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New Samarium Oxotellurites: Synthesis and Characteristic

We synthesized samarium oxotellurites $SmMTeO_{4.5}$ (M — Mg, Ba) by the method of ceramic technology with solid-phase interaction (800–1200 °C) of Sm_2O_3 and TeO_2 oxides with $MgCO_3$ and $BaCO_3$ carbonates. The compounds were characterized by X-ray and electrophysics methods. It has been established that the synthesized compounds crystallize in the tetragonal syngony with the following crystallographic characteristics: $SmMgTeO_{4.5}$ — a = 12.226; c = 5.783 Å, $V^0_{un.cell}$ = 864.38 Å^3, Z = 4, $\rho_{xray.}$ = 2.876, $\rho_{pycn.}$ = (2.874±0.002) g·cm^{-3}; $SmBaTeO_{4.5}$ — a = 12.717; c = 6.132Å, $V^0_{un.cell}$ = 991.62Å^3, Z = 4, $\rho_{xray.}$ = 3.264, $\rho_{pycn.}$ = (3.260±0.004) g·cm^{-3}; for both tellurites α = β = γ = 90°. The experimental and calculated values of 2 Th and d-sp, X-ray and pycnometric densities, as well as theoretical and experimental values of unit cell volumes are agreed satisfactorily. Thus, we can confirm correctness and reliability of the results of indexing X-ray patterns of new samarium oxotellurites. We found that increase in the ionic radii from Mg to Ba increases the lattice parameters of the synthesized oxotellurites. Oxotellurites are crystallized in the structural types of distorted perovskite P_m3_m. Based on the given study samarium oxotellurites can have semiconductor and ferroelectric properties.

Keywords: samarium oxotellurites, synthesis, crystallographic characteristics of the compounds, permittivity, resistance.

Introduction

The research object is complex oxides with the perovskite structure ABO_3 and A_2BO_4 (where A is REE and/or AEM; B is Cu, Ti, Cr, Mn, Fe, Co, Ni). It is connected with the possibility of their potential application in various fields of technology. In particular, perovskite oxides are widely used in electrocatalysis [1] and heterogeneous catalysis [2] owing to the low cost, simplicity of the synthesis process, and extraordinary ability to combine a wide range of substituting and alloying elements to modulate their properties. The oxygen sublattice is resistant to oxidizing environments and high temperatures, high electrical conductivity and mobility. Consequently, these materials are used as CO_2 laser cathodes, fuel cell electrodes, oxygen membranes, magnetoresistors and exhaust gas after burning catalysts.

Active researches are carried out to obtain materials with the required properties. The traditional way to modify the properties of inorganic compounds is to vary their composition by partial substitution of components in different sublattices. One of the approaches is based on the concept of the stereochemical effect of lone electron pairs of p-cations in medium oxidation states (I^V, Se^{IV}, Te^{IV}, As^{III}, Sb^{III}, Bi^{III}, Pb^{II}, Tl^{I}, etc.). It often results to the appearance of acentric and microporous structures [3]. In this work [4] we have predicted possible polytypes of the maximum degree of order. As well as we have discussed the crystal chemical relationships between rare earth oxyhalides and lone pair ions.

In this aspect, oxocompounds of tellurium with rare earth elements are of particular interest for research. This interest is caused because tellurium has a stereochemically active lone pair of electrons, and

also tellurium oxo compounds have semiconductor, ferroelectric properties and are nonlinear optical materials [5, 6]. In this regard, the purpose of this work is the synthesis and study of the X-ray and electrophysical properties of new phases — samarium oxotellurites of the composition $SmMTeO_{4.5}$ (M — Mg, Ba).

Experimental

Solid-phase synthesis of compounds was carried out by the ceramic technology method (800–1200 °C) with the interaction of oxides Sm_2O_3 (special purity) and TeO_2 (chemically pure) with carbonates $MgCO_3$, $BaCO_3$ (analytical grade) in stoichiometric ratios according to the method published earlier in this work [6].

X-ray diffraction patterns of the synthesized compounds using an Empyrean powder diffractometer (PANalytical) have been obtained. Data collection was performed using the Data Collector version 7.7 h. We decrypted X-ray patterns and identified phases using a specialized computer program H'Pert HighScore Plus, which provides automated quantitative phase analysis, including measurement, processing, and obtaining results, using all currently accepted analytical models. The phase composition was identified using the Crystallography Open Database and PDF-2. The pycnometric density has been determined according to the procedure [7]. Indifferent liquid was toluene.

We carried out the study of electrical properties — permittivity and resistance in the range of 293–483 K by measuring the electrical capacitance of the samples on an LCR-800 instrument (Taiwan) at an operating frequency of 1; 5 and 10 kHz continuously in dry air in thermostatic mode with holding time at each fixed temperature. The preparation of samples and their study were carried out according to the method published earlier in this work [8].

Results and Discussion

Diffraction patterns of $SmMTeO_{4.5}$ tellurites are shown in Figure 1.

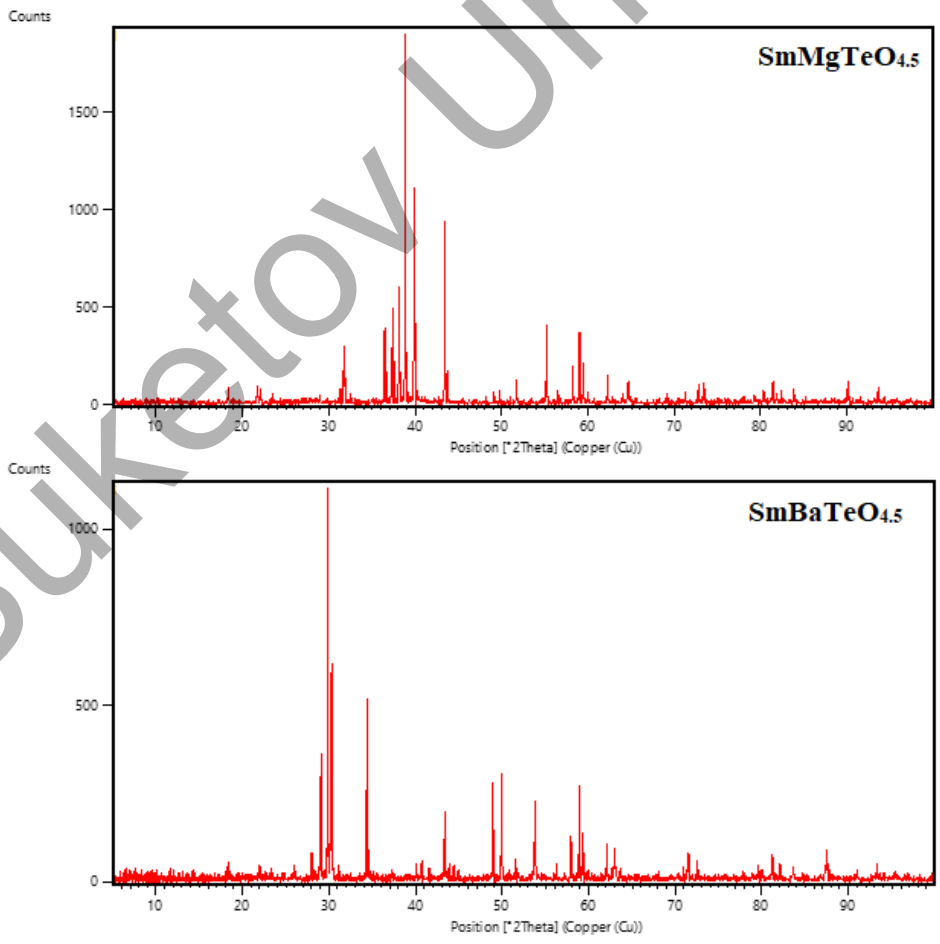

Figure 1. X-ray diffraction patterns of $SmMTeO_{4.5}$ tellurites

Table 1 shows the results of X-ray indexing of the compounds.

Table 1

The results of indexing X-ray patterns of new samarium tellurites $SmMTeO_{4.5}$ (M — Mg, Ba)

h	k	l	2Th. (c) [°]	2Th. (o) [°]	d-sp. (c) [Å]	d-sp. (o) [Å]
$SmMgTeO_{4.5}$						
1	1	1	18.4354	18.3813	4.808763	4.822810
3	0	0	21.7942	21.7940	4.074674	4.074707
1	0	2	31.7540	31.7421	2.815693	2.816724
4	2	1	36.3194	36.4737	2.471547	2.461447
5	0	0	36.7308	36.6132	2.444805	2.452383
2	2	2	37.3710	37.3907	2.404382	2.403161
5	1	0	37.4850	37.5239	2.397328	2.394932
3	0	2	38.1144	38.1731	2.359178	2.355685
3	1	2	38.8456	38.8713	2.316432	2.314963
5	0	1	40.0023	39.9413	2.252079	2.255378
4	1	2	43.6769	43.6857	2.070748	2.070349
2	2	3	51.8630	51.7294	1.761502	1.765740
6	0	2	55.0857	55.1806	1.665830	1.663189
6	4	1	56.5239	56.5130	1.626808	1.627096
4	2	3	58.5173	58.2330	1.576050	1.583064
6	5	0	58.9659	59.0455	1.565126	1.563204
7	3	1	59.7391	59.4480	1.546702	1.553577
7	0	2	62.0236	62.2384	1.495103	1.490458
6	6	0	64.6473	64.6549	1.440615	1.440466
9	0	1	71.2599	71.2336	1.322293	1.322716
4	1	4	72.6615	72.7986	1.300202	1.298091
9	3	0	73.4271	73.4631	1.288525	1.287981
8	5	2	81.2899	81.3631	1.182583	1.181705
10	3	0	82.2795	82.4266	1.170849	1.169132
5	4	4	83.8338	83.8293	1.153052	1.153103
11	1	1	90.2486	90.0962	1.087012	1.088454
10	4	2	93.6117	93.5834	1.056595	1.056841
$SmBaTeO_{4.5}$						
4	0	0	28.0191	28.0241	3.181951	3.181394
0	0	2	29.1527	29.0980	3.060747	3.066380
3	3	0	29.7568	29.8754	2.999972	2.988340
1	0	2	30.0031	30.2772	2.975909	2.949582
4	2	1	34.7326	34.4057	2.580740	2.604513
4	2	2	43.3801	43.3450	2.084222	2.085833
2	2	3	48.9760	48.9492	1.858368	1.859323
3	1	3	50.0795	49.9677	1.819974	1.823786
4	1	3	53.8114	53.7995	1.702229	1.702578
8	0	0	57.9159	57.9695	1.590976	1.589631
7	0	2	59.0446	58.8784	1.563227	1.567243
5	5	2	59.5328	59.3122	1.551568	1.556812
8	2	1	61.9532	62.0848	1.496632	1.493776
6	0	3	63.1755	63.0113	1.470586	1.474024
8	5	1	71.5574	71.4936	1.317525	1.318543
5	1	4	72.3722	72.5596	1.304685	1.301777
7	7	2	81.0590	81.1581	1.185369	1.184171
8	7	2	87.3902	87.4452	1.115049	1.114489

According to Table 1 and Table 2, the experimental and calculated values of 2Th. and d-sp. and the values of X-ray and pycnometric densities agreed satisfactorily with each other. That confirms the reliability and correctness of the indexing results.

Theoretical cell volumes of the synthesized tellurites were determined using the data on the cell volumes of the oxides contained in them according to the scheme:

$$V°_{un.cell}\,SmMTeO_{4.5} = 0.5\,V°_{un.cell}\,Sm_2O_3 + V°_{un.cell}\,TeO_2 + V°_{un.cell}\,MO\ (M — Mg, Ba). \quad (1)$$

The correctness of the indexing of X-ray diffraction patterns of the synthesized tellurites is also confirmed by the satisfactory agreement between the values calculated for the unit cell volumes of tellurites from the sum of the unit cell volumes of the initial samarium, magnesium (barium) and tellurium oxides borrowed from using the Crystallography Open Database [9] with the calculated cell volumes of the compounds from radiographic data. Thus, for $SmMgTeO_{4.5}$, $V°_{un.cell}$= 896.415 Å^3 (from the sum of $V°_{un.cell}$ of oxides) and 864.38 Å^3 (from indexing data), and for $SmBaTeO_{4.5}$, $V°_{un.cell}$= 987.790 Å^3 (from the sum of $V°_{un.cell}$ oxides) and 991.62 Å^3 (from indexing data).

It was found that with increasing ionic radii from Mg to Ba, the elementary cell volumes of the synthesized tellurites increase. Based on the indexing of X-ray diffraction patterns of the studied tellurites, we established that $SmMgTeO_{4.5}$ and $SmBaTeO_{4.5}$ compounds crystallize in the tetragonal syngony with lattice parameters, which are presented in Table 2.

Table 2

Crystallographic characteristics of $SmMTeO_{4.5}$ compounds

Compound	Type of syngony	Lattice parameters, Å			Z	α, β, γ, degree.	Density, g·cm^{-3}	
		a	*c*	$V^0_{un.cell.\ Å^3}$			xray.	pycn.
$SmMgTeO_{4.5}$	tetragonal	12.226	5.783	864.38	4	90	2.876	2.874±0.002
$SmBaTeO_{4.5}$	tetragonal	12.717	6.132	991.62	4	90	3.264	3.260±0.004

On the basis of H'Pert HighScore Plus we compared X-ray diffraction patterns of the synthesized tellurites with the X-ray parameters [I/I_0, d] of the starting materials and with possible tellurites of this system. It was revealed that the diffraction patterns of new tellurites have no analogs with them. These data further confirm that the synthesized tellurites are new compounds.

The data of X-ray studies show that the synthesized tellurites crystallize in the structural type of distorted perovskite P_m3_m. Therefore, it can be assumed that these compounds can have semiconductor and ferroelectric properties.

As a rule, the temperature dependence of electrophysical properties is observed in ceramic ferroelectrics. For this purpose, we studied the temperature dependences of the permittivity and resistance of the synthesized tellurites in the range of 293–483 K according to the procedure [8].

The value of the permittivity at each temperature was determined by the formula:

$$\varepsilon = C/C_0, \quad (2)$$

where $C_0 = E_0\,s/d$ — the capacitance of the capacitor without the test substance (air).

The dielectric constant of a standard substance, barium titanate $BaTiO_3$ has been measured at frequencies of 1; 5 and 10 kHz for the reliability of the obtained data. The experimental value of the permittivity of $BaTiO_3$ at 293 K at a frequency of 1 kHz, equal to 1296, satisfactorily agrees with its recommended value of 1400 ± 250 [10]. In addition, the observed changes in the electrical conductivity of $BaTiO_3$ at 383 K at all frequencies are also consistent with its transition from the perovskite cubic P_m3_m phase to the tetragonal (polar) ferroelectric phase with sp.gr. P4mm [10].

Increase in the operating frequency from 1 kHz to 10 kHz, only leads to some decrease in resistance and practically does not affect the dielectric constant of the connection. Therefore, we present the results of experiments only at a frequency of 1 kHz.

The results of experimental data on the study of the electrical properties of the compounds are shown in Table 3 and in Figures 2 and 3.

Table 3

Temperature dependence of the electrical properties of tellurites of $SmMTeO_{4.5}$

T, K	C, nF	Ohm	lgR	ε	lgε
$SmMgTeO_{4.5}$					
293	0.0085	370500	5.57	61	1.79
303	0.00851	444700	5.65	61	1.79
313	0.00857	485200	5.69	62	1.79
323	0.00845	225300	5.35	61	1.78
333	0.00774	1917000	6.28	56	1.75
343	0.00767	1656000	6.22	55	1.74
353	0.00745	1228000	6.09	54	1.73
363	0.00735	804900	5.91	53	1.72
373	0.00726	670300	5.83	52	1.72
383	0.00724	521000	5.72	52	1.72
393	0.00717	185100	5.27	52	1.71
403	0.00722	210700	5.32	52	1.72
413	0.00725	264700	5.42	52	1.72
423	0.00726	199700	5.30	52	1.72
433	0.00727	176300	5.25	52	1.72
443	0.00729	228400	5.36	52	1.72
453	0.00734	120800	5.08	53	1.72
463	0.00735	134400	5.13	53	1.72
473	0.0073	191700	5.28	53	1.72
483	0.0074	225700	5.35	53	1.73
$SmBaTeO_{4.5}$					
293	0.0189	146600	5.17	54	1.74
303	0.01875	93480	4.97	54	1.73
313	0.01901	282500	5.45	55	1.74
323	0.02301	1591000	6.20	66	1.82
333	0.0335	2700000	6.43	96	1.98
343	0.07076	2635000	6.42	204	2.31
353	0.12886	2129000	6.33	371	2.57
363	0.15916	1946000	6.29	458	2.66
373	0.03888	2620000	6.42	112	2.05
383	0.021105	736600	5.87	61	1.78
393	0.0197	220800	5.34	57	1.75
403	0.01967	1733000	6.24	57	1.75
413	0.01968	61580	4.79	57	1.75
423	0.01983	56940	4.76	57	1.76
433	0.01986	61680	4.79	57	1.76
443	0.02002	113200	5.05	58	1.76
453	0.02001	75830	4.88	58	1.76
463	0.02014	73200	4.86	58	1.76
473	0.02018	57020	4.76	58	1.76
483	0.02028	56020	4.75	58	1.77

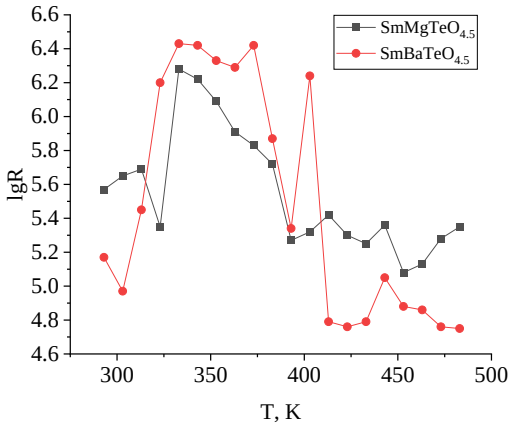

Figure 2. Temperature dependences of resistance

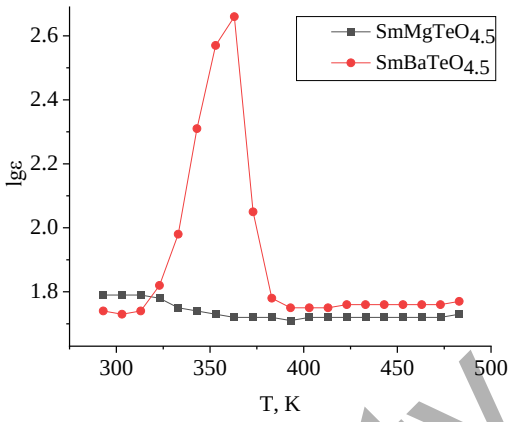

Figure 3. Temperature dependences of dielectric permittivity

An analysis of the data in Table 3 and Figures 2 and 3 shows that with increasing temperature, significant changes in the electrophysical characteristics of tellurites were found, and, as is typical for ceramic materials, such changes occur in a certain temperature range.

$SmMgTeO_{4.5}$. The dielectric constant is low. The resistances of $SmMgTeO_{4.5}$ in the interval 293–313 K and 323–333 K increase in the same way as in metals, respectively. The resistance in the range of 293–313 K increases. The rise is observed in the range of 313–323 K, at which the resistance declines with a minimum at 323 K. Then, at 333–393 K, the resistance decreases again (electrical conductivity increases), i.e. semiconductor conductivity appears.

The calculation of the band gap (ΔE) of the test compound was determined by the formula:

$$\Delta E = \frac{2kT_1T_2}{0.43(T_2 - T_1)} \lg \frac{R_1}{R_2}, \quad (3)$$

where k — the Boltzmann constant, equal to $8.6173303 \cdot 10^{-5}$ eV·K^{-1}; R_1 and R_2 — resistances at temperatures T_1 and T_2, respectively. The band gap (ΔE) calculated by formula (3) for $SmMgTeO_{4.5}$ tellurite in the range of 333–393 K is 1.04 eV and can be attributed to narrow-probe semiconductors. In the range 443-483 K we observe variable conductivity. As the frequency increases, the resistance decreases.

$SmBaTeO_{4.5}$. This compound in the range of 303–333 K exhibits metal-like tendency, and at 333–363 K, as well as at 373–393 K and at 443–483 K — semiconductor conductivity. In the range 433–483 K we observe variable conductivity. The band gap (ΔE) calculated by formula (3) for $SmBaTeO_{4.5}$ tellurite is equal to 1.65 eV within 333–363 K, and 2.27 eV within 443–483 K. The tellurite can be attributed to a narrow-probe semiconductor.

Studies of the temperature dependence of the permittivity and resistance of double samarium tellurites have shown that these compounds can have semiconductor and ferroelectric properties. As a rule, the temperature dependence of electrophysical properties is observed in ceramic ferroelectrics. The observed anomalous jumps on the curves of the temperature dependence of the resistance of the compounds indicate Type II phase transitions, conditioned by the semiconductor and ferroelectric properties of the new double samarium tellurites [11–13]. It is known that an abrupt transition is accompanied by a structure that provides an anomalously fast three-dimensional diffusion of cations [14]. Therefore we can state that these compounds have phase transitions. The curves of the temperature dependence of the permittivity of $SmBaTeO_{4.5}$ (Fig. 3) has a λ — shaped form, which corresponds, according to [15], a ferroelectric phase transition. It is known that the ferroelectric transition temperature (Curie temperature — T_k) for barium titanate $BaTiO_3$ is 406 K, and for polycrystalline ceramics of the perovskite group is in the range of 453–573 K [16]. This fact indirectly indicates that barium tellurite $SmBaTeO_{4.5}$ undergoes a ferroelectric phase transition.

Conclusions

For the first time, samarium oxotellurites $SmMgTeO_{4.5}$ and $SmBaTeO_{4.5}$ were synthesized using the ceramic technology method. Syngony types, unit cell parameters, X-ray and pycnometric densities were determined by X-ray phase analysis method. It was found that with an increase in the ionic radii from Mg to Ba, the lattice parameters of the synthesized tellurites increase.

We studied the temperature dependences of the permittivity and resistance of tellurites in the temperature range 293–483 K and calculated the band gap of the compounds.

Tellurites crystallize in the structural types of distorted perovskite $P_m\ 3_m$ and exhibit semiconductor and ferroelectric properties.

The results obtained can be used for predicting, synthesizing and studying new derivatives of tellurium and rare earth elements and are of interest for electronic technology. The synthesized samarium oxo tellurites can find application in the field of creating materials with ferroelectric properties.

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