Crystal Chemistry and Structures determination of mixed valence multiphase of a novel $Bi_{2.5}Cd_{3.125}La_{1.25}N_{0.25}O_{1.25}$ by *Ab Initio* Method via Powder XRD

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ABSTRACT

Analysis of multiphase $Bi_{2.5}Cd_{3.125}La_{1.25}N_{0.25}O_{1.25}$ samples produced under argon or in air, using laboratory X-ray powder diffraction (XRD) revealed multiphase phase compositions, together with either perovskite or rutile impurities. Investigations of these samples using diffuse reflectance and X-ray photoelectron spectroscopies revealed mixed Bi/Cd oxidation states for argon-annealed samples and Bi/La oxidation states for air-annealed samples.. Severe intensity reduction of hkl odd XRD peaks from the sample produced in air was found to be the result of Bi sharing the La site. The refined crystal structures for multiphase samples were found to be consistent with the compositions and mixed oxidation states observed by the above spectroscopic measurements. Although the normalized uranium leach rate of the argon-annealed sample was found to be approximately twice that of the air-annealed sample, in 7-day Product Consistency Test the durability of both compounds with respect to leaching by water was found to be excellent. The sample was refined by Rieveld refinement using JANA software package.The refinement parameters are a=3.8266(9) Å b=7.8185(19) Å c=9.948(2) Å α=103.759(19)° β=90.953(18)°,GOF=0.016,Rp=0.1837 and Rwp=0.2309.having triclinic crystal system and space group is P-1.and the structure is centrosymmetric.

Key-words: Crystalsystem,triclinic, ,Rietveld,parameters,JANA, centrosymmetric

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INTRODUCTION

THERE has been a renewed interest in the use of ceramics for the stabilization of long-lived waste isotopes from nuclear power and weapons programs. In particular, ceramics consisting mainly of a pyrochlore-structured titanate1–15 were chosen by the U.S. Department of Energy for the immobilization of surplus impure Pu in the U.S. and Russia in the late 1990s.16 While it has been subsequently argued[1] that pyrochlore-structured zirconates have much more structural resistance to radiation damage arising from the decay of the actinides in the structure than titanate pyrochlores, the zirconates require firing at considerably higher temperatures than the titanates and are much less amenable for the incorporation of the impurities in the surplus Pu.18 Moreover, it was found that the aqueous durability of pyrochlore-structured titanates was not significantly affected even when they were completely amorphized by a-event damage. 19 In the application of immobilizing surplus Pu, twice as much U as Pu is incorporated in the pyrochlore, and in the present work we are concentrating on the incorporation of U in pyrochlore-structured $Bi_{2.5}Cd_{3.125}La_{1.25}N_{0.25}O_{1.25}$. The general perovskite crystal structure describes materials of the type NO3P4Q5R9 structure where the N,P,Q and R species are generally Cd and La and Bi transition and nontransition metal species, respectively. The pyrochlore structure is a superstructure derivative of the simple fluorite structure NO3P4Q5R9 where the N and P cations are ordered on the two distinct cation sites, and the oxygen vacancy resides in the tetrahedral

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interstice between adjacent B-site cations. It has been found that the La-containing pyrochlore stoichiometry, nominally $Bi_{2.5}$ $Cd_{3.125}$ $La_{1.25}$ $N_{0.25}$ $O_{1.25}$. varies depending on t he starting composition, the hot consolidation temperature, and the processing atmosphere.20 The Cd content has been reported to be as high as 1.46 formula units (f.u.).20. The work by Vance et al.22 on substituting Bi for Cd in bismuth cadmium lanthanate oxide ($Bi_{2.5}$ $Cd_{3.125}$ $La_{1.25}$ $N_{0.25}$ $O_{1.25}$.) showed that the pyrochlore structure was preferred when x4B0.6. For the $Bi_{2.5}$ $Cd_{3.125}$ $La_{1.25}$ $N_{0.25}$ $O_{1.25}$ nominal stoichiometry, Vance et al.[2] found that sintering in argon at 1450^0C for several days failed to achieve complete reaction, with minor phases present and the pyrochlore yield not exceeding N 75%. This result was similar to those obtained by Solomah et al.23 and Dickson et al.[3,] although these groups used reducing atmospheres and shorter sintering times. To achieve near single-phase Bi, La and Cd oxide pyrochlore, In the first part of the current work, we target different La valences in the pyrochlore structure, with nominal compositions $Bi_{2.5}$ $Cd_{3.125}$ $La_{1.25}$ $N_{0.25}$ $O_{1.25}$ (targeting La) single oxide. Many metal-rich ternary metal carbides, nitrides, and oxides have structures belonging to a class known as the Nowotny octahedral phases. These M-M'-X (X = , N, 0) compounds are characterized by M-X octahedra which are linked either by corners, edges, or faces to form various one-,two-, or three-dimensional networks. The metal atom sub- lattice is not close-packed, in contrast to the binary M-X compounds. Typically for the Nowotny phases, the M element is an early transition metal, the M' element is a post-transition metal, and the majority are nitrides and oxide [4]. A few compounds have been discovered in which M' is a late transition metal and X is nitride . In the course of our investigations into the ternary system Bi-La-Cd [5] we discovered new ternary nitride and oxides containing alloy which can be classified as oxygen- stabilized Nowotny phases: $Bi_{2.5}$ $Cd_{3.125}$ $La_{1.25}$ $N_{0.25}$ $O_{1.25}$. During previous phase equilibria investigations on the ternary Ti-M '-0 (M' = Mn, Fe, Rh, Ir, Pt) (8-10) and Zr-M'-0 (M' = Fe, Rh, Pd, Ir, Pt) [6] systems, several stable ternary oxides with low oxygen content have been discovered. In each of the cases listed above, a ternary oxide forms which is structural with the triclinic ternary phase, but with the addition of oxygen in a subset of the octahedral holes formed by the Cd atoms. This structure type consists of a three-dimensional network of M6 octahedra linked by sharing common faces. The nominal composition is $Bi_{2.5}$ $Cd_{3.125}$ $La_{1.25}$ $N_{0.25}$ $O_{1.25}$. and there often exists a wide range of oxygen nonstoichiometry. In addition to the Bi-La-Cd structure type, there are several other structure types for ternary oxides containing an early and a late transition metal: [7] the K-carbide type phase with nominal stoichiometry M,MiO, which has been observed for the M-ON-O (M = Bi-La) systems [8] and also as high temperature phases in the La-M'-0 (M' = Bi,Cd and La) systems [9] the filled 08, type (or filled La,Cd with nominal stoichiometry M,M;O which has been found in the Nb-M'-0 (M = Ir, Pt) systems [10] the filled Re,B type with nominal stoichiometry M,M '0 which has been found in the Zr-Ni-0 and the Hf-M'-0 (M' = Co, Ni) systems and finally the perovskite structure with a nominal stoichiometry of M,M'O which has been found in the $Bi_{2.5}$ $Cd_{3.125}$ $La_{1.25}$ $N_{0.25}$ $O_{1.25}$ [11]It is interesting to note that superconductivity has been observed in some of the ternary oxide phases having the .The two new materials reported here do not superconduct above 2 K . In this paper, we report the synthesis and crystal structures of $Bi_{2.5}$ $Cd_{3.125}$ $La_{1.25}$ $N_{0.25}$ $O_{1.25}$. The structures were determined by examination of X-ray powder diffraction data and refined by the Rietveld profile analysis method [12] In this paper, we report the synthesis and crystal structures of $Bi_{2.5}$ $Cd_{3.125}$ $La_{1.25}$ $N_{0.25}$ $O_{1.25}$. by powder XRD followed by ab initio methods and study of the crystal chemistry.

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MATERIALS AND METHODS

All the chemicals used were analytical grade. Typical synthesis process was conducted as follows. The initial polycrystalline mixture of starting materials [Bi2(CO3)3, CdCO3 and La(NO3)3)] as starting materials was grounded with an agate mortar, pressed into a pellet and slowly heated up to 800 °C at a rate of 80 °C h^{-1}. The resulting pellet was reground, repressed and heated at a higher temperature with intermediate regrinding steps. After every annealing process, the resultant sample was checked by PXRD. The PXRD data for the structure determination were collected on a PANalytical X'Pert Pro Alpha-1 equipped with a PIXcel detector (Cu-Kα1 radiation).The powder spectra shown in figure 1. Energy-dispersive X-ray spectroscopy (EDS) was performed using a JEOL 2100F transmission electron microscope[8]. The relative density of the sample before the mechanical grinding was 79 %. Platinum electrodes were connected to the two faces of the pellet via a platinum paste to keep good electric contacts. Impedance spectroscopy measurements were carried out using a Hewlett-Packard 4192a Impedance Analyzer. The impedance spectra were recorded in the 5 Hz-13 MHz frequency range. Electrical conductivity measurements of representative $Bi_{2.5}\,Cd_{3.125}\,La_{1.25}\,N_{0.25}\,O_{1.25}$were carried out by complex impedance spectroscopy with a 1174 Solectron frequency response analyzer coupled to a 1286 Solartron electrochemical interface. Pellets of about 14 mm diameter and 1 mm thickness were prepared by cold pressing of a mechanically activated powder mixture with the composition: $Bi_{2.5}\,Cd_{3.125}\,La_{1.25}\,N_{0.25}\,O_{1.25}$was form the phase, the pellets were heated at 7003C during 12 h and slowly cooled to room temperature. This synthesis method was employed to improve the ceramic quality, as it has been shown for other materials [13].The formed phases and crystallinity were studied by X-ray powder diffraction. Platinum electrodes were deposited on the two faces by sputtering, and measurements were carried out in the temperature range 200-6503C, at steady temperatures, with pellets under air flow. The frequency range was fixed.The Rietveld refinement carried out JANA software package and VBS is determined by using SIR 92 package.Density was measured by Archimedes principl and ionic properties determined with help of Arrhenius relation.

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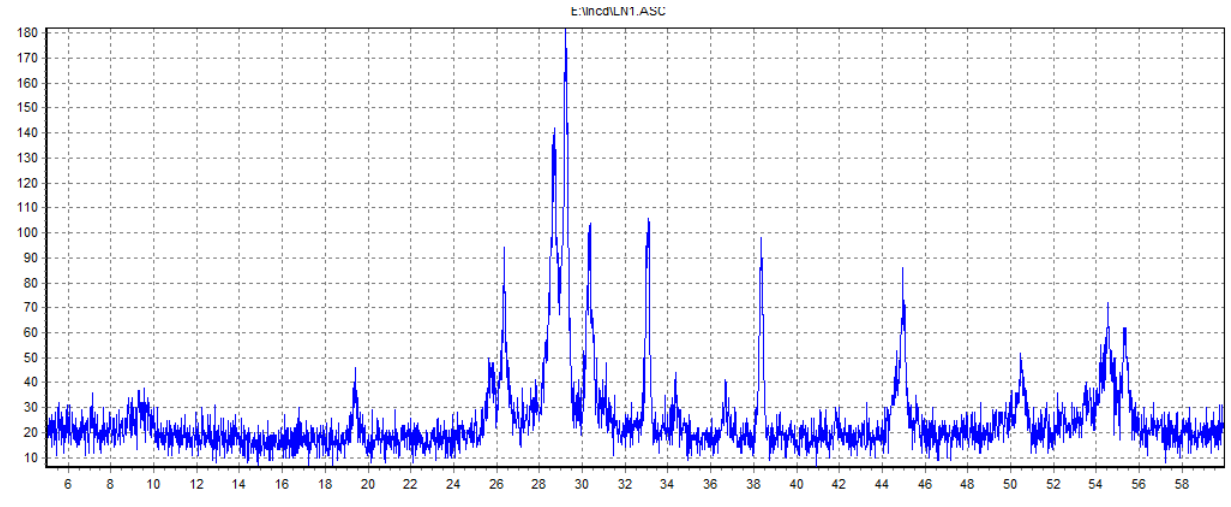


Figure1. Powder spectra of $Bi_{2.5}\,Cd_{3.125}\,La_{1.25}\,N_{0.25}\,O_{1.25}$

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RESULTS AND DISCUSSIONS

Several attempts to grow single crystals of the title compound were unsuccessful, but the powder quality had to be improved to be suitable for powder XRD analysis. The powder resulting from the reaction of $Bi_{2.5}$ $Cd_{3.125}$ $La_{1.25}$ $N_{0.25}$ $O_{1.25}$air presents an average crystal size of 60 nm, which is too small for a satisfactory structure solution. As a mineralize, La ion was found to be helpful to increase the crystal size. Small amounts of Mo improved the crystal growth slightly. Finally, an almost single phase powder of $Bi_{2.5}$ $Cd_{3.125}$ $La_{1.25}$ $N_{0.25}$ $O_{1.25}$was obtained with an average coherence length of Bi 120 nm.This sample was found to be suitable to solve the structure from conventional powder diffraction data. Indexing the 25 first reflections Figure 1 gave an tetragonal unit cell. A whole pattern fitting using the Le Bail method,[14] after crystallographic transformations, suggested the space group *P-1* as the one with the highest possible symmetry. The structure was solved in this space group by using the Diamond program package, as described in the materials and methods . The starting model was based on five bismuth ions and two rigid Cd ions octahedral. After convergence, the structure was refined with JANA (see Figure 2). The refinement parameters are summarized in Table 2 for 34 atomic coordinates and 3 isotropic temperature coefficients. Structural data are given in Table 3 and selected interatomic distances are given in Table 4 together with the calculated valence bond sum.[15] The values for Bi scatter between 1.6 and 2.7. The unusual high valence bond sum (VBS) of Mn+2 ion is probably related to the lowest coordination number 6 and short Bi–O distances. In difference to the other Bi ions in the structure, Bi3 is located in a tunnel-shaped arrangement of oxygen atoms parallel to the *b* axis. The high VBS value marks probably the limit of the stability of this structure. The VBS values for iodine are in the expected range (Table 4). Figure 3 shows the structural arrangement perpendicular to the *b* axis and Figure 4 the oxygen channel around La ion . The structure can be described by cations forming a two-dimensional network around an almost pentagonal hole, which is covered by the layers above and below. Oxygen atoms O5–O8 are located within the layer and oxygen atoms O1–O4 connect the different layers. All oxygen atoms are part of the two Bi2O3 octahedra. The average Bi–O distance is 2.69(1) Å, the shortest distance Bi3–O8 was calculated to be 2.54(1) Å. Similar short distances were found in $Bi_{2.5}$ $Cd_{3.125}$ $La_{1.25}$ $N_{0.25}$ $O_{1.25}$ [16] Coordination numbers (and average distances) are 7 for Bi2 (2.64 Å), 10 for Bi1 (2.93 Å), Mn3 (2.89 Å), and Cd (2.87 Å) and 11 for La (3.02 Å). It should be noted that the coordination number of 7 around Ba2 is unexpected. Nevertheless a geometrically similar environment was observed around zirconium in monoclinic LaO[17]. The average La–O distances of the $Bi_{2.5}$ $Cd_{3.125}$ $La_{1.25}$ $N_{0.25}$ $O_{1.25}$ octahedral are 1.87 and 1.91 Å, as observed for other bismuth ions are shown in table2

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STRUCTURE SOLUTION

REITVELD REFINEMENT

Rietveld refinement of orthorhombic La0.5.Cd0.125.Zr0.125.S.0.76.O $_{0.25}$.N $_{0.125}$ against XRD data for structural determination proved difficult, due to a combination of preferred orientation of the plate-like crystallites in flat-plate geometry[17]. The framework structure of $Bi_{16}Mn_{12}Mo_3O16$ was first examined by ab initio structure determination method using the powder XRD data. Refined by JANA software The initial lattice parameters were determined

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to be parameters $Bi_{2.5}$ $Cd_{3.125}$ $La_{1.25}$ $N_{0.25}$ $O_{1.25}$ formula weight 1070.9 g/mol,crystal system triclinic crystal system and refine parameters are a=3.8266(9) Å b=7.8185(19) Å c=9.948(2) Å α=103.759(19)° β=90.953(18) and volume=283.62 space group is P -1 by an indexing procedure using the program TREOR15 in EXPO2004.16 The most probable space group was suggested to be P -1 Next, the integrated intensities were extracted by the Le Bail method using the program Jana2006.14 A profile function and background function of the Le Bail method used in the present study were pseudo-Voigt function and 20th order Legendre function, respectively. An initial structure model was then obtained by the charge flipping (CF) method17 using the extracted integrated intensities. Although the Cd site could not be clearly determined by the CF and particle size dermined with help od Debye- Sherer relation.The particle size is 75.513 like nanoparticle.

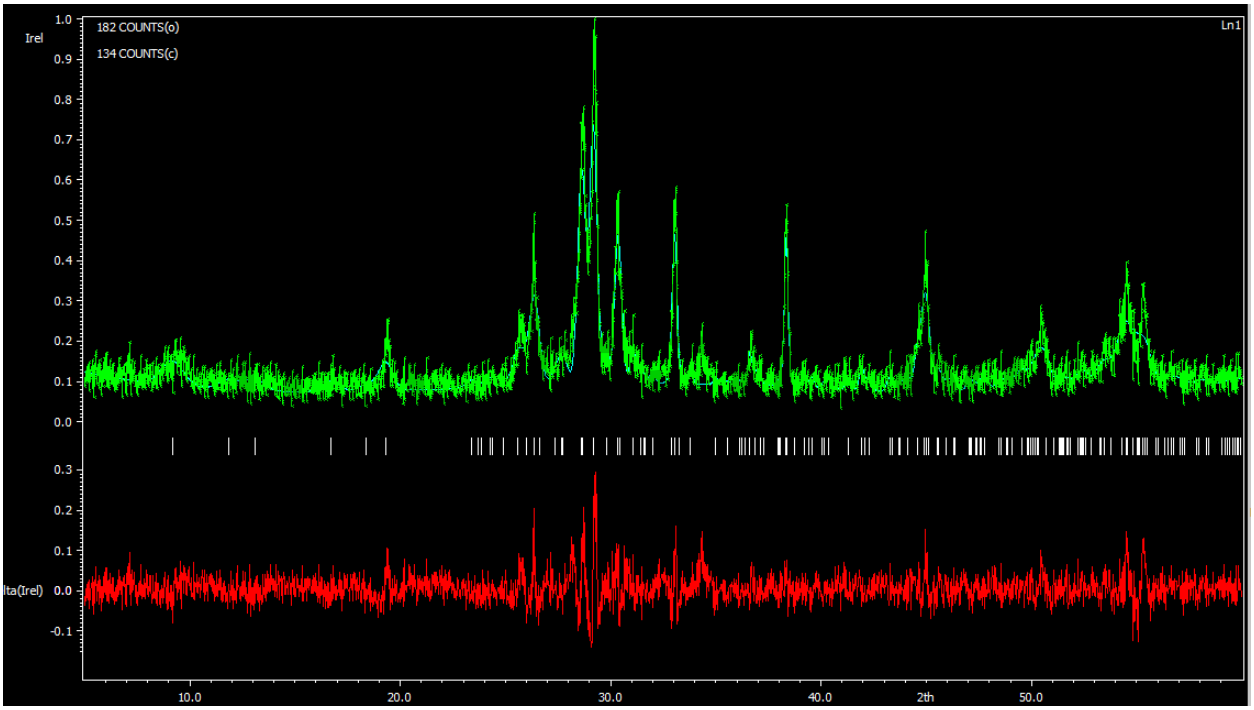


Figure 2. Graphical representation of the result from Rietveld refinement with X-ray powder data. Vertical bars indicate positions of the Bragg reflections for $Bi_{2.5}$ $Cd_{3.125}$ $La_{1.25}$ $N_{0.25}$ $O_{1.25}$ dots mark the observed intensities and the solid line gives the calculated intensity curve. The deviations between the observed and the calculated intensities from the refined model are shown by the difference plot in the lower part of the diagram.

Table 1. crystallographic Phase data

Formula sum	$Bi_{2.5}$ $Cd_{3.125}$ $La_{1.25}$ $N_{0.25}$ $O_{1.25}$
Formula weight	1070.9 g/mol
Crystal system	triclinic
Space-group	P -1 (2)

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Cell parameters	a=3.8266(9) Å b=7.8185(19) Å c=9.948(2) Å α=103.759(19)° β=90.953(18)° γ=100.566(15)°
Cell ratio	a/b=0.4894 b/c=0.7859 c/a=2.5997
Cell volume	283.62(12) $Å^3$
Z	2
Calc. density	12.5391 g/cm^3
Meas.density	12.4532g/cm^3
Pearson code	aP44
Formula type	NO3P4Q5R9
Wyckoff sequence	i22
Index	0≤h≤1,-1≤k≤2,0≤l≤2

Table 2 Fraction Atomic parameters

Atom	Ox.	Wyck.	Site	.	x/a	y/b	z/c	U [$Å^2$]
Bi1	+3	2i	1		0.27705	0.26116	0.13943	0.0380
Bi2	+3	2i	1		0.10063	0.39325	-0.00055	0.0380
Bi3	+3	2i	1		0.58915	0.46951	0.31604	0.0380
La1	+3	2i	1		0.37814	0.00054	0.66298	0.0380
Cd1	+2	2i	1		0.95885	0.68755	0.36387	0.0380
Cd2	+2	2i	1		0.40674	-0.03119	0.12348	0.0380
Cd3	+2	2i	1		0.91315	0.93903	0.50863	0.0380
Cd4	+2	2i	1		0.25488	0.80469	0.56159	0.0380
Cd5	+2	2i	1		0.05946	0.48088	0.19231	0.0380
Cd6	+2	2i	1		0.06050	0.08430	0.27026	0.0380
Cd7	+2	2i	1		0.18864	0.17131	-0.16296	0.0380
Cd8	+2	2i	1		0.53616	0.63432	0.49156	0.0380
Cd9	+2	2i	1		0.60961	0.51877	0.14364	0.0380
O1	-2	2i	1		0.53340	0.31438	-0.03675	0.0380
O2	-2	2i	1		0.95323	0.65192	0.65395	0.0380
O3	-2	2i	1		0.35484	0.19922	0.30080	0.0380
O4	-2	2i	1		-0.25740	0.20137	0.23049	0.0380
N1	-3	2i	1		-0.20104	0.18762	0.08096	0.0380
N2	-3	2i	1		0.47599	0.75459	0.38085	0.0380
N3	-3	2i	1		0.26658	0.68956	0.18338	0.0380
N4	-3	2i	1		0.90068	0.53729	0.53090	0.0380
N5	-3	2i	1		-0.13115	0.12686	-0.28319	0.0380

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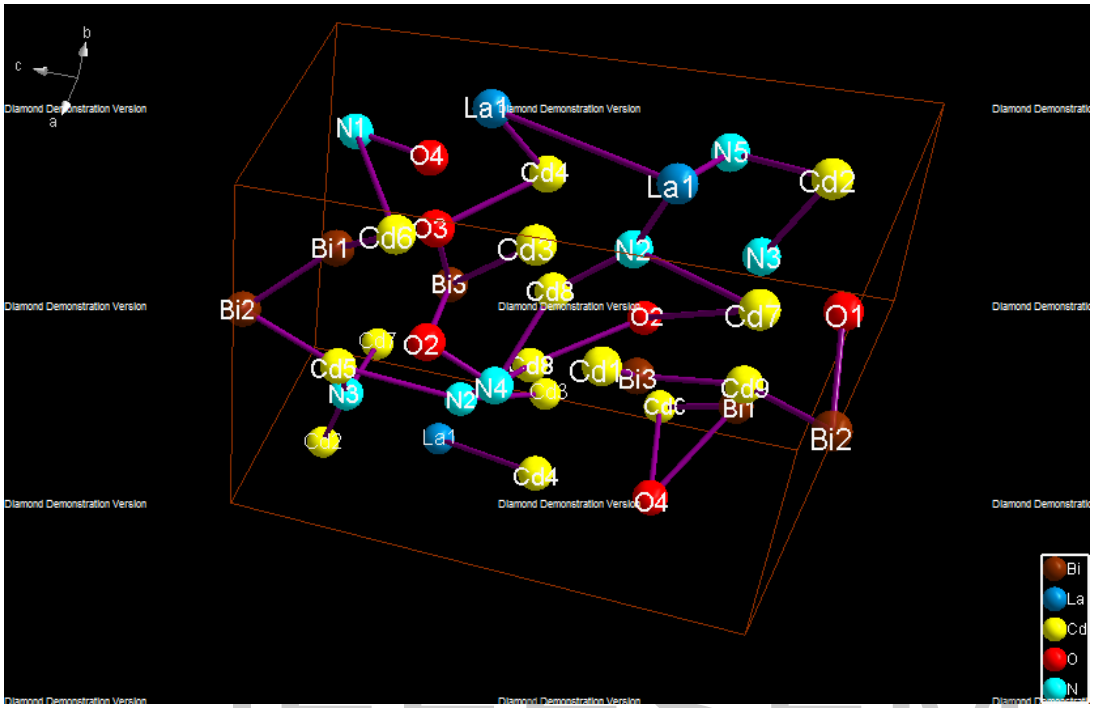


Figure 3.3D structure of $Bi_{2.5}Cd_{3.125}La_{1.25}N_{0.25}O_{1.25}$

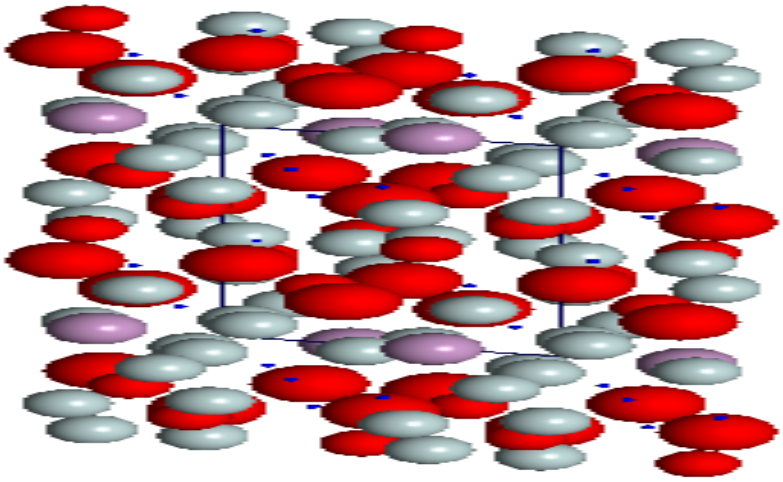


Figure 4.Projection toward the (132) plane of the $Bi_{2.5}Cd_{3.125}La_{1.25}N_{0.25}O_{1.25}$ layer type structure.

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Table 2.Selected bond angles

Atom1	Atom2	Atom3	Angles(A)
Bi1	O3	N1	109.528
	O3	Cd5	105.550
Bi2	O1	Cd5	111.591
	O1	Bi2	138.505
La1	Cd4	Cd6	77.184
	N2	O3	135.883
	N2	Cd7	78.920
	N3	Cd2	73.389
	N3	Bi2	75.792
N1	O4	Bi1	84.638
	O4	O1	122.616
	O1	N3	Cd778.642
	N3	Bi2	123.874
	N3	Cd9	66.020
	N3	N1	108.423
	N3	Cd9	127.998
	N3	Bi1	168.002
	N3	Bi2	68.383
	N3	Cd2	73.389
	N3	Bi2	75.792
	Cd7	Bi2	71.139
	Cd7	Cd9	76.162
	Cd7	N1	116.428
	Cd7	Cd9	142.161
	Cd7	Bi1	95.725
	Cd7	Bi2	142.150
	Cd7	Cd2	62.649
	Cd7	Bi2	137.572
	Bi2	Cd9	61.491
	Bi2	N1	127.172
	Bi2	Cd9	71.355
	Bi2	Bi1	62.809
	Bi2	Bi2	143.683
	Bi2	Cd2	125.890
	Bi2	Bi2	96.226
	Cd9	N1	165.764
	Cd9	Cd9	90.325
	Cd9	Bi1	123.267
	Cd9	Bi2	105.316
	Cd9	Cd2	126.537
	Cd9	Bi2	62.680
	N1	Cd9	83.153

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	N1	Bi1	64.397
	N1	Bi2	60.811
	N1	Cd2	60.165
	N1	Bi2	103.590
	Cd9	Bi1	62.323
	Cd9	Bi2	75.304
	Cd9	Cd2	142.970
	Cd9	Bi2	52.346
	Bi1	Bi2	112.823
	Bi1	Cd2	94.618
	Bi1	Bi2	114.633
	Bi2	Cd2	89.730
	Bi2	Bi2	50.822
	Cd2	Bi2	136.955
O2	N4	O4	145.366
	N4	O3	117.525
	N4	Cd4	84.815
	N4	Cd6	135.998
	N4	Cd5	110.650
	N4	Bi3	84.556
	N4	Bi3	85.834
	N4	Cd8	53.703
	N4	N4	23.750
	N4	Bi1	145.120
	O4	O3	88.999
	O4	Cd4	70.390
	O4	Cd6	55.024

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In order to locate anionic vacancies, we first assumed that vacancies were distributed on one oxygen site only. Therefore, 12 Rietveld refinements were successively performed by fixing the appropriate occupancy on oxygen atom Oi (i = 1, 12). In addition to R-factor values noted after each refinement, bond valence calculations (BVC)21 were achieved using the BondStr program implemented in the JANAsuite with the following parameters B = 0.37 Å and Ro = 2.172 and 1.921 Å for La3+−O2− , Bi^{+3}–O^{2-} and Cd^{+2}, respectively. The sum of squares of deviations (SSD) between calculated and expected (+3 for La, +2 for Cd, and +3 for Bi) bond valences over the 20 atoms of the structure was used to discriminate between the results. The best solution, which minimizes the SSD, was obtained with vacancies located on oxygen O5. The best R-factor values were also found with this atomic occupancy setting. The final combined Rietveld refinement of the appropriate atomic coordinates and temperature factors along with the cell constants, scale factors, zero-points, peak-shape parameters, and background parameters converged to the R-factor values gathered in Table 4. The complete crystallographic information on the $Bi_{2.5}$ $Cd_{3.125}$ $La_{1.25}$ $N_{0.25}$ $O_{1.25}$compound structure is shown in Table 3. Selected La−O, Bi−O and Cd-O distances and BVC results are given in Tables 5 and 6, respectively. The observed, calculated, and difference profiles are illustrated in Figure 3 Instead of the initial composition "$Bi_{2.5}$ $Cd_{3.125}$ $La_{1.25}$ $N_{0.25}$ $O_{1.25}$compound (66.7% La2O3) by taking into account that Z = 2, the final result is $Bi_{2.5}$ $Cd_{3.125}$ $La_{1.25}$ $N_{0.25}$ $O_{1.25}$compound with a slight difference in composition (68% Bi2O3). The same

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feature was previously observed in the case of the compound (47.4% La2O3), whereas the predicted composition was $Bi_{2.5}Cd_{3.125}La_{1.25}N_{0.25}O_{1.25}$compound (46.7% La2O3). The consistency of the structure of $Bi_{2.5}Cd_{3.125}La_{1.25}N_{0.25}O_{1.25}$compound was verified by considering BVC results (Table 4), which are quite satisfactory. The minimum distance between oxygen atoms is also a structural parameter that needs to be examined: some oxygen−oxygen distances were found to be smaller than 2.80 Å, which is the theoretical oxygen−oxygen distance when using the ionic radii of O2− (1.40 Å). These short oxygen−oxygen distances, and N-N distance gathered in Table 7, are commonly observed in fluorite related structures.

Table 4 Calculated Bond Valences of $Bi_{2.5}Cd_{3.125}La_{1.25}N_{0.25}O_{1.25}$compound of the Structural Model Proposed from the Refinement

Atom	coordination	BVC	Atom	Coordination	BVC
Bi1	3	2.32	O3	4	4.217(4)
Bi2	3.25	2.12	O2	4	.4.31(4)
La1	6.88	2.92	O4	4	1.92(4)
La2	6	2.97	O5	4	1.54(4)
Cd	6	1.34	N1	4	1.56(4)
Cd	4	1/53	N2	4	1.65(4)
Cd	4	1.67	O5	4	1.32

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The interaction converged with an R factor of 18.15% and the final electron density shows a P-1 (123) symmetry with a 5% error. The program of EDMA was then used to automatically assign atomic positions. Four unique heavy atomic positions were found and the heaviest one was assigned as Bi while the others were considered as La and Cd. Due to the existence of heavy atoms, all oxygen positions were ambiguous in the electron density map of this stage. To locate the oxygen atoms, a Monte-Carlo based simulated annealing process with the program Diamond package was applied. For each annealing process, various atomic coordinates were randomly assigned as the initial positions of the oxygen atoms [18]. The annealing process was restarted after finding a few oxygen positions, until all oxygen positions were found to be reasonable shown in figure 3 and perovskite structure and centrosymmetry shown in figure 4.

IONIC PROPERTY

. Represented on the Arrhenius plot figure 6 the ionic conductivity of $Bi_{2.5}Cd_{3.125}La_{1.25}N_{0.25}O_{1.25}$ is increase with increasing temperature. In addition, the curve exhibits two slopes that can be indicative of a phase transition or a change of charge carrier. On the slope at high temperatures, the activation energy calculated by Arrhenius plot shown in figure 6 (2.10 eV) is characteristic of an oxygen conduction.[19]

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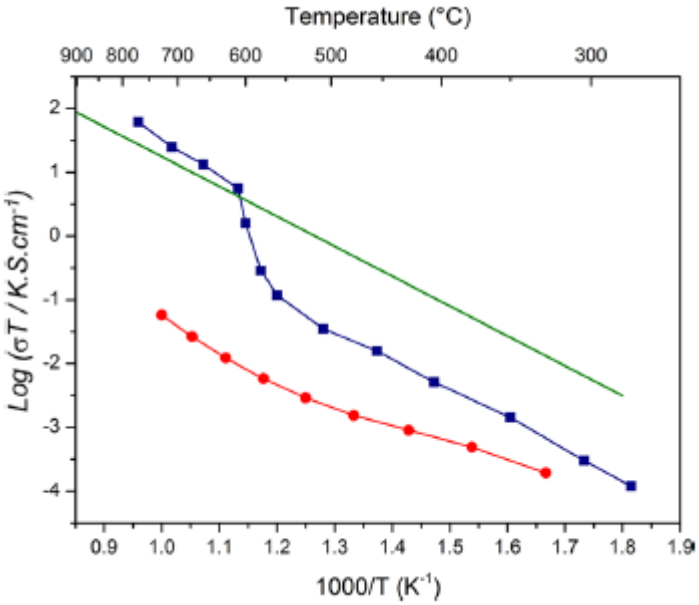


Figure 6. Arrhenius plot of $Bi_{2.5}\,Cd_{3.125}\,La_{1.25}\,N_{0.25}\,O_{1.25}$

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Observation of the pellets75%. A pure ionic conduction cannot be concluded because no electrode polarization was visible in the range of frequency studied[20,21]. Our primary results of the ionic properties of the synthesized compound should be compared and may be reinvestigated with the possibility of protonic conduction. According to the particle size the cited oxide is show nanomaterial and can be used in super conductor materials too.

CONCLUSIONS

Materials science is different from condensed matter physics in the sense that materials science deals with engineered materials that exist and are employed in the real world beyond the well-posed boundary conditions of laboratory tests or theoretical models. The microstructure of metal alloys for structural applications is of key importance for obtaining the target mix of properties. Microstructures are intricately complex as they are allowed the many degrees of freedom provided by the wide variety of lattice defects, which are the basic building blocks of the microstructural phenomenon. The scientific way of dealing with complexity is by applying statistical methods, which are capable of revealing certain patterns in the diffuse reality of experimental data. In this Article, the synthesis and the structural investigation of the so-called $Bi_{2.5}\,Cd_{3.125}\,La_{1.25}\,N_{0.25}\,O_{1.25}$compound by ab ignition methos via powder XRD pattern is reported. It exhibits a new structural type and crystallizes in the triclinic crystal system P-1 with the cell parameters: a=3.8266(9) Å b=7.8185(19) Å c=9.948(2) Å α=103.759(19)° β=90.953(18)° γ=100.566(15)° The final composition, determined from structural resolution using combined synchrotron and X-ray diffraction powder data, was found to be $Bi_{2.5}\,Cd_{3.125}\,La_{1.25}\,N_{0.25}\,O_{1.25}$ with centrosymmetric structure.This material is significantly ionic conduction with increase the temperature.

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REFERENCES

[1] T. Dan Vu, Firas Krichen, Maud Barre, Sandrine Coste, Alain Jouanneaux, EmmanuelleSuard,Andrew Fitch,(2019) and François Goutenoire , Ab Initio Structure Determination of La34Mo8O75 Using Powder X-ray and Neutron Diffraction Data, DOI: 10.1021/acs.cgd.8b01552 Cryst. Growth Des. XXXX, XXX, XXX−XXX
[2]. Kharton, V. V.; Marques, F. M. B.; Atkinson, A(2004). Transport properties of solid oxide electrolyte ceramics: a brief review. Solid State Ionics 174, 135−149.
[3] Lacorre, P.; Goutenoire, F.; Bohnke, O.; Retoux, R.; Laligant, Y(2000). Designing fast oxide-ion conductors based on La2Mo2O9. Nature 404, 856−858.
[4] Goutenoire, F.; Isnard, O.; Retoux, R.; Lacorre, P(2000). Crystal Structure of La2Mo2O9, a New Fast Oxide-Ion Conductor. Chem. Mater. 12, 2575−2580.
[5] Dubois, F.; Goutenoire, F.; Laligant, Y.; Suard, E.; Lacorre, P.(2001) Ab-Initio Determination of La2Mo4O15 Crystal Structure from X-rays and Neutron Powder Diffraction. J. Solid State Chem. 159, 228− 233.
[6] Brize, V.; Georges, S.; Kodjikian, S.; Suard, E.; Goutenoire, F. La6Mo8O33: a new ordered defect scheelite superstructure. J. Solid State Chem. 2004, 177, 2617−2627.
[7] Vu, T. D.; Krichen, F.; Barre, M.; Adil, K.; Jouanneaux, A.; Goutenoire, F(2019). Two ternary phase diagrams of La2O3-Nb2O5-WO3 and La2O3-Nb2O5-MoO3 systems. In preparation.
[8] Fournier, J. P.; Kohlmuller, R. Etude des(1970) systemes La2O3-MoO3, Y2O3-MoO3, et des phases Ln6MoO12. Bull. Soc. Chim. Fr. 12, 4277−4279.
[9]. Galven, C.; Mounier, D.; Bouchevreau, B.; Suard, E.; Bulou, A.; Crosnier-Lopez, M.-P.; Le Berre, F(2016). Phase Transitions in thRuddlesden−Popper Phase Li2CaTa2O7: X-ray and Neutron Powder Thermodiffraction, TEM, Raman, and SHG Experiments. Inorg. Chem. 55, 2309−2323.
[10]. Degen, T.; Sadki, M.; Bron, E.; König, U.; Nenert, G(2014). The HighScore suite. Powder Diffr. 29, 13−18.
[11] Werner, P. E.; Eriksson, L.; Westdahl, M. TREOR(1985), a semiexhaustive trial-and-error powder indexing program for all symmetries. J. Appl. Crystallogr. 18, 367−370.
[12] Boultif, A.; Louer, D.(2004) Powder pattern indexing with the dichotomy method. J. Appl. Crystallogr.37, 724−731.
[13] Le Bail, (2004,). Monte Carlo indexing with Mc Maille. Powder Diffr. 19, 249−254.
[14] Le Bail, A.; Duroy, H.; Fourquet, J. F(1988). Ab Initio Structure Determination of LiSbWO6 by X-ray Powder Diffraction. Mater. Res. Bull. 23, 447−452.
[15] Rodriguez, C. J.(2001) Recent Developments of the Program FULLPROF. Commission on Powder Diffraction (IUCr) Newsletter 26, 12−19.
[16]. Palatinus, L.; Chapuis, G.(2007), SUPERFLIP − a computer program for the solution of crystal structures by charge flipping in arbitrary dimensions. J. Appl. Crystallogr. 40, 786−790.
[17]. Petříček, V.; Dušek, M.; Palatinus, L.(201), Jana2006: general features. Z. Kristallogr. 229, 345−352.
[18] Parashuram Mishra(2011), Synthesis, crystal structure determination and ionic properties of novel BiCa0.5Mg0.5 O2.5 via X- ray powder diffraction data *Crystal Growth. 2041 32 (2011) 2041-204*
[19]. Bimal K. Kanth, Parashuram Mishra[2020], Synthesis and Ab Initio Determination Bi1.256 La0.53 N0.231 O0.521 Zr1.543 Triclinic Structure from Powder X-Ray Diffraction Data,Sch Int J Chem Mater Sci, July, 2020; 3(6): 6
[20] Bimal K. Kanth and Parashuram Mishra,(2020), Synthesis And AB Initio Structure Determination Of Transition Metal Mixed Valence Oxide Cu0.45 Mo1.25 O9 V2.23 By Powder XRD And Study The Physical Property, *International Journal of Engineering And Science Vol.10, Issue 7 (July 2020), PP 58-68*
[21]. Bimal K.Kanth and Parashuram Mishra(2020) Isolation and Crystal Structure determination of Tetragonal Crystal System of Mixed Valence Bi0.5Mn0.64Mo1.5 O 0.41 oxide by Powder XRD*IJES/ 9(3) 2020 56-59*,

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