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New nanostructured manganites of $LaMe^{II}CuZnMnO_6$ (Me^{II} — Mg, Ca, Sr, Ba)

The copper-zinc manganites of $LaMe^{II}CuZnMnO_6$ (Me^{II} — Mg, Ca, Sr, Ba) have been synthesized with the high-temperature interaction of alkaline earth metals carbonates with oxides of lanthanum (III), copper (II), zinc (II) and manganese (III). The synthesized polycrystalline copper-zinc manganites have been grinded on the Retsch vibration mill MM301 (Germany). As a result their nanostructured particles have been obtained. Their sizes have been determined using an electron microscope Mira3 LMU, Tescan. Methods of radiography determined that all synthesized nanostructured copper-zinc manganites crystallize in the cubic syngony with the following parameters of a lattice: $LaMgCuZnMnO_6$ — a = 13.53±0.02 Å, V^o = 2476.81±0.06 Å^3, Z = 4, $V^o_{elect.cell}$ = 619.20±0.02 Å^3, ρ_{roent} = 4.52; ρ_{pick} = 4.50±0.01 g/cm^3; $LaCaCuZnMnO_6$ — a = 13.69±0.02 Å, V^o = 2565.73±0.06 Å^3, Z = 4, $V^o_{elect.cell.}$ = 641.43±0.02 Å^3, ρ_{roent} = 4.43; ρ_{pick} = 4.41±0.01 g/cm^3; $LaSrCuZnMnO_6$ — a = 13.91±0.02 Å, V^o = 2691.42±0.06 Å^3, Z = 4, $V^o_{elect.cell}$ = 672.85±0.02 Å^3, ρ_{roent} = 4.99; $\rho_{pick.}$ = 4.96±0.01 g/cm^3; $LaBaCuZnMnO_6$ — a = 14.55±0.02 Å, V^o = 3080.27±0.06 Å^3, Z = 4, $V^o_{elect.cell}$ = 770.07±0.02 Å^3, ρ_{roent} = 4.95; ρ_{pick} = 4.94±0.01 g/cm^3. The X-ray investigations demonstrated that the values of lattice parameters of the studied copper-zinc manganites have been increased from Mg to Ba. As a result of the investigations, these compounds can be included in Pm3m spatial group.

Keywords: synthesis, copper-zinc manganite, lanthanum, alkaline-earth metals, nanostructured particles, electron microscopy, radiography.

Introduction

Cuprates, manganites, zincates of the rare-earth elements, which partially substituted by oxides of alkaline-earth metals, have the unique physical and physicochemical properties [1–10]. Abishev Chemical-Metallurgical Institute has been conducting the systematic and targeted investigation in this direction for many years. Thus their results have been summarized in monographs [11–14]. This paper presents the results of synthesis and the radiousgraphic studies of the new nanostructured copper-zinc manganites of lanthanum and the alkaline earth metals. Zincates, cuprates and manganites have been combined into a single phase as the copper-zinc manganites.

Experimental

In order to obtain the copper-zinc manganites of lanthanum and the alkaline earth metals of $LaMe^{II}CuZnMnO_6$ (Me^{II} — Mg, Ca, Sr, Ba) the stoichiometric ratios of La_2O_3 (especially pure), ZnO (analytically pure), CuO (analytically pure), Mn_2O_3 (analytically pure) and $MgCO_3$, $CaCO_3$, $SrCO_3$ and $BaCO_3$ (analytically pure) have been intensively mixed, milled in an agate mortar. Then the mixtures have been placed in the alundum crucibles and placed in a muffle furnace SNOL. The annealing has been performed at 600 °C for 10 h, 800 °C for 10 h, 1000 °C for 10 h, 1200 °C for 10 h and at 1100 °C for 20 h. After each annealing at these temperatures, the mixtures have been cooled to a room temperature and intensively milled and mixed. The low temperature annealing of the mixtures have been performed at 400 °C for 10 h to obtain the stable and equilibrium phases at low temperatures.

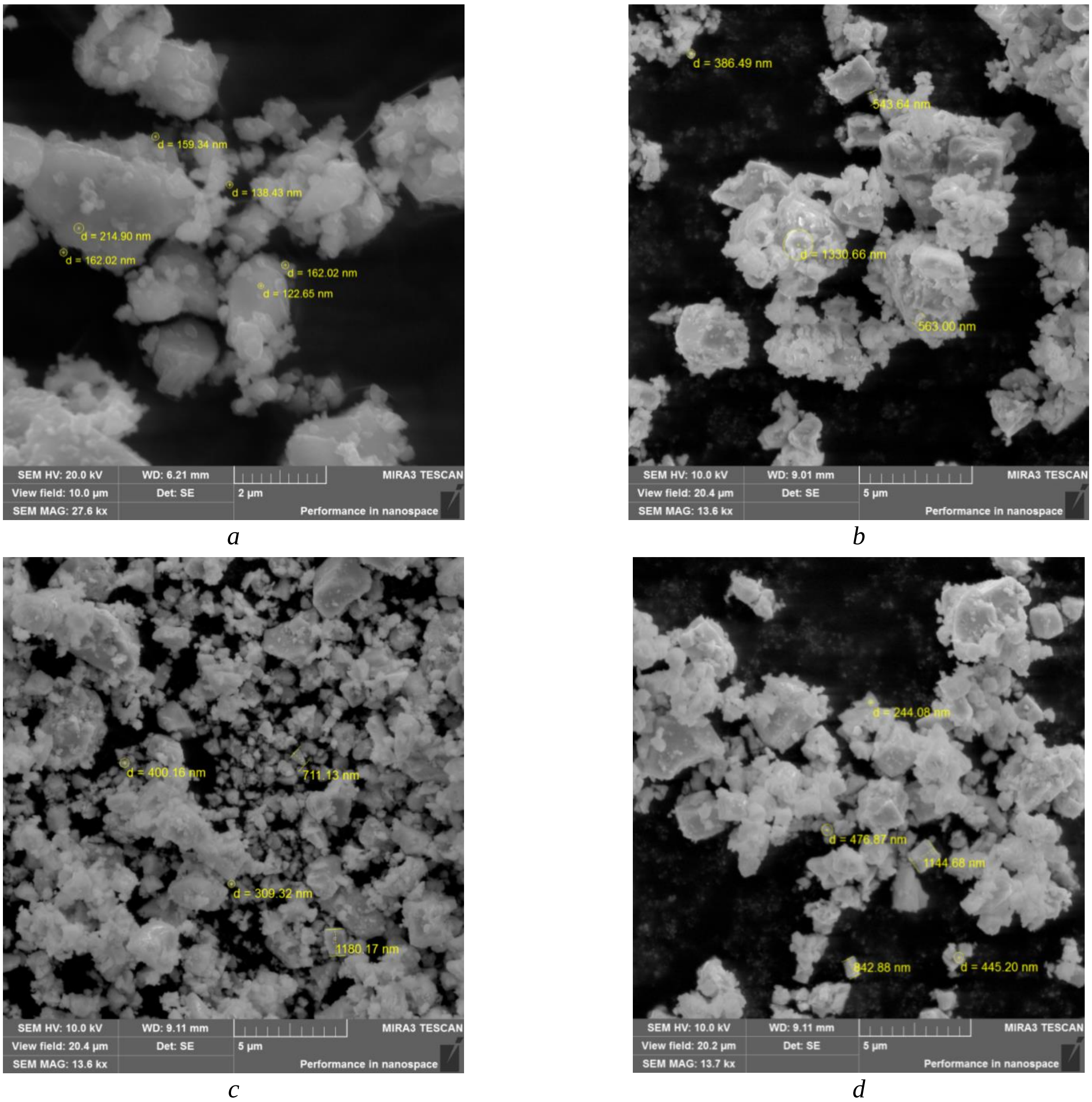
Then, the received polycrystalline samples of the copper-zinc manganites have been mixed under the special conditions to the nanostructured particles on the Retsch vibration mill (Germany). Their sizes have been determined on an electron microscope Mira3 LMU, Tescan (Fig.).

The X-ray phase analysis of the synthesized nanostructured copper-zinc manganite particles have been performed on DRON-2.0. The operating conditions: CuK_{δ}-radiation, U = 30 kV, J = mA, rotation speed is 100 pps, time constant τ = 5 s, an angle interval 2θ from 10 to 90°. The intensity of the diffraction maxima has been estimated on a100-point scale. The indexing of radiographs has been performed with the analytical method [15]. The pycnometric density has been determined with a procedure described in [16]. Toluene has been used as an indifferent liquid.

Results and discussion

It has been found that the nanoparticle sizes of the synthesized copper-zinc manganites exceed 100 nm.

Referring to [17], if a nanoparticle has a complex shape and structure, as a result the size of its structural element is studied to be characteristic. Such particles are generally referred to as the nanostructured particles, and their linear sizes can be significantly exceeded 100 nm [17].



a — $LaMgCuZnMnO_6$; *b* — $LaCaCuZnMnO_6$; *c* — $LaSrCuZnMnO_6$; *d* — $LaBaCuZnMnO_6$

Figure. The electron microscopy of samples

It should also be pointed out that this compound can be referred to nanostructured clusters.

Referring to [18], the nanoclusters formed in the solid-phase reactions are measured from one to hundreds of nanometers.

These above mentioned arguments demonstrate that copper-zinc manganites of lanthanum and alkaline earth metals can be studied as the nanostructured nanoclusters.

The Figure illustrates that the $LaMgCuZnMnO_6$ has particles within 122.6; 138.4; 159.3: 162.0: 214.9 nm; $LaCaCuZnMnO_6$ — 386.5; 543.6; 563.0; 1330.7 nm; $LaSrMgCuZnMnO_6$ — 309.3; 400.2; 711.1 and 1180.2 nm; $LaBaCuZnMnO_6$ — 244.1; 445.2; 476.9 and 842.9 nm.

The Table demonstrates results on the indexing of radiographs of $LaMe^{II}CuZnMnO_6$ (Me^{II} — Mg, Ca, Sr, Ba).

T a b l e 1

The indexing of radiographs of $LaMe^{II}CuZnMnO_6$ (Me^{II} — Mg, Ca, Sr, Ba)

I/I^0	d, Å	$10^4/d^2$exp.	hkl	$10^4/d^2$calcul.
1	2	3	4	5
$LaMgCuZnMnO_6$				
17	3.905	655.8	422	656.0
100	2.763	1310	444	1312
15	2.509	1588	730	1585
13	2.458	1655	650	1667
25	2.245	1984	661	1995
9	2.126	2212	900	2213
4	1.989	2528	8.5.3	2541
32	1.948	2635	9.4.0	2651
7	1.746	3280	10.4.2	3279
33	1.586	3975	11.5.0	3990
8	1.499	4450	991	4454
12	1.379	5259	12.7.0	5274
14	1.230	6610	15.4.1	6613
$LaCaCuZnMnO_6$				
10	3.862	670.4	500	670.0
100	2.738	1334	1341	1341
8	2.642	1433	1421	1421
13	2.480	1626	1636	1636
7	2.339	1828	1823	1823
15	2.233	2005	2011	2011
4	2.006	2485	2494	2494
35	1.928	2690	2682	2682
5	1.727	3353	3352	3352
7	1.626	3782	3781	3781
32	1.599	3911	3915	3915
15	1.365	5367	5363	5363
11	1.220	6719	6704	6704
$LaSrCuZnMnO_6$				
9	3.877	665.3	511	665.0
5	3.370	880.5	600	887.1
32	2.850	1231	550	1232
100	2.735	1337	552	1331
13	2.475	1632	741	1626
4	2.319	1859	555	1848
13	2.222	2025	910	2021
6	2.118	2229	931	2242
8	2.017	2458	10.0.0	2464
42	1.925	2699	952	2710
8	1.638	3727	12.2.2	3745
28	1.576	4026	10.8.0	4041
6	1.469	4634	13.4.2	4657

Continuation of the Table

I/I^0	d, Å	$10^4/d^2$exp.	hkl	$10^4/d^2$calcul.
1	2	3	4	5
15	1.363	5383	13.7.0	5372
12	1.216	6730	16.4.1	6727
$LaBaCuZnMnO_6$				
1	2	3	4	5
12	3.905	655.8	520	656.0
13	3.247	948.5	541	949.8
12	2.890	1197	720	1199
100	2.763	1310	730	1312
5	2.598	1481	811	1493
10	2.475	1632	822	1628
16	2.249	1977	664	1990
35	1.948	2635	10.4.1	2646
8	1.737	3314	11.5.1	3324
17	1.375	5289	12.9.3	5292
4	1.258	6319	12.10.6	6332
13	1.231	6599	17.0.0	6581

Correctness and assurance of results on the indexing have been confirmed with the experimental and calculated values of $10^4/d^2$, the X-ray and pycnometric densities (Table).

Based on the indexing of radiographs of the nanostructured $LaMe^{II}CuZnMnO_6$ (Me^{II} — Mg, Ca, Sr, Ba), it has been found that all synthesized copper-zinc manganites are crystallized in the cubic syngony with the following parameters of the lattice: $LaMgCuZnMnO_6$ — a = 13.53±0.02 Å, V^o = 2476.81±0.06 $Å^3$, Z = 4, $V^o_{unit\ cell.}$ = 619.20±0.02 $Å^3$, $\rho_{x\text{-}ray.}$ = 4.52; ρ_{pick} = 4.50±0.01 g/cm^3; $LaCaCuZnMnO_6$ — a = 13.69±0.02 Å, V^o = 2565.73±0.06 $Å^3$, Z = 4, $V^o_{unit\ cell.}$ = 641.43±0.02 $Å^3$, $\rho_{x\text{-}ray}$ = 4.43; ρ_{pick} = 4.41±0.01 g/cm^3; $LaSrCuZnMnO_6$ — a = 13.91±0.02 Å, V^o = 2691.42±0.06 $Å^3$, Z = 4, $V^o_{unit\ cell}$ = 672.85±0.02 $Å^3$, $\rho_{x\text{-}ray}$ = 4.99; $\rho_{pick.}$ = 4.96±0.01 g/cm^3; $LaBaCuZnMnO_6$ — a = 14.55±0.02 Å, V^o = 3080.27±0.06 $Å^3$, Z = 4, $V^o_{unit\ cell}$ = 770.07 ± 0.02 $Å^3$, $\rho_{x\text{-}ray}$ = 4.95; ρ_{pick} = 4.94±0.01 g/cm^3.

Referring to [19], the obtained $LaMe^{II}CuZnMnO_6$ can be included in Pm3m spatial group.

In a row from Mg to Ba, the values of “a” parameters and cell volumes have been increased.

Conclusions

The polycrystalline copper-zinc manganites of $LaMe^{II}CuZnMnO_6$ have been first synthesized with a solid-phase method. Their nanostructured particles have been obtained with further milling.

Their parameters of lattices have been determined with the radiographic methods.

The results of investigations make a certain contribution to the nanochemistry, radiography of the new inorganic oxide compounds. They are a basis for further thermodynamic and electrophysical studies of the obtained nanostructured particles.

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$LaMe^{II}CuZnMnO_6$ (Me^{II} — Mg, Ca, Sr, Ba) жаңа наноқұрылымды манганиттері

Сілтіліжер металдар карбонаттары мен лантана (III), мыс (II), мырыш (II) және марганец (III) тотықтарының жоғарытемпературалық әрекеттесуі арқылы $LaMe^{II}CuZnMnO_6$ (Me^{II} — Mg, Ca, Sr, Ba) құрамды мыс-мырышты-манганиттері синтезделініп алынды. «ММ301» маркалы «Retsch» (Германия) вибрациялық диирменінде синтезделініп алынған поликристалдық мыс-мырышты-манганиттерді үгіту арқылы олардың наноқұрылымдыбөлшектері алынып, Mira3 LMU, Tescan электрондық микроскоп көмегімен олардың өлшемдері анықталды. Рентгенография әдісімен синтезделінген наноқұрылымды мыс-мырышты-манганиттердің кубтық сингонияда келесідей тор көрсеткіштермен кристалданатыны анықталды: $LaMgCuZnMnO_6$ — a = 13,53±0,02 Å, V^o = 2476,81±0,06 Å^3, Z = 4, $V^o_{эл.ұя.}$ = 619,20±0,02 Å^3, $\rho_{рент.}$ = 4,52; $\rho_{пикн.}$ = 4,50±0,01 г/см^3; $LaCaCuZnMnO_6$ — a = 13,69±0,02 Å, V^o = 2565,73±0,06 Å^3, Z = 4, $V^o_{эл.ұя.}$ = 641,43±0,02 Å^3, $\rho_{рент.}$ = 4,43; $\rho_{пикн.}$ = 4,41±0,01 г/см^3; $LaSrCuZnMnO_6$ — a = 13,91±0,02 Å, V^o = 2691,42±0,06 Å^3, Z = 4, $V^o_{эл.ұя.}$ = 672,85±0,02 Å^3, $\rho_{рент.}$ = 4,99; $\rho_{пикн.}$ = 4,96±0,01 г/см^3; $LaBaCuZnMnO_6$ — a = 14,55±0,02 Å, V^o = 3080,27±0,06 Å^3, Z = 4, $V^o_{эл.ұя.}$ = 770,07±0,02 Å^3, $\rho_{рент.}$ = 4,95; $\rho_{пикн.}$ = 4,94±0,01 г/см^3. Рентгендік зерттеулер негізінде, яғни иондық радиустардың өсуімен Mg-ден Ba-ге зерттеліп отырған мыс-мырышты-манганиттердің тор көрсеткіштер шамасы ұлғаяды. Жасалған зерттеулерді ескере отырып, бұл қосылыстарды Pm3m кеңістіктік топқа жатқызуға болады.

Кілт сөздер: синтез, мыс-мырышты манганит, лантан, сілтіліжер металдар, наноқұрылымды бөлшектер, электронды микроскопия, рентгенография.

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Новые наноструктурированные манганиты $LaMe^{II}CuZnMnO_6$ (Me^{II} — Mg, Ca, Sr, Ba)

Высокотемпературным взаимодействием карбонатов щелочноземельных металлов с оксидами лантана (III), меди (II), цинка (II) и марганца (III) синтезированы медно-цинковые манганиты состава $LaMe^{II}CuZnMnO_6$ (Me^{II} — Mg, Ca, Sr, Ba). Измельчением синтезированных поликристаллических медно-цинковых манганитов на вибрационной мельнице «Retsch» (Германия) марки «MM301» получены их наноструктурированные частицы, размеры которых определены с помощью электронного микроскопа Mira3 LMU, Tescan. Методами рентгенографии установлено, что все синтезированные наноструктурированные медно-цинковые манганиты кристаллизуются в кубической сингонии со следующими параметрами решетки: $LaMgCuZnMnO_6$ — a = 13,53±0,02 Å, V^o = 2476,81±0,06 Å^3, Z = 4, $V^o_{эл.яч.}$ = 619,20±0,02 Å^3, $\rho_{рент.}$ = 4,52; $\rho_{пикн.}$ = 4,50±0,01 г/см^3; $LaCaCuZnMnO_6$ — a = 13,69±0,02 Å, V^o = 2565,73±0,06 Å^3, Z = 4, $V^o_{эл.яч.}$ = 641,43±0,02 Å^3, $\rho_{рент.}$ = 4,43; $\rho_{пикн.}$ = 4,41±0,01 г/см^3; $LaSrCuZnMnO_6$ — a = 13,91±0,02 Å, V^o = 2691,42±0,06 Å^3, Z = 4, $V^o_{эл.яч.}$ = 672,85±0,02 Å^3, $\rho_{рент.}$ = 4,99; $\rho_{пикн.}$ = 4,96±0,01 г/см^3; $LaBaCuZnMnO_6$ — a = 14,55±0,02 Å, V^o = 3080,27±0,06 Å^3, Z = 4, $V^o_{эл.яч.}$ = 770,07±0,02 Å^3, $\rho_{рент.}$ = 4,95; $\rho_{пикн.}$ = 4,94±0,01 г/см^3. На основании рентгенографических исследований установлено, что с повышением ионных радиусов от Mg к Ba увеличиваются величины параметров решетки исследуемых медно-цинковых манганитов. С учетом проведенных исследований можно отнести эти соединения к пространственной группе Pm3m.

Ключевые слова: синтез, медно-цинковый манганит, лантан, щелочноземельные металлы, наноструктурированные частицы, электронная микроскопия, рентгенография.

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