flux density, the induction vector) D:

$$D \cdot n = \rho = \rho_0(\chi + 1) = \varepsilon_0 \cdot E \cdot n + P \cdot n \tag{2}$$

where:
n - is the normal unit vector directed in the dielectric direction,
ε_0 - is permittivity of vacuum.
Polarisation P, directly bonded with ρp surface density of polarisation charge, can be therefore presented in the form:

$$P = D - \varepsilon_0 E = (\chi + 1)\varepsilon_0 E - \varepsilon_0 E = \varepsilon\varepsilon_0 E - \varepsilon_0 E = (\varepsilon - 1)\varepsilon_0 E = \chi\varepsilon_0 E \tag{3}$$

A basement of the impedance spectroscopy method (IS) is a measurement of a linear electrical answer of an examined material to an excitation with a relatively small voltage signal of the sinusoidal form $u(t) = Um \sin(\omega t + \psi_u)$, in a wide frequency range f ($\omega = 2\pi f$). An answer to a sinusoidal voltage signal is a sinusoidal form of current $i(t) = Im \sin(\omega t + \psi_i)$ with the same angle speed ω. This current is a sum of conductive current and polarisation (displacement) current (Walter S. Zaengl, 2003):

$$i(t) = \rho_0 E(t) + \partial D(t)/\partial t = \rho_0 E(t) + \varepsilon_0 \partial E(t)/\partial t + \partial P(t)/\partial t \tag{4}$$

Using the Fourier transform to both sides of equation (6) we get an answer of dielectric in the frequency domain (A.K. Jonscher, 1983; A. Bouaïcha et.al, 2009):

$$\underline{I}(\omega) = \rho_0 \underline{E}(\omega) + j\omega \underline{D}(\omega) = \{\rho_0 + \varepsilon_0 \omega \chi''(\omega) + j\omega\varepsilon_0 [1 + \chi'(\omega)]\}\underline{E}(\omega) \tag{5}$$

where: χ' i χ'' are respectively the real and the imaginary component of the complex electric susceptibility $\underline{\chi}$.
The nature of examination of material electrical answer is a measure of the effective current's value I and its φ phase displacement in relation to forcing voltage (of the effective value U), whereby $\varphi = \psi_u - \psi_i$.
Based on these measures the T(ω) spectral transmittance is calculated, which characterises dependence between the forcing and the answer phase displaced for the same ω pulsation:

$$\underline{T}(\omega) = \left|\underline{T}(\omega)\right| \cdot e^{j\psi(\omega)} \tag{6}$$

where: $\left|\underline{T}(\omega)\right|$ is a modulus and ψ (ω) is an argument of the spectral transmittance $\underline{T}(\omega)$.
In the impedance spectroscopy, the spectral transmittance usually has a form of the Z(ω) complex impedance or Y(ω)admittance, which are defined as:

$$\left|\underline{T}(\omega)\right| = \underline{Z}(\omega) = \frac{\underline{U}(\omega)}{\underline{I}(\omega)} = \left|\underline{Z}(\omega)\right| e^{j\phi(\omega)} = \mathrm{Re}\,\underline{Z} + j\,\mathrm{Im}\,\underline{Z} = Z'(\omega) + jZ''(\omega) \tag{7}$$

$$\left|\underline{T}(\omega)\right| = \underline{Y}(\omega) = \underline{Z}^{-1}(\omega) = \frac{1}{Z} e^{-j\varphi(\omega)} = Ye^{-j\varphi} = \mathrm{Re}\,\underline{Y} + j\,\mathrm{Im}\,\underline{Y} = Y'(\omega) + jY''(\omega) \tag{8}$$

where

$$|\underline{Z}| = Z = \sqrt{\left(Z^{'}\right)^2 + \left(Z^{''}\right)^2}\ ,\ |\underline{Y}| = Y = \sqrt{\left(Y^{'}\right)^2 + \left(Y^{''}\right)^2}\ ,\ \phi(\omega) = arctg\frac{Z^{''}(\omega)}{Z^{'}(\omega)}$$

In the IS method we are not limited only to frequency analysis of impedance or admittance of examined material (their amplitude and phase characteristics or dependence of real and imaginary parts of the complex transmittance upon frequency), but we can also use other quantities, such as the complex capacitance $\underline{C}$, or the complex permittivity $\underline{\varepsilon}$. These quantities are bonded with the complex admittance by frequency and geometrical parameters of sample.

The complex admittance, which can be associated to a simple equivalent scheme in a parallel connection configuration of two ideal elements - resistance R and capacitance C, is explained by a formula:

$$\underline{Y} = \frac{1}{R} + j\omega C = G + j\omega C \tag{9}$$

Transformation of expression (9) allows to introduce a term of the $\underline{C}$ complex capacitance:

$$\underline{C}(\omega) = \frac{\underline{Y}(\omega)}{j\omega} = C' - jC'' \tag{10}$$

If we assume, that examined material of d thickness is placed between two flat parallel electrodes of area S, that is the geometrical capacitance of capacitor electrodes configuration $Co = \varepsilon_o S/d$ is given, then a knowledge of this parameter allows to define the relative permittivity in a complex form:

$$\varepsilon(\omega) = C(\omega) / C_o = C(\omega)\, d / \varepsilon_o S = \varepsilon' + j\varepsilon'' \tag{11}$$

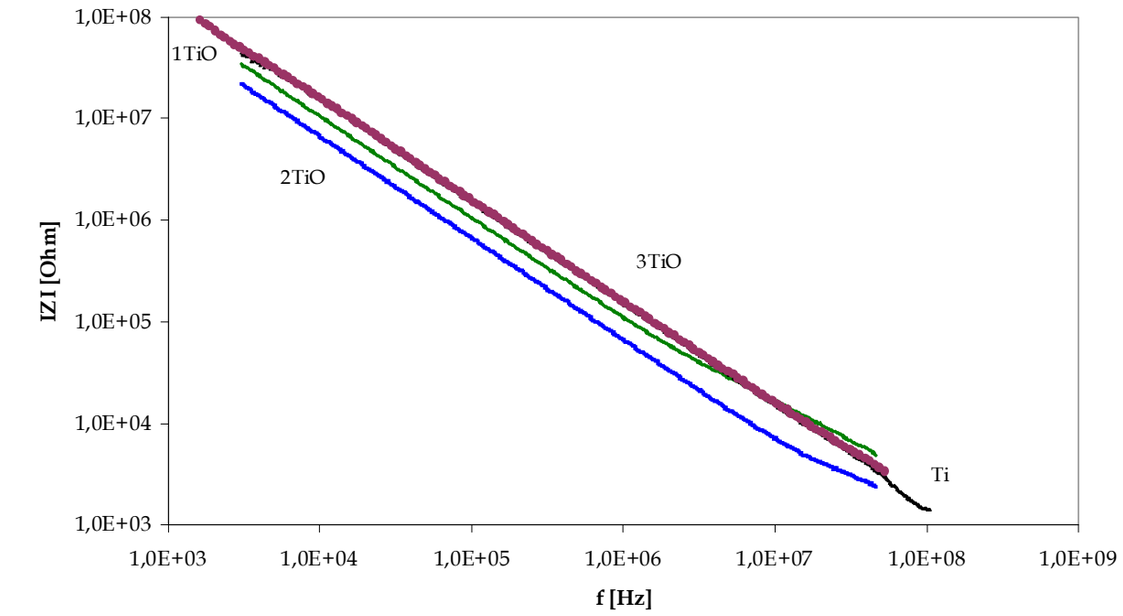
The capacitance and the relative permittivity in a complex form are the most often used form of the spectral transmittance in the IS method. Their frequency analysis shows multiple phenomena, which take place simultaneously in an examined material.

Below the examinations of an answer of dielectric composite materials made of nonwoven fabrics covered by thin plasmatic layers of titanium and titanium monoxide as a representative for composites described in this chapter are shown. For these examinations the measurement system from the High Voltage Group of Wrocław University of Technology was used. Its main elements are the precise impedance analyser Agilent 4294A and the measuring cell Agilent 16451B consisting of the three-electrode measuring capacitor. Table 1 includes the description of samples made of alternately covered titanium and titanium monoxide layers on nonwoven fabric.

Bode diagrams of examined materials are shown on figure 16. The frequency spectrum of the modulus of impedance does not indicate significant differences between examined samples. It varies for all examined samples in the same way, and additionally for samples 3TiO and Ti overlaps in almost entire spectrum of frequencies.

	Ti	TiO	Ti	TiO	Ti	TiO
3TiO	•	•	•	•	•	
2TiO		•	•	•		
1TiO		•	•	•	•	•
Ti	•					

Table 1. Description of samples made of titanium and titanium monoxide layers alternately deposited on a textile



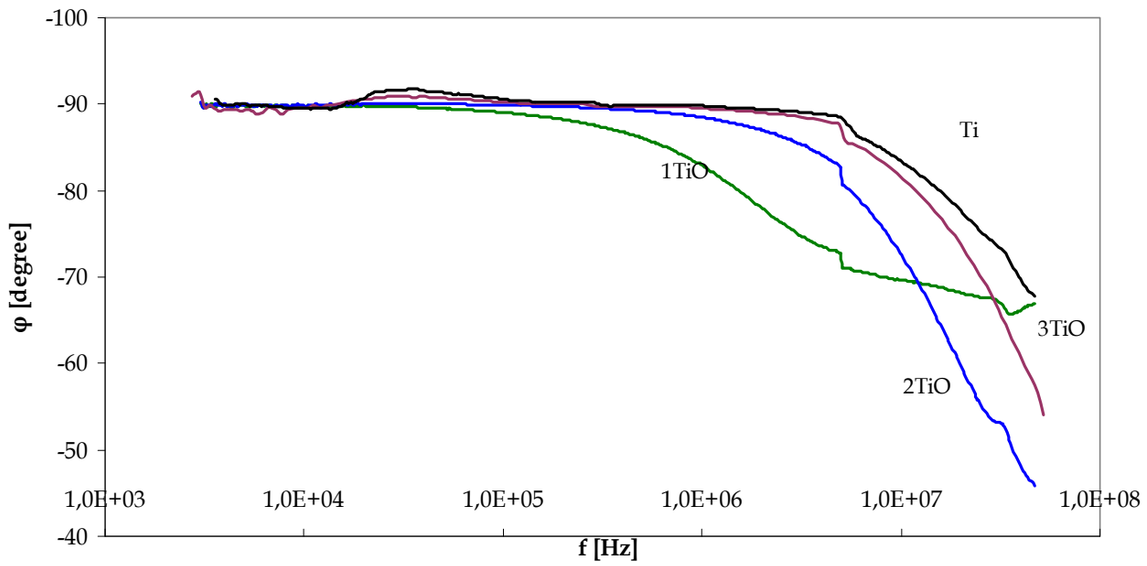


Fig. 16. The Bode diagram of examined composite structures.

The Bode diagram analysis suggests a possibility to use the equivalent scheme consisted of a parallel connection RC and also a presence of the Maxwell-Wagner polarisation to describing the polarisation phenomena. It is possible to find analogies to examinations outcomes of some laminar structures (Nitsch, 1999).

More pieces of information about dielectric properties may be given by a form analysis of the complex capacitance presented as a function of frequency and on the complex plane (the Cole-Cole diagram). Figures 17 and 18 present frequency spectrums of examined complex capacitance. An incline of real component curves of the complex capacitance for 1TiO and 2TiO samples can be seen there, what suggests a presence of at least one relaxation mechanism in the examined frequency range.

Diagram forms for Ti and 3TiO samples suggest a possibility of a presence of relaxation processes in frequencies above the measuring range. It is confirmed on diagrams of the imaginary component of the complex capacitance presented on figure 18. We can observe the presence of a relaxation pick for 1TiO sample for frequency under 10^7 Hz also for 2TiO sample for frequency approximately $5x10^7$ Hz.

The Cole-Cole diagram of the complex capacitance of examined composite structures is presented on figure 19. The diagram has a form of deformed semicircles with clearly displaced centres. Each of the examined samples distinguishes then with a specific relaxation process of different time-constants. Furthermore we can observe a presence of the second semicircle for each of examined composites, what clearly suggests the presence of another relaxation process at higher frequencies.

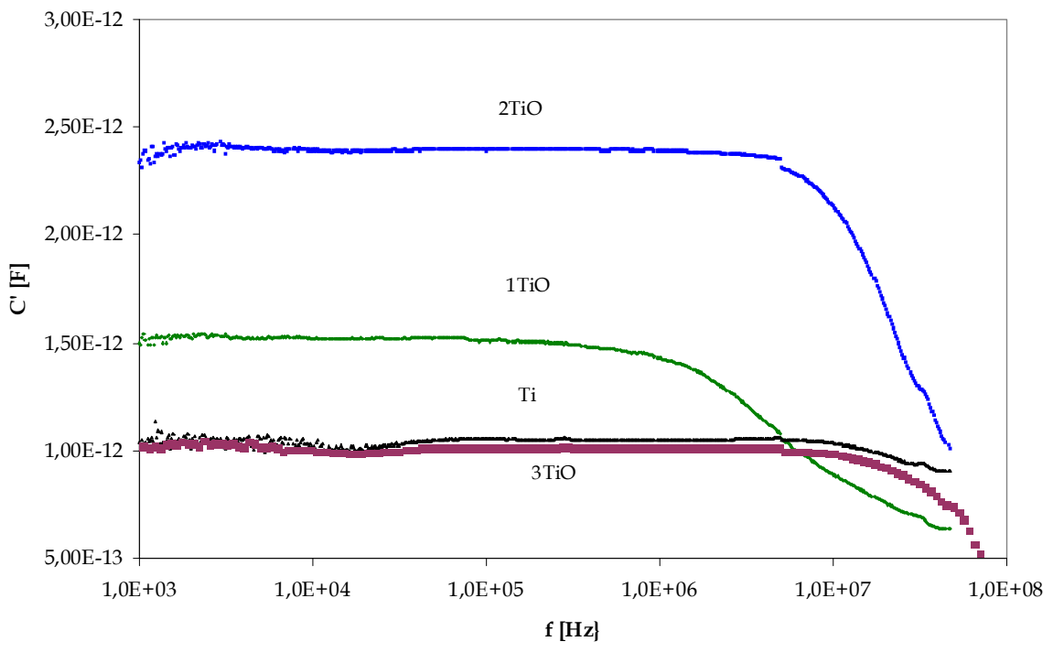


Fig. 17. Frequency spectrums of real component of complex capacitance.

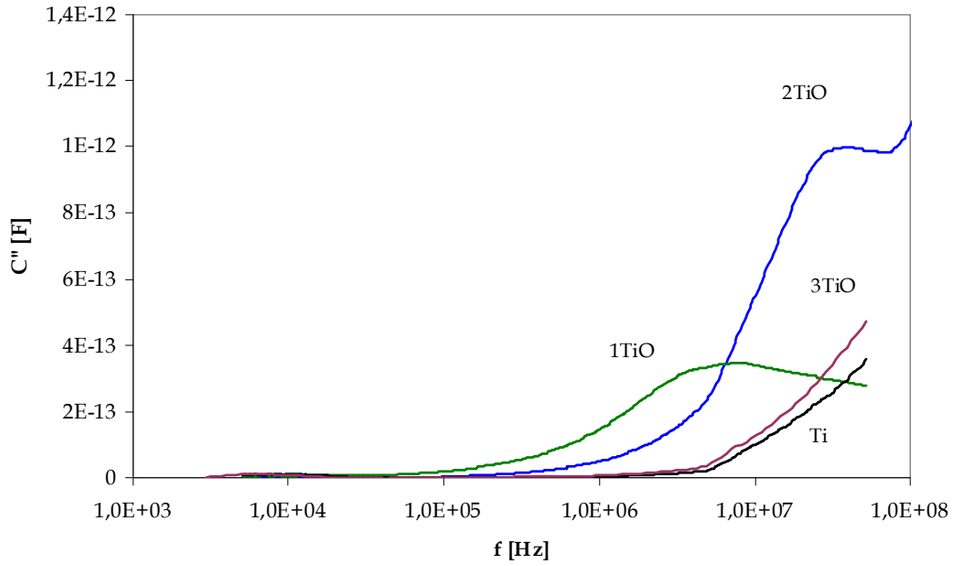


Fig. 18. Frequency spectrums of imaginary component of complex capacitance.

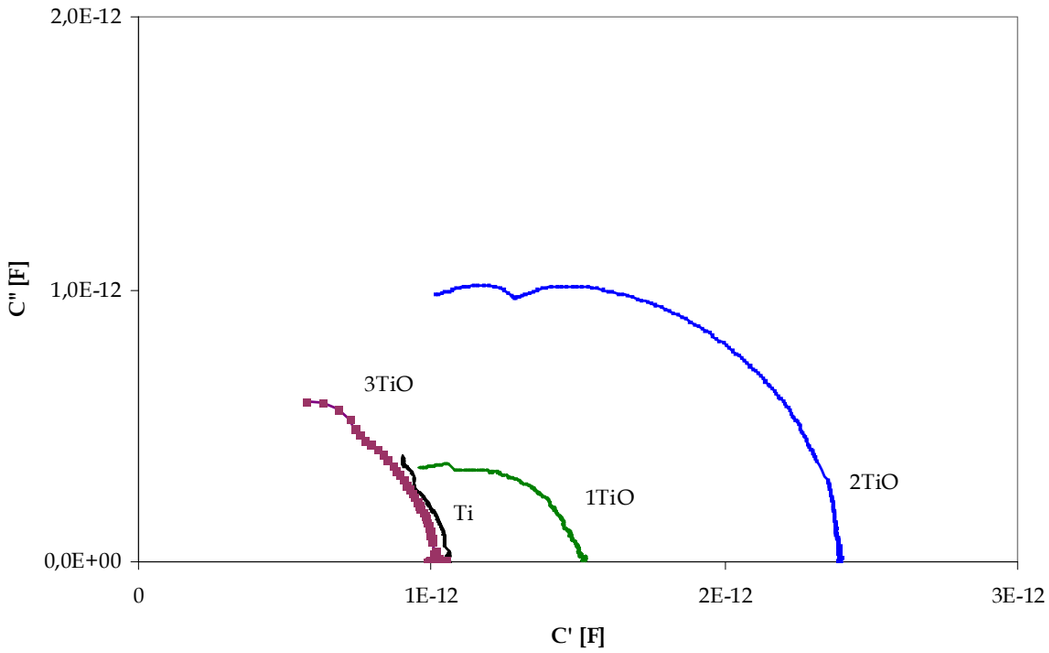


Fig. 19. The Cole-Cole diagram of the complex capacitance

In the outcomes presented above, one can notice some general rules. We can observe a clear separation between dielectric properties of composites, in which the outer layer is formed by

titanium (Ti), and of composites with the outer layer formed by a titanium monoxide (TiO). For composites with outer titanium layer (Ti and 3TiO samples) in the measuring frequency range, we observe very little differences in frequency spectrums of measured parameters and in the Cole-Cole diagrams independently from a number of layers Ti-TiO.
In the diagram form of the imaginary component of the complex capacitance as a function of frequency, we do not observe a relaxation pick. At the same time, the diagram form of this characteristic suggests the presence of a relaxation pick at frequencies higher than the measuring range. Quite a different situation for composites with the outer layer formed by a titanium monoxide (1TiO and 2TiO samples) appears. In this case we can observe the strong dependence of dielectric composite properties upon a number of formed Ti-TiO layers.
Frequency diagram forms of the imaginary component of the complex capacitance shows, in the examined measuring frequency range, the presence of a relaxation pick and a possibility of a presence of the second relaxation phenomenon at higher frequencies. The value and frequency of a relaxation pick presence are strictly depended on a number of Ti-TiO layers forming a composite. Increasing a number of layers results in reducing of a relaxation pick value and in displacement in the lower frequencies direction. It is confirmed by Cole-Cole diagrams of the complex capacitance, in which there is a clear presence of a displacement of the semicircle centre to the right for 1TiO sample.
The capability of composite materials to shield electromagnetic fields is coherently associated with their dielectric properties in a wide frequency band. The method of impedance spectroscopy allows one to connect the measured frequency characteristics with the physical structure of tested material and the alternations in the structure.
The method has been used by the authors to determine the connection between surface structure of a fabric being a substrate and dielectric properties of obtained composite fabric –carbon (Jaroszewski et al., 2010) and to evaluate the correlation between dielectric response of the system and surface resistance of the carbon layer (Pospieszna et al., 2010, Pospieszna & Jaroszewski, 2010). The possibilities to design desired electric properties of composite materials are also used to improve the shielding properties of the materials. Thus, the connection of the impedance spectroscopy method with those properties.

6. Summary

It should be noted that the performed studies and collected experience in the field of modern technologies of shielding have already solved a lot of actual problems but there is still a challenge for further work to improve the efficiency of shielding and to develop new designs of electromagnetic shields. They can also be used in the shielding of power engineering systems, where a compatibility with environment in a wide sense of this meaning is the main problem (i.e. not only in the aspect of emission and electromagnetic disturbances). In the light of the latest experiences it seems that the future in the area of EM field shielding is connected with the application of modern technologies to fabricate thin-film composite coatings, including nano-composites. The materials are capable to fulfil all conditions of effective shielding from EM fields and to eliminate all undesired occurrences associated with operation of the shielded systems. The results of our investigations, presented above, point out the possibility of industrial fabrication of the composite shielding materials with the coefficient of shielding efficiency exceeding 50 dB. Good mechanical properties and high resistance to environmental effects are additional advantages of such materials.

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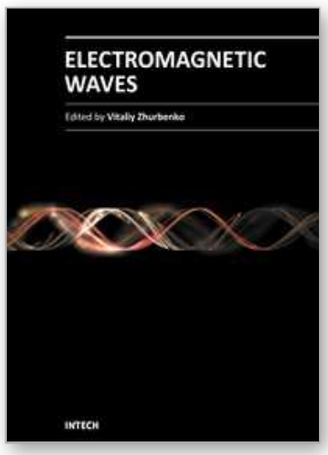
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