ISSN 0036-0244, Russian Journal of Physical Chemistry A, 2018, Vol. 92, No. 4, pp. 760–767. © Pleiades Publishing, Ltd., 2018.
Original Russian Text © B.K. Kasenov, Zh.I. Sagintaeva, Sh.B. Kasenova, K.T. Ermaganbetov, K.S. Kakenov, G.A. Esenbaeva, 2018, published in Zhurnal Fizicheskoi Khimii, 2018, Vol. 92, No. 4, pp. 637–644.

PHYSICAL CHEMISTRY OF NANOCLUSTERS AND NANOMATERIALS

Thermodynamic and Electrophysical Properties of Nanosized $LaMeFeCrMnO_{6.5}$ (Me = Li, Na, K) Ferro-Chromo-Manganites

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Received July 6, 2017

Abstract—Ferro-chromo-manganites with compositions $LaMeFeCrMnO_{6.5}$ (Me = Li, Na, K) are synthesized via ceramic technology from La_2O_3 (special purity grade), Fe_2O_3, Cr_2O_3, Mn_2O_3, and carbonates of alkali metals (analytical grade). The individuality of the resulting compounds is confirmed via X-ray phase analysis. The heat capacity of nanosized $LaMeFeCrMnO_{6.5}$ (Me = Li, Na, K) ferro-chromo-manganites is measured via dynamic calorimetry in the temperature interval of 298.15–673 K on an IT-S-400 apparatus. The values of $C_p^\circ(T)$ and thermodynamic functions $H^\circ(T) - H^\circ(298.15)$, $S^\circ(T)$, and $\Phi^{xx}(T)$ are calculated. The temperature dependences of the electrical capacity (C), dielectric permeability (ε) and electrical resistivity (R) studied in the temperature range of 293–483 K show that these materials are also of interest for microelectronics.

Keywords: lanthanum, alkali metals, ferro-chromo-manganite, dynamic calorimetry, thermodynamic functions, electrophysical characteristics

DOI: 10.1134/S0036024418040118

INTRODUCTION

The synthesis and study of the physicochemical properties of manganites, chromites, and ferrites of rare-earth elements doped with oxides of light (alkali, alkaline-earth) metals are of great theoretical and practical interest, due to their multifunctional properties [1–9]. It should be noted that manganese-based alloys are also of great importance in metallurgy as deoxidizers [10, 11].

In analogy with [12], the combination of manganites, chromites and ferrites in one compound in the form of ferro-chromo-manganites is of certain interest, especially in regard to preparing their nanoparticles and studying their physicochemical properties.

The aim of this work was therefore to study the thermodynamic and electrophysical properties of new nanosized (nanocluster) $LaMeFeCrMnO_{6.5}$ (Me = Li, Na, K) ferro-chromo-manganites.

EXPERIMENTAL

Our compounds were synthesized using ceramic technology from stoichiometric amounts of La_2O_3 (special purity grade), Fe_2O_3, Cr_2O_3, Mn_2O_3, and carbonates of alkali metals (analytical grade), calculated for the final formula $LaMeFeCrMnO_{6.5}$ (Me = Li, Na, K).

Nanosized particles of synthesized ferro-chromo-manganites were prepared by grinding on an MM301 vibrating mill (Retsch, Germany).

The sizes of ground particles were determined on a JSPM-5400 scanning electron microscope (JEOL). Nanoparticles (nanoclusters) with sizes of 40–100 nm were prepared. The results from X-ray phase analysis show that the synthesized nanosized (nanocluster) particles of lanthanum–alkali metal ferro-chromo-manganites crystallized in cubic syngony with the following lattice parameters: $LaLiFeCrMnO_{6.5}$, a = 20.181 ± 0.03 Å, V^o = 8219 ± 0.09 Å^3, Z = 8, $V^o_{\text{unit cell}}$ = 1027.40 ± 0.01 Å^3, $\rho_{\text{X-ray}}$ = 5.05; ρ_{peak} = 5.10 ± 0.06 g/cm^3; $LaNaFeCrMnO_{6.5}$, a = 20.168 ± 0.03 Å, V^o = 8203 ± 0.08 Å^3, Z = 8, $V^o_{\text{unit cell}}$ = 1025.41 ± 0.01 Å^3, $\rho_{\text{X-ray}}$ = 5.06; ρ_{peak} = 5.05 ± 0.03 g/cm^3; $LaKFeCrMnO_{6.5}$, a = 20.273 ± 0.03 Å, V^o = 8332 ± 0.08 Å^3, Z = 8, $V^o_{\text{unit cell}}$ = 1041.41 ± 0.01 Å^3, $\rho_{\text{X-ray}}$ = 5.03 g/cm^3; ρ_{peak} = 5.04 ± 0.09 g/cm^3 [13].

Table 1. Experimental values of the heat capacity of $LaMeFeCrMnO_{6.5}$ (Me = Li, Na, K) [$C^{\circ}_{p(\text{spec})} \pm \bar{\delta}$, J/(g K); $C^{\circ}_{p(\text{m})} \pm \overset{0}{\Delta}$, J/(mol K)]

T, K	$C_p \pm \bar{\delta}$	$C^{\circ}_p \pm \overset{0}{\Delta}$	T, K	$C_p \pm \bar{\delta}$	$C^{\circ}_p \pm \overset{0}{\Delta}$
		$LaLiFeCrMnO_{6.5}$			
298.15	0.6023 ± 0.0154	248 ± 17	498	0.9013 ± 0.0133	372 ± 15
323	0.6562 ± 0.0155	271 ± 18	523	0.9299 ± 0.0243	384 ± 29
348	0.8369 ± 0.0162	345 ± 18	548	0.9910 ± 0.0144	409 ± 16
373	0.8858 ± 0.0164	365 ± 19	573	1.0067 ± 0.0131	415 ± 15
398	0.9325 ± 0.0136	385 ± 16	598	1.0645 ± 0.0319	439 ± 36
423	0.9857 ± 0.0259	407 ± 30	623	1.1394 ± 0.0146	470 ± 17
448	0.9502 ± 0.0155	392 ± 18	648	1.1637 ± 0.0168	480 ± 19
473	0.9209 ± 0.0126	380 ± 14	673	1.2621 ± 0.0242	521 ± 28
		$LaNaFeCrMnO_{6.5}$			
298.15	0.5915 ± 0.0186	253 ± 22	498	0.5915 ± 0.0186	253 ± 22
323	0.6838 ± 0.0197	293 ± 23	523	0.6838 ± 0.0197	293 ± 23
348	0.7486 ± 0.0140	321 ± 17	548	0.7486 ± 0.0140	321 ± 17
373	0.7811 ± 0.0188	335 ± 22	573	0.7811 ± 0.0188	335 ± 22
398	0.8398 ± 0.0219	360 ± 26	598	0.8398 ± 0.0219	360 ± 26
423	0.9892 ± 0.0148	424 ± 18	623	0.9892 ± 0.0148	424 ± 18
448	0.9679 ± 0.0171	415 ± 20	648	0.9679 ± 0.0171	415 ± 20
473	0.9554 ± 0.0157	409 ± 19	673	0.9554 ± 0.0157	409 ± 19
		$LaKFeCrMnO_{6.5}$			
298.15	0.5633 ± 0.0140	251 ± 17	498	0.7909 ± 0.0191	352 ± 24
323	0.7651 ± 0.0181	340 ± 22	523	0.8522 ± 0.0121	379 ± 15
348	0.8614 ± 0.0126	383 ± 16	548	0.7948 ± 0.0146	354 ± 18
373	0.8789 ± 0.0137	391 ± 17	573	0.7678 ± 0.0275	342 ± 34
398	0.7411 ± 0.0086	330 ± 11	598	0.7441 ± 0.0198	331 ± 24
423	0.6850 ± 0.0207	305 ± 26	623	0.8132 ± 0.0188	362 ± 23
448	0.6101 ± 0.0130	271 ± 16	648	0.8637 ± 0.0242	384 ± 30
473	0.7527 ± 0.0099	335 ± 12	673	0.9154 ± 0.0224	407 ± 28

EXPERIMENTAL

The specific heat capacities of $LaMeFeCrMnO_{6.5}$ (Me = Li, Na, K) were measured on an IT-S-400 calorimeter in the temperature interval of 298.15–673 K, and the molar heat capacities were then calculated according to [14].

Before performing each experiment, the instrument was calibrated via experimental determination of the thermal conductivity K_T of the heat meter. Five parallel experiments with a copper sample and five with an empty ampoule were performed. The operation of the device was confirmed by determining standard heat capacity α-Al_2O_3, the value of which

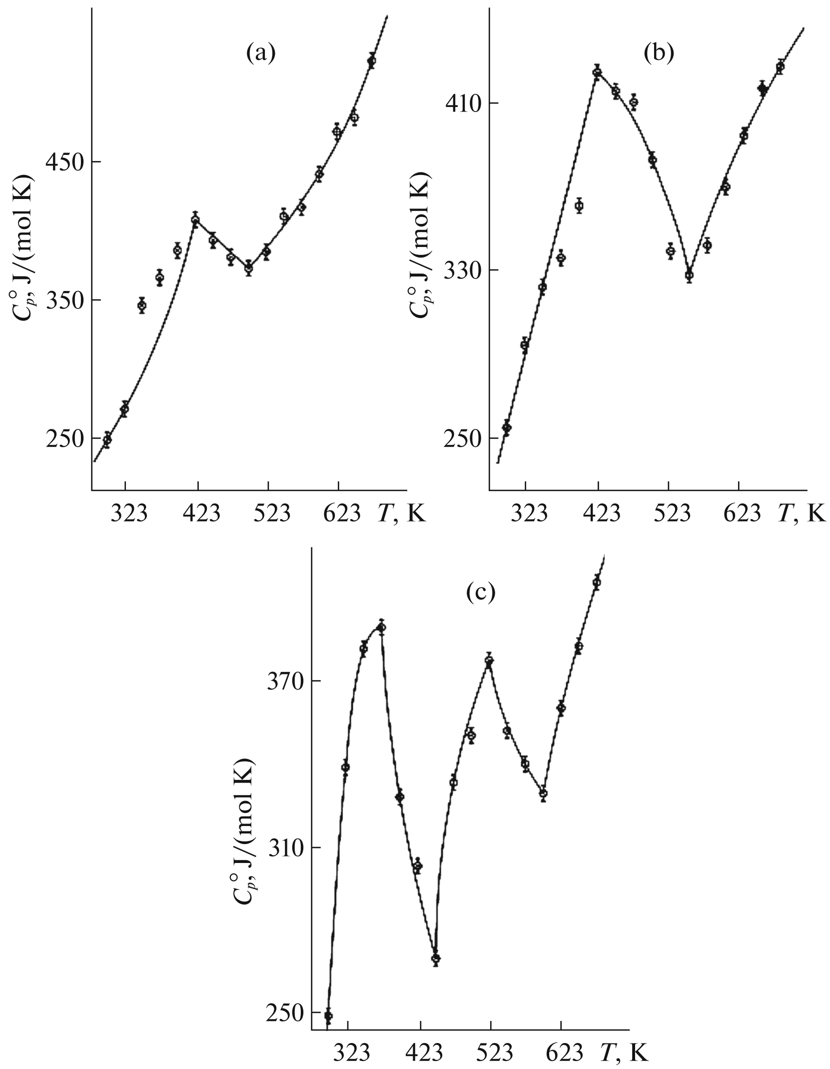

Fig. 1. Temperature dependences of the heat capacities of (a) $LaLiFeCrMnO_{6.5}$, (b) $LaNaFeCrMnO_{6.5}$, and (c) $LaKFeCrMnO_{6.5}$ in the interval 298.15–673 K.

[76.0 J/(mol K)] was in satisfactory agreement with its recommended value [79.0 J/(mol K)] [15]. Five parallel experiments were performed at each temperature with 25 K step. The results were averaged and processed by means of mathematical statistics [16] (Table 1). The rms deviation ($\bar{\delta}$) was determined for the average values of the specific heat capacities and random component errors ($\overset{0}{\Delta}$) for the molar heat capacities. It should be noted that the heat capacities of nanosized cuprate manganites, manganite ferrites, and polycrystalline cobalt manganites synthesized in our laboratory were studied in the same manner on the same calorimeter [17–19].

The results from our calorimetric studies are given in Table 1 and Fig. 1.

The electrophysical properties (dielectric permeability and electric resistivity) were studied by continuously measuring the electrical capacity of thermostatted samples in dry air on an LCR-800 unit (Taiwan) at a working frequency of 1 kHz with a time delay at each fixed temperature as in [20, 21].

A two-electrode system was used, and the electrodes were deposited via the burning of silver paste. The dielectric permittivity was determined from the electrical capacity of each sample at known values of its thickness and the area of the electrode's surface.

RESULTS AND DISCUSSION

We can see from the data in Table 1 and Fig. 1 that $LaLiFeCrMnO_{6.5}$, $LaNaFeCrMnO_{6.5}$ at 423 K and $LaKFeCrMnO_{6.5}$ at 398 K, 523 K undergo λ-like second-order phase transitions, due possibly to Schottky effects, the Curie and Neel points, changes in dielec-

Table 2. Equation of the temperature dependence of the heat capacity of ferro-chromo-manganites $C_p^\circ = a + bT + cT^{-2}$, J/(mol K)

a	$b \times 10^{-3}$	$-c \times 10^5$	ΔT, K
$LaLiFeCrMnO_{6.5}$			
−559.7 ± 29.4	2054.9 ± 108.1	−173.94 ± 9.15	298–423
603.1 ± 31.7	−464.2 ± 24.4	—	423–498
−897.2 ± 47.2	1806.2 ± 95.0	−916.68 ± 48.22	498–673
$LaNaFeCrMnO_{6.5}$			
−180.9 ± 11.7	1415.3 ± 91.9	−11.33 ± 0.73	298–423
1834.4 ± 119.1	−2255.7 ± 146.5	816.26 ± 53.00	423–548
778.8 ± 50.6	−168.6 ± 10.9	1080.85 ± 70.19	548–673
$LaKFeCrMnO_{6.5}$			
3403.5 ± 206.7	−5471.4 ± 332.3	1352.08 ± 82.11	298–373
−1803.0 ± 109.5	2922.0 ± 177.4	−1536.0 ± 93.3	373–448
4666.7 ± 283.4	−5469.1 ± 332.1	3904.0 ± 237.1	448–523
−711.4 ± 43.2	1052.2 ± 63.9	−1477.6 ± 89.7	523–598
1417.0 ± 86.0	−758.7 ± 46.1	2261.3 ± 137.3	598–673

tric permeability and electrical conductivity, and other features.

The equations for dependences $C_p^\circ \sim f(T)$ in Table 2 were calculated with allowance for the phase transition temperatures.

Since the technical limitations of the calorimeter did not allow us to calculate the standard entropies of the studied compounds from the experimental data on heat capacities, they were estimated from the ionic entropy increments [22] according to the scheme

$$S^\circ(298.15)\mathrm{LaMeFeCrMnO_{6.5}} = S^i(298.15)\mathrm{La^{3+}} + S^i(298.15)\mathrm{Li^+} + S^i(298.15)\mathrm{Fe^{3+}} + S^i(298.15)\mathrm{Cr^{3+}} + S^i(298.15)\mathrm{Mn^{3+}} + 6.5S^i(298.15)\mathrm{O^{2-}}, \quad (1)$$

where $S^i(298.15)$ is the ion entropy increment at 298.185 K, and $M^+ = Li^+, Na^+, K^+$.

The values of $S^i(298.15)$ Li^+, Na^+, K^+, La^{3+}, Fe^{3+}, Cr^{3+}, Mn^{3+}, O^{2-} needed for our calculations were taken from [22].

The temperature dependences of their thermodynamic functions in Table 3, were calculated from experimental data on heat capacities and calculated values of the standard entropies of ferro-chromo-manganites.

The standard enthalpies of formation ($\Delta_f H^\circ(298.15)$) of $LaLiFeCrMnO_{6.5}$, $LaNaFeCrMnO_{6.5}$, and $LaKFeCrMnO_{6.5}$, calculated by approximate means, were −3285.2, −3356.3, and −3438.9 kJ/mol, respectively [23].

We can see from the data in Fig. 2 and Table 4 that the studied compounds show variable types of conductivity in the interval of 293–483 K: semiconductor and metal. In addition, the compounds had high values of dielectric permeability (ε), demonstrating their promise as materials for microelectronics. It is known that the compounds with high ε values are of interest to microelectronics as capacitors [24].

$LaLiFeCrMnO_{6.5}$

This compound displays semiconductor conductivity in the interval 293–373 K, metal conductivity at 373–403 K, and semiconductor conductivity again at 403–483 K. Dielectric permittivity ε rises from 158 at 293 K to 531428 at 483 K. In this interval, ε grows by 3363 times, and electrical resistivity R is reduced by a factor of 90.

$LaNaFeCrMnO_{6.5}$

This compound displays semiconductor conductivity in the interval 293–403 K, metal conductivity at 403–463 K, and semiconductor conductivity again at 463–483 K. Dielectric permittivity ε rises from 3284 at 293 K to 4.95×10^6 at 403 K (i.e., by 1507 times). Electrical resistivity falls by a factor of 224 in the interval 293–403 K.

Table 3. Thermodynamic functions of ferro-chromo-manganites

T, K	$C_p^\circ(T) \pm \overset{0}{\Delta}$, J/(mol K)	$S^\circ(T) \pm \overset{0}{\Delta}$, J/(mol K)	$H^\circ(t) - H^\circ(298.15) \pm \overset{0}{\Delta}$, J/mol	$\Phi^{xx}(T) \pm \overset{0}{\Delta}$, J/(mol K)
		$LaLiFeCrMnO_{6.5}$		
298	248 ± 13	218 ± 6	–	218 ± 6
300	250 ± 13	220 ± 18	500 ± 30	218 ± 18
350	301 ± 16	262 ± 22	14190 ± 750	221 ± 18
400	371 ± 19	306 ± 25	30940 ± 1630	229 ± 19
450	394 ± 21	353 ± 29	50700 ± 2670	240 ± 20
500	373 ± 20	393 ± 32	69830 ± 3670	253 ± 21
550	399 ± 21	430 ± 35	89050 ± 4680	268 ± 22
600	441 ± 23	466 ± 38	110010 ± 5790	283 ± 23
650	494 ± 26	503 ± 41	133350 ± 7010	298 ± 25
675	523 ± 27	522 ± 43	146050 ± 7680	306 ± 25
		$LaNaFeCrMnO_{6.5}$		
298	253 ± 16	238 ± 7	–	238 ± 7
300	256 ± 17	240 ± 23	510 ± 30	238 ± 22
350	324 ± 21	284 ± 27	15000 ± 970	241 ± 23
400	392 ± 25	332 ± 31	32890 ± 2140	250 ± 24
450	416 ± 27	407 ± 39	64240 ± 4170	264 ± 25
500	380 ± 25	449 ± 43	84250 ± 5470	280 ± 26
550	324 ± 21	483 ± 46	101920 ± 6620	297 ± 28
600	377 ± 24	528 ± 50	127480 ± 8280	316 ± 30
650	413 ± 27	560 ± 53	147290 ± 9560	333 ± 32
675	428 ± 28	576 ± 55	157810 ± 10250	342 ± 32
		$LaKFeCrMnO_{6.5}$		
298	246 ± 15	251 ± 8	–	226 ± 8
300	260 ± 16	253 ± 23	510 ± 30	252 ± 23
350	385 ± 23	305 ± 28	17390 ± 1060	255 ± 23
400	326 ± 20	355 ± 32	35980 ± 2190	265 ± 24
450	270 ± 16	389 ± 35	50580 ± 3070	277 ± 25
500	371 ± 23	424 ± 39	67270 ± 4090	290 ± 26
550	356 ± 22	460 ± 42	85830 ± 5210	304 ± 28
575	341 ± 21	475 ± 43	94520 ± 5740	311 ± 28
600	330 ± 20	489 ± 44	102900 ± 6250	318 ± 29
650	389 ± 24	518 ± 47	121050 ± 7350	332 ± 30
675	409 ± 25	534 ± 48	131020 ± 7960	339 ± 31

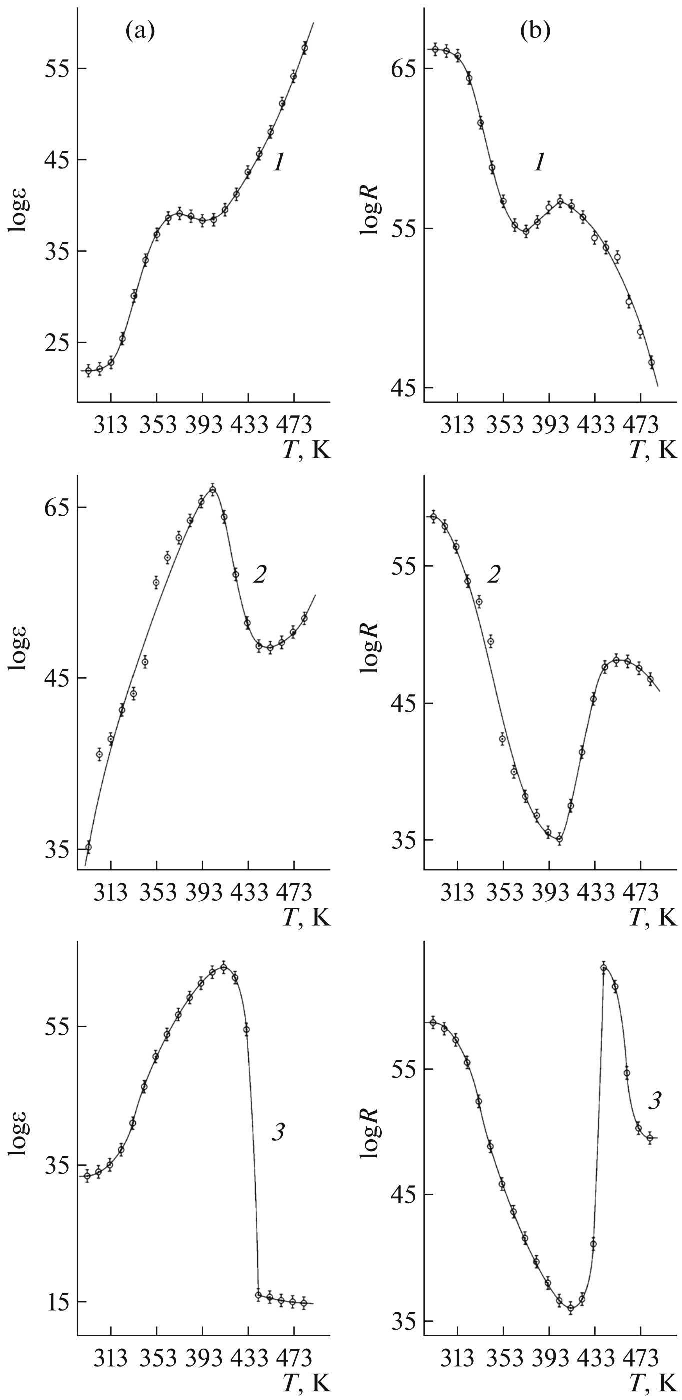

Fig. 2. Temperature dependences of (a) dielectric permeability and (b) electrical resistivity of (*1*) $LaLiFeCrMnO_{6.5}$, (*2*) $LaNaFeCrMnO_{6.5}$, and (*3*) $LaKFeCrMnO_{6.5}$.

LaKFeCrMnO$_{6.5}$

This compound displays semiconductor conductivity in the interval 293–413 K, metal conductivity at 413–443 K, and semiconductor conductivity again at 443–483 K. The electrical resistivity falls by a factor of 189 in the interval 293–413 K. Dielectric permittivity ε rises from 2082 to 2.28×10^6 (i.e., by 1097 times) in the interval 293–413 K. A sharp drop in dielectric permeability, from 2.84×10^5 to 38 (i.e., by 7462 times), is observed in the interval 433–443 K.

CONCLUSIONS

The temperature dependences of the isobaric heat capacity of $LaLiFeCrMnO_{6.5}$, $LaNaFeCrMnO_{6.5}$, and

Table 4. Temperature dependence of electrical resistivity (R), electrical capacity (C), and dielectric permeability (ε) of nanosized ferro-chromo-manganites

T, K	C, pF	E	logε	R, Ω	log$R$
		$LaLiFeCrMnO_{6.5}$			
293	0.04765	158	2.20	4061000	6.61
313	0.05953	197	2.29	3677000	6.57
333	0.31665	1048	3.02	1414000	6.15
353	1.4699	4866	3.69	454700	5.66
373	2.53	8376	3.92	295800	5.47
393	2.0988	6948	3.84	415100	5.62
403	2.1527	7127	3.85	454200	5.66
413	2.7701	9171	3.96	431400	5.63
433	7.1409	23641	4.37	269300	5.43
453	19.535	64674	4.81	202200	5.31
473	79.397	262857	5.42	68780	4.84
483	160.52	531428	5.73	45120	4.65
		$LaNaFeCrMnO_{6.5}$			
293	0.5633	3284	2.52	693000	5.84
313	1.0358	6038	3.78	414300	5.62
333	3.4688	20222	4.31	166400	5.22
353	69.278	403866	5.61	16620	4.22
373	230.81	1345540	6.13	6331	3.80
393	609.34	3552236	6.55	3493	3.54
403	848.88	4948669	6.69	3093	3.49
413	405.21	2362230	6.37	5401	3.73
433	23.305	135860	5.13	32540	4.51
453	11.997	69938	4.84	61390	4.79
463	13.776	80309	4.90	59620	4.78
473	17.956	104677	5.02	53480	4.73
483	25.7	149822	5.18	44900	4.65
		$LaKFeCrMnO_{6.5}$			
293	0.45911	2082	3.32	734800	5.87
313	0.66664	3023	3.48	537500	5.73
333	2.6938	12214	4.09	173400	5.24
353	25.086	113744	5.06	38380	4.58
373	102.28	463755	5.67	14000	4.15
393	299.75	1359117	6.13	6129	3.79
413	503.91	2284813	6.36	3882	3.59
433	62.541	283571	5.45	12470	4.10
443	0.00843	38	1.58	2008000	6.30
453	0.00774	35	1.55	1427000	6.15
473	0.0066	30	1.48	105200	5.02
483	0.00632	29	1.46	87030	4.94

$LaKFeCrMnO_{6.5}$ ferro-chromo-manganites were studied in the interval 298.15–673 K via experimental dynamic calorimetry. The λ-like effects associated with a second-order phase transition were observed on the $C_p^\circ \sim f(T)$ dependences for all of studied ferro-chromo-manganites in the investigated temperature ranges. Equations for the temperature dependences of the heat capacity of ferro-chromo-manganites were derived in light of the phase transition temperatures. The standard entropies of the studied ferro-chromo-manganites were calculated using ionic increments. The temperature dependences of $C_p^\circ(T)$ and thermodynamic functions $S^\circ(T)$, $H^\circ(T) - H^\circ(298.15)$ and $\Phi^{xx}(T)$ were calculated for the interval of 298.15–675 K. The results from these studies are of interest in the inorganic and physical chemistry of nanosized oxide materials, inorganic materials science, and physicochemical simulations of the technology for preparing ferro-chromo-manganites and similar compounds. They can serve as the initial data for the fundamental directories and databases of thermodynamic constants.

ACKNOWLEDGMENTS

This work was performed as part of project no. 0026/PTsF, "Developing Technology for Obtaining Nanosized Ferro-chromo-manganites of Alkali, Alkaline-Earth, and Rare-Earth Metals with Promising Electrophysical Properties," under the program "Scientific and Technological Support for the Rational Use of Mineral Resources and Anthropogenic Waste of Ferrous and Nonferrous Metallurgy in Manufacturing Products Demanded by Domestic Industry."

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Translated by L. Mosina