New zircon-manganites of lanthanum and alkali metals

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Zircon-manganites of the composition $LaMe^{I}_2ZrMnO_6$ (Me^{I} – Li, Na, K) were synthesized using the ceramic technology method in the range of 800-1200°C by the interaction of lanthanum (III) oxides and lithium, sodium and potassium carbonates (analytical grade). Using X-ray diffraction methods, it was established that all synthesized zircon-manganites crystallize in a cubic system with the following lattice parameters: $LaLi_2ZrMnO_6$ – a = 16.26 ±0.02 Å; V^0 = 4300.93 ±0.06 Å^3; Z = 6; $V°_{elem\ cell}$ = 716.82 ± 0.02 Å^3; $\rho_{x\text{-}ray}$ = 5.49 g/cm^3; $\rho_{picn.}$ = 5.42 ± 0.06 g/cm^3; $LaNa_2ZrMnO_6$ – a = 16.85 ± 0.02Å; V^0 = 4785.46 ± 0.02 Å^3; Z = 6; $V°_{elem\ cell}$ = 795.58 ± 0.02 Å^3; $\rho_{x\text{-}ray}$ = 5.35 g/cm^3; $\rho_{picn.}$ = 5.30 ± 0.04 g/cm^3; LaK_2ZrMnO_6 – a = 17.45 ± 0.03 Å; V^0 = 5318.85 ±0.09 Å^3; Z = 6; $V°_{elem\ cell}$ = 885.81 ± 0.02 Å^3; $\rho_{x\text{-}ray}$ = 5.16 g/cm^3; $\rho_{picn.}$ = 5.08 ± 0.02 g/cm^3. It has been established that with an increase in ionic radii in the Li→Na→K series, the values of the parameter "a" and the volumes of lattices and unit cells of zircon-manganites increase.

Keywords: lanthanum; zircono-manganite; lithium; sodium; potassium; synthesis; radiography.

Лантан мен сілтілі металдардың жаңа циркон-манганиттері

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$LaMe^{I}_2ZrMnO_6$ (Me^{I} – Li, Na, K) құрамды циркон-манганиттер керамикалық технология әдісімен 800-1200°C аралықта лантан (III) оксидтері мен литий, натрий және калий карбонаттарының әрекеттесуімен синтезделді. Рентгендік дифракция әдістерін қолдана отырып, барлық синтезделген циркон-манганиттер келесі тор көрсеткіштері бар кубтық сингонияда кристалданатыны анықталды: $LaLi_2ZrMnO_6$ – a = 16,26 ±0,02 Å; V^0 = 4300,93 ±0,06 Å^3; Z = 6; $V°_{эл.ұяш.}$ = 716,82 ± 0,01 Å^3; $\rho_{рент.}$ = 5,49 г/см^3; $\rho_{пикн.}$ = 5,42 ± 0,06 г/см^3; $LaNa_2ZrMnO_6$ – a = 16,85 ± 0,02Å; V^0 = 4785,46 ± 0,07 Å^3; Z = 6; $V°_{эл.ұяш.}$ = 795,58 ± 0,011 Å^3; $\rho_{рент.}$ = 5,35 г/см^3; $\rho_{пикн.}$ = 5,30 ± 0,04 г/см^3; LaK_2ZrMnO_6 – a = 17,45 ± 0,03 Å; V^0 = 5318,85 ±0,09 Å^3; Z = 6; $V°_{эл.ұяш.}$ = 885,81 ± 0,01 Å^3; $\rho_{рент.}$ = 5,16 г/см^3; $\rho_{пикн.}$ = 5,08 ± 0,02 г/см^3. Li→Na→K қатарындағы иондық радиустардың ұлғаюымен «а» параметрінің мәндері және циркон-манганиттердің торлары мен бірлік ұяшықтарының көлемдері өсетіні анықталды.

Түйін сөздер: лантан; циркон-манганит; литий; натрий; калий; синтез; рентгенография.

Новые цирконо-манганиты лантана и щелочных металлов

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Методом керамической технологии в интервале 800-1200°C взаимодействием оксидов лантана (III) и карбонатов лития, натрия и калия (ч.д.а.) синтезированы цирконо-манганиты состава $LaMe^{I}_2ZrMnO_6$ (Me^{I} – Li, Na, K). Методами рентгенографии установлено, что все синтезированные цирконо-манганиты кристаллизуются в кубической сингонии со следующими параметрами решетки: $LaLi_2ZrMnO_6$ – a = 16,26 ±0,02 Å; V^0 = 4300,93 ±0,06 Å^3; Z = 6; $V°_{эл.яч}$ = 716,82 ± 0,01 Å^3; $\rho_{рент.}$ = 5,49 г/см^3; $\rho_{пикн.}$ = 5,42 ± 0,06 г/см^3; $LaNa_2ZrMnO_6$ – a = 16,85 ± 0,02Å; V^0 = 4785,46 ± 0,07 Å^3; Z = 6; $V°_{эл.яч}$ = 795,58 ± 0,011 Å^3; $\rho_{рент.}$ = 5,35 г/см^3; $\rho_{пикн.}$ = 5,30 ± 0,04 г/см^3; LaK_2ZrMnO_6 – a = 17,45 ± 0,03 Å; V^0 = 5318,85 ±0,09 Å^3; Z = 6; $V°_{эл.яч}$ = 885,81 ± 0,01 Å^3; $\rho_{рент.}$ = 5,16 г/см^3; $\rho_{пикн.}$ = 5,08 ± 0,02 г/см^3. Установлено, что с повышением ионных радиусов в ряду Li→Na→K увеличиваются величины параметра «а» и объемов решеток и элементарных ячеек цирконо-манганитов.

Ключевые слова: лантан; цирконо-манганит; литий; натрий; калий; синтез; рентгенография.

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1. Introduction

Interest in the study of manganite perovskite materials with the effects of gigantic and colossal magnetic resistance (Nobel Prize, 2007) contributed to the emergence of miniature media that are used in many advanced electronic devices. These phenomena stimulated the search for new compounds exhibiting similar effects due to the wide possibilities of their application. Due to such properties as high mechanical and optical characteristics, low thermal conductivity, high ionic conductivity, chemical and biological resistance of the zirconium dioxide-based material is widely used in engineering and medicine. These properties determine the possibilities of practical use in micro- and optoelectronics in the form of dielectric substrates and thin films, and make their application in the form of solid electrolytes in the form of films and thin membranes in various electrochemical devices for oxygen sensors and solid fuel elements extremely promising [1-4].

It should also be emphasized that among the manganites with the perovskite structure, compositions have already been found in which the effect of colossal magnetic resistance reaches 10^4% or more [5]. In particular, the possibility of using manganites in a new developing branch of electronics-spintronics, where the spin of an electron is an information carrier is considered [6,7].

Conditions for the synthesis of polycrystalline layered manganites $Ln_2BaMn_2O_{7-\delta}$ (Ln = Pr, Nd) of orthorhombic structure (spatial group Fmmm) with a certain oxygen nonstoichiometry are proposed for the first time: temperature, oxygen partial pressure [8].

$La_{1-x}K_xMnO_3$ were obtained at low temperature, where x= 0.0, 0.1, 0.15 [9]. The compounds were studied by X-ray phase analysis, electron paramagnetic and ferromagnetic resonance.

The authors [10] investigated the crystal structure and phonon spectrum of the $La_2Zr_2O_7$ crystal.

Nanocrystalline zirconium dioxide (ZrO_2) doped with La_2O_3 was obtained in [11] by chemical co-deposition for various concentrations of the alloying impurity.

The structural phases were characterized by X-ray diffraction. It was found that all newly synthesized samples are in the monoclinic phase.

Nonstoichiometric composites $Nd_{2-x}Zr_{2+x}O_{7+x/2}$ (x=0, 0.1, 0.2) were synthesized by chemical co-deposition and calcination [12]. The evolution of the phase structure and the thermophysical properties of $Nd_{2-x}Zr_{2+x}O_{7+x/2}$ are investigated.

In the above works, the production of both individual manganites and individual zircons doped with alkaline and alkaline earth metals is considered. The purpose of this work is to combine manganites and zirconates into single new compounds in the form of zircono-manganites of lanthanum and alkali metals with valuable physico-chemical properties.

In the light of the above, the task in this work was to obtain new zircono-manganites of the composition $LaMe^{I}_2ZrMnO_6$ (Me^I – Li, Na, K) and their identification by X-ray phase analysis methods.

2. Experiment

Solid-phase synthesis of $LaMe_2^{I}ZrMnO_6$ (Me^I – Li, Na, K) compounds was carried out using ceramic technology from lanthanum (III) oxides of the “extra clean” qualification, zirconium (IV), manganese (III) and lithium, sodium and potassium carbonates of the “clean for analysis” brand.

The stoichiometric amounts of the starting substances, previously dehydrated at 400°C, were thoroughly mixed and ground in an agate mortar. Then they were annealed in alund

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crucibles in the “SNOL” furnace at first at 600°C for 10 h, 800°C for 10 h, 1000°C with and 1200°C for 20 h. At each temperature, the mixtures were cooled to room temperature with repeated mixing and grinding processes and reheated. To obtain equilibrium phases at low temperatures, low-temperature annealing was performed at 400°C for 10 h, followed by repetitions of mixing and grinding. In order to eliminate the probability of the formation of nonequilibrium, metastable phases at high temperatures, low-temperature annealing was carried out at 400°C for 10 h to obtain stable phases at low temperatures.

The formation of the equilibrium composition of the compounds was controlled by X–ray phase analysis on the DRON - 2.0 diffractometer (NPP Burevestnik, Russia) using CuK_{α} radiation filtered by a Ni filter (U = 30 kV, J = 10 mA, pulse counter scale 1000 imp/s, counter rotation speed 2 degrees/min, time constant = 5 sec, angle interval 2 from 10 to 90°). The intensity of the diffraction maxima was estimated on a one-hundred-point scale. Figure 1 shows the X-ray images of the obtained zircono-manganites.

The indexing of radiographs was carried out by the analytical method [13].

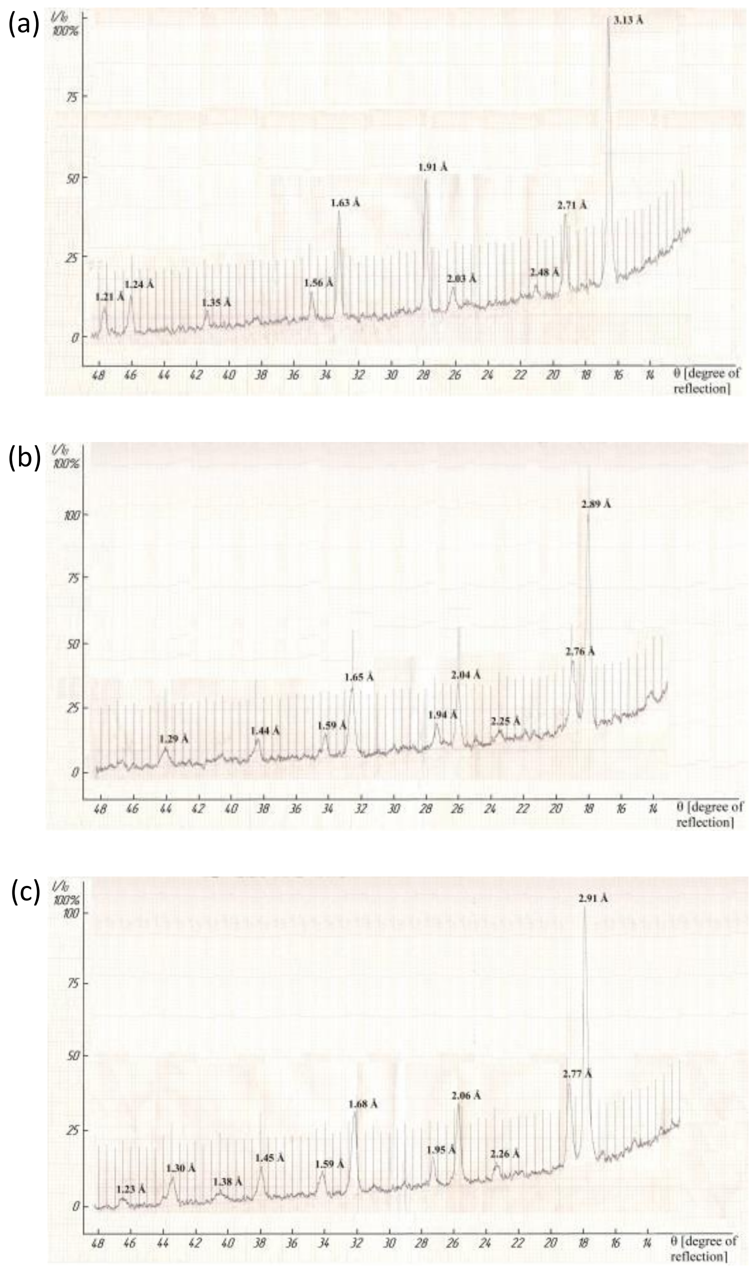

Figure 1 – X-rays $LaLi_2ZrMnO_6$ (a), $LaNa_2ZrMnO_6$, LaK_2ZrMnO_6 (c)

3. Results and Discussion

Based on the indexing of radiographs, it was found that all synthesized zircon-manganites crystallize in cubic symmetry. The main parameters of the X-ray density gratings are determined (Table 1).

The X-ray density (x-ray.) of the investigated zircono-manganites was determined by the formula [13]:

$$\rho_{x-ray} = \frac{1{,}66 \cdot Mr \cdot Z}{V^0}$$

where:
Mr – is the molecular weight of the compound,
Z – is the number of formula units in the lattice,
V° – is the volume of the unit cell.

Pycnometric densities were determined according to the method [14] in glass pycnometers with a volume of 1 mL. Toluene served as an indifferent liquid, which wets the material under study well, is chemically inert to it, and its density is stable to temperature changes.

Table 1 – Indexing of radiographs of $LaMe^{I}_2ZrMnO_6$ (Me^I – Li, Na, K) annealed at 400°C

I/I_0	d, Å	$10^4/d^2_{exp.}$	hkl	$10^4/d^2_{cal.}$
		$LaLi_2ZrMnO_6$		
100	3.13	1021	333	1021
30	2.71	1362	600	1361
6	2.48	1626	533	1626
9	2.03	2427	800	2420
50	1.91	2741	660	2723
41	1.63	3764	10.00	3781
12	1.56	4109	10.3.0	4122
7	1.35	5487	12.1.0	5483
15	1.24	6504	10.6.6	6504
12	1.21	6830	10.9.0	6845
		$LaNa_2ZrMnO_6$		
100	2.89	1197	433	1197
33	2.76	1313	610	1303
6	2.25	1975	642	1972
29	2.04	2403	820	2394
12	1.94	2657	662	2676
31	1.65	3673	10.2.0	3661
11	1.59	3956	870	3978
11	1.44	4823	10.6.1	4823
8	1.29	6009	13.1.1	6020
		LaK_2ZrMnO_6		
100	2.91	1181	442	1181
33	2.77	1303	620	1312
7	2.26	1958	553	1936
31	2.06	2356	660	2362
13	1.95	2576	752	2559
32	1.68	3543	10.2.2	3543
9	1.59	3956	11.0.0	3969
13	1.45	4756	12.1.0	4757
5	1.38	5251	12.4.0	5249
11	1.30	5917	12.6.0	5905
3	1.23	6610	10.10.1	6594

Table 2 – Lattice parameters of zircono-manganites $LaLi_2ZrMnO_6$ (I), $LaNa_2ZrMnO_6$ (II), LaK_2ZrMnO_6 (III)

Zircono-manganite	*a*, Å	V^0, Å³	Z	$V°_{elem\ cell.}$, Å³	(ρ),g/cm³	
					$\rho_{x-ray.}$	$\rho_{picn.}$
I	16,26 ±0,02	4300,93 ±0,06	6	716,82 ± 0,02	5,49	5,42 ± 0,06
II	16,85 ± 0,02	4785,46 ± 0,07	6	795,58 ± 0,02	5,35	5,30 ± 0,04
III	17,45 ± 0,03	5318,85 ±0,09	6	885,81 ± 0,02	5,16	5,08 ± 0,02

Table 2 shows the parameters of the elementary cells, X-ray and pycnometric densities of the obtained new zircono-manganites.

The reliability, correctness and reliability of the results of indexing and determination of lattice parameters are confirmed by a satisfactory agreement of experimental and calculated values of $10^4/d^2$, X-ray and pycnometric densities. Based on the conducted studies, the obtained zirconate-manganites can be attributed to the spatial group of perovskite Pm3m.

It can be assumed that, by analogy with other double manganites of rare earth and alkali metals [15], the La^{3+} ion is located in the center of the unit cell and has a coordination number for oxygen of 12, and in the nodes of the unit cells there is an Mn^{3+} ion with an oxygen coordination number equal to 6. Considering also the fact that Zr is in the same group with Ti in the periodic table and, by analogy with $LaLi_2TiMnO_6$ $LaNa_2TiMnO_6$ [16], it should be assumed that $LaLi_2ZrMnO_6$, $LaNa_2ZrMnO_6$ and LaK_2ZrMnO_6 can be attributed to a cubic perovskite structure with a tolerance factor t>0.89 [17]. It was revealed that with an increase in ionic radii in the range from Li to K, the values of parameter "a" and the volumes of lattices and elementary cells of synthesized zirconate-manganites increase.

4. Conclusions

For the first time, new zirconate-manganites of the composition $LaMe_2^IZrMnO_6$ (Me^I – Li, Na, K) were obtained by high-temperature synthesis. The types of their symmetry and lattice parameters were established.

It was revealed that the lattice parameters of zirconate-manganites change symbatically with an increase in ionic radii in the Li→Na→K series.

The results obtained are of interest for the directed synthesis of similar compounds in inorganic materials science and chemical informatics as new data on the radiographic characteristics of previously unexplored compounds.

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