Millimeter Wave Resistance of Metal-Dielectric $Co_x(SiO_2)_{1-x}$ and $Co_x(Al_2O_3)_{1-x}$ Films

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***Abstract*—Transmission and reflection of millimeter waveband electromagnetic waves have been studied for thin-film metal-dielectric $Co_x(SiO_2)_{1-x}$ and $Co_x(Al_2O_3)_{1-x}$ nanocomposite materials, where cobalt nanoparticles are placed inside SiO_2 or Al_2O_3 films of 100 nm thickness. The microwave properties of the nanocomposite samples with different cobalt content have been measured in the frequency ranges from 26 to 38 GHz and from 53 to 77 GHz. Frequency dependencies of transmission and reflection coefficients have been measured. Power loss in the samples has been determined. An algorithm for recovering the conductivity from the frequency dependencies of the transmission and reflection coefficients has been worked out. It has been found that the microwave conductivity increases with increasing cobalt content and differs drastically from the dc conductivity. The obtained results have been compared to the actual measurements of magnetic properties.**

***Index Terms*—Microwave measurement, microwave and dc conductivity, thin-film nanocomposite, waveguide.**

I. Introduction

INVESTIGATION of the electrodynamic properties of thin-film nanocomposite materials is one of the modern topics. The film materials composed of metal nanoparticles embedded into a dielectric matrix have unusual high-frequency electrodynamic properties. Particularly, a $Co-SiO_2$ nanocomposite has an appropriate permeability in the GHz frequency range, which makes it a good antenna candidate. In this context, the paper [1] deals with tunneling magnetoresistance in ultrathin $Co-SiO_2$ films. Magnetic correlations in non-percolated $Co_x(SiO_2)_{1-x}$ granular films were studied in [2], [3]. The phenomenon of injection magnetoresistance was observed in heterostructures of GaAs/granular SiO_2 films with Co nanoparticles. Exchange interaction between electrons in the different layers of the ferromagnetic/semiconductor heterostructure causes a potential barrier for spin-polarized electrons. Once electrons are injected into the semiconductor layer, giant injection magnetoresistance is observed. Tunneling magnetoresistance in ultrathin $Co-SiO_2$ films is especially important at low temperatures [4]–[6]. The films with $x = 0.32$ exhibit evidence of magnetic interactions but no extended magnetic correlation. The magnetic correlation length estimated corresponds to the particle size.

To date, the microwave properties of granular nanocomposite systems are not studied enough. Spin-wave modes in granular $(SiO_2)Co_xGaAs$ films were observed using the Brillouin light scattering method [7]. The measured value of the effective exchange constant turned out to be three times less than its value for bulk cobalt. Microwave spin-dependent tunneling was studied in several ferromagnetic metal-dielectric nanocomposites such as $Co_{51.5}Al_{19.5}O_{29}$, $Co_{50.2}Ti_{9.1}O_{40.7}$, $Co_{52.3}Si_{12.2}O_{35.5}$, $(Co_{0.4}Fe_{0.6})_{48}(MgF)_{52}$, which possess tunneling magnetoresistance [8]. It was shown that there is correlation between the DC magnetoresistance and a change of the microwave transmission coefficient in a magnetic field.

A few papers report on the possibility of measuring conductivity and other characteristics of metallic and metal-semiconductor films [9], [10]. If a thickness of the film is a predefined value, the film conductivity can be calculated.

The problem of interaction between an electromagnetic wave and small magnetic metallic particles attracts substantial interest, because the granular metal-dielectric films discussed are promising materials for tunable microwave media. The investigation of the transmission (reflection) frequency spectra allows one to find the degree of frequency dispersion of the material constants and to determine the best possible frequency range for the film nanocomposites to be applied in practice.

The present paper investigates the frequency dependences of electromagnetic waves and transmission (reflection) coefficients in the frequency ranges from 26 to 38 GHz and from 53 to 77 GHz. The above coefficients make it possible to estimate a portion of electromagnetic losses. Microwave measurements on the series of metal-dielectric $Co_x(SiO_2)_{1-x}$ and $Co_x(Al_2O_3)_{1-x}$ thin films with different Co content were carried out. The method of measurements implements the transmission of microwaves through the sample consisting of the film and a dielectric substrate. The sample is placed across a rectangular waveguide that operates in a normal TE_{10} mode. The variations of the microwave signal either passed through the film nanocomposite or reflected from it are caused mostly

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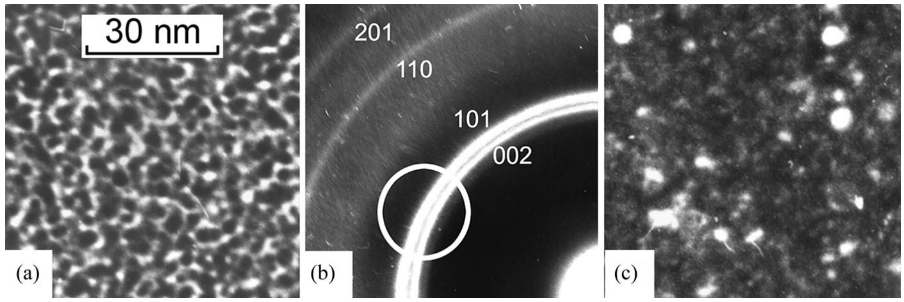

Fig. 1. TEM-image of the microstructure of a $Co_{40}(SiO_2)_{60}$ sample after annealing at 500 $^\circ$C, obtained in the "light field" (a) and diffraction image (b) regimes. The circle in the figure (b) indicates the position of the diaphragm in the implementation of the "dark field" regime (c). The digits on the diffraction lines (b) are the Miller indices of the corresponding reflecting atomic planes.

by a variation of surface impedance of the nanocomposite and by dissipation of the electromagnetic wave. The paper is organized as follows. The first part covers preparation and characterization data of the sample. Here, the magnetic properties of the samples are briefly discussed. Further, the second part describes the methods of taking the microwave measurements and analyzes the frequency dependences of the transmission and reflection coefficients in zero magnetic field. The microwave conductivity findings are presented below. The third part discusses the magnetic field dependences of the transmission coefficients.

II. Structure and Magnetic Properties

$Co - SiO_2$ and $Co - Al_2O_3$ composite films of 100 nm thick were deposited by RF-sputtering technique onto "Corning" glass substrates, which had a thickness of 0.2 mm and a size of 24 $\times$ 24 mm^2 in the plane. The target was a cobalt disc with a diameter of 100 mm, on the surface of which 3.8 mm diameter SiO_2 or Al_2O_3 plates were placed. Nominal Co-content in the compositions was varied by changing the area ratio of cobalt and the dielectric. Pressure before the deposition was $2 \cdot 10^{-6}$ Torr and during the process it was kept at 1 mTorr, the chamber being filled with Ar. The deposition rate of the composite films was about 0.05 nm/s.

An electron microscopic study was carried out using a JEM-200CX transmission microscope. The samples were precipitated at NaCl chips covered by SiO_2. The typical transmission image of the microstructure and the electron-diffraction pattern are shown in Fig. 1 for $Co_{40}(SiO_2)_{60}$ sample. Annealing of the films up to $T_a \sim 500$ $^\circ$C does not vary the amorphous structure of SiO_2 as a whole. However, as can be seen from the diffraction images, there is a tendency for lines to appear and be localized with increasing T_a. After annealing at 500 $^\circ$C, it is possible to identify several broadened diffraction lines (see Fig. 1(b)), which correspond to the following interplanar spacing: 0.200, 0.187, 0.124, and 0.105 nm. The dark field image of the microstructure is given in Fig. 1(c) for the limitation of the electron beam, as is shown with a circle in Fig. 1(b). The light spots in the image of Fig. 1(c) belong to the crystallites whose locations promote high reflection. Apparently, essential dispersion in the particle size takes place, but in any case, their diameters do not exceed 8 nm. Small-scale irregularity in the dark-field TEM images (see Fig. 1), as well as the lack of clear diffraction lines in the electron diffraction pattern confirm that the typical size of the Co-particles is less than 8 nm.

Geometry of the surface was studied with a NanoScan scanning atomic force microscope from NanoScan AG. For the sample $Co_{0.5}(SiO_2)_{0.5}$ the peak-to-peak difference is about 35 nm in the area of 1 $\times$ 1 microns whereas it equals to 60 nm in the area of 4 $\times$ 4 microns.

Magnetic measurements were carried out using a vibrating magnetometer and SQUID-magnetometer. The magnetization curves of the $Co_x(SiO_2)_{1-x}$ samples are shown in Fig. 2(a). All the curves tend to saturation in magnetic fields above 5 kOe and exhibit a strong variation of saturation magnetization with increasing Co-content. Fig. 2(b) presents the saturation magnetization dependence on cobalt content for two systems. The saturation magnetization increases as cobalt content increases. The magnetization is very low for the $Co_x(SiO_2)_{1-x}$ system when $x < 0.45$. The data obtained with the vibrating magnetometer show that a distinct hysteresis loop is only for $x \geq 0.55$ for the $Co_x(SiO_2)_{1-x}$ system. The magnetoresistance is maximal for the $Co_{0.47}(Al_2O_3)_{0.53}$ sample. Fig. 2(c) displays the magnetoresistance dependence on the magnetic field for this sample.

III. Microwave Measurement Methods and Analysis of the Frequency Dependences of Transmission and Reflection Coefficients

The problem of interaction between an electromagnetic wave and small magnetic metallic particles attracts substantial interest. To theoretically describe the wave propagation is extremely formidable task. This is explained by a simultaneous action of normal metallic conductivity inside an individual particle and tunneling conductivity between the particles. Besides, the displacement current is necessary to be taken into account [8]. The research of the transmission (reflection) frequency spectra allows one to reveal frequency dispersion and to determine the best frequency ranges for the films to be applied in practice.

This section of the paper provides a deep insight into the frequency dependences of transmission and reflection coefficients at frequencies from 26 to 38 GHz and from 53 to 77 GHz. Microwave measurements were carried out using a conventional waveguide operating with the TE_{10} mode. The sample was placed inside the waveguide, as shown in Fig. 3. The single-mode regime was realized in the above frequency ranges. An

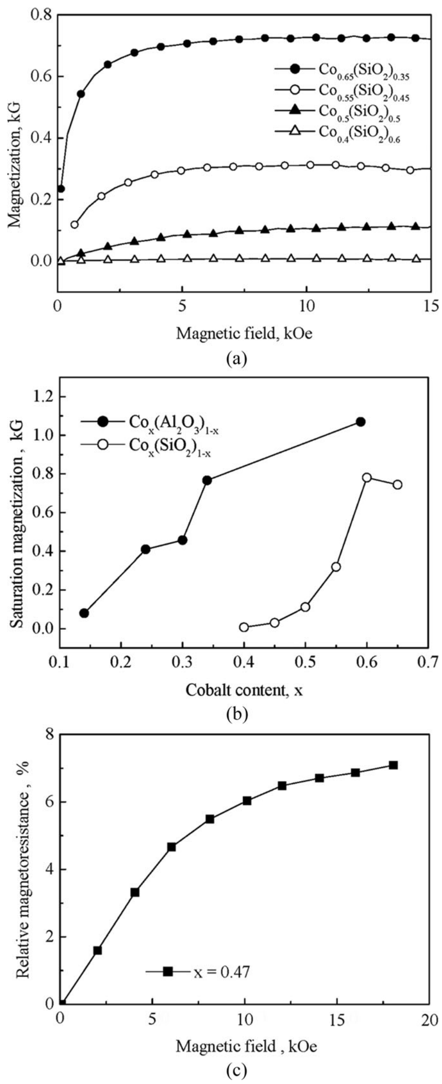

Fig. 2. Magnetization curves for $Co_x(SiO_2)_{1-x}$ samples ($0.4 \leq x \leq 0.65$) (a); saturation magnetization as a function of cobalt content for $Co_x(SiO_2)_{1-x}$ and $Co_x(Al_2O_3)_{1-x}$ systems (b); magnetoresistance for $Co_{0.47}(Al_2O_3)_{0.53}$ sample (c).

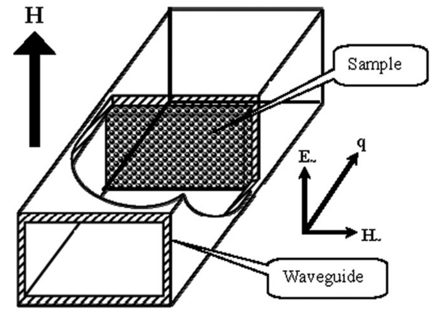

Fig. 3. Scheme of location of the samples in a waveguide.

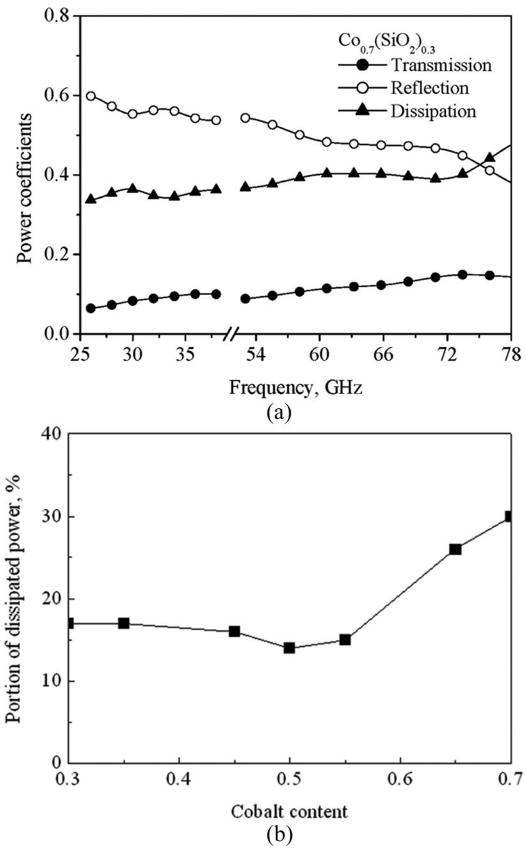

Fig. 4. Frequency dependence of transmission and reflection coefficients for a thin film sample $Co_{0.7}(SiO_2)_{0.3}$, as well as for the portion of absorbed power Δ (a);dependence of the absorbed power portion on cobalt content for the frequency f = 32 GHz (b).

external magnetic field $\mathbf{H}$ created by an electromagnet was applied perpendicularly to the wave vector of an electromagnetic wave $\mathbf{q}$. The plane of the sample was perpendicular to the plane where the microwave magnetic vector $\mathbf{H}_\sim$ lies. The microwave signal suffered changes when passing through or reflecting from the nanocomposite film. The changes were caused mostly both by the difference in surface impedances of the sample and waveguide and by dissipation of the wave. The power transmission and reflection coefficients were measured experimentally. The microwave measurements were performed at room temperature.

Microwave measurements were performed on a series of $Co_x(SiO_2)_{1-x}$ and $Co_x(Al_2O_3)_{1-x}$ thin-film nanocomposites with different content of Co nanoparticles. The frequency dependences of transmission and reflection coefficients for the sample with a $Co_{0.7}(SiO_2)_{0.3}$ composition and a 100 nm thick film deposited on a silicon substrate with a thickness of 0.2 mm are shown in Fig. 4. These coefficients are weakly frequency-dependent throughout the above frequency ranges. However, it can be noted that the transmission coefficient gradually increases, and the reflection coefficient, on the contrary, diminishes with increasing frequency. These variations are produced both by frequency dependence of the absorption coefficient and by dispersion of the TE_{10} mode. Similar results were obtained for the other $Co_x(Al_2O_3)_{1-x}$ nanocomposites studied. Shown in Fig. 4(a), a continuous line with triangle symbols represents the difference between 1 and the sum of the transmission and reflection coefficients, namely, $\Delta = 1 - D - R$. This line illustrates dissipation process, e.g. partial power absorption by the sample. As our experiments show, the higher cobalt content is, the greater the dissipated power is when $x > 0.55$ (see Fig. 4(b)). Obviously, such a tendency is a consequence of Joule losses as being a main source of losses in the objects under examination. The dissipated power portion varies from 15 to 30%, depending on Co content. Inspection of Fig. 4(b) indicates that, at higher cobalt content, the power losses grow due to sharply increasing the Joule losses induced by eddy currents. It should be noted, that there is a discontinuity between 26–38 GHz and 53–77 GHz data in Fig. 4(a). For example, the transmission coefficient at 54 GHz is smaller than the same at 38 GHz. This discontinuity is the consequence of the method of the measurements. The measurements in frequency ranges 26–38 GHz and 53–77 GHz have been carried out using the waveguides with different dimensions. Therefore, the wave impedances of the waveguides are different that leads to the discontinuities in Fig. 4(a), see [11] for details.

A theoretical analysis gives the possibility to calculate the transmission and reflection coefficients for the structure placed into the waveguide [12]. In the general case, the sample is a system consisting of two layers. One of them is a conducting ferromagnetic film and the second one is a dielectric substrate. Let us discuss a procedure of determining the effective conductivity from the frequency dependences of the transmission and reflection coefficients. We designate the experimentally measured transmission and reflection coefficients as $D^*(\omega, \sigma)$ and $R^*(\omega, \sigma)$, respectively. It should be emphasized that D and D^* , R and R^* are accordingly functions of frequency ω and microwave conductivity σ. As for the transmission coefficient, the difference between the theoretically calculated D and actually measured values can be represented as $\Delta_D = D(\omega, \sigma) - D^*(\omega, \sigma)$. Analogously, the same difference for the reflection coefficient appears as $\Delta_R = R(\omega, \sigma) - R^*(\omega, \sigma)$. Suppose the effective conductivity σ of the film to be an unknown quantity. To estimate σ, we use a least squares fit. In doing so, we obtain the minimal squared difference between the calculated and measured transmission coefficient values:

$$\Delta(\sigma^*) = \min_{\sigma=\sigma^*} \left[\sqrt{(\Delta_R)^2 + (\Delta_D)^2} \right]. \quad (1)$$

The value of the effective conductivity obtained as a result of the minimization procedure can be regarded as an estimation of the microwave conductivity. Generally speaking, it is necessary to take into account variation of magnetic permeability when extracting dielectric permittivity or conductivity. For ferromagnetic system a possibility to use the above described simplified method is confirmed here by low saturation magnetization of the samples (see Fig. 2(b)) and slow variations of microwave transmission in low magnetic fields (see Section IV below). These data point out that the dynamical magnetic permeability in zero field is close to 1. This method is successfully applied to estimate the dielectric permittivity of ceramic and nanocomposite titanates of transition metals [13] and to analyze the millimeter waveband dielectric properties of nanocomposite materials based on opal matrices with particles of spinels [14]. The experimentally measured and calculated frequency dependences of the power transmission coefficient for the sample $Co_{0.7}(SiO_2)_{0.3}$ are shown in Fig. 5.

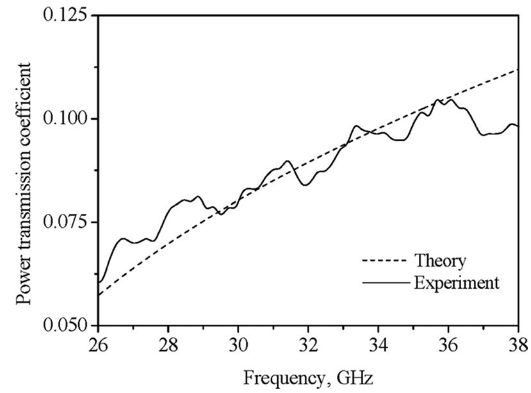

Fig. 5. Comparison between the experimental (a solid line) and calculated (a dashed line) frequency dependences of transmission coefficient for a thin film sample $Co_{0.7}(SiO_2)_{0.3}$. The calculated dependence is presented for $\sigma = 1.2 \cdot 10^5$ S/m.

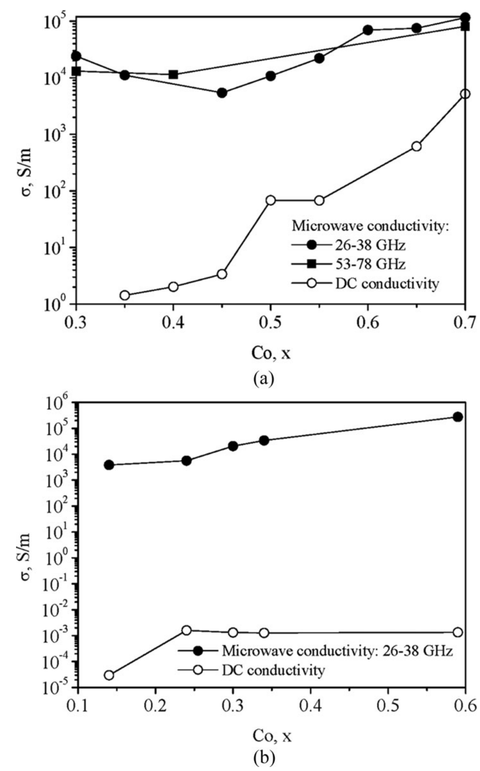

Fig. 6. Comparison between dc and microwave conductivity for thin films $Co_x(SiO_2)_{1-x}$ (a) and $Co_x(Al_2O_3)_{1-x}$ (b).

The comparison between the experimental and calculated frequency dependences of transmission coefficient shows a fairly good fit even for a chosen unique value of the effective microwave conductivity over the entire frequency range studied (see Fig. 5). Hence it follows that the effective microwave conductivity undergoes no substantial changes within the frequency range. As can be seen from Fig. 6, the microwave conductivity is several orders of magnitude greater than DC conductivity. This fact is not surprising for granular metal-dielectric systems. In our case, the change in the cobalt-content-dependent microwave conductivity is less pronounced than that in the dc conductivity for both $Co_x(SiO_2)_{1-x}$ and $Co_x(Al_2O_3)_{1-x}$ systems. It is

TABLE I
EFFECTIVE CONDUCTIVITY OF $Co_x(SiO_2)_{1-x}$ AND $Co_x(Al_2O_3)_{1-x}$ FILM NANOCOMPOSITES

Sample content, $Co_x(SiO_2)_{1-x}$	Specific conductivity of film nanocomposites, S/m	Sample content, $Co_x(Al_2O_3)_{1-x}$	Specific conductivity of film nanocomposites, S/m
$Co_{0.3}(SiO_2)_{0.7}$	$2.4 \cdot 10^4$	$Co_{0.14}(Al_2O_3)_{0.86}$	$3.9 \cdot 10^3$
$Co_{0.35}(SiO_2)_{0.65}$	$1.1 \cdot 10^4$	$Co_{0.24}(Al_2O_3)_{0.76}$	$5.6 \cdot 10^3$
$Co_{0.65}(SiO_2)_{0.35}$	$7.6 \cdot 10^4$	$Co_{0.3}(Al_2O_3)_{0.7}$	$2.1 \cdot 10^4$
$Co_{0.7}(SiO_2)_{0.3}$	$1.2 \cdot 10^5$	$Co_{0.34}(Al_2O_3)_{0.66}$	$3.4 \cdot 10^4$
		$Co_{0.59}(Al_2O_3)_{0.41}$	$2.8 \cdot 10^5$

evident that the displacement current plays an essential role in the process. Certainly, the strong variation of the DC conductivity is closely associated with percolation. Table I summarizes the values of the effective conductivity of $Co_x(SiO_2)_{1-x}$ and $Co_x(Al_2O_3)_{1-x}$ film nanocomposites, obtained from the transmission and reflection coefficients measured in zero magnetic field.

IV. MICROWAVE MEASUREMENTS IN MAGNETIC FIELD

Our microwave experiments involve the measurements of relative variations $d_m = \frac{D(H)-D(0)}{D(0)}$ and $r_m = \frac{R(H)-R(0)}{R(0)}$ of the power transmission coefficient $D(H)$ and power reflection coefficient $R(H)$, respectively, in an external magnetic field. The external magnetic field was applied in the direction shown in Fig. 3. The results of the measurements undertaken are shown in Fig. 7. All the $Co_x(SiO_2)_{1-x}$ nanocomposites exhibit weak monotonic variations caused, probably, by microwave magnetoresistance. Against the $Co_x(SiO_2)_{1-x}$ system, some nanocomposites of the $Co_x(Al_2O_3)_{1-x}$ system demonstrate a magnetic resonance line. The variations caused by the magnetic resonance are present for the $Co_{0.59}(Al_2O_3)_{0.41}$ and $Co_{0.34}(Al_2O_3)_{0.66}$ samples, see Fig. 7(b) and for the $Co_{0.34}(Al_2O_3)_{0.66}$ sample the resonance line is observed in higher field in accordance to the magnetization data in Fig. 2 where is seen that the magnetization of the $Co_{0.59}(Al_2O_3)_{0.41}$ sample is greater. Also, it is worth noting that a ferromagnetic phase in the samples is the phase that gives rise to the resonant-type variations in the magnetic field. The amplitude of the ferromagnetic resonance line is dependent on several factors, namely, the saturation magnetization, the size and shape of particles, the constant of magnetic damping [15]. Really, the magnetization for $Co_{0.59}(Al_2O_3)_{0.41}$ and $Co_{0.34}(Al_2O_3)_{0.66}$ samples is greater than that for other samples. From comparison between these microwave measurements and the conductivity data in Fig. 6 for the $Co_x(Al_2O_3)_{1-x}$ system it can be concluded that the demagnetizing factors for Co particles plays a valuable role because the resonance line presents only for $x = 0.59$ and $x = 0.34$ samples with high DC conductivity, therefore close to percolation. At the same time this reason is not determinative because the samples with $x = 0.3$ and $x = 0.24$ with high DC conductivity do not exhibit the resonance line. For the $Co_x(SiO_2)_{1-x}$ system, there is a

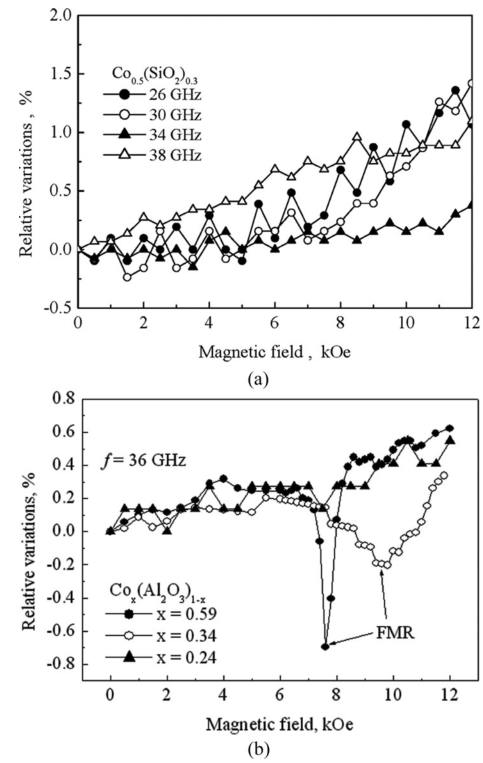

Fig. 7. Magnetic field dependences of transmission coefficients for a $Co_{0.5}(SiO_2)_{0.5}$ nanocomposite, measured at different frequencies (a) and resonant-type dependences of transmission coefficients for $Co_x(Al_2O_3)_{1-x}$ nanocomposite at frequency of 36 GHz for different cobalt contents (b).

ferromagnetic phase in the samples at least with cobalt content greater than $x = 0.55$. In order to make clear the reason why there is no distinct ferromagnetic resonance line in this system, structural investigations with high resolution are necessary that allow to determine the real size and shape of particles.

V. CONCLUSION

The paper presents experimental research of transmission and reflection coefficients for thin film metal-dielectric $Co_x(SiO_2)_{1-x}$ and $Co_x(Al_2O_3)_{1-x}$ nanocomposites. It has been found that the portion of dissipated power in the $Co_x(SiO_2)_{1-x}$ systems with different Co-content varies from 13 to 30% in the frequency range from 26 to 38 GHz.

Knowing a thickness of the films, the authors have succeeded in working out an algorithm of recovering the microwave effective film conductivity from frequency dependences of the transmission and reflection coefficients. DC conductivity of the film metal-dielectric nanocomposites is much less than their effective microwave conductivity. Therefore, displacement currents are very important for millimeter waveband electromagnetic wave to penetrate through these films. The frequency dependency of the transmission coefficient of the films is quite well described with the calculated conductivity. The DC conductivity of the film metal-dielectric nanocomposites is hundreds of times less than the continuous metallic film conductivity. There is a tendency: the microwave conductivity decreases with decreasing cobalt content in the nanocomposites.

It has been established that the frequency dependences measured in zero magnetic field for the above coefficients coincide very well with calculated ones providing that variations of the microwave conductivity are small in the frequency range aforementioned. Therefore it can be concluded that the frequency dependences of the transmission coefficient are mostly controlled by dispersion properties of a TE_{10} waveguide mode. When measured in a magnetic field, the $\mathrm{Co_x(Al_2O_3)_{1-x}}$ samples with cobalt content exceeding 34% demonstrate a ferromagnetic resonance line. However, the $\mathrm{Co_x(SiO_2)_{1-x}}$ systems show only weak monotonic variations of the transmission coefficient in magnetic field.

In principle, the results obtained offer the prerequisites for development of millimeter waveband devices containing these thin film materials.

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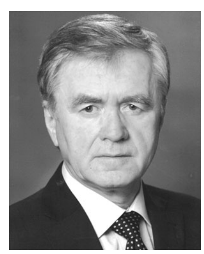

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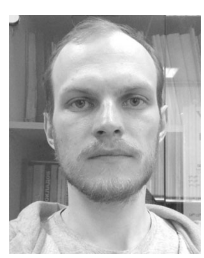

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