Synthesis of New Complex Ferrite $Li_{0.5}MnFe_{1.5}O_4$, Chemical – Physical and Electrophysical Research

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Article

Synthesis of New Complex Ferrite $Li_{0.5}MnFe_{1.5}O_4$, Chemical – Physical and Electrophysical Research

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Abstract: In this article, the sol-gel method was used as a synthesis method, which shows the physico –chemical nature of the synthesis of a new complex material ferrit Li0.5MnFe1.5O4. The synthesized structure and structure were determined by the method of X-ray phase analysis. According to the analysis indicators, it was found that our compound is single-phase, spinel-structured, and syngony - cubic type. The microstructure of the compound and the quantitative composition of the elements contained were analyzed under a scanning electron microscope (SEM). Through a scanning electron microscope, Microsystems were taken from different parts of l Li0.5MnFe1.5O4-type crystallite, the elemental composition of crystals was analyzed, and the general type of surface layer of complex ferrite was shown. As a result, the fact that the compound consists of a single phase, the clarity of its construction was determined by the topography and chemical composition of the compound. As a result, it was found that the newly synthesized complex ferrites correspond to the Formula Li0.5MnFe1.5O4. The particles of the formed compounds have a large size (between 50.0 μm, 20.0 μm and 10.0 μm). Electro physical measurements were carried out on the LCR - 800 unit at intervals of 293-483 K and at frequencies of 1.5 and 10 kHz. The increase in frequency to 10 kHz led to a decrease in the value ε in the range of the studied temperature (293-483 K).

Keywords: Sol-gel method; ferrites; X-ray; pycnometric density; elementary cell parameters; scanning electron microscope:

1. Introduction

Research in nanotechnology is a fast-growing field at the fore-front of physics and is attracting special interest from the scientific community [1]. Multifunctional materials, in particular, are investigated for their advantageous properties that are combined in one smart compound [2,3]. A broad range of intelli gent materials have been proposed and among them, ferrites have drawn significant attention and are deemed to be promoter hosts due to their extremely prominent features [4,5].

Doped mixed ferrites with impactful features like permeability, magnetic behavior, and magnetic transition at high temperatures are the soft ferrimagnetic compounds, which already mark their presence in the technology field. These materials are significant due to their imprudent magnetization at room environment [6,7].

The extensive use of spinel ferrites is due to their low price, easy fabrication and abundant use in technological and industrial applications [8]. The technological and industrial applications of these magnetic materials is because of their excellent electrical and magnetic properties, which leads to their use in magnetic memory devices, transformers, high frequency applications and electronic devices like cellular phones, video cameras, notebook computers [9].

Many researcher studied the structural, electrical and magnetic properties of various Li based ferrites like Li-Mn [10], Li-Ni [11], Li-Co [12], Li-Mg [13], Li-Cr [14], Li-Zn [15] and Li-Ti [16] prepared by ceramic method. However, Ferrite prepared by a ceramic method involves high calcination

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temperature synthesis for the completion of solid-state reaction between the constituent oxides or carbonates and the particles are obtained rather bigger and non-uniform in size. These non-uniform particles, on compacting, result in the formation of voids and subsequently the low density ferrites. To overcome these difficulties, the sol-gel method is the preferred method for synthesizing nanoferrit on a volumetric scale by obtaining homogeneous particles and the most convenient method for synthesizing nanoparticles. This is because the simplicity of this method, the low cost precursors, the fact that the annealing process is carried out in a short time, good control of the amount of crystallite and other properties of materials [17].

Nanoscale manganese ferrites exhibit interesting structural and magnetic properties. They have a wide range of applications, from basic research to industrial applications. In this work, the composition of ferrite nanoparticles is studied.

2. Materials and Methods

The sol - gel method was used as an effective way to synthesize a new mixed ferrite of a complex mixed composition. in order to determine the composition of a new complex mixed ferrite obtained by the sol - gel method, an X-ray phase study was carried out, and in order to conduct quantitative and qualitative analysis, an examination under a scanning electron microscope, a study of electrophysical properties (dielectric conductivity and electrical resistance) was carried out.

As a primary raw material, distilled water of the manganese (III) oxide (“chemical pure”) brand, Iron (III) oxide (“chemical pure”) brand and lithium conbanate (“chemical pure”) brand were used. Trihydric alcohol – glycerin and citric acid were used as gel-forming reagents. The raw materials obtained by stoichiometric calculation were weighed on an analytical balance with the highest accuracy of 0.0001 grams, mixed, ground in an agate sieve, placed in an Alund crucible and fired in a muffle furnace at a temperature of 1100ºC.

The resulting dried gel was fired in a muffle oven for 10 hours at a temperature of 600 ºC until a black powder was obtained. Finally, the resulting powder was fired in stages for 7 hours at a temperature of 700-1100 ºC in the air [18].

The pycnometric density of ferrites was determined by the method [19]. Toluene and distilled water were used as indifferent liquids. The density of composite materials was measured 5 times and the average values were calculated.

3. Results

3.1. X-ray Analysis

X-ray analysis was carried out at the Kazakh National Women’s Pedagogical University on the diffractometer Miniflex/600 (Rigaku). Analysis using a sika beam (U=30 KV, J=10 MA, rotation speed 1000 pulses per second, time constant t=5 sec., 2θ with an angle interval between 5 and 900) was carried out on the Miniflex 600 RIGAKU, filtered by a filter.,

During the X-ray observation of the synthesized mixed complex ferrite, it was observed that the amorphous state of the samples decreased, the crystallization process was complete, and the kinetics of the sol-gel reaction was low. In addition, it is proved from the diffractogram shown in Figure 1 below that the samples have changed from the amorphous state to the fully polycrystalline state, and the independent phase has been completely formed.

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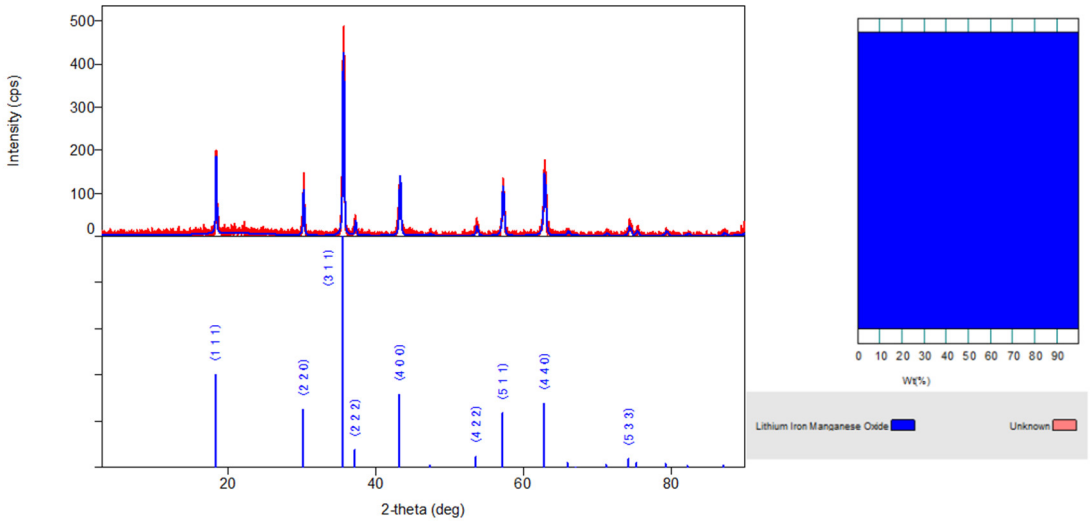

Figure 1. X-ray diffractogram of the complex ferrite Li0,5MnFe1.5O4. Insert: phase ratio diagram.

Below are the results of the Rietveld method of indexing the diffractogram of complex ferrite studied by X-ray analysis.

According to Scherrer's formula, the average size of crystallites can be written as follows:

$$d = \frac{K\lambda}{\beta \cos\theta} \quad (1)$$

where

d - average size of crystals;

K - dimensionless particle shape coefficient (Scherrer constant);

λ - is the wavelength of X-ray radiation;

β - half-height reflex width (2θ in radians and units)

θ - diffraction angle (Bragg angle)

A new complex mixed ferrite synthesized by X-ray phase analysis method has unit number Z = 8, Li0,5MnFe1.5O4 is crystallized in cubic wall - centered cell, and space group is Fd $\bar{3}$m. Cell parameters a = 8.3677, b = 8.3677, c = 8.3677. The average size of crystallites according to Scherrer's formula through the X-ray wavelength of ferrite is 40.6 μm, and the correctness of the results of X-ray studies was confirmed by the coincidence of the values of X-ray and pycnometric densities.

3.2. Scanning Electron Microscope

A study was carried out using a scanning electron microscope (SEM) (APPLICATION Note team, Brooker, Germany) to study the spectrum of distribution of the element, quantitative and qualitative analysis, and the percentage content of emenets.

A scanning electron microscope (SEM) is designed to obtain a magnified image of an object by scanning it with an electron beam directed at it and recording the signal generated by the interaction of electrons with a detector. The small diameter of the probe, even at low accelerating voltages and high currents, allows for elemental analysis of samples with dimensions of the analyzed area of several tens of nanometers. The beam current detector is located on the microscope column below the aperture of the objective lens, so that the beam current can be monitored at any time during the analysis.

In order to study the morphology of the surface layer of the new complex mixed ferrite samples synthesized by the sol-gel method, a study was carried out using an electron microscope scanning the microstructures of the electric diffraction image. Electron monographs of the compound taken in an imaging electron microscope are given in Figure 2 a), b), c).

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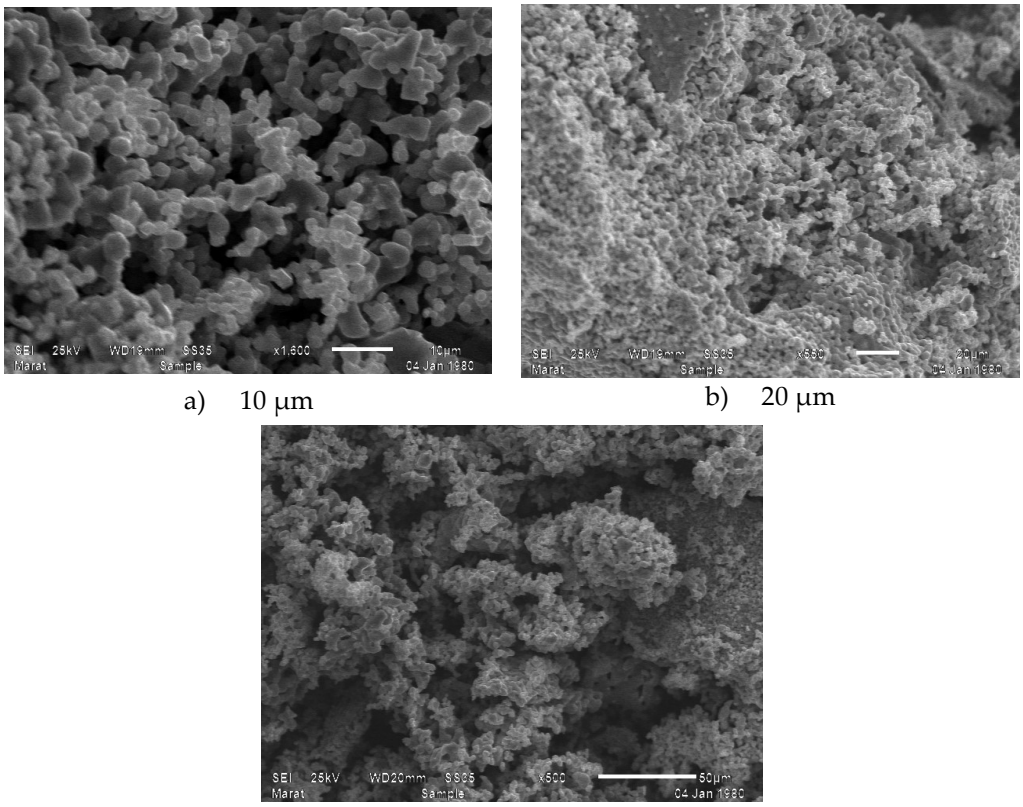
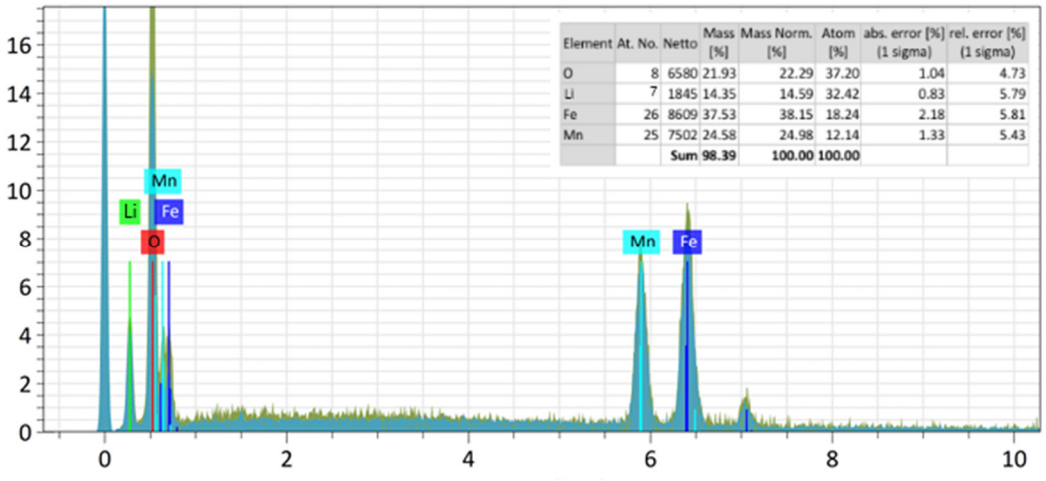


a) 10 μm b) 20 μm

c) 50 μm

Figure 2. Image of the new mixed complex ferrite $Li_{0,5}MnFe_{1.5}O_4$ measured with three different micrometer accuracy.

The above images show the results of micrographs taken at magnifications of 10.0μm, 20.0μm and 50.0μm, and also show the general appearance of the complex ferrite surface layer. As a result, the compound consists of one phase, the clarity of its structure was determined by the topography and chemical composition of the compound. Figure 3 shows the spectrum patterns of the newly synthesized mixed complex ferrite $Li_{0,5}MnFe_{1.5}O_4$ and the elemental analysis results of the $Li_{0,5}MnFe_{1.5}O_4$ compound.



Element	At. No.	Netto	Mass [%]	Mass Norm. [%]	Atom [%]	abs. error [%] (1 sigma)	rel. error [%] (1 sigma)
O	8	6580	21.93	22.29	37.20	1.04	4.73
Li	7	1845	14.35	14.59	32.42	0.83	5.79
Fe	26	8609	37.53	38.15	18.24	2.18	5.81
Mn	25	7502	24.58	24.98	12.14	1.33	5.43
		Sum	98.39	100.00	100.00		

Figure 3. Spectrum samples of the $Li_{0,5}MnFe_{1.5}O_4$ compound. The results of the element analysis are built-in.

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Based on the distribution map of elements, on the basis of solving the nature of crystallization, the chemical composition with microstructure and distribution zones of chromium, iron, lithium, and oxygen atoms were studied. As a result of the numerical elemental composition study, it can be concluded that iron, chromium, lithium metals, oxygen, carbon atoms are distributed in the 10.0 μm regions (Figure 2 a). In an imaging electron microscope, it is possible to obtain nanoscale measurements of solids in powder form.

To study the spectrum of distribution of the element, quantitative and qualitative analysis, and the percentage content of emenets, a study was carried out using a scanning electron microscope. The spectrum samples of the synthesized new mixed complex Ferrite and the results of elemental analysis are shown in Figure 3.

3.3. Electrophysical Research

Measurements of electrophysical properties were carried out according to the methods [20,21].

The study of electrophysical properties (dielectric constant and electrical resistance) was carried out by measuring the electrical capacity of samples on a serial device LCR-800 (Taiwan) at an operating frequency of 1 kHz continuously in dry air in a thermostatic mode with a holding time at each fixed temperature.

Plane-parallel samples in the form of disks with a diameter of 10 mm and a thickness of 2-6 mm with a binder additive (⌀1,5 %) were pre-manufactured. The pressing was carried out at a pressure of 20 kg/cm2. The resulting discs were fired in a laboratory furnace "SNOL" at 400 ° C for 6 hours. Then they were carefully double-sided sanded.

The dielectric constant was determined from the electrical capacity of the sample at known values of sample thickness and electrode surface area. The Sawyer-Tower scheme was used to obtain the relationship between the electric induction D and the electric field strength E. Visual observation of the D (E hysteresis loop) was carried out on an oscilloscope C1-83 with a voltage divider consisting of a resistance of 6 MOm and 700 kOm and a reference capacitor of 0.15 mcF. The frequency of the generator is 300 Hz. In all temperature studies, the samples were placed in an oven, the temperature was measured by a chromel-alumel thermocouple connected to a voltmeter B2-34 with an error of 0.1 mV. The rate of temperature change is ⌀5 K/min. The value of the dielectric constant at each temperature was determined by the formula:

$$\varepsilon = \frac{C}{C_0} \tag{2}$$

where $C_0 = \frac{\varepsilon_0 \cdot S}{d}$ is the capacitance of the capacitor without the test substance (air).

Calculation of the width of the forbidden zone (ΔE) of the substance under investigation is determined by the formula:

$$\Delta E = \frac{2kT_1T_2}{0.43(T_2 - T_1)} \lg \frac{R_1}{R_2}, \tag{3}$$

where k is the Boltzmann constant equal to 8,6173303·10-5 эВ·K-1, R1 is the resistance at T1, R2 is the resistance at T2.

To ensure the reliability of the data obtained, the dielectric constant of a standard substance, barium titanate BaTiO3, was measured at frequencies equal to 1 kHz, 5 kHz and 10 kHz. Table 1 below shows the results of measurements of the electrophysical characteristics of BaTiO3.

Table 1. Symmetry type and unit cell parameters of $Li_{0,5}MnFe_{1.5}O_4$.

Sample	**$Li_{0,5}MnFe_{1.5}O_4$**
Space group	$Fd\bar{3}m$, cubic side centered
Z	8

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Parameter cell (Å)	
a =	8.3677(9)
b =	8.3677(9)
c =	8.3677(9)
V(A^3)	585.90(11)
α	90
β	90
γ	90
According to Scherrer's formula, the average size of crystallites is	40.6 μm
X-ray density (g/cm3)	4.678
Pycnometric density (g/cm3)*	4.675

Table 2. Dependence of electrical resistance (R), electrical capacity (C) and dielectric constant (ε) on $BaTiO_3$ temperature.

T, K	C, nF	R, Ом	ε	lgε	lgR
Measurement frequency at 1 kHz					
293	0,27278	13400	1296	3,11	4,13
303	0,27426	13270	1303	3,11	4,12
313	0,27715	12910	1316	3,12	4,11
323	0,28125	12560	1336	3,13	4,10
333	0,28772	11890	1367	3,14	4,08
343	0,29313	11210	1392	3,14	4,05
353	0,29916	10290	1421	3,15	4,01
363	0,30751	9383	1461	3,16	3,97
373	0,31202	8831	1482	3,17	3,95
383	0,31702	9061	1506	3,18	3,96
393	0,32255	8814	1532	3,19	3,95
403	0,32967	7881	1566	3,19	3,90
413	0,3423	7098	1626	3,21	3,85
423	0,35119	6902	1668	3,22	3,84
433	0,36668	6153	1742	3,24	3,79
443	0,38018	6317	1806	3,26	3,80
453	0,39802	6010	1891	3,28	3,78
463	0,4169	5584	1980	3,30	3,75
473	0,43147	5149	2050	3,31	3,71
483	0,45456	4656	2159	3,33	3,67
Measurement frequency at 5 kHz					
293	0,25678	29630	1220	3,09	4,47
303	0,2683	21650	1274	3,11	4,34
313	0,2775	13080	1318	3,12	4,12
323	0,28638	5236	1360	3,13	3,72
333	0,29667	4301	1389	3,14	3,63
343	0,30226	4733	1409	3,15	3,68
353	0,30787	3296	1436	3,16	3,52
363	0,31283	2966	1462	3,17	3,47
373	0,31843	2805	1486	3,17	3,45
383	0,32148	2529	1513	3,18	3,40
393	0,32578	2669	1527	3,18	3,43
403	0,32976	3172	1547	3,19	3,50
413	0,33303	4434	1566	3,19	3,65
423	0,33948	6377	1582	3,20	3,80
433	0,35613	9644	1613	3,21	3,98
443	0,3713	11520	1692	3,23	4,06
453	0,3925	10430	1764	3,25	4,02
463	0,41682	8021	1864	3,27	3,90
473	0,44245	5978	1980	3,30	3,78

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483		3799	2102	3,32	3,58
Measurement frequency at 10 kHz					
293	0,11814	152300	561	2,75	5,18
303	0,18494	70790	878	2,94	4,85
313	0,22927	32200	1089	3,04	4,51
323	0,25954	11870	1233	3,09	4,07
333	0,27501	4842	1306	3,12	3,69
343	0,28531	3312	1355	3,13	3,52
353	0,29302	2689	1392	3,14	3,43
363	0,29988	2257	1424	3,15	3,35
373	0,30652	1946	1456	3,16	3,29
383	0,31215	1689	1483	3,17	3,23
393	0,31667	1737	1504	3,18	3,24
403	0,32294	3130	1534	3,19	3,50
413	0,32779	5945	1557	3,19	3,77
423	0,33406	8231	1587	3,20	3,92
433	0,34256	8805	1627	3,21	3,94
443	0,35658	8052	1694	3,23	3,91
453	0,378	5967	1796	3,25	3,78
463	0,39475	4604	1875	3,27	3,66
473	0,41687	3343	1980	3,30	3,52
483	0,44203	2353	2100	3,32	3,37

As can be seen from the data in Table 1, the values of dielectric constant at 293 K at frequencies of 1 kHz and 5 kHz are in satisfactory agreement with its recommended value of 1400 ± 250. In addition, the observed changes in the electrical conductivity of BaTiO3 at 383 K at all frequencies (1 kHz, 5 kHz and 10 kHz) are also consistent with its transition from the perovskite cubic phase Pm3m to the tetragonal (polar) ferroelectric phase with space group P4mm [26–28].

It should be noted that despite the reduced values of the dielectric constant of BaTiO3 at a frequency of 10 kHz, and at T equal to 293 K, 303 K, 313 K, all values of ε BaTiO3 at all three frequencies (1 kHz, 5 kHz and 10 kHz) in the range 313-483 K have approximately the same values up to 2150, which indicates that that the frequency change does not particularly affect the temperature dependence of the dielectric constant of BaTiO3 in the range 313-483 K.

Electrophysical measurements of $Li_{0.5}MnFe_{1.5}O_4$ in the range of 293-483 K and frequencies equal to 1, 5 and 10 kHz were carried out at the LCR installation (Table 3, Figure 4)

Table 3. Dependence of electrical capacity (C), electrical resistance (R) and dielectric constant (ε) of material 2 on temperature and frequency.

T, K	**C, nF**	**R, Ом**	**ε**	**lgε**	**lgR**
Measurement frequency at 1 kHz					
293	7,7977	165700	22448	4,35	5,22
303	10,302	135800	29658	4,47	5,13
313	15,68	101700	45140	4,65	5,01
323	12,489	131900	35954	4,56	5,12
333	8,1684	203600	23515	4,37	5,31
343	14,516	121200	41789	4,62	5,08
353	86,933	32510	250266	5,40	4,51
363	176,65	19300	508547	5,71	4,29
373	265,14	14110	763295	5,88	4,15
383	378,56	10520	1089813	6,04	4,02
393	495,27	8184	1425802	6,15	3,91
403	312,87	10720	900702	5,95	4,03
413	11,039	85500	31779	4,50	4,93
423	1,8105	379400	5212	3,72	5,58
433	0,95809	649900	2758	3,44	5,81
443	0,68585	843600	1974	3,30	5,93

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453	0,70009	874900	2015	3,30	5,94
463	0,89737	796100	2583	3,41	5,90
473	1,3483	666100	3882	3,59	5,82
483	2,1329	511600	6140	3,79	5,71
Measurement frequency at 5 kHz					
293	1,3926	140100	4009	3,60	5,15
303	1,8886	115400	5437	3,74	5,06
313	2,728	89690	7853	3,90	4,95
323	1,517	128100	4367	3,64	5,11
333	0,9064	184900	2609	3,42	5,27
343	2,5719	93070	7404	3,87	4,97
353	15,47	29090	44536	4,65	4,46
363	31,031	17900	89333	4,95	4,25
373	45,909	13060	132165	5,12	4,12
383	64,608	9799	185996	5,27	3,99
393	82,944	7702	238782	5,38	3,89
403	40,501	11420	116596	5,07	4,06
413	1,7403	79660	5010	3,70	4,90
423	0,28075	293500	808	2,91	5,47
433	0,13233	455500	381	2,58	5,66
443	0,09429	533500	271	2,43	5,73
453	0,09084	555900	262	2,42	5,74
463	0,10483	536400	302	2,48	5,73
473	0,13985	477500	403	2,60	5,68
483	0,19866	401900	572	2,76	5,60
Measurement frequency at 10 kHz					
293	0,62512	126300	1800	3,26	5,10
303	0,85581	103700	2464	3,39	5,02
313	1,188	83510	3420	3,53	4,92
323	0,50394	127500	1451	3,16	5,11
333	0,3238	169200	932	2,97	5,23
343	1,2082	76460	3478	3,54	4,88
353	7,2162	26640	20774	4,32	4,43
363	14,584	16680	41985	4,62	4,22
373	21,676	12440	62402	4,80	4,09
383	30,192	9304	86918	4,94	3,97
393	39,355	7426	113297	5,05	3,87
403	14,483	12940	41694	4,62	4,11
413	0,64942	83960	1870	3,27	4,92
423	0,12128	243700	349	2,54	5,39
433	0,06145	329700	177	2,25	5,52
443	0,04865	351900	140	2,15	5,55
453	0,04694	362500	135	2,13	5,56
463	0,05137	360400	148	2,17	5,56
473	0,06369	339200	183	2,26	5,53
483	0,08415	306100	242	2,38	5,49

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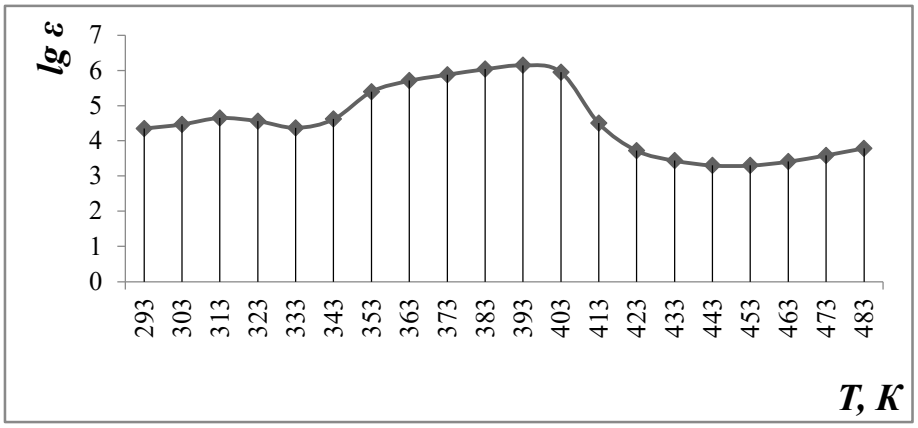

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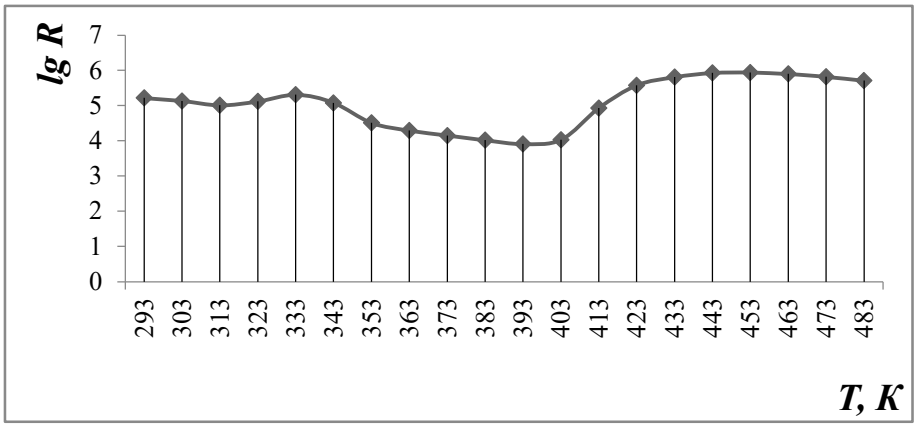

Figure 4. Dependence of dielectric constant (a) and electrical resistance (b) on temperature and frequency equal to 1 kHz.

The data in Tables 3 and Figure 4 show that the value of the dielectric constant (ε) reaches a maximum value at 383 K, equal to 1.43·106 (1 kHz). Then they decrease to 6140 at 483 K (frequency 1 kHz). Increasing the frequency to 5 and 10 kHz leads to a decrease in ε in the entire temperature range under study (293-483 K).

The study of the temperature dependence of electrical resistance shows the complex nature of conductivity: at ΔT = 293-313 K – semiconductor, at ΔT = 313-333 K – metallic, at 333-393 K – semiconductor, at ΔT = 393-453 K – metallic and at ΔT = 453-483 K – again semiconductor.

a) Calculation of the width of the forbidden zone (ΔE) in the range 293-313 K:

T, K	*lg R*
293	5,22
313	5,01

$$\Delta E = \frac{2 \times 0{,}000086173 \times 293 \times 313}{0{,}43(313-293)} lg\frac{5{,}22}{5{,}01} = 1{,}91 \text{ эВ}$$

b) Calculation of the width of the forbidden zone (ΔE) in the range 333-393 K:

T, K	*lg R*
333	5,31
393	3,91

$$\Delta E = \frac{2 \times 0{,}000086173 \times 333 \times 393}{0{,}43(393-333)} lg\frac{5{,}31}{3{,}91} = 1{,}19 \text{ эВ}$$

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c) Calculation of the width of the forbidden zone (ΔE) in the range 453-483 K:

T, K	*lg R*
453	5,94
483	5,71

$$\Delta E = \frac{2 \times 0{,}000086173 \times 453 \times 483}{0{,}43(483-453)} lg\frac{5{,}94}{5{,}71} = 3{,}04 \text{ эВ}$$

The band gap of the material 2 at ΔT = 293-313 K and 333-393 K are equal to 1.91 and 1.19 eV, respectively (narrow-band semiconductor), and at ΔT = 453-483 K is equal to 3.04 eV (wide-band semiconductor).

4. Conclusions

Summing up the results of the study, a new mixed complex ferrite with the composition $Li_{0.5}MnFe_{1.5}O_4$ was synthesized for the first time by the Sol – gel method. In order to determine the composition of the resulting new complex mixed ferrite, an X-ray phase study was carried out, and in order to conduct quantitative and qualitative analysis, an examination under a scanning electron microscope, a study of electrophysical properties (dielectric conductivity and electrical resistance) was carried out.

For the first time, syngony types and parameters of elementary cells of a complex mixed Ferriter synthesized by the method of X-rayophase analysis were determined. $Li_{0.5}MnFe_{1.5}O_4$ (cubic, a=8.3677(9), B= 8.3677(9), C=8.3677(9) Å , Z=8, denX-ray = 4.678 g/cm3, denPycno = 4.675 g/cm3); through the X-ray wavelength of Ferrite, the average size of crystallites according to Scherrer's formula is 40.6 μm. The results of radiographic research showed that the synthesized compound is polycrystalline. The accuracy of crystallochemical data is evidenced by a satisfactory correspondence of X-ray and pycnometric densities.

Through a scanning electron microscope, Microsystems were taken from different parts of $Li_{0.5}MnFe_{1.5}O_4$ type crystallite, the elemental composition of crystals was analyzed, and the general type of surface layer of complex ferrite was shown. As a result, the fact that the compound consists of a single phase, the clarity of its construction was determined by the topography and chemical composition of the compound. As a result, it was found that the newly synthesized complex ferrites correspond to the formula $Li_{0.5}MnFe_{1.5}O_4$. The particles of the formed compounds have a large size (between 50.0 μm, 20.0 μm and 10.0 μm). The results of the elemental analysis were presented in the form of a table.

Electrophysical measurements of $Li_{0.5}MnFe_{1.5}O_4$ on the LCR - 800 unit were carried out at intervals of 293-483 K and frequencies of 1.5 and 10 kHz.

The fact that 293 K is equal to a relatively high value of 2.69*105 at 1 kHz, the maximum value of 383 K reached 1.43*106. Next, they are reduced to 6140 at 483 K (frequency of 1 kHz). An increase in frequency to 5 and 10 kHz leads to a decrease in the entire test temperature interval (293-483 K).

The study of the dependence of electrical resistance on temperature shows the complex nature of conductivity: t = 293-313 K-semiconductor, t = 313-333 K – Metal, 333-393 K-semiconductor, t = 393-453 K-Metal and T = 453-483 K – semiconductor again.

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