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Novel Titanium-Manganites of Lanthanum and Alkali Metals

The titanium-manganites of $LaMe^I_2TiMnO_6$ (Me^I — Li, Na, K) have been 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 ºC. The X-ray diffraction methods demonstrated that all of them have been crystallized in the cubic syngony with the lattice parameters such as $LaLi_2TiMnO_6$ — $a = 13.48 \pm 0.02$ Å, $V^o = 2449.46 \pm 0.06$ Å^3, Z = 4, $V^o_{el.cell} = 612.87 \pm 0.02$ Å^3, $\rho_{roent.} = 3.81$; $\rho_{pick.} = 3.78 \pm 0.03$ g/cm^3; $LaNa_2TiMnO_6$ — $a = 14.06 \pm 0.02$ Å, $V^o = 2779.43 \pm 0{,}06$ Å^3, Z = 4, $V^o_{el.cell} = 694.96 \pm 0.02$ Å^3, $\rho_{roent.} = 3.67$; $\rho_{pick.} = 3.65 \pm 0.01$ g/cm^3; LaK_2TiMnO_6 — $a = 14.74 \pm 0.02$ Å, $V^o = 3202.52 \pm 0.06$ Å^3, Z = 4, $V^o_{el.cell} = 800.52 \pm 0.02$ Å^3, $\rho_{roent.} = 3.45$; $\rho_{pick.} = 3.43 \pm 0.01$ g/cm^3. Correctness and authenticity of the results on the indexing of X-ray photographs of titanium-manganite have been 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 has been 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 has been 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 $La_{1-x}(Ca, Ba)_xMnO_3$ 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 $(La_{0.5+x}Sr_{0.5-x})_{1-y}Mn_{0.5}Ti_{0.5}O_{3-\delta}$ ($x = 0$–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 %) have been the initial reagents to synthesize the titanium-manganites of $LaMe^{I}_{2}TiMnO_6$ (Me^{I} — Li, Na, K). These substances have been pre-annealed at 300 °C to remove the adsorption moisture. Then, the stoichiometric amounts of these substances have been thoroughly mixed and ground in an agate mortar to obtain the titanium-manganites of $LaMe^{I}_{2}TiMnO_6$ (Me^{I} — Li, Na, K). Further, the mixtures have been placed to the alundum crucibles pre-calcined at 600 °C. Then they have been annealed at 600 °C in a SNOL furnace for 5 h. Mixtures have been cooled to a room temperature with the repeated processes of mixing and grinding. Then the mixtures have been heat-treated at 800 °C for 5 h with processes of cooling to a room temperature, grinding and stirring. Further the analogous procedures have been 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 has been performed to obtain the equilibrium phases at a low temperature.

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

Results and Discussion

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

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

Table 1

The indexing of X-ray photographs of $LaMe^{I}_{2}TiMnO_6$ (Me^{I} — Li, Na, K) powders

I/I_o	d/Å	$10^4/d^2_{exp.}$	*hkl*	$10^4/d^2_{calc.}$
$LaLi_2TiMnO_6$				
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
$LaNa_2TiMnO_6$				
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
LaK_2TiMnO_6				
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 have been determined with using the actual literature data on the cell volumes of the oxides included in their composition [10, 11] under the scheme:

$$V^0_{el.cell}\,LaMe^I_2TiMnO_6 = 0.5\,V^0_{el.cell}La_2O_3 + V^0_{el.cell}\,Me_2O + V^0_{el.cell}\,TiO_2 + 0.5\,V^0_{el.cell}\,Mn_2O_3, \quad (1)$$

where Me^I — are Li, Na, K.

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

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

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 have been performed in 1-mL glass pycnometers under the procedure [12]. Toluene has been used as an indifferent liquid i.e. it wetted these compounds well. It has been chemically inactive towards them and its density has been 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 $LaLi_2TiMnO_6$ (I), $LaNa_2TiMnO_6$ (II) and LaK_2TiMnO_6 (III)

Titanium-manganite	a, Å	$V°$, Å^3	Z	$V°_{el.cell}$, Å^3	ρ, g/cm^3	
					$\rho_{roent.}$	$\rho_{pick.}$
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 have been 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 $LaLi_2TiMnO_6$ ($V°_{theor.}$ = 622.10 Å^3, $V°_{exp.}$ = 612.36 Å^3), $LaNa_2TiMnO_6$ ($V°_{theor.}$ = 693.71 Å^3, $V°_{exp.}$ = 694.86 Å^3) and LaK_2TiMnO_6 ($V°_{theor.}$ = 790.97 Å^3, $V°_{exp.}$ = 800.63 Å^3). All experimental data have been processed with methods of the mathematical statistics.

Based on the described above studies, the obtained titanium-manganites can be attributed to the perovskite *Pm3m* space group.

The pattern has been 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 have been observed with increasing in the ionic radii from Li to K.

Conclusions

Titanium-manganites of lanthanum and alkali metals of $LaMe^I_2TiMnO_6$ (Me^I — Li, Na, K) have been first obtained with the ceramic technology, and their syngony types and lattice parameters have been determined. It has been found that the lattice parameters of the synthesized titanium-manganites are 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 La_2O_3, TiO_2, Mn_2O_3 тотықтарының Li_2CO_3, Na_2CO_3, K_2CO_3 карбонаттарымен 800–1200 ºС аралықта жоғары температуралы әрекеттесуімен $LaMe^I{}_2TiMnO_6$ (Me^I — Li, Na, K). құрамды титан-манганиттері синтезделді. Рентгенография әдістері олардың барлығы келесідей тор параметрлерімен кубтық сингонияда кристалданатындығы анықтады: $LaLi_2TiMnO_6$ — a = 13,48±0,02 Å, V^o = 2449,46±0,06 Å^3, Z = 4, $V^o_{эл.ұяш.}$ = 612,87±0,02 Å^3, $\rho_{рент.}$ = 3,81; $\rho_{пикн.}$ = 3,78± ± 0,03 г/см^3; $LaNa_2TiMnO_6$ — a = 14,06±0,02 Å, V^o = 2779,43±0,06 Å^3, Z = 4, $V^o_{эл.ұяш.}$ = 694,96±0,02 Å^3, $\rho_{рент.}$ = 3,67; $\rho_{пикн.}$ = 3,65±0,01 г/см^3; LaK_2TiMnO_6 — a = 14,74±0,02 Å, V^o = 3202,52± ± 0,06 Å^3, Z = 4, $V^o_{эл.ұяш.}$ = 800,52±0,02 Å^3, $\rho_{рент.}$ = 3,45; $\rho_{пикн.}$ = 3,43±0,01 г/см^3. Титано-манганиттердің рентгенограммаларын индицирлеудің нәтижелерінің дәлділігі мен сенімділігі $10^4/d^2$ 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.

Б.К. Касенов, Ш.Б. Касенова, Ж.И. Сагинтаева, С.О. Байсанов,
Н.Ю. Лу, Е.Е. Куанышбеков, М.О. Туртубаева, М.А. Исабаева

Новые титано-манганиты лантана и щелочных металлов

Методом керамической технологии с высокотемпературным взаимодействием оксидов La_2O_3, TiO_2, Mn_2O_3 с карбонатами Li_2CO_3, Na_2CO_3, K_2CO_3 в интервале 800–1200 ºС синтезированы титано-манганиты состава $LaMe^I{}_2TiMnO_6$ (Me^I — Li, Na, K). Методами рентгенографии установлено, что все они кристаллизуются в кубической сингонии со следующими параметрами решетки: $LaLi_2TiMnO_6$ — a = 13,48±0,02 Å, V^o = 2449,46±0,06 $Å^3$, Z = 4, $V^o_{эл.яч.}$ = 612,87±0,02 $Å^3$, $\rho_{рент.}$ = 3,81; $\rho_{пикн.}$ = 3,78±0,03 г/см^3; $LaNa_2TiMnO_6$ — a = 14,06±0,02 Å, V^o = 2779,43±0,06 $Å^3$, Z = 4, $V^o_{эл.яч.}$ = 694,96±0,02 $Å^3$, $\rho_{рент.}$ = 3,67; $\rho_{пикн.}$ = 3,65±0,01 г/см^3; LaK_2TiMnO_6 — a = 14,74±0,02 Å, V^o = 3202,52±0,06 $Å^3$, Z = 4, $V^o_{эл.яч.}$ = 800,52± ±0,02 $Å^3$, $\rho_{рент.}$ = 3,45; $\rho_{пикн.}$ = 3,43±0,01 г/см^3. Корректность и достоверность результатов индицирования 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, индицирование, сингония, элементарная ячейка, параметры решеток.

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