# Homogeneity Ranges of Tl<sub>5</sub>Te<sub>2</sub>Cl and Tl<sub>5</sub>Te<sub>2</sub>Br<sup>\*</sup>

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**Abstract**—This paper systematizes phase-diagram data for the Tl–TlCl(Br)–Te systems and presents their 500-K subsolidus phase diagrams.  $Tl_5Te_2Cl$  and  $Tl_5Te_2Br$  ( $Tl_5Te_3$  structure) are shown to be nonstoichiometric compounds with wide homogeneity ranges, which have been accurately determined using emf measurements, x-ray diffraction, and microhardness tests. Using emf data for reversible concentration cells with a thallium electrode, we have evaluated the partial thermodynamic functions of the thallium in the alloys studied, the standard thermodynamic functions of formation of  $Tl_5Te_2Cl$  and  $Tl_5Te_2Br$ , and their standard entropies. The crystal chemistry of these phases of variable composition is discussed in relation to the  $Tl_5Te_3$  structure.

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#### **INTRODUCTION**

The Tl–X–Hal (X = Se, Te; Hal = Cl, Br, I) systems have been studied extensively by several groups [1–8]. The pseudobinary joins TlHal–Tl<sub>2</sub>Se and TlI–Tl<sub>2</sub>Te were shown to contain ternary compounds of the Tl<sub>5</sub>X<sub>2</sub>Hal stoichiometry [1–3], which crystallize in the Tl<sub>5</sub>Te<sub>3</sub> structure (tetragonal symmetry, sp. gr. *I4/mcm*) [4]. The above ternary systems were studied in the composition regions Tl–TlHal–Se [5–7] and Tl–TlI–Te [3]. The results were used to construct *T–x* phase diagrams along a number of joins, several isothermal sections, and liquidus diagrams.

The ternary compounds  $Tl_5Se_2Hal$  and  $Tl_5Te_2I$  are known to have wide homogeneity ranges, which extend well beyond the TlHal–Tl<sub>2</sub>Se and TlI–Tl<sub>2</sub>Te pseudobinary systems. In earlier studies [5, 6, 8], emf measurements were used to evaluate the standard thermodynamic functions of formation of these thallium chalcogenides.

The lack of data on thallium telluride chlorides and thallium telluride bromides led our group to undertake a detailed study of the phase equilibria in the Tl–Te– Cl(Br) systems along several joins [9–15]. The pseudobinary systems TlCl(Br)–Tl<sub>2</sub>Te were shown to contain ternary compounds with the compositions Tl<sub>5</sub>Te<sub>2</sub>Cl and Tl<sub>5</sub>Te<sub>2</sub>Br, which melt incongruently by syntectic reactions at 708 and 750 K, respectively [9]. Both compounds are phases of variable composition.

According to powder x-ray diffraction (XRD) data, these compounds crystallize in tetragonal symmetry

 $(Tl_5Te_3 \text{ structure, sp. gr. } I4/mcm)$  with the following lattice parameters:

$$a = 8.921$$
 Å,  $c = 12.690$  Å,  $z = 4$  (Tl<sub>5</sub>Te<sub>2</sub>Cl);

 $a = 8.926 \text{ Å}, \quad c = 12.801 \text{ Å}, \quad z = 4 \text{ (T1}_5 \text{Te}_2 \text{Br)}.$ 

The  $Tl_5Te_2Cl(Br)$  compounds were shown to form a continuous series of solid solutions with  $Tl_5Te_3$  [10]. It is of interest to note that the TlCl(Br)-Tl<sub>2</sub>Te and  $Tl_5Te_3$ - $Tl_5Se_2Cl(Br)$  systems contain not only wide solid-solution ranges but also liquid-liquid miscibility gaps. The T-x phase diagrams of the TlCl(Br)–Te systems feature monotectic and degenerate eutectic equilibria [11]. The other joins studied, TlCl(Br)- $TITe(Tl_2Te_3)$  and  $Tl_5Te_2Cl(Br)$ -TITe are not pseudobinary: at least one end-member in these systems melts incongruently [12–15]. The subsolidus phase relations in these systems are, however, stable: the alloys consist of mixtures of the end-members. Only in the last two systems were limited solid solutions identified (based on Tl<sub>5</sub>Te<sub>2</sub>Cl(Br), with a limit at  $\approx$ 15 mol % TlTe [12, 131.

This paper describes our attempt to obtain a complete picture of the phase equilibria in the Tl–TlCl(Br)– Te systems, to more accurately determine the homogeneity ranges of the ternary compounds  $Tl_5Te_2Cl(Br)$ , and to evaluate the thermodynamic functions of their formation.

#### **EXPERIMENTAL**

For our experiments, we synthesized the compounds TlCl, TlBr, Tl<sub>2</sub>Te, TlTe, Tl<sub>5</sub>Te<sub>3</sub>, Tl<sub>2</sub>Te<sub>3</sub>, Tl<sub>5</sub>Te<sub>2</sub>Cl, and Tl<sub>5</sub>Te<sub>2</sub>Br. The thallium tellurides were prepared by melting high-purity (99.99%) thallium and tellurium at 750 K in silica tubes sealed off under a vac-

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uum of ~ $10^{-2}$  Pa. Given that TITe and Tl<sub>2</sub>Te<sub>3</sub> form peritectically at 573 and 511 K, respectively [16], these compounds were homogenized at 550 and 500 K for 200 and 300 h, respectively.

TICl and TIBr were prepared by an indirect process, as described elsewhere [17]. First, metallic thallium was dissolved in dilute (5%)  $H_2SO_4$  at  $\approx 350$  K to give a  $Tl_2SO_4$  solution. To a boiling 2%  $Tl_2SO_4$  solution was added dilute HCl or HBr until complete precipitation. After cooling, the TICl was separated from the solution, washed with distilled water, and dried for a long time at 110–120°C in a drying cabinet. Since TIBr is difficult to recrystallize, the mother liquor was removed by repeated boiling with water, followed by suction filtration. The material was dried in a desiccator over KOH at 390–400 K and stored in the dark (to avoid its decomposition).

The ternary compounds  $Tl_5Te_2Cl$  and  $Tl_5Te_2Br$  were synthesized by vacuum-melting a stoichiometric mixture of TlCl(Br) and Tl<sub>2</sub>Te. Since these compounds decompose into two immiscible liquids on melting [9], the ampule containing the melt was cooled very slowly (over a period of 5 h) from 800 to 700 and 740 K, respectively (temperatures intermediate between those of the syntectic and eutectic equilibria in the TlCl–Tl<sub>2</sub>Te and TlBr–Tl<sub>2</sub>Te systems), and held there for  $\approx 200$  h.

All of the synthesized compounds were identified by differential thermal analysis (DTA) and XRD.

Tl–TlCl(Br)–Te samples were prepared by melting the above compounds with Tl and Te in evacuated silica tubes, followed by prolonged (400–500 h) annealing at temperatures 30–50 K below the solidus. The samples were characterized by DTA (NTR-70 pyrometer, Chromel–Alumel thermocouples), XRD (DRON-2 powder diffractometer,  $CuK_{\alpha}$  radiation), microhardness measurements (PMT-3 tester) [18], and emf measurements using concentration cells of the type

$$(-)Tl(s) | pglycerol + KHal + TlHal | (TlHal-Tl2Te-Te)(s)(+)$$
(1)

at temperatures from 300 to 430 K. The emf was measured by a compensation technique with a V-7-34A high-resistance digital voltmeter. The procedures used to set up electrochemical cells and measure their emf were similar to those described earlier [5, 6, 9].

### **RESULTS AND DISCUSSION**

To check the earlier reported phase diagrams of the TlCl(Br)-Tl<sub>2</sub>Te systems [9], we studied these systems using emf and microhardness measurements. The results are presented in Figs. 1 and 2, together with the T-x phase diagrams.

Figure 1 demonstrates that, in the TlCl–Tl<sub>2</sub>Te system, the microhardness and the emf of cell (1) are continuous

functions of composition in the range 66.7-100 mol % Tl<sub>2</sub>Te, confirming that Tl<sub>5</sub>Te<sub>2</sub>Cl and Tl<sub>2</sub>Te form a continuous series of solid solutions.

According to XRD data, the solid solutions containing 66.7–88 mol % Tl<sub>2</sub>Te ( $\delta$ –phase) are isostructural with Tl<sub>5</sub>Te<sub>2</sub>Cl, and those containing  $\geq$ 90 mol % Tl<sub>2</sub>Te ( $\alpha$  phase) are similar in XRD pattern to Tl<sub>2</sub>Te (monoclinic structure, sp. gr.  $C_2/c$  [20]). The  $\alpha + \delta$  two-phase region is essentially degenerate (Fig. 1a, dashed lines). We assume that, at  $\approx$ 90 mol % Tl<sub>2</sub>Te, there is an  $\alpha \implies \delta$  morphotropic transition.

In going from 98 mol %  $Tl_2Te$  to  $Tl_5Te_2Cl$ , the microhardness of the solid solutions decreases, suggesting that the stoichiometric compound  $Tl_5Te_2Cl$  has the least distorted crystal lattice [18].

In the composition range <66.7 mol % Tl<sub>2</sub>Te, the microhardness of the  $\delta$  phase and the emf are composition-independent, in accordance with the constant phase composition of the system and the constant compositions of the coexisting phases.

In the TlBr–Tl<sub>2</sub>Te system, we found other solidstate phase relations (Fig. 2). The XRD, emf, and microhardness data for this system indicate the existence of an  $\alpha + \delta$  two-phase region ( $\approx 80-92 \text{ mol } \%$ Tl<sub>2</sub>Te).

Detailed characterization of thoroughly annealed alloys, especially in the composition regions  $Tl_2Te_-Tl_5Te_2Cl(Br)$ -TITe, enabled us to construct the 500-K subsolidus phase diagrams of the Tl-TlCl(Br)-Te systems. It can be seen in Fig. 3 that TlCl, the most thermodynamically stable compound in the Tl-TlCl-Te system, plays a key role in determining the phase relations in this system. The field of the  $Tl_5Te_2Cl$ -based phase ( $\delta$ ) is bounded by the  $Tl_5Te_{2-x}Cl_x$  (0 < x < 1) compositions on the Te-rich side and by the TlCl-Tl\_2Te join on the Tl-rich side. The  $Tl_2Te-Tl_5Te_2Cl-Tl_5-Te_3$  composition triangle contains an  $\alpha + \delta$  two-phase region, which narrows down in going from the Tl-Te constituent system to the morphotropic phase boundary in the TlCl-Tl\_2Te join.

In Fig. 3, we indicate the 300-K emf values (mV) for some of the three-phase regions. The emf is composition-independent within each of these regions and changes jumpwise in going from one region to another. Note that the emf in the TICl–TITe–TI<sub>2</sub>Te<sub>3</sub> and TICl– TI<sub>2</sub>Te<sub>3</sub>–Te three-phase regions coincides to within  $\pm 3$  mV with the values reported by Vasil'ev et al. [21] for TITe and TI<sub>2</sub>Te<sub>3</sub>, respectively. This lends support to the subsolidus phase diagram under consideration and indicates that there are no significant ranges of TITe- or TI<sub>2</sub>Te<sub>3</sub>-based solid solutions.

The subsolidus phase diagram of the Tl–TlBr–Te system is similar to that in Fig. 3, with the only difference that the  $\alpha + \delta$  two-phase region in the Br system is somewhat broader.



Fig. 1. (a) T-x, (b) H-x, and (c) E-x diagrams of the TlCl-Tl<sub>2</sub>Te system.

Thus, the thallium telluride halides with the  $Tl_5Te_3$  structure are typical nonstoichiometric compounds. To gain insight into the mechanism underlying the formation of the crystal lattice of these compounds and the formation of  $Tl_5Te_{2-x}Hal_x$  solid solutions, consider the salient features of the  $Tl_5Te_3$  structure.

 $Tl_5Te_3$  crystallizes in tetragonal symmetry and forms its own structure type [4], with four formula units per unit cell. The basic structural component of  $Tl_5Te_3$ is a tellurium octahedron with a thallium atom in its center (Fig. 4). The octahedra share corners to form a  $Tl_4Te_{12}$ , or (TITe<sub>3</sub>)<sub>4</sub>, framework. The other 16 thallium atoms per unit cell reside between the octahedra and link them along the *C* axis, forming the  $Tl_{16}$ (TITe<sub>3</sub>)<sub>4</sub> unit cell.

The crystal-chemical formula of the  $Tl_5Te_2Hal$  compounds can be represented in the form

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 $Tl_{16}(TlTe_2Hal)_4$  [9]. Their unit cell results from halogen substitution for the Te(2) atoms residing in the opposite corners of the octahedron along the C axis (Fig. 4). The difference in charge between the X<sup>2-</sup> and Hal<sup>-</sup> ions is compensated by the transition of all the thallium atoms located in the center of the octahedra to the oxidation state 1+. It is easy to show that such anion substitutions may, in principle, lead not only to the boundary composition Tl<sub>5</sub>Te<sub>2</sub>Hal of Tl<sub>5</sub>Te<sub>3</sub>-structured chalcogenides but also to all  $Tl_5Te_{3-x}Hal_x$  (0 < x < 1) intermediate compositions. The electroneutrality of each composition is ensured through the transition of an appropriate fraction of the thallium cations to the oxidation state 1+. This leads to the formation of continuous or wide series of Tl<sub>5</sub>Te<sub>3</sub>-structured solid solutions in the Tl–TlHal–Te systems.

Using emf data, we were able not only to more accurately determine the boundaries of phase fields in the



**Fig. 2.** (a) *T*–*x*, (b) *H*–*x*, and (c) *E*–*x* diagrams of the TlBr–Tl<sub>2</sub>Te system.



Fig. 3. 500-K section of the TI-TICI-Te phase diagram; numbers in parentheses indicate the 300-K emf (mV) for the phase region.

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Fig. 4. Basic structural component of Tl<sub>5</sub>Te<sub>3</sub>.

Tl-TlCl(Br)-Te phase diagrams but also to evaluate the thermodynamic functions of  $Tl_5Te_2Cl$  and  $Tl_5Te_2Br$ .

Table 1 presents linear equations for the temperature-dependent emf of cell (1) in some of the phase fields in the Tl–TlCl(Br)–Te systems. The equations were obtained by least squares fitting our experimental data [22].

From the data in Table 1, we evaluated partial thermodynamic functions of thallium in the alloys using well-known relations [19] (Table 2).

It can be seen from Fig. 3 that partial molar values of thallium in the three-phase regions TlHal– $Tl_5Te_2Hal$ –TlTe are the thermodynamic functions of potential-forming solid-state reactions of the form

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## $TI + 0.5TIHal + TITe = 0.5Tl_5Te_2Hal.$

Therefore, the standard thermodynamic functions of formation of the ternary compounds can be found using relations of the form

$$\Delta G^{0}(\mathrm{Tl}_{5}\mathrm{Te}_{2}\mathrm{Hal})$$
  
=  $2\overline{\Delta G}(\mathrm{Tl}) + \Delta G^{0}(\mathrm{Tl}\mathrm{Hal}) + 2\Delta G(\mathrm{Tl}\mathrm{Te}),$ 

where  $\Delta G^0$  is the standard Gibbs energy, and their standard entropy can be found as

$$S^{0}(\mathrm{Tl}_{5}\mathrm{Te}_{2}\mathrm{Hal}) = 2\overline{\Delta S}(\mathrm{Tl}) + 2S^{0}(\mathrm{Tl}) + S^{0}(\mathrm{Tl}\mathrm{Hal}) + S^{0}(\mathrm{Tl}\mathrm{Te}).$$

Phase field in Fig. 3 $E (mV) = a + bT \pm 2S_E(T)$ TlCl-Tl2Te3-Te $461.2 + 0.032T \pm 2[(1.6/22) + 5 \times 10^{-5}(T - 361.6)^2]^{1/2}$ TlCl-TlTe-Tl2Te3 $431.8 + 0.035T \pm 2[(0.9/22) + 3 \times 10^{-5}(T - 361.6)^2]^{1/2}$ TlCl-TlTe-Tl5Te2Cl $420.4 - 0.012T \pm 2[(2.4/22) + 7.5 \times 10^{-5}(T - 360.4)^2]^{1/2}$ TlBr-Tl2Te3-Te $464.8 + 0.013T \pm 2[(2.2/22) + 6 \times 10^{-5}(T - 359.5)^2]^{1/2}$ TlBr-TlTe-Tl2Te3 $434.2 + 0.030T \pm 2[(3.7/22) + 9 \times 10^{-5}(T - 359.5)^2]^{1/2}$ TlBr-TlTe-Tl5Te2Br $435.5 - 0.003T \pm 2[(1.7/22) + 5.3 \times 10^{-5}(T - 360.4)^2]^{1/2}$ 

Table 1. Temperature-dependent emf of cell (1) for some of the phase fields in the Tl-TlCl(Br)-Te systems

**Table 2.** Relative partial thermodynamic functions of thalli-um in Tl–TlCl(Br)–Te alloys at 298 K

Phase field in Fig. 3	$-\Delta \overline{G}(\mathrm{Tl})$	$-\Delta \overline{H}(\mathrm{Tl})$	$-\Delta \bar{S}(\mathrm{Tl})$
C	kJ/mol		J/(mol K)
TICI-TITe-TI <sub>5</sub> Te <sub>2</sub> Cl	$40.920 \pm \pm 0.122$	$40.563 \pm \pm 0.605$	1.198 ± ± 1.671
TlBr–TlTe–Tl <sub>5</sub> Te <sub>2</sub> Br	$41.819 \pm 20.093$	$41.733 \pm 1.392$	$0.289 \pm \pm 1.41$

**Table 3.** Standard thermodynamic functions of thallium telluride halides and input reference data

Compound	$-\Delta G^0$ (298 K)	$-\Delta H^0(298 \text{ K})$	<i>S</i> <sup>0</sup> (298 K),
Ĩ	kJ/mol		J/(mol K)
TlCl [23]	$185.0 \pm 0.8$	$208.3\pm3.0$	$111.5\pm0.2$
TIBr [23]	$167.4 \pm 0.6$	$172.7\pm0.7$	$122.6\pm0.2$
TITe [21]	$44.5\pm0.4$	$43.9\pm0.5$	$115.9\pm1.6$
Tl <sub>5</sub> Te <sub>2</sub> Cl	$355.9 \pm 1.1$	$377.1\pm5.0$	$474.1\pm6.8$
Tl <sub>5</sub> Te <sub>2</sub> Br	$340.1 \pm 1.0$	$343.9\pm4.4$	$483.4\pm6.2$
$Tl_5Te_2I$ [8]	$296.5 \pm 5.3$	$286.8\pm3.7$	$511 \pm 8.0$

In calculations by these equations, we used reference data for TICl, TIBr, TITe, and Tl (Table 3). Uncertainties were evaluated using error propagation analysis. The results are presented in Table 3.

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