SYNTHESIS AND PROPERTIES OF $NdMe^{II}TeO_{4.5}$ (Me^{II} – Ca,Ba) TELLURITES

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ABSTRACT

The aim of this work was to synthesize and investigate the roentgenographic and electro-physical properties of new phases, namely double tellurites of neodymium. Oxotellurites of the composition $NdMe^{II}TeO_{4.5}$ (Me^{II} - Ca, Ba) were synthesized by the method of ceramic technology with high-temperature interaction of Nd_2O_3, TeO_2 oxides with carbonates of alkaline earth metals in the interval of 800°C - 1200°C. Roentgenograms of the synthesized compounds were obtained using an Empyrean powder diffractometer by PANalytical and indication was determined using the X'Pert HighScore Plus program. It was found that the synthesized tellurites were crystallized in tetragonal syngony. The unit cell parameters, roentgen and pycnometric densities of the compounds were determined. It was revealed that with increasing ionic radii from Ca to Ba, the lattice parameters and unit cell volumes of synthesized tellurites increase. The assumed structure of the synthesized double tellurites of neodymium is a distorted perovskite structure. A study of the temperature dependence of the dielectric permittivity and electrical resistance of tellurites of neodymium-alkaline earth metals established that they can possess semiconducting properties at 293 K - 483 K. Temperature coefficients of resistance and forbidden zone width of tellurites were calculated.

Keywords: neodymium, tellurites, synthesis, X-ray phase analysis, dielectric permittivity, electrical resistance.

INTRODUCTION

Compounds based on rare-earth elements (REEs) oxides due to the electronic structure of lanthanides have a unique combination of electrical, magnetic, thermal, optical and other properties, which can be widely used in modern microelectronics and many areas of modern technology in creating multifunctional systems [1 - 6]. The variety of these properties depends on the composition, structure, and production method of the oxide.

At present, active research is being carried out with the aim of obtaining materials with the required properties [7 - 9]. The traditional way to modify the properties of inorganic compounds consists in changing their composition by partial substitution of components in different sublattices.

In this aspect, tellurium oxo-compounds with rare-earth elements are of some interest for research. This interest is caused by the fact that tellurium has a stereochemically active lone pair of electrons, as well as that tellurium oxo-compounds are nonlinear optical materials and possess semiconducting, ferroelectric properties [10 - 13].

However, by the time this paper was written, there were virtually no data on the study of such systems involving tellurium. It should be noted that [14, 15] described the synthesis and characteristics of some tellurites mixed with metals, but without the participation of REEs. Double tellurites, in which

expensive REEs are partially replaced by cheaper metals (alkali and alkaline earth metals) are of particular interest. Obtaining such tellurites with similar electrical and magnetic properties would be more economical and affordable.

Our previous radiographic, thermodynamic, and electro-physical studies of double and ternary tellurites of REEs and alkali (alkaline earth) metals showed that these compounds exhibit semiconductor and ferroelectric properties [16 - 18]. The above review of the literature shows that compounds based on tellurites of rare earth elements with a perovskite-like structure, alloyed with s-elements oxides, are of great scientific and practical interest. In this regard, the aim of this work was to synthesize and to study the radiographic and electro-physical properties of the new phases, namely double neodymium tellurites of the composition $NdMe^{II}TeO_{4.5}$ (Me^{II} - Ca, Ba).

EXPERIMENTAL

Materials and methods

Synthesis of tellurites of neodymium-alkaline earth metals $NdMe^{II}TeO_{4.5}$

$NdMe^{II}TeO_{4.5}$ samples were prepared using oxides of neodymium Nd_2O_3 (extra pure grade), tellurium TeO_2 (chemically pure grade) and carbonates of alkaline earth metals $CaCO_3$, $BaCO_3$ (chemically pure grade) as initial components, previously calcined to remove adsorbed moisture and gases. The initial oxides and metal carbonates for the solid-phase reaction were mixed in an appropriate ratio corresponding to the stoichiometric composition of $NdMe^{II}TeO_{4.5}$ compounds and homogenized in an agate mortar. The samples for the study were synthesized according to the standard ceramic technology. The mixtures in alundumina crucibles were subjected to a step-by-step heat treatment for solid-phase interaction in air in the "SNOL" furnace. The following heat treatment regime was used, namely stage I for 15 hours at 400°C, stage II at 800°C for 20 hours, and stage III at 1200°C for 20 hours. The samples were then slowly cooled to room temperature. Further, annealing was performed at 400°C for 20 hours to produce compounds that were stable at low temperatures. After each stage, the mixtures were cooled, stirred and thoroughly milled.

Powder characteristics

Roentgenograms of the synthesized compounds were obtained using an Empyrean powder diffractometer from PANalytical. There were used X - ray tube "Empyrean Cu LFF HR (9430 033 7310x) DK407912", "PIXcel3D - Medipix3 1x1 detector", Cu as anode material, 30 kV anode voltage, 10 mA current. The measurements were made with the help of a goniometer with a radius of 240 mm according to the Bragg-Brentano method. Data collection was performed using Data Collector software version 7.7h. Approximation and subtraction of the background, determination of peak positions and intensity on a 100 - point scale as well as indication were determined using the X'Pert HighScore Plus program.

The interpretation of the obtained roentgenograms and phase identification were carried out with the help of the specialized computer program X'Pert HighScore Plus, which provides an automated quantitative phase analysis, including measurement, processing and obtaining results, applying all currently accepted analytical models. The Crystallography Open Database and the PDF - 2 database were used to identify the phase composition.

The individuality and distribution of atoms determine the intensity of the diffracted rays. The powder diffraction pattern is an individual characteristic of a crystalline substance. The pycnometric density of tellurites was determined by the method given in [19]. Toluene served as an indifferent liquid. The density of each sample was measured 3 - 5 times and the data were averaged.

The study of electro-physical properties, namely dielectric permittivity and electrical resistance in the range of 293 K - 483 K was carried out by measuring the electrical capacity of the samples on the device LCR - 781 at a working frequency of 1 kHz continuously in dry air in thermostatic mode with exposure time at each fixed temperature. Preparation of samples and their imaging were carried out according to the method described in [20].

RESULTS AND DISCUSSION

This section presents the results of the study and interpretation of X-ray diffraction analysis and electrophysical properties of the synthesized neodymium tellurites.

X-ray diffraction

Fig. 1 shows roentgenograms of the synthesized double neodymium tellurites.

The indication results of roentgenograms of the synthesized neodymium tellurites are shown in Table 1.

Reliability and correctness of the indication results were confirmed by satisfactory agreement between the values of experimental and calculated values of 2 Th. and d-sp. (Table 1) as well as values of radiographic and pycnometric densities (Table 2).

The theoretical cell volumes of the synthesized tellurites were determined using data on the cell volumes of their constituent oxides according to the scheme:

$$V°_{unit\ cell}NdMe^{II}TeO_{4.5} = 0.5\ V°_{unit\ cell}Nd_2O_3 + V°_{unit\ cell}TeO_2 + V°_{unit\ cell}Me^{II}O\ (Me^{II} – Ca, Ba) \quad (1)$$

Satisfactory coincidence of the calculated unit cell volumes of tellurites from the sum of unit cell volumes of the initial neodymium oxides (+3), tellurium (+4) and calcium (barium) oxides, borrowed from the Crystallography Open Database with the calculated cell volumes of compounds from the X-ray data, also confirms the correct indication of the radiographs of new tellurites. Thus, for $NdCaTeO_{4.5}$ $V^0_{unit\ cell} = 331.62$ Å^3 (from the sum of the $V^0_{unit\ cell}$ of oxides) and 352.10 ± 0.01 Å^3 (from the indication data) and for $NdBaTeO_{4.5}$ $V^0_{unit\ cell} = 387.49$ Å^3 (from the sum of the $V^0_{el.cell}$ of oxides) and 388.40 ± 0.02 Å^3 (from indication data) [21].

It was revealed that the lattice parameters and unit cell volumes of the synthesized tellurites increase with increasing ionic radii from Ca to Ba. Based on the indication of the roentgenograms of the tellurites under study, it was found that the compounds $NdCaTeO_{4.5}$

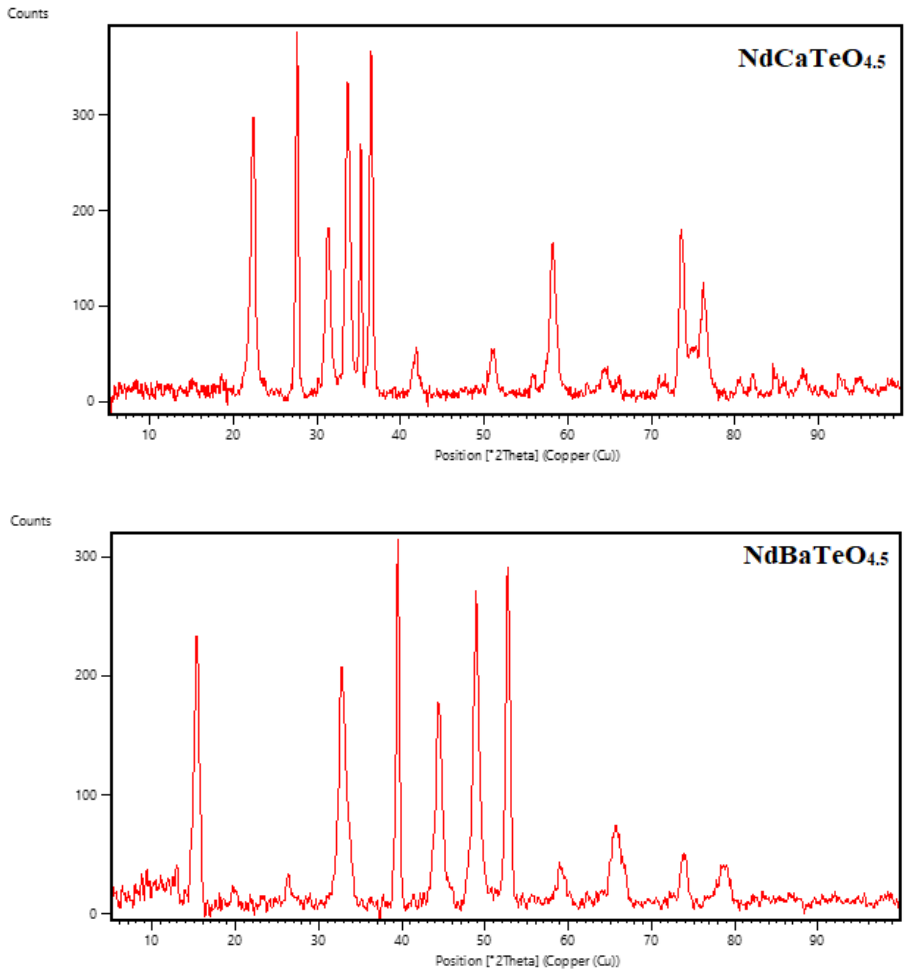

Fig.1. Roentgenograms of the synthesized double tellurites of neodymium.

Table 1. The indication results of radiographs of the synthesized tellurites of neodymium-alkaline earth metals.

h	k	l	2Th. (c) [°]	2Th. (o) [°]	d-sp. (c) [A]	d-sp. (o) [A]	I/I_0
$NdCaTeO_{4.5}$							
1	1	1	22.6876	22.3342	3.916198	3.977352	75.45
2	0	1	27.5803	27.5577	3.231575	3.234173	100
2	2	0	31.2441	31.1674	2.860474	2.867339	43.27
0	0	2	33.3298	33.6410	2.686086	2.661945	86.28
1	0	2	35.1754	35.1868	2.549263	2.548464	68.06
1	1	2	36.9402	36.3949	2.431423	2.466593	93.05
2	1	2	41.8517	41.8164	2.156739	2.158476	12.81
0	0	3	50.9555	51.0038	1.790724	1.789141	11.19
4	1	2	58.1753	58.1705	1.584498	1.584618	40.04
4	1	3	71.2328	71.2330	1.322730	1.322726	2.55
6	1	1	73.2561	73.5621	1.291109	1.286492	44.36
6	2	1	76.4847	76.2471	1.244447	1.247732	28.46
6	1	2	80.5182	80.5375	1.191961	1.191724	4.01
5	5	0	84.6332	84.6080	1.144189	1.144466	6.97
4	1	4	88.0580	88.0810	1.108308	1.108078	5.78
$NdBaTeO_{4.5}$							
0	0	1	15.4376	15.4023	5.735187	5.748251	72.14
3	0	0	32.7111	32.648	2.735466	2.740605	58.45
3	2	0	39.5634	39.4695	2.276045	2.281242	100
2	2	2	44.3804	44.3169	2.039542	2.042319	50.38
1	0	3	48.8777	48.8822	1.861876	1.861715	82.08
2	0	3	52.7853	52.6801	1.732877	1.736088	89.61
3	0	3	58.8891	59.0071	1.566982	1.564130	9.55
5	4	0	73.8879	73.9098	1.281624	1.281299	12.33

Table 2. Syngony types and unit cell parameters of double neodymium tellurites.

Compound	Syngony type	Lattice parameters, Å		$V^0_{\text{unit cell. Å}^3}$	Z	Density, g·cm^{-3}	
		a	c			radio-graphic	pycnometric
$NdCaTeO_{4.5}$	tetragon	8.099 ± 0.002	5.368 ± 0.003	352.10 ± 0.01	2	3.62	3.61 ± 0.01
$NdBaTeO_{4.5}$	tetragon	8.223 ± 0.003	5.745 ± 0.003	388.40 ± 0.02	2	4.17	4.15 ± 0.02

For both tellurites $\alpha = \beta = \gamma = 90$ deg.

and $NdBaTeO_{4.5}$ crystallize in tetragonal syngony with the lattice parameters, which are presented in Table 2.

The phase composition of the compound was confirmed by calculation of the cell parameters of the studied sample using the X'Pert HighScore Plus diffractometer software from PANalytical. Based on the X'Pert HighScore Plus program databases, roentgenograms of synthesized tellurites were compared with roentgenographic indices [I/I_0, d] of the initial substances and with possible tellurites of this system. It was revealed that the diffractograms of new tellurites have no analogues with them. These data further confirm that the synthesized tellurites are new compounds that crystallize in the perovskite structural type. According to [22], there are compounds in the structure of perovskite (or its slightly distorted versions) that obey the equation taking into account the "tolerance factor" t:

$$r_A + r_0 = t \cdot \sqrt{2 \cdot (r_B + r_0)} \quad (2)$$

from where

$$t = \frac{r_A + r_0}{\sqrt{2(r_B + r_0)}} \quad (3)$$

where r_A, r_B, r_0 are the radii of the ions that make up the compound. In our case, A is the Nd^{3+} ion, B is the set of Te^{4+}, Ca^{2+} and O is the O^{2-} ion for $NdCaTeO_{4.5}$ and A is the Nd^{3+} ion, B is the set of Te^{4+}, Ba^{2+} and O is the O^{2-} ion for $NdBaTeO_{4.5}$. The ionic radii of the above elements according to the modern classification of Shannon and Prewitt were taken from [23]. It was established that for all compounds with perovskite-type structure the "tolerance factor" t lies approximately in the range of 0.8 - 1.0. In our case, t for $NdCaTeO_{4.5}$ and $NdBaTeO_{4.5}$ are 0.96 and 0.91, respectively, which to a certain extent can be attributed to the perovskite type structure [22]. Therefore, we can assume that these compounds may possess semiconductor and ferroelectric properties. To confirm this assumption, the study of the compounds' electro-physical properties was carried out.

Electro-physical properties

The temperature dependences of electrical capacity, dielectric permittivity and electrical resistance of synthesized tellurites were investigated on the device LCR - 781 in order to reveal the physical properties in the range of 293 K - 483 K.

The accuracy of electrical capacitance and electrical resistance measurements, according to the instrument's data sheet is ± 0.05% [20]. Similar studies were carried out earlier in [24, 25]. It is known that, as a rule, there is a temperature dependence of the electro-physical properties in ceramic ferroelectrics. The dielectric permittivity of the standard substance, namely barium titanate $BaTiO_3$, was measured to verify the obtained data. Thus, the experimental value of dielectric permittivity at 293 K at a frequency of 1 kHz, equal to 1296 satisfactorily agrees with its recommended value of 1400 ± 250 [26]. In addition, the observed change in the electrical conductivity of $BaTiO_3$ at 383 K is consistent with its transition from the perovskite cubic phase Pm3m to the tetragonal (polar) ferroelectric phase with the space group P4mm [27].

The dependence of electrical resistance and dielectric permittivity of neodymium tellurites on temperature are shown in Figs. 2 and 3.

The dielectric permittivity was determined from the sample electrical capacitance at known values of the sample thickness and electrode surface area. The value of dielectric permittivity at each temperature was determined by the formula:

$$\varepsilon = C/C_0, \quad (4)$$

where $C_0 = \varepsilon_0 S/d$ is capacitance of the condenser without the test substance (air).

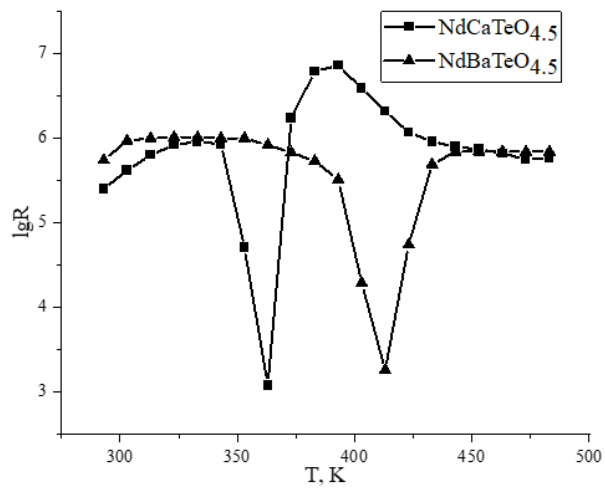

Fig. 2. Temperature dependences of electrical resistance of double neodymium tellurites.

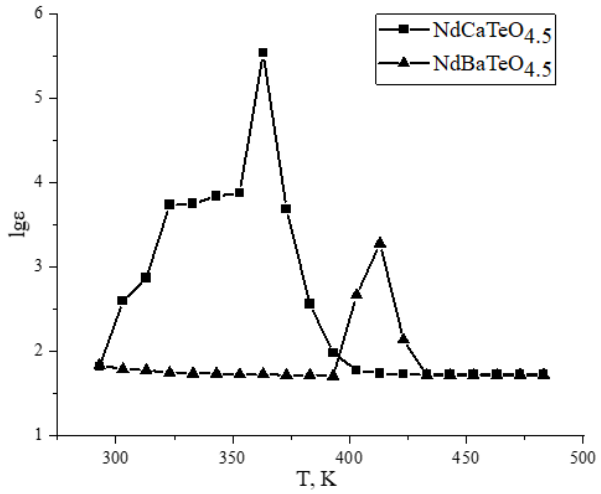

Fig. 3. Temperature dependences of dielectric permittivity of double neodymium tellurites.

Analysis of the data in Figs. 2 and 3 shows that with increasing temperature significant changes in the electro-physical characteristics of tellurites are found, and, as is typical for ceramic materials, such changes occur in a certain temperature range.

Errors of measurement of electro-physical characteristics are within the accuracy of the device (± 0.05%).

$NdCaTeO_{4.5}$

The electrical resistance dependence of $NdCaTeO_{4.5}$ between 293 K - 333 K and 373 K - 393 K is metal-like (Fig. 2) with a high temperature coefficient of resistance of 0.12 K^{-1} and 0.06 K^{-1}, respectively. The temperature coefficient α was calculated by the formula:

$$\alpha = \frac{R_T - R_0}{R_0 \cdot \Delta T}, \quad (5)$$

where R_0 and R_T are resistance at the initial and the final temperature, respectively, ΔT is the temperature difference.

Electrical resistance increases in the range of 293 K - 333 K and in the range of 343 K- 363 K a jump is observed, in which electrical resistance decreases with a minimum at 363 K (see Fig. 2), then an inverse change occurs at 373 K - 393 K that is an increase in electrical resistance followed by a smooth decrease in resistance at 393 K - 473 K.

In the range of 333 K - 363 K and 393 K - 473 K the sample reveals semiconducting properties, i.e., electrical resistance decreases with increasing temperature (electrical conductivity increases). The forbidden zone width calculated by the formula (6) for the studied compound was 1.63 eV:

$$\Delta E = \frac{2kT_1 \cdot T_2}{0.43 \cdot (T_2 - T_1)} \cdot \lg \frac{R_1}{R_2}, \quad (6)$$

where k is the Boltzmann coefficient, equal to 8.6173303 $\cdot 10^{-5}$ eV·k^{-1}, R_1 and R_2 are resistance at temperatures T_1 and T_2, respectively.

$NdBaTeO_{4.5}$

For tellurite $NdBaTeO_{4.5}$ the electrical resistance in the interval of 293 K- 333 K increases, and in the range of 343 K - 413 K there is a jump in which the electrical resistance decreases with a minimum at 413 K, further at 423 - 483 K there is a change, i.e. increase in electrical resistance (Fig. 2). The temperature coefficients of resistance in the temperature range of 293 K - 333 K and 423 K - 483 K are 0.04 K^{-1} and 0.69 K^{-1}, respectively. In the interval 343 K - 413 K, the sample exhibits semiconductor properties, that is, the electrical resistance decreases with increasing temperature (electrical conductivity increases). The forbidden zone width ΔE = 1.84 eV.

Studies of the temperature dependence of the dielectric permittivity and electrical resistance of tellurites of neodymium-alkaline earth metals showed that these compounds can possess semiconductor and ferroelectric properties. As a rule, the temperature dependence of the electro-physical properties is observed in ceramic ferroelectrics. The anomalous jumps observed on the temperature dependence curves of electrical resistance and dielectric permittivity of compounds, probably indicate phase transitions of type II, due to the semiconducting and ferroelectric properties of new double neodymium tellurites [16 - 18]. It is known that a jump transition takes place if it is accompanied by the appearance of a structure that provides an anomalously fast three-dimensional diffusion of cations [27], so we can assert the presence of phase transitions in these compounds. The temperature dependence curves of the dielectric permittivity of tellurites have the "lambda" form, which corresponds, in agreement with [28], to a ferroelectric phase transition.

CONCLUSIONS

As a result of the research carried out on triple systems of Nd_2O_3 - TeO_2 - $Me^{II}CO_3$ (Me^{II} - Ca, Ba) for the first time two new tellurites of the composition $NdMe^{II}TeO_{4.5}$ were identified and isolated in the individual state. The optimal conditions for the solid-phase synthesis of the newly identified double tellurites were developed. Syngony types, unit cell parameters, radiographic and pycnometric densities of the synthesized compounds were determined. It was found that the synthesized tellurites crystallize in the distorted perovskite structural type.

In the temperature range of 293 K - 483 K, electro-physical properties (electrical capacitance, dielectric permittivity and electrical resistance) of the compounds were studied. Temperature coefficients of resistance and the forbidden zone width were determined. New double tellurites of neodymium were found to possess semiconducting and ferroelectric properties.

The results of the work are of a fundamental nature and the obtained data (radiographic, electro-physical) on double tellurites will be useful in the study of complex oxide systems and in identifying regularities in the structure and properties of the compounds. Radiographic characteristics of new s - f - elements tellurites are source materials for inclusion in fundamental data banks and reference books. The obtained double neodymium tellurites are promising as solid electrolytes. Since these compounds according to the temperature dependence of electrical resistance can be referred to ceramic materials, such compounds can find application in the field of creating materials with ferroelectric properties. The results obtained can be used for prediction, synthesis, and study of new derivatives of tellurium and rare - earth elements that have important electro-physical properties and are of interest for electronic technology.

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