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Investigation of the frequency dispersion of the complex permittivity of lithium-iron spinel doped with La, Y

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The temperature-frequency dependences of the complex dielectric permittivity of $Li_2Fe_{2.5-x}Me_xO_4$ (Me = La; Y, x = 0.01; 0.03; 0.05) spinels, synthesized by the «sol-gel» autocombustion technology, were obtained by the method of impedance spectroscopy, in temperature range 298-473K. Their analysis indicates the presence of a fractal structure in the studied samples, the influence of which is manifested in the entire studied temperature interval. To study the electric polarization phenomenon associated with this structure, Josher's generalized law has been used.

Keywords: impedance spectroscopy, spinel, fractal structure, frequency dispersion of dielectric permittivity, dielectric susceptibility, dielectric loss angle, hopping mechanism of conduction.

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Introduction

Spinel ferrites, due to their dielectric and magnetic properties, are widely used in radio-electronic devices of various technical purposes. At the same time, the ability of such materials to intercalate-deintercalate lithium ions into their structure allows them to be considered as a promising material for the manufacture of the cathode matrix of portable lithium current sources [1].

The electrical properties of ferrites depend significantly on the method of synthesis, preparation conditions, chemical composition, cation distribution and microstructure of the material. The doping method is one of the most common in chemistry and technology as a way to control the structure of complex oxides and create new functional materials. Aluminum-doped lithium-iron spinels-ferrites with the general formula $Li_{0.5}Fe_{2.5-x}Al_xO_4$, synthesized by ceramic technology, have attracted the attention of researchers as stable ferrite materials widely used in modern technological systems [2,3]. The morphology, content of phases, crystal structure of the spinel phase of synthesized Al-substituted lithium-iron spinels depending on the composition and regime of heat treatment at the final stage of synthesis, and their electrophysical characteristics were investigated in works [4,5]. The research in work [6] of the temperature dependence of the conductivity of the synthetic material showed that in the temperature range lower than 475 K, the electronic component of the conductivity of these disordered systems dominates, which can be realized with the help of activation mechanism and of hopping mechanism. It was shown in work [7] that in the region of temperatures higher than 475 K in synthesized $Li_{0.5}Fe_{2.5-x}Al_xO_4$ ceramics, the Li^+-ion mechanism of conductivity becomes predominant. In work [8], using the generalized Josher law, the dielectric properties of these materials were investigated and it was shown that their polarization significantly depends on the aluminum content in them.

In recent years, in order to expand the range of electrophysical properties of lithium-iron spinels, which can be useful in various fields of technology, in addition to isovalent substitution of iron ions with aluminum ions, attempts are being made to substitute ions of other elements. In this regard, the doping of lithium-iron spinels with ions of rare earth metals may be a promising trend. A number of papers [9-11] have been published in scientific journals in which the structure, morphology, and electromagnetic properties of several nanocrystalline iron spinels doped with rare earth metals using the "sol-gel" synthesis technology are investigated.

The work [12] investigated the temperature-frequency dependences of the conductivity of $Li_2Fe_{2.5-x}Me_xO_4$ (Me = La; Y, x = 0.01; 0.03; 0.05) spinels synthesized by the "sol-gel" autocombustion technology, in temperature range 298-473 K. On the basis of their analysis, it was determined that the main mechanisms of conductivity of these materials in the studied temperature range are hopping and activation. The effect of doping lithium-iron spinels with impurities of rare earth metals on these conductivity mechanisms was investigated. It was determined that the presence of these impurities in small concentrations in the synthesized samples significantly reduces their conductivity mainly due to the destruction of the electronic conductivity hopping mechanism.

The purpose of this work is to investigate the frequency dispersion of the complex permittivity of lithium-iron spinel doped with rare earth metals in the temperature range of 298-473K based on the generalized Josher law.

I. Research methodology

The procedure of «sol-gel» autocombustion synthesis, which used for the synthesis of the samples, was as follows: for each composition, according to the formula, the necessary amounts of starting compounds were calculated, which were selected as crystal hydrates of iron nitrates $Fe(NO_3)_3 \cdot 9H_2O$, lithium $LiNO_3$, lanthanum $La(NO_3)_3 \cdot 9H_2O$ and yttrium $Y(NO_3)_3 \cdot 9H_2O$. Citric acid acted as a chelating agent, and an aqueous ammonia the addition of citric acid. Ammonia solution (10%) was added dropwise to the precursors solution to adjust the required pH level (≈7). The resulting solution was kept in a drying cabinet at a temperature of 343 K until the water was completely removed. After that, the dry gel was placed in an oven and heated to a temperature of 523-553 K at which the mixture ignited and the final product was formed. For conducting impedance studies, briquettes were created by pressing the obtained powder with the addition of a 10% solution of polyvinyl alcohol (PVA). The obtained samples with a diameter of 1 cm and a height of about 0.4 cm were subjected to sintering at a temperature of 873 K for 4 hours in an air atmosphere with slow cooling.

Dielectric characteristics of the synthesized compounds were calculated on the basis of experimental impedance spectra obtained on Autolab PGSTAT 12/FRA-2 spectrometer in the frequency range of 0.01 Hz – 100 kHz and the temperature range of 293-473 K. Temperature recordings were carried out with isothermal exposure every 20 K.

II. The results of the experiment and their discussion

Dielectric losses, which characterize the conversion of a part of electrical energy into thermal energy, are an important electrophysical parameter of the material. The magnitude of these losses, as well as their frequency dependence, is determined by the features of the polarization mechanism. Complex dielectric permittivity is a particularly convenient parameter for describing the dependence of dielectric loss on frequency:

$$\varepsilon^*(\omega) = \varepsilon'(\omega) + i\varepsilon''(\omega). \quad (1)$$

The value of dielectric losses is mostly characterized by the tangent of the dielectric loss angle:

$$\tan\delta(\omega) = \frac{\varepsilon''(\omega)}{\varepsilon'(\omega)}. \quad (2)$$

Dielectric losses usually change significantly when various impurities are introduced into the material. Depending on the concentration of impurities or structural defects, the value of dielectric losses can vary by tens or hundreds of times, while the change in the value of the real part of the complex permeability ε' can be relatively small. Therefore, dielectric losses are the most sensitive indicator of changes in the material structure. The study of dielectric losses and their dependence on impurities, structural defects, and other factors (temperature, intensity and frequency of the electric field, etc.) is of great interest for solid state physics.

In this work, we will investigate how the presence of rare earth metal impurities La and Y in small quantities affects the frequency dependence of the loss tangent, and therefore the structure of lithium-iron spinels.

The temperature-frequency dependences of the complex dielectric constant of the studied samples were calculated using experimental Nyquist diagrams in work [12]. The frequency dispersion of the tangent of the dielectric loss angle $tg\delta$ in the synthesized samples of lithium-iron spinel without impurities for the range of investigated temperatures is shown in figure 1. In work [13], this behavior of the dispersion curves of the tangent of the dielectric loss angle with well-defined maxima is associated with the manifestation of the fractal structure of the material.

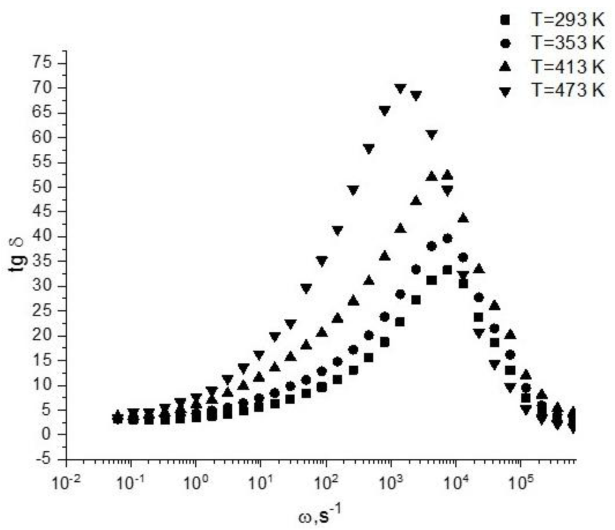

Fig. 1. Experimental dependencies of the tangent of the dielectric loss angle on frequency for several temperature values in $Li_{0,5}Fe_{2,5}O_4$ spinel samples.

As can be seen from the figure, when the temperature increases, the local maximum of the dependence, which characterizes the resonant frequency of dielectric losses, shifts to the region of low frequencies. At the same time, the value of the maximum increases. Such a temperature dependence of the tangent of the dielectric loss angle is evidence that with increasing temperature, the electronic hopping mechanism is gradually destroyed and replaced by an activation one [12].

According to [13], in the generalized Josher law, which satisfactorily describes the contribution of various mechanisms to the complex conductivity as a function of frequency for a wide class of different heterogeneous substances, regardless of their specific structure, except for the ordinary current caused by free current carriers $\vec{J}_0 = \sigma_0 \vec{E}$, and the polarization current $\vec{J}_\mathrm{p} = \frac{\partial \vec{P}}{\partial t}$, caused by the emergence of electric polarization dipoles, we consider the current that is associated with the fractal structure $\vec{J}_{frac} = \sigma_{frac}(\omega)\vec{E}$ and is caused by local polarization with the resulting field of microstructural formations of the material. Then the total current is equal to:

$$\vec{J} = \vec{J}_0 + \vec{J}_\mathrm{p} + \vec{J}_{frac} = \sigma(\omega)\vec{E}, \tag{3}$$

where the generalized conductivity normalized to the constant $\frac{\varepsilon_0}{4\pi}$ $(\varepsilon_0 = 8{,}85 \cdot 10^{-12}\,\frac{\Phi}{\text{м}})$

$$\sigma(\omega) = -i\omega(\varepsilon(\omega) - \varepsilon_\infty) \tag{4}$$

is expressed through the complex dielectric constant, which takes the form:

$$\varepsilon(\omega) = \varepsilon_\infty + \frac{\sigma_0}{i\omega} + \frac{\chi\tau^{-\nu}}{i\omega + (i\omega)^{1-\nu}\tau^{-\nu}} + R(i\omega) \tag{5}$$

The penultimate term of this expression reflects the contribution to the dielectric permittivity of the fractal structure, and the last term represents the contribution of relaxation processes, which in the case of migration polarization is expressed by the Cole-Cole formula:

$$R(i\omega) = \frac{\varepsilon_0 - \varepsilon_\infty}{1 + (i\omega\tau)^\nu} \ . \tag{6}$$

In formula (5), the value χ determines the dielectric susceptibility of the fractal structure.

Using simple algebraic transformations from formula (5), expressions for calculating the real and imaginary part of the complex dielectric constant can be found:

$$\varepsilon'(\omega) = Re[\varepsilon(\omega)] = \varepsilon_\infty + \frac{\chi\omega^{-\nu}\tau^{-2\nu}\sin\left(\frac{\nu\pi}{2}\right)}{\omega\left[1 + 2\cos\left(\frac{\nu\pi}{2}\right)(\omega\tau)^{-\nu} + (\omega\tau)^{-2\nu}\right]} + \frac{(\varepsilon_0 - \varepsilon_\infty)\left[1 + \cos\left(\frac{\nu\pi}{2}\right)(\omega\tau)^\nu\right]}{1 + 2\cos\left(\frac{\nu\pi}{2}\right)(\omega\tau)^\nu + (\omega\tau)^{2\nu}}, \tag{7}$$

$$\varepsilon''(\omega) = Im[\varepsilon(\omega)] = \frac{\sigma_0}{\omega} + \frac{\chi\tau^{-\nu}\left[1 + \cos\left(\frac{\nu\pi}{2}\right)(\omega\tau)^{-\nu}\right]}{\omega\left[1 + 2\cos\left(\frac{\nu\pi}{2}\right)(\omega\tau)^{-\nu} + (\omega\tau)^{-2\nu}\right]} + \frac{(\varepsilon_0 - \varepsilon_\infty)\left[1 + \sin\left(\frac{\nu\pi}{2}\right)(\omega\tau)^\nu\right]}{1 + 2\cos\left(\frac{\nu\pi}{2}\right)(\omega\tau)^\nu + (\omega\tau)^{2\nu}} \ . \tag{8}$$

Figure 2 shows the experimental frequency dependences of the tangent of the dielectric loss angle, together with their approximating theoretical curves according to formulas (2), (7) and (8) for four temperature values. The frequency experimental dependences at these temperatures correspond to the values of the parameters of

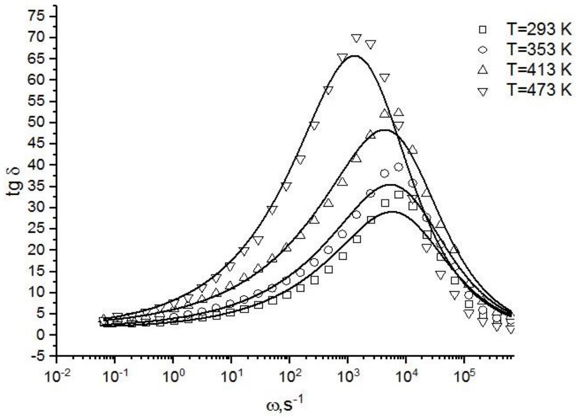

Fig. 2. Approximation of the experimental dependences of the tangent of the dielectric loss angle on the frequency for several temperatures by theoretical curves obtained on the basis of formulas (2), (7) and (8) for a sample of $Li_{0.5}Fe_{2.5}O_4$ spinel free of impurities.

Table 1.

T, K	ε_∞	ε_0	σ_0, s^{-1}	ν	τ, s	χ
293	120	$3{,}0 \cdot 10^8$	$2{,}7 \cdot 10^{10}$	0,38	0,83	$3{,}91 \cdot 10^{12}$
353	113	$4{,}8 \cdot 10^8$	$3{,}4 \cdot 10^{10}$	0,36	2,0	$5{,}94 \cdot 10^{12}$
413	130	$7{,}9 \cdot 10^8$	$5{,}0 \cdot 10^{10}$	0,33	9,83	$9{,}79 \cdot 10^{12}$
473	180	$3{,}9 \cdot 10^8$	$2{,}3 \cdot 10^{10}$	0,38	30,8	$3{,}44 \cdot 10^{12}$

the approximating curves presented in Table 1.

Let's investigate how the presence in $Li_{0.5}Fe_{2.5}O_4$ spinel of lanthanum impurity, which is the most common representative of rare earth metals, affects the dielectric losses and dielectric susceptibility of the fractal structure. Figure 3 shows the experimental frequency dependences of the tangent of the dielectric loss angle, together with their approximating theoretical curves obtained according to formulas (2), (7) and (8) for lanthanum-doped $Li_2Fe_{2.5-x}La_xO_4$ spinel at temperature of T = 293 K for different impurity contents. The experimental frequency dependences at this temperature correspond to the values of the parameters of the approximating curves presented in Table 2.

As follows from Figure 3, as the content of lanthanum impurity in the samples increases, the value of the maximum of tangent of the dielectric loss angle decreases and it shifts to lower frequencies. Obtained theoretically, by approximating experimental frequency dependences with theoretical curves, the dielectric susceptibility χ of the fractal structure decreases significantly, by orders of magnitude.

There is a close correlation between the theoretical parameters ν, τ, χ, the fractal structure and the conductivity mechanism of the synthesized samples. By studying the effect on these parameters of the increase in the impurity content in the samples, we can predict how their fractal structure changes. The fact that the value of the exponent of the power of the frequency in the Josher equation is within $0{,}31 \leq \nu \leq 0{,}42$ for all values of x indicates the realization of the hopping mechanism of conductivity in all samples at temperature T=293K. The hopping mechanism of electrical conductivity in these ceramics is mainly realized by the hopping of an electron between ions of the same element (in this case, these are ions Fe^{2+} and Fe^{3+}), which can be in more than one valence state, randomly distributed in crystallographically equivalent octohedral sites of the lattice [14].

The parameter τ means of the polarization relaxation time. As the temperature of the samples increases, it increases, which indicates a decrease in the mobility of electrons that participate in the hopping mechanism of conduction within the grains. Conversely, with an increase in the content of the lanthanum impurity in the samples, the relaxation time decreases, which is a consequence of the fact that the sizes of boundaries between grains increase. An exception to this rule for the

$Li_2Fe_{2.5-x}La_xO_4$ spinel sample with x = 0.03 can be explained by the fact that groups of closely spaced grains formed in it due to the non-uniformity of the fractal structure.

The magnitude of the dielectric susceptibility χ of the fractal structure of the sample is determined by the concentration of free charge carriers and their ability to move throughout the entire volume of the sample, which in turn depends on the features of the fractal structure (its homogeneity, grain sizes and grain boundaries). A sharp decrease, by orders of magnitude, in the dielectric susceptibility indicates that with an increase in the impurity content in the samples, the concentration of electrons participating in conductivity decreases, and the dispersion of grain sizes and the average distance between them increases. This is also evidenced by the decrease in the maximum value of the tangent of the dielectric loss angle and its shift to the low frequency region.

The effect of replacing iron ions with yttrium ions Y^{+3} on the electrical properties of lithium-iron spinels has the same character as the effect of replacing it with lanthanum ions La^{+3} [12]. An impurity of yttrium, as well as an aimpurity of lanthanum, leads to heterogeneity of the structure of the synthesized samples, which is characterized by a dispersion of grain sizes and an increase in the average distance between them. However, the presence of an yttrium impurity in the synthesized samples reduces their conductivity much more strongly than the presence of a lanthanum impurity.

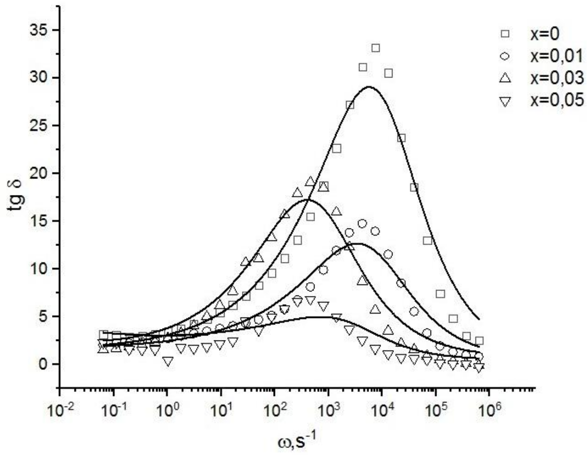

Fig. 3. Experimental dependencies of the tangent of the dielectric loss angle on the frequency at the temperature T=293K and their approximating curves for samples of $Li_2Fe_{2.5-x}La_xO_4$ spinel with different values of x.

Table 2

x	ε_∞	ε_0	σ_0, s^{-1}	ν	τ, s	χ
0	120	$3{,}0 \cdot 10^8$	$2{,}7 \cdot 10^{10}$	0,38	0,83	$3{,}91 \cdot 10^{12}$
0,01	87	$5{,}6 \cdot 10^7$	$6{,}0 \cdot 10^9$	0,37	0,14	$2{,}8 \cdot 10^{11}$
0,03	98	$2{,}0 \cdot 10^7$	$1{,}2 \cdot 10^9$	0,42	1,84	$2{,}0 \cdot 10^{10}$
0,05	140	$1{,}0 \cdot 10^6$	$2{,}3 \cdot 10^{10}$	0,31	0,02	$1{,}5 \cdot 10^9$

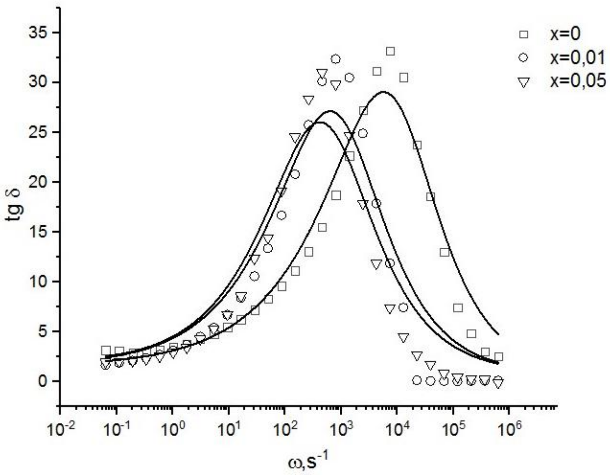

Fig. 4. Experimental dependencies of the tangent of the dielectric loss angle on the frequency at the temperature T = 293 K and their approximating curves for samples of $Li_2Fe_{2.5-x}Y_xO_4$ spinel at different values of x.

Table 3

x	ε_∞	ε_0	σ_0, s^{-1}	ν	τ, s	χ
0	120	$3{,}0 \cdot 10^8$	$2{,}7 \cdot 10^{10}$	0,38	0,83	$3{,}91 \cdot 10^{12}$
0,01	167	$6{,}3 \cdot 10^7$	$3{,}1 \cdot 10^9$	0,42	3,8	$1{,}3 \cdot 10^{11}$
0,05	172	$3{,}0 \cdot 10^7$	$1{,}8 \cdot 10^9$	0,44	3,8	$3{,}5 \cdot 10^{10}$

Figure 4 shows the experimental frequency dependences of the tangent of the dielectric loss angle, together with their approximating theoretical curves according to formulas (2), (7) and (8) for yttrium-doped lithium-iron spinel at a temperature of T = 293K with different impurity content. The experimental frequency dependences at this temperature correspond to the values of the parameters of the approximating curves given in Table 3.

As can be seen from Figure 4, the maxima of the approximating curves shift to lower frequencies with increasing yttrium impurity content in the samples, but unlike the case of doping spinel with lanthanum, their values decrease slightly. This means that both active and polarization components of the current through the samples decrease to the same extent. At the same time, there is a sharp decrease in the dielectric susceptibility χ and a sharp increase in the relaxation time already at x=0.01 and its invariance with a further increase in the content of the yttrium impurity. From all this, we can come to the conclusion that the yttrium impurity destroys the electronic hopping mechanism of the surface layer conductivity more strongly than the lanthanum impurity, significantly reducing the concentration of ions in the surface layers of monocrystalline grains of spinel.

Conclusions

The generalized Josher's law, which we applied to the study of polarization processes, quite correctly describes the experimental temperature-frequency dependences of the tangent of the dielectric loss angle of lithium-iron spinel synthesized by the «sol-gel» autocombustion technology and doped with rare earth metal impurities. On its basis, it is possible to analyze the evolution of the fractal structure with a change in the temperature of the studied samples and the concentration of the impurity, estimate the average size of its grains, obtain a value of its dielectric susceptibility, and also draw a conclusion about the dominant conduction mechanism in the given temperature range.

It has been determined that an increase in the content of lanthanum and yttrium impurities in the samples leads to an increase in the inhomogeneity of the fractal structure (an increase in the dispersion of the sizes of single-crystal grains and intergrain boundaries) and the destruction of the electronic hopping conduction mechanism. Moreover, in the presence of yttrium impurity, this destruction is stronger than in the presence of lanthanum. This is most likely due to the fact that the radius of the yttrium ion is smaller than the radius of the lanthanum ion and it more easily replaces iron ions in the crystal lattice of the surface layers of monocrystalline grains of lithium-iron spinel.

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Дослідження частотної дисперсії комплексної діелектричної проникності літій-залізної шпінелі, легованої La, Y

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Методом імпедансної спектроскопії отримані температурно-частотні залежності комплексної діелектричної проникності $Li_2Fe_{2,5-x}Me_xO_4$ (Me = La; Y, x = 0,01; 0,03; 0,05) шпінелей, синтезованих за технологією «золь-гель» автоспалювання, в інтервалі температур 298-473 К. Їх аналіз вказує на присутність фрактальної структури в досліджуваних зразках, вплив якої проявляється у всьому досліджуваному інтервалі температур. Для дослідження явища електричної поляризації, пов'язаного із цією структурою, використаний узагальнений закон Джошера.

Ключові слова: імпедансна спектроскопія, шпінель, фрактальна структура, частотна дисперсія діелектричної проникності, діелектрична сприйнятливість, кут діелектричних втрат, стрибковий механізм провідності.