International Journal on
"Technical and Physical Problems of Engineering"
(IJTPE)
Published by International Organization of IOTPE

ISSN 2077-3528
IJTPE Journal
www.iotpe.com
ijtpe@iotpe.com

June 2023 | Issue 55 | Volume 15 | Number 2 | Pages 322-329

FERRITE NANO-FILMS ON FD-RESISTOR SHELL

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Abstract- The spectra of micro powders and nano thin films of $Ni_{1-x}Zn_xFe_2O_4$ ferrospinels in the region of super high frequencies, including terahertz frequencies, have been studied with the aim of their specific practical application in the structure of the shell of a frequency-dependent resistor to eliminate high-frequency over voltages in networks and devices in power engineering, electronics, computer systems for receiving/processing information, etc. The interpretation and analysis of the research results confirm the effectiveness of the use of Ni-Zn ferrites as an element of the absorbing shell of a frequency-dependent resistor for filtering high-frequency over voltages that occur in the high and super high frequencies.

Keywords: Ferrospinel, Super High Frequency, FD-Resistor, Overvoltage, Domain Wall.

1. INTRODUCTION

The perspective of using frequency-dependent resistors for filtering external and internal high-frequency interference arising from external, natural, or other factors leading to the occurrence of high-frequency over voltages in power networks and systems, in circuits of electronic modules, systems for receiving/transmitting and processing information, etc., as studies have shown, stimulated the study of electrical, thermal, magnetic, optical and other properties, the role of geometric factors, the presence of a layered structure of the ferromagnetic layer, the quality of the surfaces of the layers, etc. resistors of this type [1-10].

Already the first experimental studies and subsequent analysis of the processes of electric current flow through a frequency-dependent resistor under conditions of high-frequency interference were carried out in the framework of computer simulation based on the percolation model of the resistor shell, created on the basis of [11] indicated that the resistive properties and features of the flow of alternating current in the resistor are determined, first of all, the frequency dependence of the magnetic permeability of the ferromagnetic shell, the observed skin effect, the ratio of the concentrations of components in the structure, the grain sizes of micro- and nano powders of ferrite used in the shell of the resistor.

The promising materials for the shell of frequency-dependent resistors include $Ni_{1-x}Zn_xFe_2O_4$ ferrites, which, as studies have shown, intensively absorb electromagnetic radiation in the frequency range from 10 MHz to 1000 MHz. The absorption of electromagnetic radiation in these ferrites in the frequency range above 300 MHz is determined by ferromagnetic resonance. The results of studying the EPR spectra and AFM profiles of $Ni_{1-x}Zn_xFe_2O_4$ ferrites, published earlier in [12-15], established the presence of features associated with changes in spin concentrations in the octahedral and tetragonal sublattices in various compositions of $Ni_{1-x}Zn_xFe_2O_4$ and the temperature range (3-300 K). The observed effects were determined by the occupation of tetrahedral and octahedral positions by Zn, Ni, and Fe ions, that is, by changes in the sublattice magnetization. The absence of the identity of the $Ni_{1-x}Zn_xFe_2O_4$ sublattices causes non-zero total mechanical and magnetic moments and, since the values of the g-factors of the sublattices are different, the compensation points for the magnetic and mechanical moments do not coincide with each other. Thus, at the points of intersection of the dependences of the spin concentrations of the magnetic sublattices, in all probability, compensation points were observed. The conducted studies allowed us to establish that the reason for the spin reorientation observed in the EPR spectra of $Ni_{1-x}Zn_xFe_2O_4$ ferrites, as well as the generation of 26.4 THz (880 cm^{-1}) terahertz radiation observed in Raman scattering, is the appearance of magnetic inhomogeneities and a non-collinear magnetic structure as a result of the influence temperature on the concentration and orientation of spins of Fe^{3+} ions in canted and superparamagnetic states. The value of the energy gap equal to 0.10914 meV agrees in value with that found in experiments on small-angle neutron scattering.

The results of studies of the Raman scattering spectra [16] in $Ni_{1-x}Zn_xFe_2O_4$ nano powders and thin films made it possible to assume the presence of magnetic inhomogeneities in them localized in the surface layer of the ferrite film, the thickness of which is comparable to the length of the exchange spin waves. This assumption was based on a physical model of the coexistence of two subsystems in magnetite: one associated with cations (Fe^{2+} and Fe^{3+}) and the second with hopping 3d electrons between cations $Fe^{2+} \rightleftarrows Fe^{3+}$, leading to magnetic ordering.

In addition to the strong electrostatic interaction between cations, the theoretical description of the effect included taking into account the influence of the Vonsovskii s-d exchange interaction, which, in the case of a positive s-d exchange, took into account the magnetopolaron states, and in the case of a negative s-d exchange, not only the localization of conduction electrons but also partial screening of the magnetic moments of atoms as a result of the orientation of the spins of conduction electrons antiparallel to these moments. An analysis of the MFM profiles of (Zn-Ni) ferrites' thin films [14] confirmed the presence of magnetic clusters in them, the size of which depended on the composition of the ferrites. Note that the assumption about the presence and nature of magnetic excitations in $Ni_{0.4}Zn_{0.6}Fe_2O_4$ ferrite was previously expressed by the authors of [17], who performed an energy analysis of small-angle neutron scattering.

In the compositions x(Zn) = 0.60; 0.68; 0.75, at low temperatures, a high-field susceptibility characteristic of a noncollinear magnetic structure, as well as the occurrence of intensive small-angle scattering, were detected. The results of the above studies were reflected in the implementation of various types of shells for frequency-dependent resistors operating in the optical, low-frequency (up to 1 MHz), terahertz ranges. This publication presents the results of microwave studies (from 1 MHz to 20 GHz) of $Ni_{1-x}Zn_xFe_2O_4$ ferrites, the purpose of which was to expand the frequency range of operation of frequency-dependent resistors.

2. EXPERIMENTAL METHODS AND TECHNIQUE

Synthesis of micro powders and thin films of the compositions $Ni_{1-x}Zn_xFe_2O_4$, where, x = 0.0; 0.25; 0.4; 0.5; 0.6; 0.75; 1.0, with their further annealing at a temperature of 960 °C was carried out according to the high-temperature technology published in [14-16], the quality of which was controlled by X-ray diffraction patterns. The surface quality of $Ni_{1-x}Zn_xFe_2O_4$ films, where x=0.25, 0.4, 0.5, 0.6, 0.75 deposited on high-quality surfaces of leucosapphire substrates 2 mm thick and 12 mm in diameter, were studied on SmartSPM confocal and atomic force microscopes with an AIST-NT measuring head (Tokyo Instruments, Japan) [14]. The Raman spectra of $Ni_{1-x}Zn_xFe_2O_4$ micro powders and thin films were studied on a 3D Laser Raman Micro spectroscopy System Nano finder 30 Raman spectrometer (Tokyo Instruments, Japan) [16, 18]. The excitation source was a YAG Nd laser (λ=532 nm), with the possibility of changing the radiation power from 0.1 mW to 10 mW. Note that these spectra already refer to high terahertz frequencies. EPR data obtained with the aid of a ELEXSYS E500 spectrometer (Bruker, Germany) at 300 K and down to 4K [14].

The high-frequency spectra of ferrites were studied using a "Deepace KC901V" vector network analyzer by the coaxial method in a short-circuited line. Granular thin films consisting of nano- or submicron magnetic particles embedded in the technological process in an immiscible, non-conductive or metallic matrix represent a special class of artificially structured materials that, due to the possibility of modeling and forming microstructures with a predetermined geometry that provides the necessary spatially modulated magnetic and electrical profiles, high coercivity, giant magnetoresistance, spin-orientation transitions, and other macroscopic quantum effects, turned out to be promising for new technical solutions. At the same time, the rigidity of the bond between the particles and the matrix turned out to be an important factor in the stability of the created structure. As it turned out, the tendency to conglomerate free particles over time led to the degradation of some key properties of granular magnetic systems, which significantly depended on the volume fraction x_v and sizes 2r of embedded magnetic particles, where the parameter x_v can continuously vary from 0 to 1, and the parameter 2r has the value, only for small values of x_v. The evolution of the granular microstructure with a change in the x_v parameter can be represented as follows: for any granular systems with a uniform dispersion of ferrite fillers, there is a so-called percolation volume fraction x_p, whose values lie in the range of 0.5-0.6. Isolated ferrite beads exist at $x_v<x_p$, and a continuous network of interacting beads appears at $x_v>x_p$.

In a real granular system, the particles are not spherical. If the average distance between particles is of the order of the particle size, then giant magneto-transport properties appear. In granular systems, the single-domain behavior of magnetic particles is observed at low volume concentrations ($x_v<x_p$), and the usual magnetic properties appear at $x_v>x_p$. In this case, all magnetic moments are coaxial and rotate synchronously with the external field as one giant moment, which corresponds to extreme ferromagnetic characteristics. Granular $Ni_{0.5}Zn_{0.5}Fe_2O_4$ (one of the Ni-Zn ferrites) embedded in a fused quartz matrix exhibits superparamagnetic properties even after being heated at 1100 °C for 2 hours. In the (Ni-Zn-ferrite/SiO_2) system, magnetic properties can be changed in a wide range by changing the volume fraction and diameter of ferromagnetic particles. At x<34%, the formation of pure stoichiometric Ni-Zn ferrite in the SiO_2 matrix is observed. Note that a characteristic feature of Ni-Zn ferrites is the presence of electronic transitions in those initiated by super exchange interaction of the $Fe^{2+}\rightarrow O^{2-}\rightarrow Fe^{3+}$ type.

The combination of various concentrations of Ni-Zn ferrite and its heat treatment made it possible to obtain samples with a saturation magnetization from 1.3 to 68 emu/g and a coercive force from 0 to 123 Oe, a value that is higher than the coercivity of bulk Ni-Zn ferrite. It was also found that $NiFe_2O_4$ films ($Ni_{1-x}Zn_xFe_2O_4$ composite at x=0) deposited on a sapphire substrate had a high coercive force, which was ~32.5 mT in out-of-plane and in-plane geometries. The calculation of deformations at the film-substrate interface, taking into account the parameters of crystal lattices and elastic coefficients C_{11}=174 GPa and C_{12}=67 GPa, showed that: coercive field, during compression: in-plane - 32.5 mT and out-of-plane - 32.5 mT; in tension: in-plane - 0 and out-of-plane - 25 mT. Magnetization: when compressed: in-plane - 10 G and out-of-plane - 5 G; in tension: in-plane - 26 G and out-of-plane

- 23 G. Thus, in thin $NiFe_2O_4$ films, there are coercive fields in the out-of-plane geometry and there is anisotropy of the "easy" plane type, which is characteristic of two-dimensional layers.

On the other hand, the technology of obtaining ultrathin films by high-temperature evaporation of the initial components in a vacuum chamber is very difficult. That is why it was decided to use PLD (pulsed laser deposition) - a method in which granular ferrite microstructures with spatially modulated magnetic and electrical profiles are created by the action of pulsed laser radiation on submicron $Ni_{0.4}Zn_{0.6}Fe_2O_4$ powder particles deposited on a glass substrate (optical glass K8, a similar glass produced by the German company Schott AG, is designated as BK7).

It has been established [19], that the thin films of ferrite obtained by deposition were observed to overlap the crystalline islands with an amorphous phase. The subsequent annealing of the obtained films led to their texturing and enhancement of the magnetic properties, and despite the fact that the magnetization of the films was close to the magnetization of the bulk ferrite, the value of the coercive magnetic field in the films was greater than in the bulk material, i.e. confirms the conclusion of the authors of the publication [20] that this feature is associated with the anisotropy of crystallites observed in the microstructure of the film of this ferrite. The scheme of the experiment is shown in Figure 1a. Modification of $Ni_{0.4}Zn_{0.6}Fe_2O_4$ ferrite nano powder was carried out by radiation from a wavelength-tunable Spectra-Physics Tsunami laser. With irradiation parameters: radiation wavelength 1.064 μm, pulse duration 10 ns, and energy density 480 mJ/cm^2 were obtained, shown in Figure 1, photomicrographs of areas of the substrate with particles of ferrite powder without exposure (b) and under exposure to a constant magnetic field (c, d), with a resolution better than 250 nm. Under the action of laser pulses with a duration of ~10 ns, conglomerates of submicron particles melted and combined into drop-like structures.

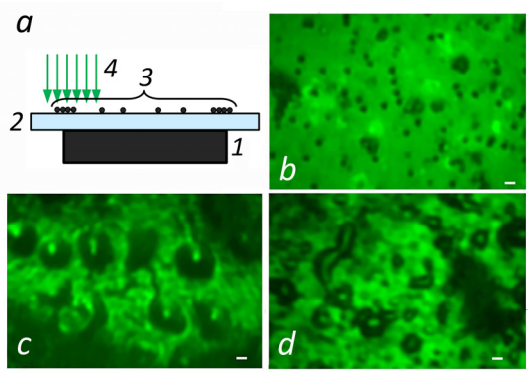

Figure 1. Experimental scheme, (a) 1. permanent magnet, 2. glass substrate; 3. submicron powder particles $Ni_{0.4}Zn_{0.6}Fe_2O_4$, 4. laser radiation, (b) radiation without influence, (c) at the influence of magnetic field, scale = 1 μm [20]

Analysis of the microcrystalline structure shown in Figure 1 showed that the nano thin ferrite film is formed by spheroids of various sizes, unevenly distributed in a fine-crystalline granular mass with rare crystallographically irregular, isometric inclusions. A complete AFM analysis of the resulting nanofilms was published in [14, 20].

Technological control of the process, previously published in [20], was carried out to achieve the desired effect for the reproducibility of parameters and the efficient operation of devices. It can be considered established that the melt of this ferrite wets the glass surface. This fact is easy to explain if you pay attention to the property of the components that make up the ferrite composition to react with silicon dioxide, namely:

Reaction $SiO_2 + 2Fe_3O_4 \rightarrow 3Fe_2SiO_4 + O_2$, in which SiO_2 is an acid and Fe_3O_4 - alkali, passes with the formation of iron orthosilicate, that is, the mineral fayalite:

$2O^{-II} - 4e^- \rightarrow 2O^0$ (oxidation)

$6Fe^{8/3} + 4e^- \rightarrow 6Fe^{II}$ (recovery)

On the other hand, this reaction is reversible;

$3Fe_2SiO_4 + O_2 \rightarrow 2Fe_3O_4 + 3SiO_2$

Similarly, the reaction $2NiO + SiO_2 \rightarrow Ni_2SiO_4$ of the interaction of nickel oxide with SiO_2 leads to the formation of nickel silicate, and the reaction $ZnO + SiO_2 = ZnSiO_3$ leads to the formation of zinc metasilicate. At about 1200 °C, all three ferrite components react with SiO_2 Detailed studies of the strength characteristics of wetting with Ni-Zn-ferrites in junctions with glasses, published in [20], indicated that in the case of, for example, permalloy (i.e., Fe-Ni alloy) or zinc-nickel ferrite, the maximum stress compensation was achieved at glass thicknesses and permalloy 1-1.5 μm and 0.5-0.75 μm, respectively.

The parameters of the optimal mode for obtaining junctions were set, namely: temperature rise to 900 °C at a rate of 10 °C/min, cooling from 900 °C to 500 °C at a rate of 10 °C/min., at a temperature of 500 °C isothermal exposure 40 min. With subsequent cooling of the junction at a rate of 3 °C/min. The influence of an external magnetic field on the nature of the change in macros tresses and the role of the mutual orientation of the domain and external magnetic fields were also established. Thus, it becomes obvious that the created ferrite microstructures, due to the chosen geometry of the macrostructure, will have specific spatially modulated magnetic and electrical profiles.

3. EXPERIMENTAL RESULTS AND THEIR DISCUSSION

As stated in [21], in the frequency range up to 300 MHz, domain wall resonance plays an important role. The condition for the occurrence of domain wall resonance is their rigid fixation at grain boundaries, pores, or other defects, which ensures their displacement relative to the equilibrium position. The frequency of natural oscillations of domain walls is determined by the stiffness coefficient of the quasi-elastic force, reduced to a unit of the effective mass of the domain wall. In a real ferrite, there are many domain boundaries of various sizes, and each such boundary has its own oscillation frequency, so the dispersion region μ'' is composed of many peaks of resonances of individual boundaries. The resonance frequency of the boundary depends on its type. The harder and shorter the boundary, the higher the resonance frequency. Thus, the resonance frequency of domain walls

is determined by the grain sizes of ferrite micro powders, and the actual spread of ferrite grain sizes will lead to an increase in the width of the domain wall resonance curve.

Assuming grains of ferrite spherical diameter d, the mass of the domain wall m can be determined from Equation (1) [22].

$$m = \frac{d^2}{2\gamma^2}\left[\frac{2kT_C}{aK}\left(1+\frac{\mu_0 H_A}{I_0}\right)\right]^{-1/2} \quad (1)$$

where, γ is the gyromagnetic ratio, k is the Boltzmann constant, a is the lattice constant, K is the crystallographic anisotropy constant, T_C is the Curie temperature of $Ni_{1-x}Zn_xFe_2O_4$ ferrites, H_A is anisotropy field, which is related to the natural ferromagnetic resonance frequency ω_{EFMR} by the $H_A = \omega_{EFMR}/\gamma$. The value of the stiffness coefficient or quasi-elastic coefficient from the Dering Equation $\xi = \frac{4\pi r I_0^2}{3\mu\mu_0} + 2\pi\sigma$.

The I_0 is saturation magnetization = 0.2÷0.3 Tl; 2r is major axis of the ellipsoid of revolution; μ is initial magnetic permeability; surface tension force $\sigma = \left(\frac{2kT_C K}{a}\right)^{\frac{1}{2}}$.

The δ_ω is domain wall thickness $\sigma_\omega = \sqrt{\frac{2kT_C}{aK}}$ and the natural frequency of oscillations of domain walls ω_0 is expressed in terms of the effective mass and the generalized quasi-elastic coefficient by the expression $\omega_0 = \sqrt{\frac{\xi}{m_{eff}}}$.

Figures 2 and 3 show the calculated (within the framework of the percolation model) and experimental spectra of the frequency dependences of the imaginary and real parts of the magnetic permeability for $Ni_{0.5}Zn_{0.5}Fe_2O_4$ in the range from 1 to 20000 MHz. As can be seen, the region of the spectrum caused by the vibrations of the boundaries has the form of a wide frequency decay band μ' and its corresponding resonant maximum μ'', the high-frequency "tail" of which can reach frequencies much higher than the frequency of the natural resonance of the boundaries. The nature of these spectra is obviously influenced by the processes of super exchange interaction, etc. [21], which lead to recessions μ' and maxima μ'' at frequencies coinciding with their relaxation frequencies, for $1/\tau$ close to the oscillation frequency of the boundaries ω_0, an expansion of the resonance region is observed, and for $1/\tau \leq \omega_0$ a relaxation maximum μ'' and the corresponding decline μ' form a separate area [21]. An estimate of the oscillation frequency of domain walls ω_0 for $Ni_{0.5}Zn_{0.5}Fe_2O_4$ ferrite micro powder gives a value of 280 MHz.

As is known, the conductivity of $Ni_{1-x}Zn_xFe_2O_4$ ferrites is associated with the presence in them of two and three-charged ions of the transition elements of iron and nickel. In the conduction process, the participation of nickel is difficult, while, between iron ions of different valence, the process of electron hopping through the intermediate oxygen ion (indirect exchange) is observed.

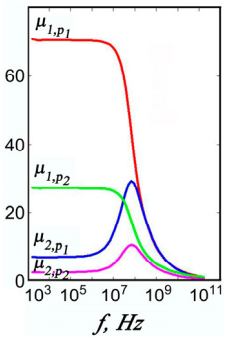

Figure 2. Calculated frequency dependences of the imaginary and real parts of the magnetic permeability of the control percolated medium with filling factors p= 0.5 and 0.8, particle radius an equal to 2·10-3 cm, magnetic permeability of the ferromagnetic substance (100-10 j), electrical conductivity σ= 105 $Om^{-1}cm^{-1}$, Filler: $Ni_{0.5}Zn_{0.5}Fe_2O_4$

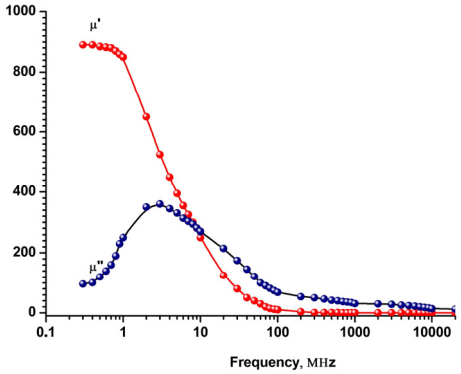

Figure 3. Experimental frequency dependences of the imaginary and real parts of the magnetic permeability of $Ni_{0.5}Zn_{0.5}Fe_2O_4$

The predominant directions of electron hopping can be caused by the precession of magnetization, which is associated with the fact that ions of different valence occupy crystallographically nonequivalent positions, and there are four types of such octahedral knots in spinels. Their local axes form different angles with the magnetization, and the energy of the system depends on where, at what site, at a given moment of time, the "extra" electron is located. Thus, during the precession of the magnetization, the angles of the moment vector with the local axes and the energy levels of the system corresponding to the doubly charged iron ion change, providing the preferential directions of transitions with the frequency of precession.

And, taking into account that the transitions of electrons between octahedral sites may lag behind the change in energy, the energy will be transferred from the magnetic subsystem to the subsystem of ions with variable valence, and from it to the crystal lattice with which the ions are associated.

$$\tau = \tau_0 \exp\frac{W}{kT} \quad (2)$$

where, W is the activation energy (constant value); for spinels: $\tau_0 \sim 10^{-14}$, $W \sim 0.25$ eV and usually, the achievement of relaxation time to $1/\omega$ occurs at temperatures of 300-400 K. As can be seen, this maximum in the compositions x=0.5 and 0.75 is reached at a temperature of 200 K.

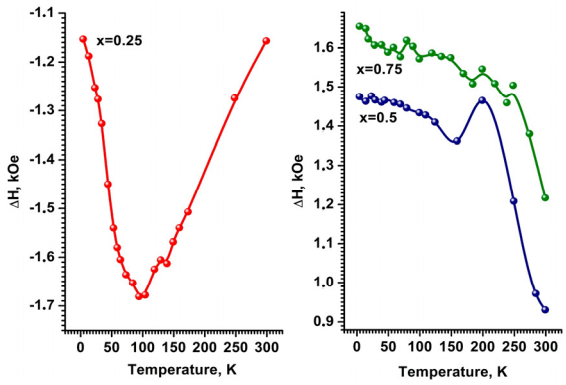

Figure 4. Experimental temperature dependences of the resonant field of $Ni_{1-x}Zn_xFe_2O_4$ (x=0.25; 0.5; 0.75) micro powders

For the composition, the relaxation frequencies of the excited states of Fe^{3+} are such that one can assume very fast energy transfer from the lattice and magnetic sublattices, and it is natural that ions strongly bound to the magnetic subsystem will be excited during its vibrations. The temperature dependences of resonant fields are determined by the temperature dependences of relaxation times and equilibrium populations. The relaxation time usually decreases rapidly according to the law (2). Note that the theory of Clogston [21] even in a rough approximation confirms the observed frequency and temperature dependences of the resonance curves and dynamic shifts of the resonance field, which are shown in Figure 4 for various compositions of $Ni_{1-x}Zn_xFe_2O_4$ (x=0.25; 0.5 and 0.75) ferrites.

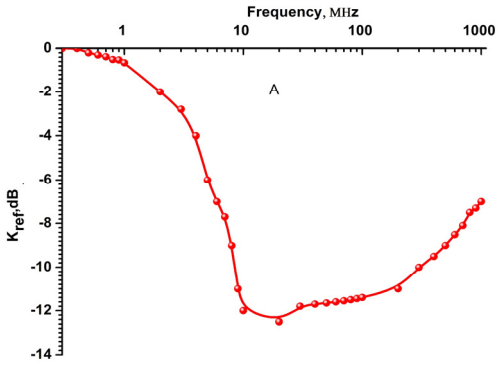

Figure 5. Reflection coefficient spectrum of $Ni_{0.5}Zn_{0.5}Fe_2O_4$ at T=300 K in the range from 1 to 1000 MHz

An analysis of the absorption of the THz spectra (Figures 5 and 6) of a frequency-dependent resistor made in the form of thin granular films of $Ni_{0.4}Zn_{0.6}Fe_2O_4$ ferrite confirmed their possibility of using interference in the terahertz frequency range [20]. Observed split sequences: (0.343 and 0.483; 0.571 and 0.727; 0.815 and 0.967; 1.05 and 1.211; 1.288 and 1.444; 1.516 and 1.680; 1.763 and 1.955; 2.012 and 2.131; 1.35; 2; 14 THz) correlates with acoustic modes with intervals: ~0.24 THz (0.9 meV), within the group ~0.15 THz (0.6 meV), between the limits ~0.08 THz (0.33 meV). The positions of the maxima in the THz spectra of granular $Ni_{0.4}Zn_{0.6}Fe_2O_4$ ferrite films of different thicknesses practically do not change, which, as indicated in [20, 23], can occur due to unlimited acoustic phonon modes.

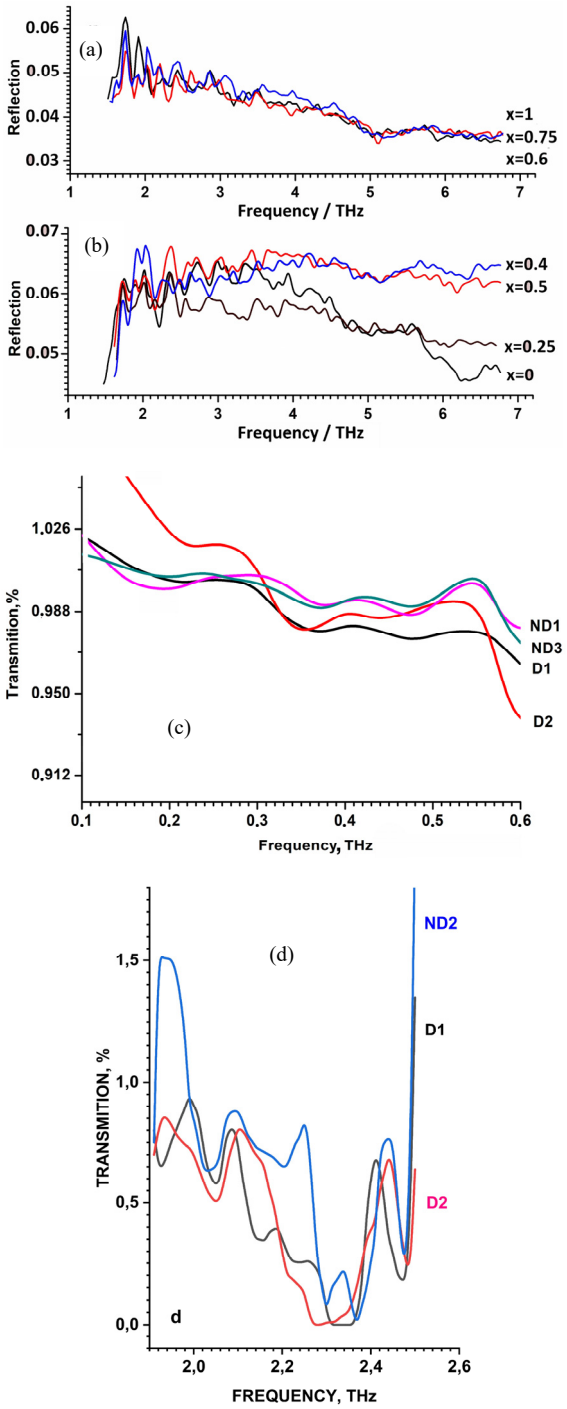

Figure 6. Terahertz reflection spectra, (a, b) $Ni_{1-x}Zn_xFe_2O_4$ ferrite nano powders in the 1-7 THz region, (c) transmission of the $Ni_{0.4}Zn_{0.6}Fe_2O_4$ granular film in the range of 0.1-2.5 THz [19, where, D1, D2 are sections of the film that differ in density (dense), (d) ND2 is a section of low density (not dense)

As noted in [20], in the range from 0.4 to 2 THz, the absorption of electromagnetic radiation in magnetite due to spin ordering is extremely low, since the frequency of spin reorientation in a magnetic material does not exceed 0.02 THz [23]. Thus, the process of absorption of electromagnetic radiation in the THz range is mainly associated with dielectric losses. The transmission spectra of the selected sections of the $Ni_{0.4}Zn_{0.6}Fe_2O_4$ films in the THz region show oscillations associated with acoustic

modes. The change in transmission demonstrates a weak dependence on the frequency of the terahertz field. Comparing the results of terahertz studies with the data of [24], it is easy to notice their similarity, indicating the possibility of studying ferrites in all $Ni_{1-x}Zn_xFe_2O_4$ compositions in the THz frequency range of the $Fe^{2+} \rightarrow O^{2-} \rightarrow Fe^{3+}$ super exchange interaction.

Analyzing the results of research in the field of microwave frequencies, as well as the practical use of Ni-Zn ferrites as an effective absorbing sheath of a frequency-dependent resistor for filtering high-frequency over voltages, it is pleasant to note a new confirmation - the publication of [25] confirming the complete suppression of the Ni-Zn ferrite absorber. distortion at frequencies of 10, 2.4, 3.65, and 5 GHz.

ACKNOWLEDGEMENTS

The work is carried out in limits within the framework of the Azerbaijan-Belorussian project at financial support of Belorussian and Azerbaijan Funds of fundamental investigations (project Ф18А3-003 Belarus) and (projects: EIF-2013-9(15)-46/05/1-M-12 and EIF-BGM-3-BRFTF-2^+/2017 Azerbaijan.

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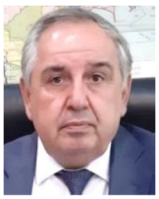

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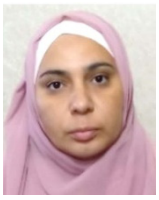

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