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# SYNTHESIS AND X-RAY OF NEW NANOSIZED (NANOCLUSTER) NICKELITE-CUPRATE-MANGANITES OF LANTHANUM AND ALKALINE METALS

**Abstract:** The main tendency in the development of microelectronics is miniaturization and increase in the speed of various devices. For storage devices, such as dynamic and static RAM, based on capacitive components (capacitors), this means that as the size of the capacitor decreases, its capacitance must remain the same [1].

The cuprates of REE are actively studied primarily as objects of high-temperature superconductivity (HTSC) compounds, cathode materials and catalysts. Lanthanum nickelites are promising materials as fuel cell cathodes.

The problems of synthesis and X-ray analysis of new nano-sized nickelite-cuprate-manganites of the composition $LaMe_2^INiCuMnO_6$ where ($Me^I$-Li, Na, K) are considered for the first time in this paper.

The nickelite-cuprate-manganites of the composition $LaMe_2^INiCuMnO_6$ (Me – Li, Na, K) are synthesized by solid-phase interaction in the range 800-1200 ° C from the oxides of lanthanum (III), nickel (II), copper (II), manganese (III) and lithium, sodium and potassium carbonates. Their nanoscale (nanoclusters) particles were obtained by grinding them on a vibratory mill "MM301" from Retsch (Germany). On the electron microscope "JSPM-5400" Scanning Probe Microscope "JEOL" (Japan) their sizes are determined. By X-ray diffraction analysis of compounds on the DRON-2.0 diffractometer and the indication of their X-ray diffraction patterns, analytical methods were used to determine the types of syngony and the parameters of the grids of the synthesized new phases: $LaLi_2NiCuMnO_6$ (cub.) – $a$=13,83±0,02 Å, $V^o$=2644,16±0,06 Å$^3$, $Z$=4, $V^o_{el.cell}$=661,04±0,02 Å$^3$, $\rho_{X\text{-}ray}$=4,03 g/cm$^3$; $LaNa_2NiCuMnO_6$ (cub.) – $a$=14,19±0,02 Å, $V^o$=2859,42±0,06 Å$^3$, $Z$=4, $V^o_{el.cell}$=714,86±0,01 Å$^3$, $\rho_{X\text{-}ray}$=3,38 g/cm$^3$; $LaK_2NiCuMnO_6$ (cub) – $a$=15,17±0,02 Å, $V^o$=3492,0±0,06 Å$^3$, $Z$=4, $V^o_{el.cell}$=873,0±0,01 Å$^3$, $\rho_{X\text{-}ray}$=3,70 g/cm$^3$.

**Key words:** nickelite, cuprate, manganite, lanthanum, alkaline metals.

Complex oxides of transition 3d- and 4f-elements with a perovskite structure closely related, e.g. manganites, nickelites, nickelates and cuprates of rare-earth elements doped with oxides of alkaline and alkaline-earth metals, due to the presence of excellent physico-chemical properties, like large values of electrical conductivity, dielectric permittivity , semiconductor conductivity, magnetic and superconducting properties [1-16].

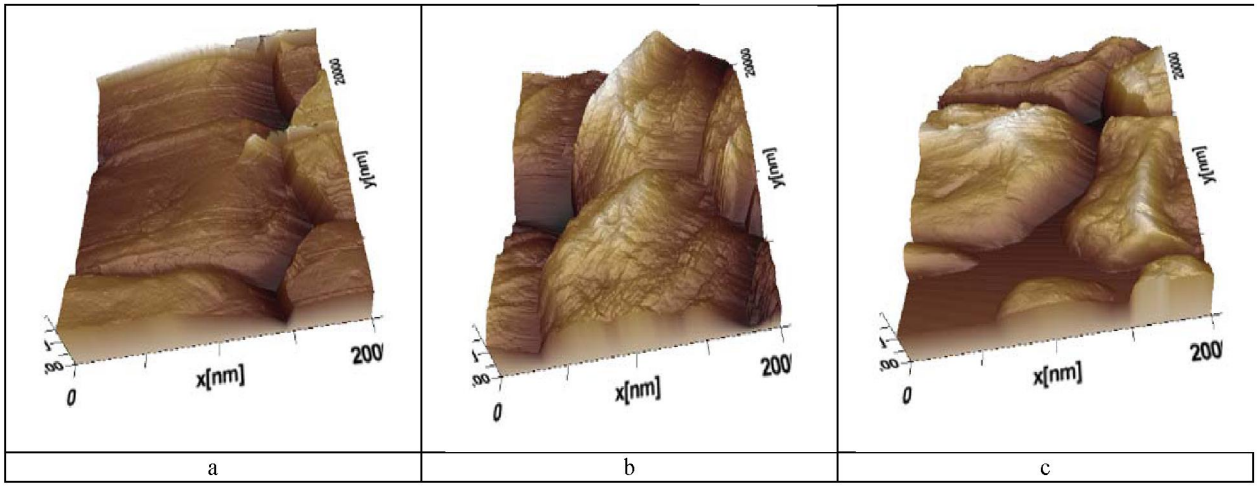
In the laboratory of thermochemical processes of the Zh. Abishev Chemical-Metallurgical Institute conducted systematic studies on the synthesis and study of crystal-chemical, thermodynamic and electrophysical properties of manganites, chromites and ferrites doped with oxides of alkali and alkaline-earth metals. Based on the results of the research, numerous articles have been published in peer-reviewed journals with nonzero impact factors, a number of security documents have been obtained and the main results have been summarized in the following papers [17, 18].

In our opinion, for the inorganic materials science, chemistry and technology of polyfunctional compounds, it is of certain importance to obtain nickelites, cuprates and manganites in one complex, as nickelite-cuprate-manganites.

To solve this problem, we present here the results of the synthesis and X-ray analysis of new nano-sized nickelite-cuprate-manganites of the composition $LaMe^{I}_2NiCuMnO_6$ where $Me^{I}$-Li, Na, K).

For the synthesis of the compounds have been used lanthanum (III), mark "high purity", nickel oxide (III), copper oxide (II), manganese oxide (III), lithium, sodium and potassium carbonates of mark "analytical grade". Their stoichiometric amounts in recalculation on the formula units of the obtained nickelite-cuprate-manganites, which were thoroughly mixed and ground. The mixture was annealed at 400 ° C for 10 hours to obtain stable at low-temperature modifications.

Nano-sized (nanoclusters) particles of synthesized nickelite-cuprate-manganites were obtained by grinding polycrystalline samples on a vibratory mill "MM301" from Retsch (Germany). The dimensions of the nanoclusters are determined using the "JSPM-5400" Scanning Probe Microscope "JEOL" electron microscope (Japan). Nanoparticles, nanoclusters with dimensions from 50 to 150 nm were obtained. The electron microscopic images of the connections are shown below.




a b c

Figure – Electron microscopy $LaLi_2NiCuMnO_6$ (a), $LaNa_2NiCuMnO_6$ (b), $LaK_2NiCuMnO_6$ (c)

X-ray analysis was performed on samples of nanoscale DRON 2.0. The intensity of the diffraction maxima was estimated from a 100-point scale. The X-ray diffraction of new nanosized compounds was carried out by an analytical method [19].

The table shows the results of the indication of the new phases obtained.

Table - Indication of the X-ray radiographs of nickelite-cuprate-manganites

| $I/I^o$ | d, Å | $10^4/d^2_{exp.}$ | hkl | $10^4/d^2_{calc.}$ |
|---|---|---|---|---|
| 1 | 2 | 3 | 4 | 5 |
| $LaLi_2NiCuMnO_6$ | | | | |
| 20 | 4,777 | 438,2 | 400 | 438,0 |
| 28 | 3,862 | 670,5 | 500 | 684,7 |
| 100 | 2,721 | 1351 | 700 | 1342 |
| 14 | 2,522 | 1572 | 722 | 1561 |
| 11 | 2,431 | 1692 | 651 | 1628 |
| 11 | 2,321 | 1856 | 820 | 1862 |
| 21 | 2,235 | 2002 | 830 | 1999 |
| 20 | 2,211 | 2046 | 751 | 2054 |
| 19 | 2,071 | 2332 | 920 | 2328 |
| 33 | 1,932 | 2679 | 853 | 2684 |
| 20 | 1,911 | 2738 | 10.0.0 | 2739 |
| 10 | 1,720 | 3380 | 11.1.1 | 3369 |
| 27 | 1,580 | 4006 | 11.5.0 | 3999 |
| 25 | 1,565 | 4083 | 10.7.0 | 4081 |
| 11 | 1,465 | 4659 | 13.1.0 | 4656 |

*Table continuation*

| 1 | 2 | 3 | 5 | 6 |
|---|---|---|---|---|
| 7 | 1,422 | 4945 | 10.9.0 | 4957 |
| 9 | 1,371 | 5320 | 13.5.0 | 5313 |
| 10 | 1,220 | 6719 | 15.4.2 | 6710 |
| 8 | 1,210 | 6830 | 10.10.7 | 6819 |
| $LaNa_2NiCuMnO_6$ | | | | |
| 21 | 3,862 | 670,5 | 510 | 670,0 |
| 5 | 3,599 | 772,0 | 521 | 773,7 |
| 6 | 3,458 | 836,3 | 440 | 825,2 |
| 5 | 3,061 | 1067 | 621 | 1057 |
| 100 | 2,735 | 1337 | 640 | 1341 |
| 10 | 2,520 | 1575 | 650 | 1573 |
| 14 | 2,4310 | 1692 | 811 | 1702 |
| 11 | 2,324 | 1851 | 660 | 1857 |
| 7 | 2,201 | 2064 | 840 | 2063 |
| 10 | 2,102 | 2263 | 664 | 2269 |
| 47 | 1,9300 | 2685 | 862 | 2682 |
| 4 | 1,8550 | 2906 | 870 | 2914 |
| 8 | 1,736 | 3318 | 881 | 3327 |
| 9 | 1,718 | 3388 | 11.3.1 | 3378 |
| 36 | 1,582 | 3996 | 975 | 3997 |
| $LaK_2NiCuMnO_6$ | | | | |
| 14 | 3,865 | 669,4 | 520 | 669,0 |
| 100 | 2,742 | 1330 | 730 | 1339 |
| 15 | 2,526 | 1567 | 644 | 1570 |
| 12 | 2,420 | 1708 | 750 | 1708 |
| 13 | 2,329 | 1844 | 840 | 1847 |
| 18 | 2,240 | 1993 | 655 | 1985 |
| 15 | 2,094 | 2281 | 755 | 2285 |
| 35 | 1,935 | 2671 | 10.4.0 | 2678 |
| 4 | 1,858 | 2897 | 10.5.1 | 2908 |
| 5 | 1,730 | 3341 | 12.1.0 | 3347 |
| 28 | 1,582 | 3996 | 12.5.2 | 3993 |
| 9 | 1,479 | 4572 | 13.5.2 | 4570 |
| 4 | 1,263 | 6269 | 16.4.0 | 6279 |
| 10 | 1,224 | 6675 | 17.0.0 | 6671 |
| 4 | 1.210 | 6830 | 16.6.2 | 6832 |

Satisfactory agreement between experimental and calculated values of $10^4/d^2$ confirms the correctness of the indexing results (table). Based on the indication of the X-ray patterns of the new nanoscale (nanocluster) phases established that they crystallize in a cubic system with the following lattice parameters: $LaLi_2NiCuMnO_6$ (cub.) – $a$=13,83±0,02 Å, $V^o$=2644,16±0,06 Å$^3$, $Z$=4, $V^o_{el.cell}$=661,04±0,02 Å$^3$, $\rho_{X\text{-}ray}$=4,03 g/cm$^3$; $LaNa_2NiCuMnO_6$ (cub.) – $a$=14,19±0,02 Å, $V^o$=2859,42±0,06 Å$^3$, $Z$=4, $V^o_{el.cell}$=714,86±0,01 Å$^3$, $\rho_{X\text{-}ray}$=3,38 g/cm$^3$; $LaK_2NiCuMnO_6$ (cub.) – $a$=15,17±0,02 Å, $V^o$=3492,0±0,06 Å$^3$, $Z$=4, $V^o_{el.cell}$=873,0±0,01 Å$^3$, $\rho_{X\text{-}ray}$=3,70 g/cm$^3$. According to [20, 21], it can be assumed that the ions $La^{3+}$ and $Me^{+}$ ($Li^+$, $Na^+$, $K^+$) are in the centers of the unit cells and have coordination numbers (kp) in oxygen equal to 12, and in the nodes of the elementary cells there are $Ni^{2+}$, $Cu^{2+}$ and $Mn^{3+}$, which c.n. in oxygen are equal to 6.

There is a pattern which is that with increasing of ionic radius among Li→Na→K also increasing the values of the lattice parameters ($a$, $V^o$, $V^o_{el.cell.}$).

Summarizing the foregoing, we can say that the nickele-cuprate-manganites $LaMe_2^INiCuMnO_6$ ($Me^I$ – Li, Na, K) were synthesized for the first time by the method of ceramic technology. Their nanosized (nanoclusters) particles were obtained, and their lattice parameters were also determined.

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**ЖАҢА НАНОӨЛШЕМДІ (НАНОКЛАСТЕРЛІК) НИКЕЛИТ-КУПРАТ-МАНГАНИТТЕРДІҢ СИНТЕЗІ ЖӘНЕ РЕНТГЕНОГРАФИЯСЫ**

**Аннотация.** Микроэлектрониканың дамуындағы негізгі тенденция болып түрлі құрылғылардың тезәсеретуін ұлғайту мен оларды миниатюризациялау болып табылады.

Динамикалық және статикалық оперативтік жадылар сияқты сыйымдылық компоненттерге (конденсаторларға) негізделген жадыда сақтау құрылғыларда, бұл деген конденсатордың өлшемі кішірейтілген кезде оның сыйымдылық шамасы бұрынғыдай қалу керек [1].

СЖЭ купраттары бәрінен бұрын жоғарытемпературалы асқынөткізгішті (ЖТАӨ) үлгілер, катодты материалдар және катализаторлар ретінде белсенді зерттелуде.

Лантан никелиттері жану ұяшықтарында катод ретінде келелі материалдар болып табылады.

Берілген жұмыста алғаш рет $LaMe_2^INiCuMnO_6$ мұндағы ($Me^I$ – Li, Na, K) құрамды жаңа наноөлшемді никелит-купрат-манганиттердің синтезі және рентгенографиялық талдау сұрақтары қарастырылған.

Лантан (III), никель (II), мыс (II), марганец (III) тотықтары және литий, натрий және калий карбонаттарының қатты фазалы әрекеттесуі арқылы 800-1200°С аралықта $LaMe_2^INiCuMnO_6$ (Me – Li, Na, K) құрамды никелит-купрат-манганиттері синтезделініп алынды.

Retsch (Германия) компаниясының «MM301» маркалы вибрациялық диірменінде үгіту жолымен олардың наноөлшемді (нанокластерлік) бөлшектері алынды.

«JSPM-5400» Scanning Probe Microscope «JEOL» (Япония) электрондық микроскопында олардың өлшемдері анықталды.

ДРОН-2,0 дифрактометрінде қосылыстарға рентгенофазалық талдау жүргізілді және олардың рентгенограммаларын аналитикалық әдіспен индицирлеу барысында синтезделініп алынған жаңа фазалардың сингония типі мен тор көрсеткіштері анықталды: $LaLi_2NiCuMnO_6$ (куб.) – $a$=13,83±0,02 Å, $V^o$=2644,16±0,06 Å$^3$, $Z$=4, $V^o_{эл.ұя.}$=661,04±0,02 Å$^3$, $\rho_{рент.}$=4,03 г/см$^3$; $LaNa_2NiCuMnO_6$ (куб.) – $a$=14,19±0,02 Å, $V^o$=2859,42±0,06 Å$^3$, $Z$=4, $V^o_{эл.ұя.}$=714,86±0,01 Å$^3$, $\rho_{рент.}$=3,38 г/см$^3$; $LaK_2NiCuMnO_6$ (куб.) – $a$=15,17±0,02 Å, $V^o$=3492,0±0,06 Å$^3$, $Z$=4, $V^o_{эл.ұя.}$=873,0±0,01 Å$^3$, $\rho_{рент.}$=3,70 г/см$^3$.

**Түйін сөздер:** никелит, купрат, манганит, лантан, сілітілі металдар.

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## СИНТЕЗ И РЕНТГЕНОГРАФИЯ НОВЫХ НАНОРАЗМЕРНЫХ (НАНОКЛАСТЕРНЫХ) НИКЕЛИТО-КУПРАТО-МАНГАНИТОВ ЛАНТАНА И ЩЕЛОЧНЫХ МЕТАЛЛОВ

**Аннотация:** Основной тенденцией в развитии микроэлектроники является миниатюризация и увеличение быстродействия различных устройств. Для запоминающих устройств, вроде динамической и статической оперативной памяти, основанных на емкостных компонентах (конденсаторах), это означает, что при уменьшении размеров конденсатора величиина его емкости должна оставаться прежней [1].

Купраты РЗЭ активно исследуются прежде всего как объекты высокотемпературной сверхпроводимости (ВТСП) соединений, катодные материалы и катализаторы.

Никелиты лантана являются перспективными материалами в качестве катодов топливных ячеек.

В данной работе впервые рассматриваются вопросы синтеза и рентгенографического анализа новых наноразмерных никелито-купрато-манганитов состава $LaMe_2^INiCuMnO_6$ где $Me^I$ – Li, Na, K).

Твердофазным взаимодействием в интервале 800-1200°С из оксидов лантана (III), никеля (II), меди (II), марганца (III) и карбонатов лития, натрия и калия синтезированы никелито-купрато-манганиты состава $LaMe_2^INiCuMnO_6$ (Me – Li, Na, K). Путем измельчения на вибрационной мельнице марки «MM301» компании Retsch (Германия) получены их наноразмерные (нанокластерные) частицы. На электронном микроскопе «JSPM-5400» Scanning Probe Microscope «JEOL» (Япония) определены их размеры. Проведением рентгенофазового анализа соединений на дифрактометре ДРОН-2,0 и индицированием их рентгенограмм аналитическим методом определены типы сингонии и параметры решеток синтизированных новых фаз: $LaLi_2NiCuMnO_6$ (куб.) – $a$=13,83±0,02 Å, $V^o$=2644,16±0,06 Å$^3$, $Z$=4, $V^o_{эл.яч.}$=661,04±0,02 Å$^3$, $\rho_{рент.}$=4,03 г/см$^3$; $LaNa_2NiCuMnO_6$ (куб.) – $a$=14,19±0,02 Å, $V^o$=2859,42±0,06 Å$^3$, $Z$=4, $V^o_{эл.яч.}$=714,86±0,01 Å$^3$, $\rho_{рент.}$=3,38 г/см$^3$; $LaK_2NiCuMnO_6$ (куб.) – $a$=15,17±0,02 Å, $V^o$=3492,0±0,06 Å$^3$, $Z$=4, $V^o_{эл.яч.}$=873,0±0,01 Å$^3$, $\rho_{рент.}$=3,70 г/см$^3$.

**Ключевые слова:** никелит, купрат, манганит, лантан, щелочные металлы.

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