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# ВЕСТНИК

НАЦИОНАЛЬНОЙ АКАДЕМИИ НАУК
РЕСПУБЛИКИ КАЗАХСТАН

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# THE COMPOSITION AND STRUCTURE OF BISMUTH-DOPED DYSPROSIUM MANGANITE

**Abstract.** The task of this work is to further study the composition and morphology of the powders of manganite synthesized by Sol-gel method using oxides of dysprosium, manganese and bismuth. It is shown that when using nitric acid as a precipitator, a single-phase powder can be obtained. Were identified methods of obtaining perovskitelike powders of dysprosium manganite. Structural and morphological properties of the samples were studied by x-ray phase analysis (X-ray) and scanning electron microscopy (SEM) in combination with optical microscopy. By the method of X-ray phase analysis has previously been used to determine the structure and parameters of elementary cells of the synthesized manganite. According to X-ray data, the manganite has an orthorhombic structure with the following parameters: a=5.2793, b= 5.83, C=7.382 Å, Z=4. Methods of optical and electronic microscopy defined shapes and sizes of the obtained powders of the manganite. It is shown that the powders of manganite are formed in the dendritic structure and it has particle sizes from 1 micron to 30 microns.

**Key words:** double bismuth manganite, Sol-gel method, structure, optical microscope, electronic microscope.

**Introduction.** The development of spintronic devices based on nanoscale boundaries of magnetic materials is a complex task. In [1], heterostructures of complex materials with the use of manganites, multiferroics, compounds of rare earth elements, etc.for non-volatile storage devices were considered. Recently, a structure for memory cells has been proposed, providing reliable spin manipulation and switching of magnetization between two stable positions in intermetallic structures [2]. The solution is based on the superlattice of single-bonded rare earth compounds $TbCo_2$ and 3d transition metal FeCo. The structure is characterized by a giant magnetostriction and demonstrates the transitions of spin reorientation by an external magnetic field and/or elastic deformation [3, 4]. In transition metal oxides $R_{1-x}A_xMnO_3$ (manganites), where R is one of the rare earth elements La or Pr, A is one of the alkaline earth metals Sr or Ca, the new state electric and magnetic phases can occur when electric fields and strains in thin films or on surfaces with dielectrics or other oxides. [5].

**Methods.** In this method of synthesis, trivalent metal oxides were used: dysprosium, manganese and bismuth. The required amount of oxides was dissolved in distilled water. Citric acid and glycerol (2:3) were added to the obtained solution as gelling agents. Then the solution was heated using an electric stove with constant stirring at 80°C to remove excess water and obtain viscous gels. The gel was dried at 250°C and annealed at 500°C for 10h to obtain the desired powder. The powders were crushed in an agate mortar to obtain a homogenous mixture. Then they were placed in crucibles and annealed at temperatures of 600-1000°C for 19 hours [6].

The finished single-phase powder of the manganite was studied using XRF to determine the structure, parameters of electronic cells and methods of optical and electron microscopy.

**Results**

*X-ray diffraction.* It was found that the manganite synthesized by the Sol – gel method is single-phase (figure 1).

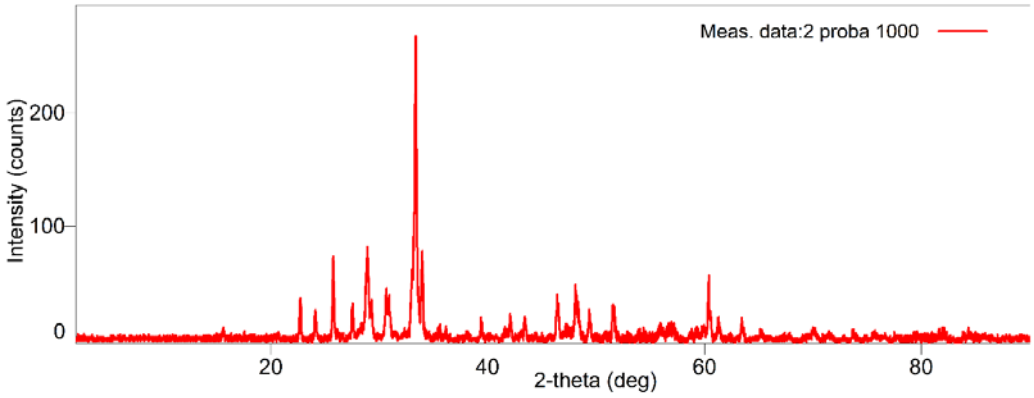



Figure 1 – The diffraction pattern of the manganite synthesized by the sol-gel method

The formation of new phases was controlled by the method of x-ray phase analysis produced by X-ray diffractometer Miniflex 600 (Rigaku) using CuK α-radiation filtered by the Ni filter (U = 30 кV, J = 10 мА, the rotation speed of 1000 pulses per second, time constant = 5 sec., the range of angles 2θ from 5 to 900). Radiographs of the synthesized polycrystalline powders were indicated by the homology method (homologue is a distorted structure type of perovskite). The density of manganites were determined by the pycnometric method according to GOST 2211-65. Toluene served as indifferent liquid. The density of the manganite was measured 4–5 times and data were averaged [6].

Table 1 – The results on indexing of radiographs of manganite

| # | [°2Th.] | d[Å] | Int. [%] | $10^4/d^2$эксп. | hkl | $10^4/d^2$теор. |
|---|---|---|---|---|---|---|
| 1 | 11.38 | 7.769 | 0.2 | 165.67 | (1,1,0) | 165.67 |
| 2 | 16.12 | 5.494 | 1.8 | 331.3 | (2,0,0) | 331.3 |
| 3 | 19.78 | 4.486 | 6.5 | 496.91 | (2,1,1) | 496.91 |
| 4 | 22.87 | 3.885 | 2.7 | 662.54 | (2,2,0) | 662.54 |
| 5 | 25.62 | 3.474 | 2.2 | 828,59 | (0,1,3) | 828,59 |
| 6 | 28.11 | 3.172 | 100.0 | 993,88 | (2,2,2) | 993,88 |
| 7 | 30.42 | 2.936 | 3.0 | 1160,08 | (1,2,3) | 1160,08 |
| 8 | 32.57 | 2.747 | 37.0 | 1325,2 | (4,0,0) | 1325,2 |
| 9 | 34.61 | 2.590 | 3.9 | 1490,74 | (4,1,1) | 1490,74 |
| 10 | 36.54 | 2.457 | 1.7 | 1656,49 | (0,2,4) | 1656,49 |
| 11 | 38.40 | 2.342 | 2.3 | 1823,16 | (3,3,2) | 1823,16 |
| 12 | 40.18 | 2.243 | 0.6 | 1987,65 | (4,2,2) | 1987,65 |

The results of the synthesized manganite radiograph indexing by different methods show that the manganites have the orthorhombic structure with the following unit cell parameters (table 2).

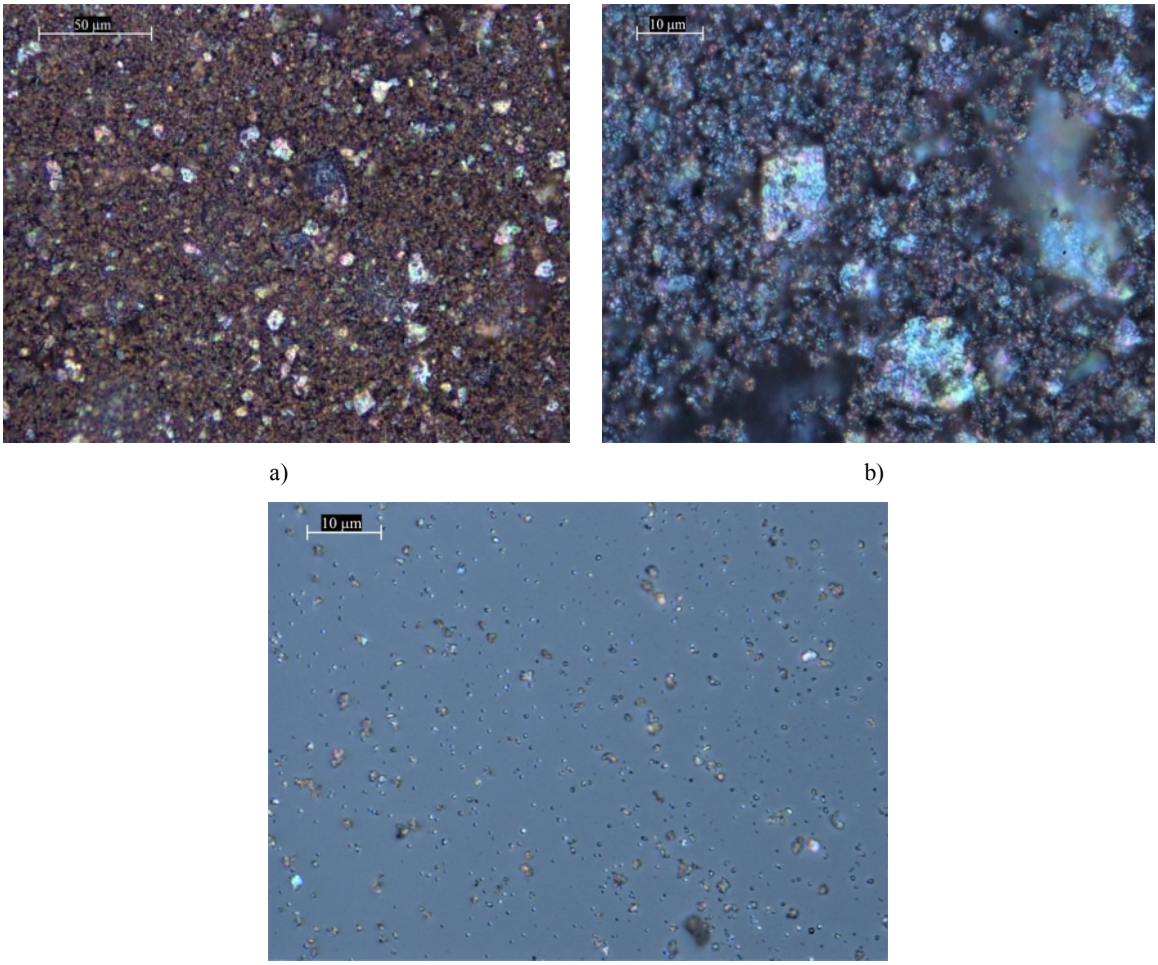
The reliability of the indexing results is controlled by a satisfactory coincidence of experimental and calculated values of the inverse squares of the interplanar spacing's ($10^4/d^2$), and the coincidence degree of the x-ray and pycnometric densities values of the studied compounds.

Thus, the double bismuth-manganite BDMO was synthesized by the sol-gel method, where glycerin was used as the stabilizer of the sols. The use of this method made it possible to obtain single-phase crystalline particles with a much shorter time [6].

Table 2 – The unit cell parameters of the manganite obtained by sol-gel method

| № | Compound | a | в | c | $V_{un.cell.}$, Å3 | Z | $P_{X\text{-}ray}$, g/cm$^3$ | $P_{рус.}$, g/cm$^3$ |
|---|---|---|---|---|---|---|---|---|
| 1 | $Bi_{0,1}Dy_{0,9}MnO_3$ | 5.2793 | 5.83 | 7.382 | 227.26 | 4 | 7.765 | 7.761 |

*Morphological study.* Figure 2 shows a micrograph of manganite powders taken from a digital material science microscope Leica DM 6000.




a) b)

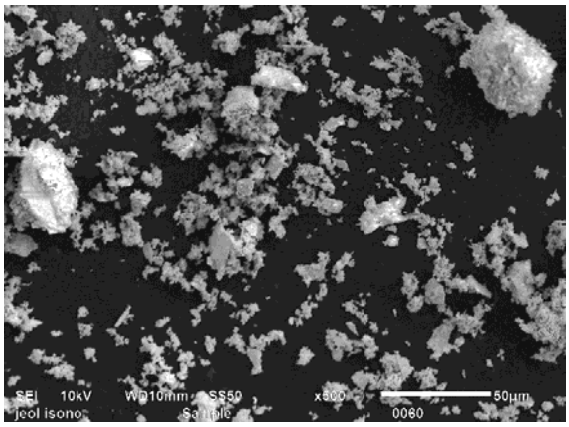
c)

Figure 2 – A micrograph of powders of manganite with different magnifications:
a) an increase of 50, b) an increase of 100, c) an increase of 150

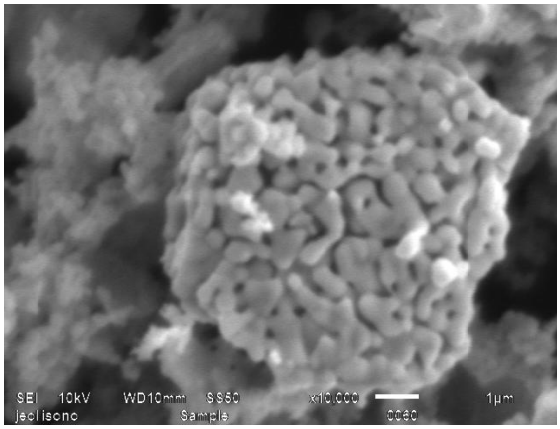
Microstructure of bulk samples was studied by scanning electron microscopy (SEM) JOEL JED-2300 with approaching up to x2000 and the ability to conduct elemental analysis. Photographs of the coatings obtained are shown in figure 3.

This increase in particle size with the level of doping is apparently due to a change in the melting point of the samples, which reduces the increase in the content of cations of alkaline earth metals. According to Harton et al. (2002), this effect leads to a liquid-phase process, which is facilitated by sintering and increasing grain growth. On the surface, it can be seen that the resulting coating has a dense structure consisting of 50 µm crystals.

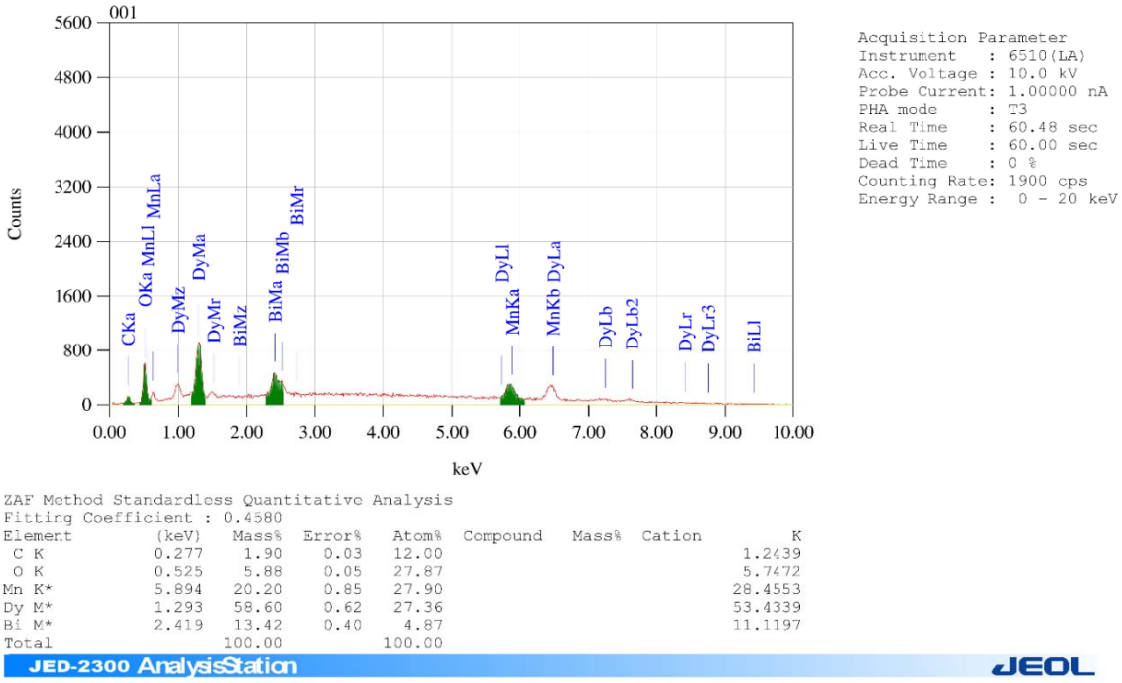
The elemental analysis performed on an electron-scanning microscope (insert in figure 4) showed that the atomic fractions of the elements practically coincide, which corresponds to the formula of bismuth-dysprosium manganite – BDMO. As can be seen from figure 3, the powders obtained by this technology are practically monodisperse, which is a great advantage of the method.




a)




b)




c)

Figure 3 – A micrograph of manganite powders taken on an electron microscope with different magnifications: a) an increase of 100, b) an increase of 2 000, c) EDS microanalysis

**Discussion.** The manganite powders obtained by the sol-gel method have a single-phase structure. According to micrographs, manganite has particle sizes from 1 micron to 30 microns.

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## ВИСМУТПЕН ЛЕГИРЛЕНГЕН ДИСПРОЗИЙ МАНГАНИТІНІҢ ҚҰРАМЫ ЖӘНЕ ҚҰРЫЛЫСЫ

**Аннотация.** Бұл жұмыстың мақсаты диспрозий, марганец және висмуттың оксидтерін пайдаланып золь-гель әдісімен синтезделген манганит ұнтағын ары қарай құрамы мен морфологиясын зерттеу болып табылады. Азот қышқылын тұнбаға түсіруші ретінде пайдалану арқылы бір фазалы манганит ұнтағын алуға болатындығы көрсетілген. Перовксит типті диспрозий манганитінің ұнтағын алу әдісі анықталған. Үлгілердің құрылыстық және морфологиялық қасиеттері рентгенофазалық талдау (РФТ) және оптикалық микроскопиямен бірге сканирлеуші электронды микроскопия (СЭМ) әдістерімен зерттелді. Бұрын рентгенофазалық талдау әдісі арқылы синтезделген манганиттің құрылысы мен қарапайым ұяшық параметрлері анықталған. РФТ мәліметтері бойынша манганит орторомбты құрылысқа ие екені көрсетілді және параметрлері келесідей: a=5.2793, в= 5,83, c=7.382Å, Z=4. Оптикалық және электронды микроскопия әдістерімен алынған манганит ұнтақтарының көлемі мен өлшемдері анықталды. Манганит ұнтақтарының дендритті құрылысқа ие екені көрсетілді және оның өлшемдері 1 микроннан 30 микронға аралығында.

**Түйін сөздер:** висмуттың қосарланған манганиті, золь-гель әдісі, құрылыс, оптикалық микроскоп, электронды микроскоп.

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## СОСТАВ И СТРУКТУРА ЛЕГИРОВАННОГО ВИСМУТОМ МАНГАНИТА ДИСПРОЗИЯ

**Аннотация.** Задачей данной работы является дальнейшее исследование состава и морфологию порошков манганита, синтезированного золь-гель методом с использованием оксидов диспрозия, марганца и висмута. Показано, что при использовании азотной кислоты в качестве осадителя можно получить однофазный порошок. Были определены методы получения перовскитоподобных порошков манганита диспрозия. Структурные и морфологические свойства образцов изучали методами рентгенофазового анализа (РФА) и сканирующей электронной микроскопии (СЭМ) в сочетании с оптической микроскопией. Методом рентгенофазового анализа ранее были определены структура и параметры элементарных ячеек синтезированного манганита. По данным РФА установлено, что манганит имеет орторомбическую структуру со следующими параметрами: a=5.2793, в= 5,83, c=7.382Å, Z=4. Методами оптической и электронной микроскопий определены формы и размеры полученных порошков манганита. Показано, что порошки манганита складываются в дендритную структуру и он имеет размеры частиц от 1 микрона до 30 микронов.

**Ключевые слова:** двойной манганит висмута, золь-гель метод, структура, оптический микроскоп, электронный микроскоп.

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