



# Condensed Matter and Interphases (Kondensirovannyye sredy i mezhfaznyye granitsy)

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## New Thallium Tellurides with Rare Earth Elements

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### Abstract

Compounds of the  $Tl_4LnTe_3$  (Ln-Nd, Sm, Tb, Er, Tm) composition were synthesized by the direct interaction of stoichiometric amounts of thallium telluride  $Tl_2Te$  elementary rare earth elements (REE) and tellurium in evacuated ( $10^{-2}$  Pa) quartz ampoules. The samples obtained were identified by differential thermal and X-ray phase analyses. Based on the data from the heating thermograms, it was shown that these compounds melt with decomposition by peritectic reactions. Analysis of powder diffraction patterns showed that they were completely indexed in a tetragonal lattice of the  $Tl_5Te_3$  type (space group  $I4/mcm$ ). Using the Le Bail refinement, the crystal lattice parameters of the synthesized compounds were calculated. It was found that when the thallium atoms located in the centres of the octahedra were substituted by REE atoms, there occurred a sharp decrease in the a parameter and an increase in the c parameter. This was due to the fact that the substitution of thallium atoms with REE cations led to the strengthening of chemical bonds with tellurium atoms. This was accompanied by some distortion of octahedra and an increase in the c parameter. A correlation between the parameters of the crystal lattices and the atomic number of the lanthanide was revealed: during the transition from neodymium to thulium, there was an almost linear decrease in both parameters of the crystal lattice, which was apparently associated with lanthanide contraction. The obtained new compounds complement the extensive class of ternary compounds - structural analogues of  $Tl_5Te_3$  and are of interest as potential thermoelectric and magnetic materials.

**Keywords:** thallium tellurides – REE, structural analogues of  $Tl_5Te_3$ , differential thermal analysis, X-ray phase analysis, crystal structure.

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## 1. Introduction

One of the rational ways to develop new functional materials is the search for complex structural analogues of already known compounds with the same properties and to optimize their characteristics by directional alloying [1–5].

Subtelluride  $\text{Tl}_5\text{Te}_3$  is one of the most suitable matrix compounds for the preparation of new ternary compounds – structural analogues and multi-component phases. According to the phase diagram of the Tl-Te system [6, 7], this compound melts congruently at 725 K and is a variable composition phase with a wide range of homogeneity (34.5–38 at% Te). Due to the peculiarities of the crystal structure [8, 9],  $\text{Tl}_5\text{Te}_3$  has a number of ternary cation- and anion-substituted structural analogies.

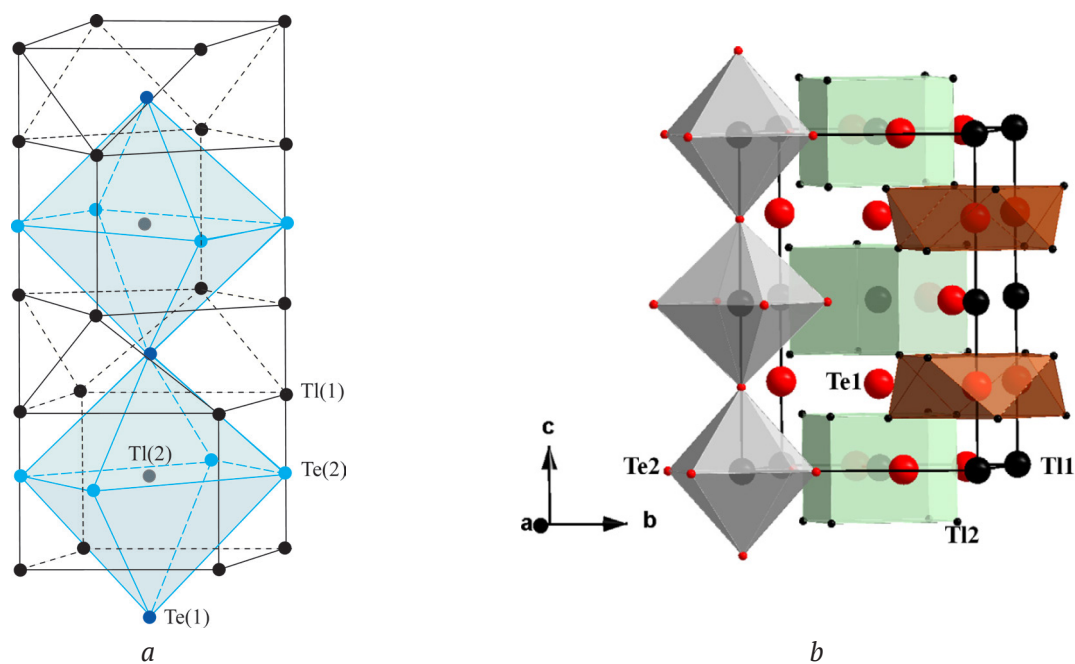
In studies [10, 11] a new class of compounds – thallium tellurides of REE type  $\text{Tl}_9\text{LnTe}_6$ , which are ternary structural analogues of  $\text{Tl}_5\text{Te}_3$  was obtained. These compounds complement the extensive class of ternary compounds with general formulas  $\text{Tl}_9\text{AX}_6$  and  $\text{Tl}_4\text{BX}_3$  (A – Sb, Bi, Au, In; B – Sn, Pb, Mo, Cu; X – Se, Te) [13–19]. The described compounds are thermoelectric with anomalously low thermal conductivity [20–23], and some of them exhibit optical [24–26] and magnetic [27, 28] properties, as well as the properties of topological insulators [29–31].

As was shown above,  $\text{Tl}_5\text{Te}_3$  crystallizes in a tetragonal structure of the  $\text{Cr}_5\text{B}_3$  type (Sp.Gr.

$I4/mcm$ ) (Fig. 1a) [8, 9]. The crystal structure of  $\text{Tl}_5\text{Te}_3$  was described in detail in studies [5, 8, 9, 32]. In the crystal lattice of  $\text{Tl}_5\text{Te}_3$ , thallium atoms occupy two different positions and exhibit oxidation states +1 and +3. Some of the thallium atoms (Tl2) are located in octahedral voids, and others (Tl1) are located in the voids of the anionic tellurium cage. Replacement of half of the thallium atoms located at the centres of octahedra (Tl2) with  $\text{B}^{+3}$  cations led to the formation of compounds of the  $\text{Tl}_9\text{BTe}_6$  type, and their complete replacement by  $\text{A}^{+2}$  cations led to the formation of compounds of the  $\text{Tl}_4\text{ATe}_3$  type.

It is known that lanthanides exhibit oxidation states +2 and +3. In compounds of the  $\text{Tl}_9\text{LnTe}_6$  type, lanthanides exhibit an oxidation state of +3. Considering the existence of compounds of the  $\text{Tl}_4\text{A}^{\text{IV}}\text{X}_3$  type, we assumed the possibility of the formation of compounds with the composition  $\text{Tl}_4\text{LnTe}_3$ , in which the REE will exhibit an oxidation state of +2. In the study [12], for the first time we synthesized and identified  $\text{Tl}_4\text{GdTe}_3$  and  $\text{Tl}_4\text{DyTe}_3$  compounds - representatives of the specified class, their isostructurality with  $\text{Tl}_5\text{Te}_3$  was confirmed and the parameters of their crystal lattices were calculated.

In this study, we continued our research in the field of REE thallium chalcogenides and report on the synthesis of some new compounds of the  $\text{Tl}_4\text{LnTe}_3$  type.



**Fig. 1.** Crystal structure of  $\text{Tl}_5\text{Te}_3$ . Main structural element (a) [5], projected onto a plane  $b, c$  (b) [28]

## 2. Experimental

### 2.1. Materials and synthesis

High-purity elements purchased from Alfa Aesar were used for the studies: thallium (catalogue number 7440-28-0), tellurium (13494-80-9), neodymium (7440-00-8), samarium (7440-19-9), terbium (7440-27-9), erbium (7440-52-0), thulium (7440-30-4).

Thallium was stored in water in order to prevent oxidation in air, therefore it was dried immediately before use. Due to the toxicity of thallium and its compounds, protective gloves were used during performed work.

Considering the previous experience in the synthesis of  $Tl_9LnTe_6$  and  $Tl_4LnTe_3$  compounds [31–34], namely, the incongruent nature of their melting and the refractoriness of the REE,  $Tl_4LnTe_3$  compounds were synthesized by a ceramic method using a special technique. In the synthesis, not elementary components, but stoichiometric amounts of thallium telluride  $Tl_2Te$ , lanthanide, and tellurium were used since lanthanides with thallium form thermodynamic stable compounds that prevent further synthesis of ternary compounds. Fusion was performed in the evacuated ( $10^{-2}$  Pa) quartz ampoules. After fusion at 1000 K, to bring the alloys to a state as close as possible to equilibrium, the cast non-homogenized ingots were ground into powder in an agate mortar, thoroughly mixed, pressed into a cylindrical tablet, and annealed at 700 K for 1000 h.

In order to prevent the interaction of lanthanides with the inner walls of the quartz ampoule, the synthesis of the compounds was carried out in graphitized ampoules. Graphitization was performed by the thermal decomposition of toluene.

The single-phase of the synthesized compounds was monitored by DTA and XRD methods.

### 2.2. Methods

The studies were carried out by the methods of differential thermal (DTA) and X-ray phase diffraction analyses (XRD). Heating curves were recorded using differential scanning calorimeter DSC NETZSCH 404 F1 Pegasus system and multi-channel DTA device based on an electronic TC-08 Thermocouple Data Logger in the temperature range from room temperature to ~ 1300 K. Powder diffraction patterns of the initial compounds and intermediate alloys were recorded on a

diffractometer D2 Phaser with  $CuK_{\alpha}$ -radiation within an angle range of  $2\theta = 10\div 70^{\circ}$ . The crystal lattice parameters of the initial compounds and intermediate alloys were determined by the indexing of powder diffraction patterns using the Topas 4.2 software by the *Le Bail method*.

## 3. Results

A comparison of powder diffraction patterns of synthesized samples of composition  $Tl_4LnTe_3$  with  $Tl_5Te_3$  and its typical triple analogue  $Tl_4PbTe_3$  showed that they all have qualitatively the same diffraction pattern (Fig. 2). All reflection lines were fully indexed in the  $Tl_5Te_3$  structure type (Sp.Gr. *I4/mcm*).

The parameters of the tetragonal lattices of  $Tl_4LnTe_3$  that were determined using the computer program Topas V4.2 using the Le Bail refinement are shown in the Table. The Table also contains data for other members of this class of compounds. Data in the Table demonstrate that the substitution of thallium (Tl2) atoms located at the centres of tellurium octahedra (Fig. 1) with REE atoms led to a sharp decrease in parameter *a* and an increase in parameter *c*. Probably, this was due to the fact that such a substitution led to the strengthening of chemical bonds between the REE and Te atoms (2) and a decrease in the corresponding interatomic distances, determining the value of parameter *a*. As a result, some distortion of tellurium octahedra occurred, which led to an increase in parameter *c*.

The dependences of the crystal lattice parameters of  $Tl_4LnTe_3$  compounds from the atomic number of the lanthanide is shown in Fig. 3. As can be seen, there is a clear correlation: during the transition from neodymium to thulium, there is an almost linear decrease in both crystal lattice parameters, which is probably associated with a decrease in the crystallographic radius of REE due to lanthanide contraction [33].

The thermogram of heating each of the synthesized compounds in the temperature range from room temperature to 1300 K contained one clear endothermic effect at 760–775 K (Table). Taking into account the difficulty of homogenizing the samples obtained by fusion and the complex picture on the DTA cooling curves, these effects cannot be attributed to the congruent melting point. Probably, these compounds melt with decomposition by peritectic reactions, and their complete transition to a liquid state occurs at temperatures above 1300 K.

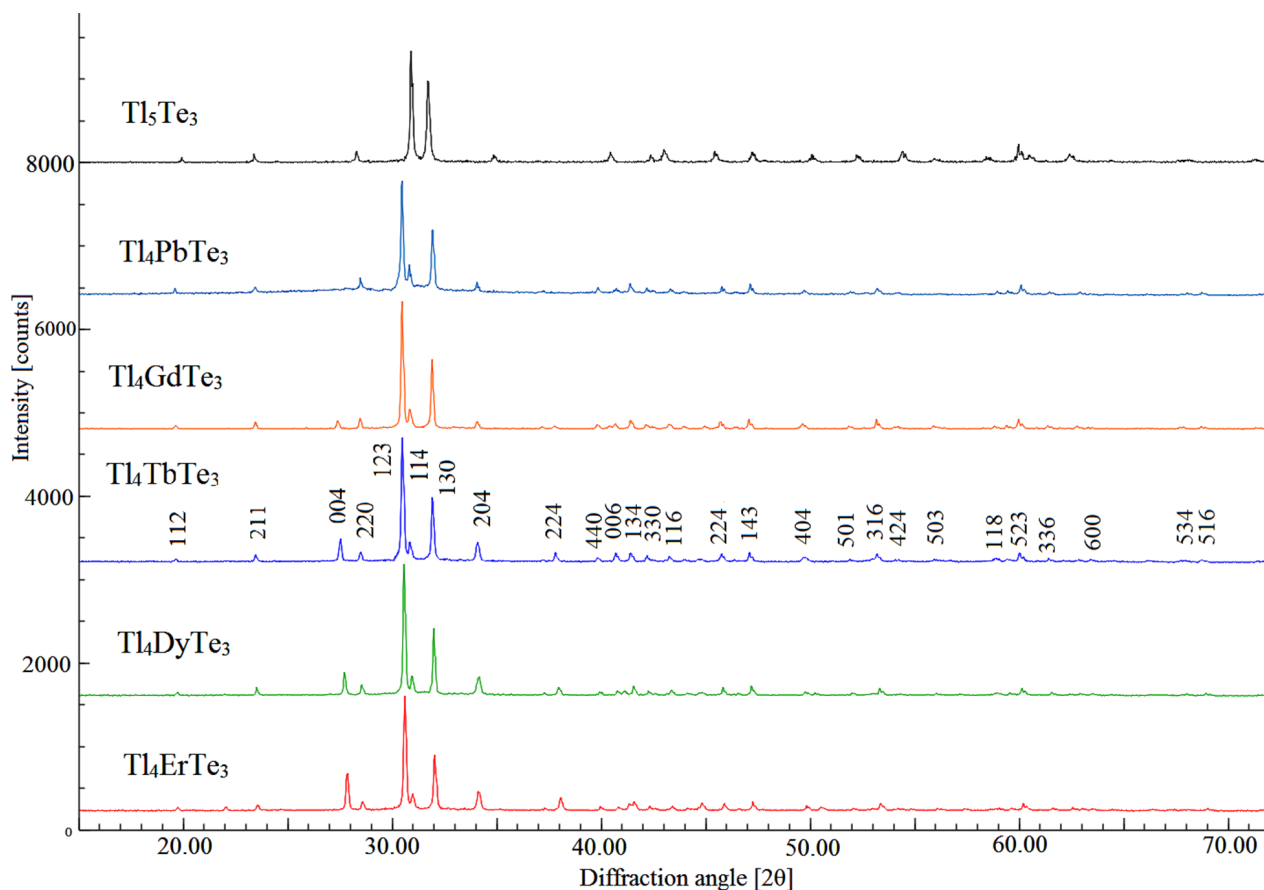
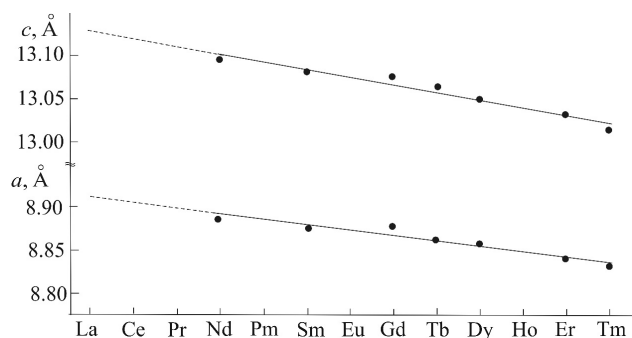


Fig. 2. Powder diffraction patterns of some compounds of the  $Tl_4LnTe_3$  type

Table. Crystallographic parameters of  $Tl_5Te_3$  and its ternary structural analogues of the  $Tl_4A^{IV}Te_3$  type

Phase	Tetragonal lattice parameters, Sp.Gr. $I4/mcm$ , $Z = 4$		Melting points	Source
	$a$ , Å	$c$ , Å		
$Tl_5Te_3$	8.930	12.598	725	[8]
$Tl_4SnTe_3$	8.820	13.010	823	[15]
$Tl_4PbTe_3$	8.841	13.056	893	[15]
$Tl_4CuTe_3$	8.929	12.603	–	[17]
$Tl_4MoTe_3$	8.930	12.575	–	[18]
$Tl_4NdTe_3$	8.8885(7)	13.0952(12)	775	present study
$Tl_4SmTe_3$	8.8752(6)	13.0784(11)	772	present study
$Tl_4GdTe_3$	8.8766(7)	13.0756(13)	770	[12]
$Tl_4TbTe_3$	8.8652(7)	13.0653(12)	768	present study
$Tl_4DyTe_3$	8.8588(7)	13.0524(16)	767	[12]
$Tl_4ErTe_3$	8.8421(6)	13.0334(11)	760	present study
$Tl_4TmTe_3$	8.8354(7)	13.015(15)	760	present study



**Fig. 3.** Correlation between crystal lattice parameters of the  $Tl_4LnTe_3$  type compounds and the atomic number of the lanthanide

#### 4. Conclusion

In this study, we reported the synthesis of new ternary compounds of the  $Tl_4LnTe_3$  (Ln-Nd, Sm, Tb, Er, Tm) type, which are structural analogues of  $Tl_5Te_3$ . Powder diffraction patterns of all synthesized compounds were completely indexed in the tetragonal structure (Sp.Gr.  $I4/mcm$ ), the parameters of their crystal lattices were calculated. According to DTA data, it was found that the synthesized compounds melt with decomposition by peritectic reactions. A comparison of the results obtained with the literature data was carried out. A correlation between the parameters of the crystal lattice and the atomic number of the lanthanide was revealed. The obtained new compounds complement a wide class of compounds, which are structural analogues of  $Tl_5Te_3$ , and are of interest as potential thermoelectric and magnetic materials.

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#### Conflict of interests

The author declares that she has no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

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