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Novel Titanium-Manganites of Lanthanum and Alkali Metals

The titanium-manganites of $\text{LaMe}_2\text{TiMnO}_6$ (Me^I — Li, Na, K) were synthesized by the ceramic technology with the high-temperature reaction of oxides of La_2O_3 , TiO_2 , Mn_2O_3 with carbonates of Li_2CO_3 , Na_2CO_3 , K_2CO_3 within 800–1200 °C. The X-ray diffraction methods demonstrated that all of them were crystallized in the cubic syngony with the lattice parameters such as $\text{LaLi}_2\text{TiMnO}_6$ — $a = 13.48 \pm 0.02$ Å, $V^0 = 2449.46 \pm 0.06$ Å³, $Z = 4$, $V_{\text{el.cell}}^0 = 612.87 \pm 0.02$ Å³, $\rho_{\text{roent.}} = 3.81$; $\rho_{\text{pick.}} = 3.78 \pm 0.03$ g/cm³; $\text{LaNa}_2\text{TiMnO}_6$ — $a = 14.06 \pm 0.02$ Å, $V^0 = 2779.43 \pm 0.06$ Å³, $Z = 4$, $V_{\text{el.cell}}^0 = 694.96 \pm 0.02$ Å³, $\rho_{\text{roent.}} = 3.67$; $\rho_{\text{pick.}} = 3.65 \pm 0.01$ g/cm³; $\text{LaK}_2\text{TiMnO}_6$ — $a = 14.74 \pm 0.02$ Å, $V^0 = 3202.52 \pm 0.06$ Å³, $Z = 4$, $V_{\text{el.cell}}^0 = 800.52 \pm 0.02$ Å³, $\rho_{\text{roent.}} = 3.45$; $\rho_{\text{pick.}} = 3.43 \pm 0.01$ g/cm³. Correctness and authenticity of the results on the indexing of X-ray photographs of titanium-manganite were confirmed with the good experimental and calculated values ($10^4/d^2$), the pycnometric and X-ray densities, and also the theoretical and experimental values of cell volumes. The rising of values of the lattice parameters of the synthesized titanium-manganites was determined with increasing in the ionic radii from Li to K.

Key words: titanium-manganite, lanthanum, alkali metals, synthesis, X-ray diffraction, indexing, syngony, unit cell, lattice parameters.

Introduction

To date the sustained interest in the ferroelectric materials was observed. This is caused by the ability to develop electrically controlled ultrahigh-frequency (UHF) devices on their basis. The ferroelectric materials have anomalously high nonlinearity of the dielectric properties. As a result they are very attractive to use in the ultrahigh-frequency electronics [1]. Manganites of a fixed composition are materials with the high spin polarization to research some fundamental issues and to use in the spintronic devices and in the magnetic tunnel structures. The interest in using of manganites is caused by some unique properties such as the high spin polarization reaching 100 %, high Curie temperature, chemical stability, etc. [2]. Attention is also drawn to the fact that the semiconductor titanium oxides with transition metal impurities are able to be as the advancing materials to use in the spin electronics and catalysis [3, 4]. For instance, barium titanate is a traditional electroceramic material and it has the ferro-, ferroelectric — and pyroelectric properties. It ought to be noted that the high values of dielectric permittivity in the ferroelectric materials near a transition temperature permit to use them in the miniature capacitors [5].

It should be pointed out that a field of the potential applications of materials with the colossal magnetic resistance such as $\text{La}_{1-x}(\text{Ca}, \text{Ba})_x\text{MnO}_3$ manganites include magnetic field sensors, the reading heads for high-density magnetic recording, displacement sensors, temperature sensors, bolometers, etc. [6]. Data on the fuel cells based on perovskite-like solid solutions of $(\text{La}_{0.5+x}\text{Sr}_{0.5-x})_{1-y}\text{Mn}_{0.5}\text{Ti}_{0.5}\text{O}_{3-\delta}$ ($x = 0-0.25$, $y = 0-0.03$) are described in [7, 8].

The abovementioned literature review demonstrates that compounds based on manganites and titanates of the rare-earth elements doped with oxides of s-elements are of the great scientific and practical interest. As a result, the purpose of this paper is to synthesize and study the X-ray diffraction characteristics of the novel titanium-manganites of lanthanum and alkali metals.

Experimental

Oxides of lanthanum (III) (“puriss. spec.” 99.99 %), titanium (IV) and manganese (III), and carbonates of lithium, sodium and potassium (production Russia) (“p.a.” 99.0 %) were the initial reagents to synthesize

the titanium-manganites of $\text{LaMe}^{\text{I}}_2\text{TiMnO}_6$ (Me^{I} — Li, Na, K). These substances were pre-annealed at 300 °C to remove the adsorption moisture. Then, the stoichiometric amounts of these substances were thoroughly mixed and ground in an agate mortar to obtain the titanium-manganites of $\text{LaMe}^{\text{I}}_2\text{TiMnO}_6$ (Me^{I} — Li, Na, K). Further, the mixtures were placed to the alundum crucibles pre-calcined at 600 °C. Then they were annealed at 600 °C in a SNOL furnace for 5 h. Mixtures were cooled to a room temperature with the repeated processes of mixing and grinding. Then the mixtures were heat-treated at 800 °C for 5 h with processes of cooling to a room temperature, grinding and stirring. Further the analogous procedures were performed at 1000 °C for 10 h twice and at 1200 °C for 4 h. After repeating the mixing and grinding, a low-temperature annealing at 400 °C for 10 h was performed to obtain the equilibrium phases at a low temperature.

Formation of the equilibrium compositions of titanium-manganites was identified with the X-ray phase analysis on DRON-2.0 diffractometer (production Russia). The recording conditions: $\text{CuK}\alpha$ -radiation, $U = 30$ kV, $J = 10$ mA, rotation speed — 1000 pps, time constant $\tau = 5$ sec and angles 2θ from 10 to 90°. Intensity of diffraction peaks was graded on a 100-point scale.

Results and Discussion

The indexing of X-ray photographs of titanium-manganites demonstrated that all synthesized titanium-manganites were crystallized in the cubic syngony.

The lattice parameters and X-ray densities were determined. The indexing of X-ray photographs was performed with the analytical method [9]. Table 1 below demonstrates the indexing results.

Table 1

The indexing of X-ray photographs of $\text{LaMe}^{\text{I}}_2\text{TiMnO}_6$ (Me^{I} — Li, Na, K) powders

I/I_0	$d/\text{Å}$	$10^4/d^2_{\text{exp.}}$	hkl	$10^4/d^2_{\text{calc.}}$
LaLi₂TiMnO₆				
6	3.89	660.8	410	661.0
100	2.75	1322	433	1322
12	2.50	1600	621	1597
23	2.24	1993	7.1.1	1982
11	2.06	2356	6.5.0	2368
44	1.94	2657	8.2.0	2643
40	1.57	4057	10.2.0	4075
7	1.41	5030	10.5.2	5011
18	1.37	5328	10.6.1	5341
14	1.22	6719	13.2.0	6718
LaNa₂TiMnO₆				
10	3.90	657.5	320	658.0
100	2.76	1313	510	1315
11	2.40	1736	530	1720
21	2.23	2011	620	2013
38	1.93	2685	720	2685
33	1.59	3956	752	3945
13	1.37	5328	10.2.1	5311
10	1.23	6610	11.3.1	6625
LaK₂TiMnO₆				
7	3.94	644.2	321	644.0
100	2.82	1257	333	1242
20	2.25	1975	533	1978
30	1.97	2577	642	2577
5	1.74	3305	660	3313
27	1.59	3956	921	3957
12	1.38	5251	871	5246
5	1.23	6610	12.0.0	6626

It should be stated that the theoretical cell volumes of the synthesized titanium-manganites were determined with using the actual literature data on the cell volumes of the oxides included in their composition [10, 11] under the scheme:

$$V_{\text{el.cell}}^0 \text{LaMe}^{\text{I}}_2\text{TiMnO}_6 = 0.5 V_{\text{el.cell}}^0 \text{La}_2\text{O}_3 + V_{\text{el.cell}}^0 \text{Me}_2\text{O} + V_{\text{el.cell}}^0 \text{TiO}_2 + 0.5 V_{\text{el.cell}}^0 \text{Mn}_2\text{O}_3, \quad (1)$$

where Me^{I} — are Li, Na, K.

The X-ray density of compounds ($\rho_{\text{roent.}}$) was calculated with the formula [1]:

$$\rho_{\text{roent}} = \frac{1,66 \cdot Mr \cdot Z}{V^0}, \quad (2)$$

where Mr is the molecular weight of a compound; Z is the number of formula units in the lattice; V^0 is an elementary cell volume.

Four to five parallel measurements of manganite density were performed in 1-mL glass pycnometers under the procedure [12]. Toluene was used as an indifferent liquid i.e. it wetted these compounds well. It was chemically inactive towards them and its density was stable to the temperature changes.

Table 2 below demonstrates the cell parameters, X-ray and pycnometric densities of the synthesized titanium-manganites.

Table 2

**Lattice parameters of titanium-manganites of $\text{LaLi}_2\text{TiMnO}_6$ (I),
 $\text{LaNa}_2\text{TiMnO}_6$ (II) and $\text{LaK}_2\text{TiMnO}_6$ (III)**

Titanium-manganite	$a, \text{Å}$	$V^{\circ}, \text{Å}^3$	Z	$V_{\text{el.cell}}^{\circ}, \text{Å}^3$	$\rho, \text{g/cm}^3$	
					$\rho_{\text{roent.}}$	$\rho_{\text{pick.}}$
I	13.48 ± 0.02	2449.46 ± 0.06	4	612.37 ± 0.02	3.81	3.78 ± 0.03
II	14.06 ± 0.02	2779.43 ± 0.06	4	694.96 ± 0.02	3.67	3.65 ± 0.01
III	14.74 ± 0.02	3202.52 ± 0.06	4	800.63 ± 0.02	3.45	3.43 ± 0.01

Correctness and authenticity of the results on the indexing and determination of the lattice parameters for titanium-manganites were confirmed with the good experimental and calculated values ($10^4/d^2$), the X-ray and pycnometric densities, and also the theoretical and experimental data on cell volumes of $\text{LaLi}_2\text{TiMnO}_6$ ($V_{\text{theor.}}^{\circ} = 622.10 \text{ Å}^3$, $V_{\text{exp.}}^{\circ} = 612.36 \text{ Å}^3$), $\text{LaNa}_2\text{TiMnO}_6$ ($V_{\text{theor.}}^{\circ} = 693.71 \text{ Å}^3$, $V_{\text{exp.}}^{\circ} = 694.86 \text{ Å}^3$) and $\text{LaK}_2\text{TiMnO}_6$ ($V_{\text{theor.}}^{\circ} = 790.97 \text{ Å}^3$, $V_{\text{exp.}}^{\circ} = 800.63 \text{ Å}^3$). All experimental data was processed with methods of the mathematical statistics.

Based on the described above studies, the obtained titanium-manganites can be attributed to the perovskite $Pm\bar{3}m$ space group.

The pattern was observed in change of the lattice parameters of the synthesized titanium-manganites in transition from Li to K.

The symbate changes i.e. the rising in values of parameters “a”, the lattice volumes and the elementary cell volumes of titanium-manganites were observed with increasing in the ionic radii from Li to K.

Conclusions

Titanium-manganites of lanthanum and alkali metals of $\text{LaMe}^{\text{I}}_2\text{TiMnO}_6$ (Me^{I} — Li, Na, K) were first obtained with the ceramic technology, and their syngony types and lattice parameters were determined. It was found that the lattice parameters of the synthesized titanium-manganites were able to change symbatically with increasing in the ionic radii of alkali metals. The results are a basis to perform the thermodynamic and electrophysical investigations, to determine the advancing physical-chemical and physical properties of the obtained novel titanium-manganites of lanthanum and alkali metals.

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Лантан және сілтілі металдардың жаңа титан-манганиттері

Керамикалық технология әдісімен La₂O₃, TiO₂, Mn₂O₃ тотықтарының Li₂CO₃, Na₂CO₃, K₂CO₃ карбонаттарымен 800–1200 °C аралықта жоғары температуралы әрекеттесуімен LaMe¹₂TiMnO₆ (Me¹ — Li, Na, K), құрамды титан-манганиттері синтезделді. Рентгенография әдістері олардың барлығы келесідей тор параметрлерімен кубтық сингонияда кристалданатындығын анықтады: LaLi₂TiMnO₆ — $a = 13,48 \pm 0,02 \text{ \AA}$, $V^0 = 2449,46 \pm 0,06 \text{ \AA}^3$, $Z = 4$, $V^0_{\text{эл.ұяш.}} = 612,87 \pm 0,02 \text{ \AA}^3$, $\rho_{\text{рент.}} = 3,81$; $\rho_{\text{пикн.}} = 3,78 \pm 0,03 \text{ г/см}^3$; LaNa₂TiMnO₆ — $a = 14,06 \pm 0,02 \text{ \AA}$, $V^0 = 2779,43 \pm 0,06 \text{ \AA}^3$, $Z = 4$, $V^0_{\text{эл.ұяш.}} = 694,96 \pm 0,02 \text{ \AA}^3$, $\rho_{\text{рент.}} = 3,67$; $\rho_{\text{пикн.}} = 3,65 \pm 0,01 \text{ г/см}^3$; LaK₂TiMnO₆ — $a = 14,74 \pm 0,02 \text{ \AA}$, $V^0 = 3202,52 \pm 0,06 \text{ \AA}^3$, $Z = 4$, $V^0_{\text{эл.ұяш.}} = 800,52 \pm 0,02 \text{ \AA}^3$, $\rho_{\text{рент.}} = 3,45$; $\rho_{\text{пикн.}} = 3,43 \pm 0,01 \text{ г/см}^3$. Титан-манганиттердің рентгенограммаларын индицирлеудің нәтижелерінің дәлділігі мен анықтылығы $10^4/d^2$ тәжірибелік пен теориялық мәндерінің, пикнометрлік және рентгендік тығыздықтарының және элементарлы ұяшықтардың теориялық және тәжірибелік көрсеткіштерінің жақсы үйлесімділігімен дәлелденді. Иондық радиустың Li-ден K-ге жоғарылауымен синтезделген титан-манганиттер тор параметрлері шамаларының артатындығы анықталды.

Кілт сөздер: титан-манганит, лантан, сілтілі металдар, синтез, рентгенография, индицирлеу, сингония, элементарлы ұяшық, тор көрсеткіштері.

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Новые титано-манганиты лантана и щелочных металлов

Методом керамической технологии с высокотемпературным взаимодействием оксидов La₂O₃, TiO₂, Mn₂O₃ с карбонатами Li₂CO₃, Na₂CO₃, K₂CO₃ в интервале 800–1200 °C синтезированы титано-манганиты состава LaMe¹₂TiMnO₆ (Me¹ — Li, Na, K). Методами рентгенографии установлено, что все

они кристаллизуются в кубической сингонии со следующими параметрами решетки: $\text{LaLi}_2\text{TiMnO}_6$ — $a = 13,48 \pm 0,02 \text{ \AA}$, $V^0 = 2449,46 \pm 0,06 \text{ \AA}^3$, $Z = 4$, $V^0_{\text{эл.яч.}} = 612,87 \pm 0,02 \text{ \AA}^3$, $\rho_{\text{рентг.}} = 3,81$; $\rho_{\text{пикн.}} = 3,78 \pm 0,03 \text{ г/см}^3$; $\text{LaNa}_2\text{TiMnO}_6$ — $a = 14,06 \pm 0,02 \text{ \AA}$, $V^0 = 2779,43 \pm 0,06 \text{ \AA}^3$, $Z = 4$, $V^0_{\text{эл.яч.}} = 694,96 \pm 0,02 \text{ \AA}^3$, $\rho_{\text{рентг.}} = 3,67$; $\rho_{\text{пикн.}} = 3,65 \pm 0,01 \text{ г/см}^3$; $\text{LaK}_2\text{TiMnO}_6$ — $a = 14,74 \pm 0,02 \text{ \AA}$, $V^0 = 3202,52 \pm 0,06 \text{ \AA}^3$, $Z = 4$, $V^0_{\text{эл.яч.}} = 800,52 \pm 0,02 \text{ \AA}^3$, $\rho_{\text{рентг.}} = 3,45$; $\rho_{\text{пикн.}} = 3,43 \pm 0,01 \text{ г/см}^3$. Корректность и достоверность результатов индирования рентгенограмм титано-манганитов подтверждались с хорошим согласием опытных и расчетных значений $10^4/d^2$, пикнометрических и рентгеновских плотностей, а также теоретических и опытных значений объемов элементарных ячеек. Выявлено, что повышением ионных радиусов от Li к K увеличиваются величины параметров решеток синтезированных титано-манганитов.

Ключевые слова: титано-манганит, лантан, щелочные металлы, синтез, рентгенография, индирование, сингония, элементарная ячейка, параметры решеток.

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