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SYNTHESIS AND STRUCTURE OF MANGANITE $Bi_{0.3}Dy_{0.7}Mn_7O_{12}$

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Manganite with mixed complex oxides $Bi_{0.3}Dy_{0.7}Mn_7O_{12}$ was synthesized by the furnace method. The Pechini method used urea as a complexing agent. The composition and structure of the synthesized manganite were determined experimentally. For the first time, the structure, the type of the syngony, the parameters of the unit cells, the x-ray diffraction density of the manganite were determined by X-ray phase analysis: a = 7.5472, b = 8.5244, c = 5.7836 Å, $V_{un.cell.}$ = 372.090Å^3, Z = 4, $\rho_{rad.}$ = 7,120 g / cm^3, $\rho_{pyc.}$ = 7.09 g / cm^3. The pycnometric density was measured 5-6 times and the data were averaged. Toluene served as an indifferent fluid.

Keywords*:manganite, crystal system, radiographic and pycnometric density, unit cell parameters*

Perovskite-type complex manganite $Ln_{1-x}A_xMnO_3$ were Ln is a rare earth (Ln = La, Pr, Sm, ..) and A is a divalent element (A = Ca, Sr, Ba, ..) have attracted considerable attention because of their interesting electrical, magnetic and catalytic properties [1–8]. With advances in information technology, more sensitive and high magnetoresistive materials are required for high-speed electronic and magnetic devices. Manganites are of vital interest to a wide community of materials scientists not only because of the fascinating phenomena and properties exhibited, but also due to their potential technological applications in magnetic ordering and sensors. These manganite have been attracting the focus of materials researchers for over more than a decade due to huge decrease in resistivity on application of magnetic field, called as colossal magnetoresistance (CMR) and behavior around Curie temperature (Tc) near room temperature and owing to their properties. In the present paper, we report synthesis, structure, of $Bi_{0.3}Dy_{0.7}Mn_7O_{12}$nanopowders elaborated by Pechini method.

Synthetic method Pechini was used for the synthesis of manganite $Bi_{0.3}Dy_{0.7}Mn_7O_{12}$ (Figure 1), which has the potential for their use in different applications.

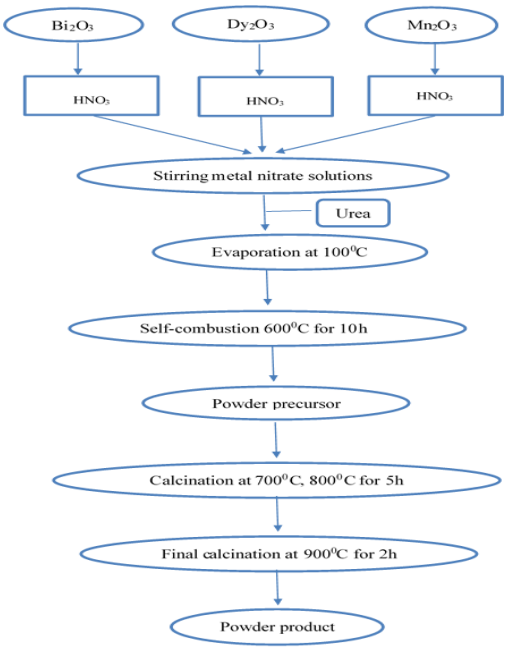

Figure 1. Flowchart for the pechini method used to obtain the perovskite compound shown in the present work

The selection of method and composition for perovskite was based on the desired application that are described later.

In the method, the standardized oxides were mixed according to the stoichiometry of the final products: $Bi_{0.3}Dy_{0.7}Mn_7O_{12}$ (BDMO).

The starting materials were Dy_2O_3 (99.9%) Bi_2O_3 (99.9%) Mn_2O_3 (99.9%) which had to be dissolved in nitric acid before the addition of the other compound (urea). A suitable amount of urea was added to the mixture as a coordinate agent. The solution was then allowed to dry to form a dried gel in an electric oven at 100 C. The resulting dried gel was annealed in a muffle furnace to give a black powder at 600°C for 10 hours. Finally, the resulting powder was heated in air at 700-900°C for 7 hours.

Results and discussion

X-ray diffraction. Powder X-ray diffraction patterns (refer with: Figure 2-3) show that the samples show single phase and indexed (refer with:Table 1) in the cubic structure with Fm-3m (225) group space.

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 filter (U = 30 кV, J = 10 мA, 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 was 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.

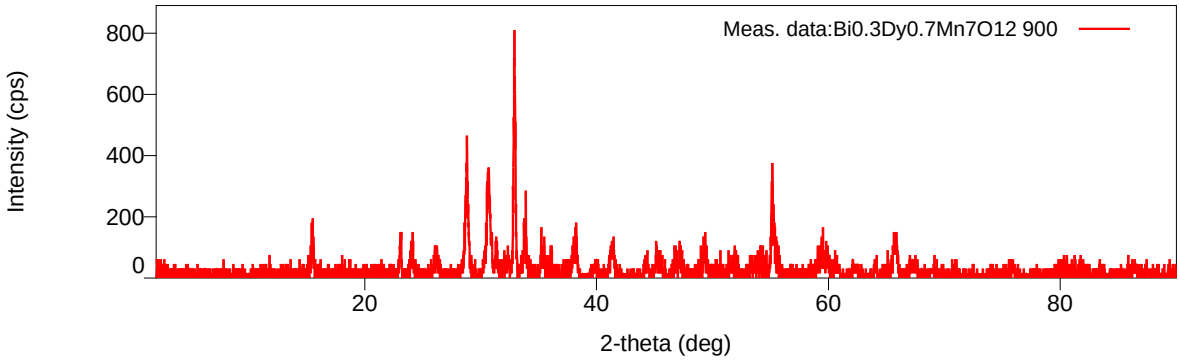

Figure 2. X-ray powder of BDMO.

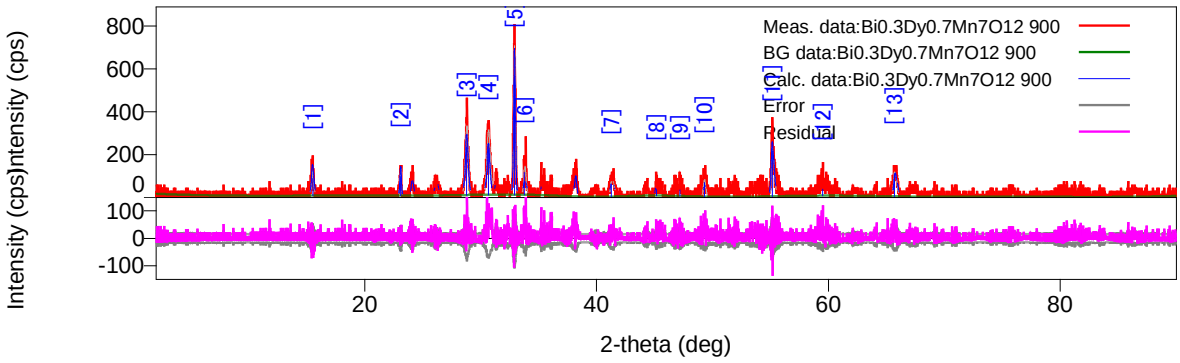

Figure 3. Observed (red symbols) and calculated (blue lines) X-ray diffraction pattern for BDMO sample and the peaks marked with pink are the remaining after the refinement of the phase by the Rietveld method.

Table 1. The results on indexing of radiographs of manganite

№	[°2Th.]	d[Å]	Int. [%]	$10^4/d^2_{exp.}$	hkl	$10^4/d^2_{theory}$
1	15.31	5.784	56.6	298,9	(0,0,1)	298,08
2	15.67	5.651	1.6	313,14	(1,1,0)	313
3	20.82	4.262	2.9	550,52	(0,2,0)	550,83
4	21.97	4.042	3.8	612,08	(1,1,1)	612,2
5	23.56	3.774	0.3	702,09	(2,0,0)	702,1
6	23.96	3.711	27.5	726,13	(1,2,0)	726,12
7	25.80	3.451	17.2	839,67	(2,1,0)	839,07
8	25.95	3.431	13.9	849,49	(0,2,1)	849,11
9	28.21	3.160	21.3	1001,44	(2,0,1)	1000
10	28.55	3.124	94.3	1024,65	(1,2,1)	1025
11	30.13	2.963	100.0	1139,03	(2,1,1)	1140
12	30.90	2.892	20.6	1195,65	(0,0,2)	1196
13	31.64	2.825	10.7	1253,03	(2,2,0)	1253
14	33.68	2.659	30.0	1414,97	(1,3,0)	1415
15	34.82	2.574	22.3	1509,33	(1,1,2)	1510
16	35.33	2.539	2.5	1551,22	(2,2,1)	1551
17	37.23	2.413	11.5	1717,45	(1,3,1)	1717
18	37.56	2.393	13.0	1746,28	(0,2,2)	1746
19	39.22	2.295	13.3	1898,60	(2,0,2)	1899
20	39.47	2.281	9.1	1921,98	(1,2,2)	1922
21	39.67	2.270	1.2	1970,65	(2,3,0)	1971
22	40.48	2.227	10.4	2016,32	(3,1,1)	2016
23	40.67	2.216	27.6	2036,39	(2,1,2)	2036,57
24	41.65	2.166	0.2	2131,49	(3,2,0)	2131,15
25	42.38	2.131	1.3	2202,08	(0,4,0)	2202
26	42.76	2.113	0.3	2239,76	(2,3,1)	2240
27	44.12	2.051	12.5	2377,22	(1,4,0)	2378
28	44.63	2.029	5.3	2429,05	(3,2,1)	2429
29	44.81	2.021	1.4	2448,32	(2,2,2)	2448,45
30	45.31	2.000	1.5	2500	(0,4,1)	2500,6
31	46.35	1.957	13.6	2611,07	(1,3,2)	2611,7
32	46.97	1.933	19.0	2676,31	(1,4,1)	2676,4
33	47.10	1.928	6.1	2690,21	(0,0,3)	2691

The results of the synthesized manganite radiograph indexed by this method show that the manganite haves the cubic structure with the following unit cell parameters (refer with:Table 2):

Table 2. The unit cell parameters of the manganite obtained by pechini method

№	Compound	a	в	c	$V_{un.cell.}$, Å^3	Z	$D_{X-ray.}$ g/cm^3	$D_{рус.}$ g/cm^3
1	BDMO	7.5472	8.5244	5.7836	372.090	4	7.120	7.09

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

Thus, the double bismuth– manganite BDMO was synthesized by various methods. Using the ceramic technology, considering the Tamman's conditions, the authors defined temperature regime of the synthesis of the dual mixed manganite BDMO. The type of crystal system and unit cell parameters were determined by the radiographic method. It is established that a complex mixed manganite is crystallized in the orthorhombic crystal system, the correctness of the results of x-ray studies of the manganite is confirmed by the good concordance between the experimental and calculated values ($10^4/d^2$), concordance between the values of x-ray and picnometer densities. The comparative analysis of parameters between the lattice parameters of the source δ-Bi_2O_3 shows that the values of the parameters *a* and *b* satisfactorily coincide with the lattice parameters δ-Bi_2O_3, the parameter *c* is distorted from the value of the *a* parameter on $\sqrt{2}$.

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СИНТЕЗ И СТРУКТУРА МАНГАНИТА $Bi_{0.3}Dy_{0.7}Mn_7O_{12}$

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Манганит со смешанными комплексными оксидами $Bi_{0.3}Dy_{0.7}Mn_7O_{12}$был синтезирован методом Печини. В методе Печини использовалась мочевина в качестве комплексообразователя. Экспериментально определены состав и структура синтезированного манганита. Впервые рентгенофазным анализом были определены структура, тип сингонии, параметры элементарных ячеек, рентгенографическая плотность манганита: a=7.5472, b= 8.5244, c=5.7836 Å, $V_{un.cell.}$ = 372.090Å^3, Z=4, $\rho_{rad.}$= 7,120 g/cm^3, $\rho_{рус.}$ = 7,09 g/cm^3. Пикнометрическая плотность измерялась 5-6 раз и данные усреднялись. Индифферентной жидкостью служил толуол.

Ключевые слова:*ванганит, кристаллическая система, радиографическая и пикнометрическая плотность, параметры элементарной ячейки*

$Bi_{0.3}Dy_{0.7}Mn_7O_{12}$ МАНГАНИТІНІҢ СИНТЕЗІ ЖӘНЕ ҚҰРЫЛЫСЫ

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Күрделі аралас оксидті $Bi_{0.3}Dy_{0.7}Mn_7O_{12}$ манганиті печини әдісі арқылы синтезделініп алынды. Печини әдісінде кешен түзуші ретінде мочевина қолданылды. Тәжірибелік түрде синтезделген манганиттің құрамы мен құрылысы анықталды. Алғаш рет рентген фазалық талдау арқылы манганитттің құрылысы, сингония типі, қарапайым ұяшық параметрлері, ренгендік тығыздығы анықталды: a=7.5472, b= 8.5244, c=5.7836 Å, $V_{un.cell.}$ = 372.090Å^3, Z=4, $\rho_{rad.}$= 7,120 g/cm^3, $\rho_{рус.}$ = 7,09 g/cm^3. Пикнометрлік тығыздығы 5-6 рет өлшеніп, мәліметтердің орташа мәні алынды. Индифферентті сұйықтық ретінде толуол қолданылды.

Түйін сөздер: *манганит, кристалдық құрылыс, радиографиялық және пикнометрлік тығыздық, қарапайым ұяшық параметрлері*