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**PHASE RELATIONS IN  $Tl_9GdTe_6$ - $Tl_9SbTe_6$  AND  $Tl_9TbTe_6$ - $Tl_9SbTe_6$  SYSTEMS****S.Z. Imamaliyeva<sup>1</sup>, T.M. Hasanly<sup>2</sup>, V.A. Gasymov<sup>1</sup>, D.M. Babanly<sup>1</sup>, F.M. Sadygov<sup>2</sup>**<sup>1</sup>*Institute of Catalysis and Inorganic Chemistry, ANAS**H.Javid ave., 113, Baku AZ 1143, Azerbaijan Republic;**e-mail: [dunyababanly2012@gmail.com](mailto:dunyababanly2012@gmail.com)*<sup>2</sup>*Baku State University**Z.Xalilov str., 23, Baku AZ 1148, Azerbaijan Republic : e-mail: [info@bsu.az](mailto:info@bsu.az)*

*Phase equilibriums in the  $Tl_9GdTe_6$ - $Tl_9SbTe_6$  and  $Tl_9TbTe_6$ - $Tl_9SbTe_6$  systems have been examined by means of differential thermal analysis, X-ray diffraction and microhardness measurements over equilibrium alloys. Phase diagram and concentration dependence of the unit cell parameters and microhardness of both systems plotted. It found that the systems are non-quasi-binary due to incongruent melting of  $Tl_9Gd$  ( $Tb$ )  $Te_6$  compositions but proved to be stable below the solidus. Systems are characterized by formation of continuous solid solutions with  $Tl_5Te_3$  structure. Solid solutions obtained may be of interest as thermoelectric and magnetic materials.*

**Keywords:** *thallium-terbium telluride, thallium-gadolinium telluride, thallium-antimony telluride, phase equilibriums, solid solutions, crystal structure.*

**1. INTRODUCTION**

A number of works are illustrative of the growing interest in new multinary chalcogenide materials. This is due to their specific functional properties, such as thermal, electrical and optical [1-3]. Furthermore, recent studies have shown that some of them exhibit topological insulator properties [4,5]. Doping by rare-earth elements may improve their properties to provide them with additional functionality [6-8].

Thallium subtelluride,  $Tl_5Te_3$  is suitable "matrix" for production of novel complex materials. This composition is crystallized in tetragonal structure (Sp.gr.  $I4/mcm$ ) [9, 10] and has a number of ternary cation- [11-14] and anion-substituted [15-18] ternary structural analogs. Cation-substituted compositions of  $Tl_4A^{IV}Te_3$  [ $A^{IV}Sn, Pb$ ] and  $Tl_9B^VTe_6$  [ $B^V-Sb, Bi$ ] types form an important class of thermoelectric materials with anomalous low thermal conductivity [19-21]. Particularly,  $Tl_9BiTe_6$  shows high ZT value comparable to the state-of-the-art thermoelectric materials [21]. On the other hand, according to recent investigations, anion-substituted  $Tl_5Se_2I$  composition is a prospective material for efficient X-ray and  $\gamma$ -ray detection [22].

A new substitution variant of  $Tl_5Te_3$ ,

thallium lanthanide tellurides,  $Tl_9LnTe_6$  ( $Ln$ -Ce, Nd, Gd, Tm, Tb) has been obtained first by authors [23-25] to ensure their melting property and crystal lattice parameters. Moreover, according to [25, 26], ytterbium does not form the composition  $Tl_9YbTe_6$ . Later, a number of tellurides,  $Tl_{10-x}Ln_xTe_6$ , were synthesized, structurally characterized and their thermoelectric properties identified by authors [27-29].

Earlier, with the purpose of obtaining a solid solution with  $Tl_5Te_3$  structure the phase relations in the  $Tl_9NdTe_6$ - $Tl_9BiTe_6$ ,  $Tl_9TbTe_6$ - $Tl_9BTe_6$  and  $Tl_9GdTe_6$ - $Tl_9BTe_6$  systems had been studied in [30-32]. Authors showed the formation of continuous areas of solid solutions with  $Tl_5Te_3$  structure.

The goal of the present work is to determine phase equilibria in the  $Tl_9GdTe_6$ - $Tl_9SbTe_6$  and  $Tl_9TbTe_6$ - $Tl_9SbTe_6$  systems and thus obtain phase relationships and provide more accurate experimental data for preparation of pure and high quality materials.

$Tl_9SbTe_6$  melts congruently at 798 K [11] and has a low symmetry crystal structure of  $Tl_5Te_3$  (Sp.gr. $I4/m$ ),  $a = 8.829 \text{ \AA}$  and  $c = 13.001 \text{ \AA}$ ,  $Z = 2$  [33].

$Tl_9GdTe_6$  and  $Tl_9TbTe_6$  melt with decomposition by peritectic reactions at 800

and 780K with the following lattice parameters:  $a = 8.870$ ,  $c = 13.027$  Å,  $Z = 4$  [31]

and  $a = 8.871$ ,  $c = 12.973$  Å,  $Z = 4$  [32].

## 2. EXPERIMENTAL

### 2.1. Materials and syntheses

Thallium (granules, 99.999 mass%), antimony (granules, 99.999 mass%), gadolinium (powder, 99.9%), terbium (powder, 99.9%) and tellurium (broken ingots 99.999 mass%) were used as starting materials. The elements were weighed to total about 20 g ( $Tl_9SbTe_6$ ) and 10 g ( $Tl_9GdTe_6$ ,  $Tl_9TbTe_6$ ) as per the molar ratio of the corresponding ternary composition, and placed in silica tubes, 20 cm long, and then sealed under a vacuum of  $10^{-3}$  Pa. The synthesis was carried out by heating in one zone an electric furnace at 850K ( $Tl_9SbTe_6$ ) and 1200K ( $Tl_9GdTe_6$ ,  $Tl_9TbTe_6$ ), followed by cooling in the switched-off furnace. To prevent a reaction between rare-earth elements and tubes, the silica tubes were coated with a carbon film via the decomposition of ethanol.

In considering that the equilibrium state could not be obtained even after a long-time (1000 h.) annealing [30-32], intermediate ingots of  $Tl_9GdTe_6$  and  $Tl_9TbTe_6$  were powdered in agate mortar, pressed into pellets and annealed at 730K within ~700h.

The purity of the synthesized compositions was examined by the differential thermal analysis (DTA) and X-ray diffraction analysis (XRD).

Just one endothermic effect was revealed for  $Tl_9SbTe_6$  (790K), and two effects for  $Tl_9GdTe_6$  (800 and 1190 K) and  $Tl_9TbTe_6$  (780 and 1110 K) showed the completion of the synthesis.

Powder XRD pattern for the  $Tl_9SbTe_6$ ,  $Tl_9GdTe_6$  and  $Tl_9TbTe_6$  were similar to that of  $Tl_5Te_3$ . The lattice parameters were refined using the Topas V3.0 software (Table 1). They are practically equal to those shown in [34] for  $Tl_9SbTe_6$ , and slightly differ from [28] for  $Tl_9TbTe_6$ .

The samples of the  $Tl_9GdTe_6$ - $Tl_9SbTe_6$  and  $Tl_9TbTe_6$ - $Tl_9SbTe_6$  systems were prepared by melting from pre-synthesized ternary compositions in evacuated silica ampoules. Total mass of the ingot was 1 g. The synthesis was carried out by heating an electric furnace on a zone. Initially ampoules were heated from room temperature to 1200 K at a rate of 5 K/min and complied with this temperature within 3 h, then slowly cooled to 730K and kept at this temperature within 200 h. DTA and XRD analyses showed that alloys containing >60mol%  $Tl_9(Gd)TbTe_6$  proved to be non-homogeneous after the heating. Therefore, the samples were powdered and pressed into pellets and then reheated in fused silica tubes at 730 K for a 500h in order to complete the homogenization.

### 2.2. Methods

Differential thermal analysis (DTA), X-ray powder diffraction (XRD), and microhardness measurements were made to analyze the samples. DTA was performed using a NETZSCH 404 F1 Pegasus differential scanning calorimeter. Measurements were carried out at room temperature and ~1400 K. Temperatures of thermal effects were read mainly from the heating curves. But in some samples thermal effects were read from cooling curves in order to establish the onset of crystallization.

X-ray powder diffraction (XRD) data were collected at room temperature in reflection mode using a Bruker D8 ADVANCE powder diffractometer and  $CuK_{\alpha}$  radiation within  $2\theta = 10$  to  $70^\circ$ .

Microhardness measurements were performed with a microhardness meter PMT-3 with typical loading reaching 20 g.

## 3. RESULTS AND DISCUSSION

The  $Tl_9GdTe_6$ - $Tl_9SbTe_6$  and  $Tl_9TbTe_6$ - $Tl_9SbTe_6$  systems (Table 1, Fig.1) are non-quasi-binary section of the Tl-Gd(Tb)-Sb-Te quaternary system due to peritectic melting of  $Tl_9Gd(Tb)Te_6$  compositions. However, they are characterized by the formation of continuous solid solutions ( $\delta$ ).

Note that the  $\delta$ -solid solutions are primarily crystallized in 0-63 mol%  $Tl_9GdTe_6$  composition area. Primary crystallization of X-phase occurs in the range of >63 mol%  $Tl_9GdTe_6$ . The mono-variant peritectic  $L+X \leftrightarrow \delta$  reaction takes place below 800K and leads to the formation of three-phase area  $L+X+\delta$ . This area is not experimentally fixed due to narrow temperature interval and shown by dotted line (Fig.1).

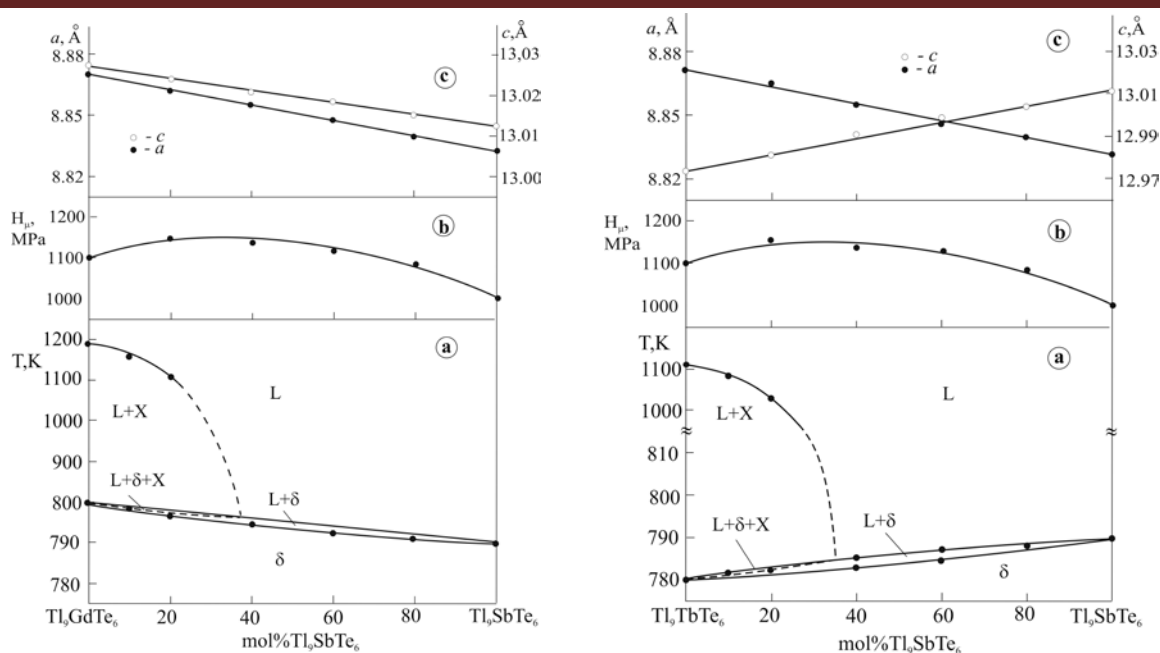
We have assumed that the X phase has a composition of  $TlGdTe_2$ . This assumption is confirmed by the presence of the most intense reflection peaks of  $TlGdTe_2$  [34] on diffractograms of cast alloys from an area exceeding 63 mol%  $Tl_9GdTe_6$ .

Note that the nature of phase equilibriums in the  $Tl_9TbTe_6$ - $Tl_9SbTe_6$  system is qualitatively identical.

It should be noted that irrespective of the very close melting temperature of  $Tl_9SbTe_6$  (790K) and peritectic decomposition of  $Tl_9GdTe_6$  (800 K) and  $Tl_9TbTe_6$  (780 K) compositions, the liquids and solidus curves of both studied systems have no extremum points.

**Table 1.** Properties of initial compositions and alloys of the  $Tl_9SbTe_6$ - $Tl_9GdTe_6$  and  $Tl_9SbTe_6$ - $Tl_9TbTe_6$  systems

Phase	Temperature of melting, K	Microhardness, MPa	Parameters of tetragonal lattice, Å	
			a	c
$Tl_9TbTe_6$	780; 1100	1100	8.8713	12.9737
$Tl_9Sb_{0,1}Tb_{0,9}Te_6$	781; 1080	-	-	-
$Tl_9Sb_{0,2}Tb_{0,8}Te_6$	782; 1030	1160	8.8626	12.9786
$Tl_9Sb_{0,4}Tb_{0,6}Te_6$	784	1140	8.8542	12.9842
$Tl_9Sb_{0,6}Tb_{0,4}Te_6$	786; 1030	1130	8.8458	12.9898
$Tl_9Sb_{0,8}Tb_{0,2}Te_6$	788	1080	8.8374	12.9954
$Tl_9SbTe_6$	790	1000	8.8312	13.0132
$Tl_9Sb_{0,8}Gd_{0,2}Te_6$	791	1050	8.8412	13.0152
$Tl_9Sb_{0,6}Gd_{0,4}Te_6$	793	1120	8.8482	13.0181
$Tl_9Sb_{0,4}Gd_{0,6}Te_6$	794	1140	8.8563	13.0211
$Tl_9Sb_{0,2}Gd_{0,8}Te_6$	796; 1100	1160	8.8631	13.0242
$Tl_9Sb_{0,1}Gd_{0,9}Te_6$	798; 1160	-	-	-
$Tl_9GdTe_6$	800; 1190	1100	8.8703	13.0276



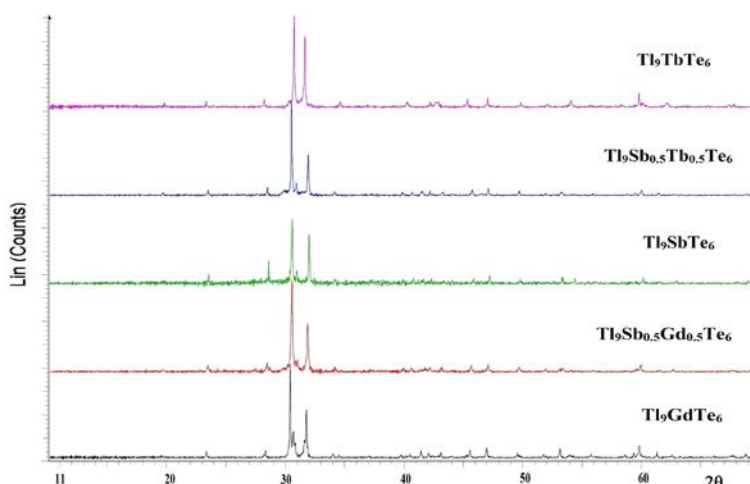
**Fig.1.** Phase diagrams of the  $Tl_9SbTe_6$ - $Tl_9GdTe_6$  and  $Tl_9SbTe_6$ - $Tl_9TbTe_6$  systems.

Both examined systems go to show that the temperature interval of the crystallization of the  $\delta$ -phase is less than 3 K. The fact makes it possible to characterize the  $\delta$ -solid solutions as quasi-ideal solution.

Results of microhardness measurements are in line with the plotted phase diagrams (Figs.1b). Curves have a flat maximum which is typical for systems with continuous solid solutions.

Phase diagrams of the above-mentioned systems are confirmed by powder X-ray

analysis (Fig.2). Powder diffraction patterns of starting compositions and intermediate alloys are qualitatively identical with slight displacement of reflections from one composition to another. For example, we provide the powder diffraction pattern of alloy with compositions 50mol% $Tl_9SbTe_6$ +50 mol% $Tl_9Gd(Tb)Te_6$ . Note that the lattice parameters of the solid solutions depend linearly on composition, i.e. subject to the Vegard's law.



**Fig.2.** XRD patterns for different compositions in the  $Tl_9GdTe_6$ - $Tl_9SbTe_6$  and  $Tl_9TbTe_6$ - $Tl_9SbTe_6$  systems

Plotted T-x diagrams afford ample opportunity to select compositions for growing

monocrystals of  $\delta$ -solid solution with given composition from the melt.

#### 4. CONCLUSION

The phase diagrams of the  $Tl_9GdTe_6$ - $Tl_9SbTe_6$  and  $Tl_9TbTe_6$ - $Tl_9SbTe_6$  systems have been plotted using various experimental methods. A continuous series of the substitutional solid solutions which are crystallized in  $Tl_5Te_3$  crystal type were found

in both systems. Proceeding from respective characteristics of the starting compositions one can assume that the  $Tl_9Sb_{1-x}Gd(Tb)_xTe_6$  ( $0 < x < 1$ ) phases possibly have thermoelectric and magnetic properties.

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**$Tl_9SbTe_6$ - $Tl_9GdTe_6$  VƏ  $Tl_9SbTe_6$ - $Tl_9TbTe_6$  SİSTEMLƏRİNDƏ FAZA TARAZLIQLARI**

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DTA, RFA və mikrobərkliyin ölçülməsi üsulları ilə  $Tl_9SbTe_6$ - $Tl_9GdTe_6$  və  $Tl_9SbTe_6$ - $Tl_9TbTe_6$  sistemlərində faza tarazlıqları öyrənilmiş, onların faza diaqramları, həmçinin kristal qəfəs parametrlərinin və mikrobərkliyinin tərkibdən asılılıq qrafikləri qurulmuşdur. Müəyyən edilmişdir ki, hər iki sistem  $Tl_9Gd(Tb)Te_6$  birləşmələrinin inkongruent əriməsi səbəbindən qeyri-kvazibinardır, lakin solidusdan aşağıda onlar stabildirlər və  $Tl_5Te_3$  tipli kristal quruluşa malik arasıkəsilməz məhlullar əmələ gəlməsilə xarakterizə olunurlar. Alınmış bərk məhlullar termoelektrik və maqnit materialları kimi maraq kəsb edirlər.

**Açar sözlər:** tallium–qadolinium telluridləri, tallium–terbium telluridləri, tallium–stibium telluridləri, faza tarazlıqları, bərk məhlullar, kristal quruluş.

**ФАЗОВЫЕ РАВНОВЕСИЯ В СИСТЕМАХ  $Tl_9SbTe_6$ - $Tl_9GdTe_6$  И  $Tl_9SbTe_6$ - $Tl_9TbTe_6$**

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Методами ДТА и РФА, а также измерением микротвердости изучены фазовые равновесия в системах  $Tl_9SbTe_6$ - $Tl_9GdTe_6$  и  $Tl_9SbTe_6$ - $Tl_9TbTe_6$ . Построены их фазовые диаграммы, а также концентрационные зависимости микротвердости и параметров кристаллической решетки. Показано, что обе системы неквазибинарны в силу инконгруэнтного плавления соединений  $Tl_9Gd(Tb)Te_6$ , однако ниже солидуса стабильны и характеризуются образованием непрерывных рядов твердых растворов со структурой  $Tl_5Te_3$ . Полученные твердые растворы представляют интерес как термоэлектрические и магнитные материалы.

**Ключевые слова:** теллуриды таллия-гадолиния, теллуриды таллия-тербия, теллуриды таллия-сурьмы, фазовые равновесия, твердые растворы, кристаллическая структура.

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