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Thermodynamic Properties of Terbium Tellurides

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Abstract

The paper presents the results of a study of solid-phase equilibria in the Tb–Te system and the thermodynamic properties of terbium tellurides obtained by the methods of electromotive forces and X-ray diffraction analysis. Based on the experimental data, it was established that the TbTe, Tb_2Te_3 , $TbTe_2 \ \mu$ TbTe₃ compounds are formed in the system. For the investigations of the alloys from the two-phase regions $TbTe_3+Te$, $TbTe_2+TbTe_3$, and $Tb_2Te_3+TbTe_2$, the EMF of concentration cells relative to the TbTe electrode was measured. The EMF of concentration cells relative to the terbium electrode was measured for the TbTe+Tb_2T_3 region. The partial thermodynamic functions of TbTe and Tb in alloys were determined by combining the EMF measurements of both types in the 300–450 K temperature range, based on which the standard thermodynamic functions of formation and standard entropies of the indicated terbium tellurides were calculated.

Keywords: terbium tellurides, electromotive forces method, thermodynamic functions

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1. Introduction

Rare earth element (REE) compounds are among the promising functional materials widely used in aerospace system applications, high-power radiofrequency sources, computer hard drives, battery electrodes for high-power batteries, etc. [1, 2]. Among them, REE chalcogenides, possessing high thermal stability, resistance to changes in environmental conditions, unique magnetic, optical, and thermoelectric properties, which are used in modern electronic technology [3–10].

The development and optimization of methods for the directed synthesis of new phases are based on data about phase equilibria in the corresponding systems and the thermodynamic properties of intermediate phases [11–13].

Although the phase diagrams of most Ln-Te type binary systems have been studied in detail and presented in a number of monographs and handbooks [3, 10, 14] phase diagram of the Tb-Te system has not yet been constructed. According to [3], terbium with tellurium forms following compounds: TbTe, Tb_2Te_3 , $TbTe_{1.8}$, Tb_2Te_5 , and TbTe, Tb_2Te_3 , TbTe, Tb_2Te_5 , and TbTe, Tb_2Te_3 , TbTe, Tb_2Te_3 , TbTe, Tb_2Te_5 , and TbTe, Tb_2Te_3 , TbTe, Tb_2Te_3 , more confirmed. However, we have not found any information on the crystal structure and properties of Tb_2Te_5 .

In the literature, the experimental data on the thermodynamic properties of terbium tellurides are very limited. The handbook [17] contains the estimated data on the standard enthalpy of formation and entropy of TbTe and Tb_2Te_3 . In the recently published studies [18, 19], the thermodynamic functions of formation and the entropy of the Tb_2Te_3 compound were estimated by the "tetrad effect" method.

This study presents the results of an investigation of solid-phase equilibria in the Tb-Te system and the thermodynamic properties of terbium tellurides.

2. Experimental

For research, alloys of the Tb-Te system with compositions > 50 at% Te (each weighing 0.5 g) were synthesized. The elements and reagents purchased from Alfa Aesar were used. The synthesis was carried out by direct interaction of elemental terbium (CAS No. 7440-27-9) and tellurium (CAS No. 13494-80-9) in evacuated $(10^{-2} Pa)$ quartz ampoules. In order to prevent the interaction of terbium with the inner walls of quartz ampoules, the synthesis of alloys was performed in graphitized ampoules. The ampoules were graphitized by the thermal decomposition of toluene.

After keeping the ampoules at 1000 K for 24 h, the alloys were ground into a powder, mixed, pressed into tablets, and annealed at 800 K (alloys with compositions 50–75 at% Te) or 700 K (alloys with compositions > 75 at% Te) for 1000 hours. Then the alloys were cooled in the switched-off furnace and were investigated by XRD (Bruker D8 diffractometer, CuK α_1 radiation). The XRD results confirmed the existence of the TbTe, Tb₂Te₃, TbTe₃, and TbTe₃ compounds.

For the investigation of the thermodynamic properties of phases of the Tb-Te system by EMF method, the following concentration cells of types (1) and (2) were assembled and their EMF were measured in the temperature range of 300–450 K.

(-) Tb (s.) | glycerol + KCl + TbCl₃ | (TbTe1_{+x})(s.) (+) (1)

(-)TbTe(s.)|glycerol+KCl+TbCl₃|(TbTe1_{+x})(s.)(+) (2)

Terbium was used as the left electrode in a type (1) cell, and terbium monotelluride with a slight excess of tellurium (composition $\text{TbTe}_{1.01}$) was used in a type (2) cell. Synthesized equilibrium alloys with different compositions from the two-phase regions $\text{TbTe+Tb}_2\text{Te}_3$ (50.3 and 55 at% Te), Tb_2Te_3 +TbTe₂ (61 and 65 at% Te), TbTe_2 +TbTe₃ (68 and 72 at% Te) and TbTe_3 +Te (77 and 90 at% Te) were used as the right electrodes. A sample with the composition 50.3 at% Te (TbTe_{1.01}) was used in a type (1) cell as the right electrode and reproducible results were obtained.

The phase compositions of all the indicated alloys were confirmed by XRD analysis. As an example, powder X-ray diffraction patterns of an alloy with a composition of 55 at% Te are shown in Fig. 1. As can be seen, this sample is two-phase and consists of a mixture of TbTe+Tb₂Te₃ compounds.

The terbium electrode was prepared by fixing a piece of metallic terbium on a molybdenum wire (down conductor), and all other samples were prepared by pressing the corresponding powder alloys on down conductors in the form of cylindrical tablets (diameter ~7 mm and thickness 2–3 mm)

In both electrochemical cells, a glycerol (CAS No. 56-81-5) solution of KCl (CAS No. 7447-40-7) with a small addition (0.1%) amount of TbCl_3 (CAS No. 10042-88-3) served as the electrolyte.



Fig. 1. The powder X-ray diffraction pattern of an alloy from TbTe₃+Tb₂Te₃ two-phase regions

Since the electrolyte should not contain moisture and oxygen, the glycerol was thoroughly dried and degassed by evacuation at \sim 450 K.

The EMF method with glycerol electrolyte has been successfully used for many years for the thermodynamic study of a number of binary and ternary chalcogenide systems [20–26].

The detailed descriptions of the methods for the preparation of the electrodes and electrolyte and assembly of the electrochemical cell were described in studies [20, 21, 25].

The EMF measurements were carried out using a Keithley Model 193 high-resistance digital voltmeter. The temperature of the electrochemical cells was measured with chromel-alumel thermocouples and a mercury thermometer with an accuracy of 0.5 K.

The first equilibrium EMF values were obtained after keeping the cell at ~400 K for 40–60 h, while the subsequent EMF values were obtained after 3–4 h when reaching the desired temperature. The EMF values were considered equilibrium values if they did not differ from each other at repeated measurements at a given temperature by more than 0.2 mV, regardless of the direction of the temperature change. During the experiments, the EMF of each sample was measured 2–3 times at two selective temperatures in order to control the reversibility of the cell.

Taking into account the results of our previous studies of Ln-Te systems by the EMF method [22, 26], we used the cell of type (1) only for study alloys from the TbTe + Tb_2Te_3 region, and reproducible results for both electrode-alloys were obtained. For other phase regions, type (2) cells were used and the reproducible results were obtained. From each heterogeneous region, two alloys were examined. The EMF measurements for two alloys from the same heterogeneous region coincided with an accuracy of 0.5 mV.

3. Results and discussion

The obtained temperature dependences of the EMF for all studied alloys of the Tb-Te system were linear (Fig. 2), which allowed performing thermodynamic calculations using the leastsquares method. Calculations were performed using the Microsoft Office Excel 2003 computer program. The obtained linear equations are presented in Table 1 in the form:

$$E = a + bT \pm t \left[\frac{S_E^2}{n} + \frac{S_E^2 (T - \overline{T})^2}{\sum (T_i - \overline{T})^2} \right]^{\frac{1}{2}},$$
 (3)



Fig. 2. Temperature dependencies of EMF for alloys of the TbTe+ Tb_2Te_3 (cell of type (1)) and $Tb_2Te_3+TbTe_2$, $TbTe_2+TbTe_3$, $TbTe_3+Te$ (cell of type (2)) phase regions of the Tb-Te system

Table 1. Relations between EMF and the temperature for type $(1)^*$ and (2) cells in some phase regions of the Tb-Te system in the temperature range of 300–450 K

N⁰	Phase region	$E, \mathrm{mV} = a + bT \pm 2[S_E^2 / n + S_b^2 (T - \overline{T})]^{1/2}$
1	TbTe ₃ +Te	$348.29 + 0.01664T \pm 2 \left[\frac{0.67}{30} + 1.13 \cdot 10^{-5} (T - 375.62) \right]^{1/2}$
2	TbTe ₂ +TbTe ₃	$386.99 + 0.03379T \pm 2 \left[\frac{0.62}{30} + 1.05 \cdot 10^{-5} (T - 375.62) \right]^{1/2}$
3	Tb ₂ Te ₃ +TbTe ₂	$425.19 + 0.05492T \pm 2 \left[\frac{0.53}{30} + 8.9 \cdot 10^{-6} (T - 375.62) \right]^{1/2}$
4	*TbTe+Tb ₂ Te ₃	$910.32 - 0.07214T \pm 2 \left[\frac{0.69}{30} + 1.18 \cdot 10^{-5} (T - 374.89) \right]^{1/2}$

where *a* and *b* – empirical constants; *n* – is the number of pairs of T and E values; S_E – the error variance of the EMF measurements; \overline{T} – the average of the absolute temperature; *t* – the Student's t-test. At the confidence level of 95 % and *n* = 30, the Student's t-test ≤ 2 .

From the obtained equations (Table 1) according to the relations [20, 21]:

$$\Delta \overline{G}_i = -zFE, \qquad (4)$$

$$\Delta \overline{S}_i = zF \left(\frac{\partial E}{\partial T}\right)_p = zFb, \qquad (5)$$

$$\Delta \overline{H}_i = -zF \left[E - T \left(\frac{\partial E}{\partial T} \right)_p \right] = -zFa.$$
(6)

the partial molar Gibbs free energy, enthalpy, and entropy of TbTe in two-phase alloys $Tb_2Te_3+TbTe_2$, $TbTe_2+TbTe_3$, and $TbTe_3+Te$ (Table 2) and terbium in alloys $TbTe+Tb_2Te_3$ (Table 3) were calculated.

The values given in Table 2 represent the difference between the corresponding partial molar functions of terbium for the right and left electrodes of the type (2) cell. For example,

Фазовая	$-\Delta \overline{G}_{ ext{TbTe}}$	$-\Delta \overline{H}_{ ext{TbTe}}$	$\Delta \overline{S}$ TbTe	
область	кJ/m	J/(mol·K)		
TbTe ₃ +Te	127.82±0.15	123.08±0.65	15.90±1.72	
TbTe ₂ +TbTe ₃	114.94±0.17	112.02±0.71	9.78±1.87	
Tb ₂ Te ₃ +TbTe ₃	102.26±0.17	100.82±0.74	4.82±1.95	

Table 2. Relative partial molar functions of TbTe in the alloys of the Tb-Te system at 298 K

Table 3. Relative partial molar functions of terbium in the alloys of the Tb-Te system at 298 K

Phase region	$-\Delta \overline{G}_{\text{Tb}}$	$-\Delta \overline{H}_{Tb}$	$\Delta \overline{S}_{Tb}$ I/(mol·K)
	KJ,	<i>"</i> "(mor ity	
TbTe ₃	385.10±0.33	386.58±1.40	-4.96 ± 3.71
TbTe ₂	372.22±0.35	375.52±1.46	-11.07 ± 3.86
Tb ₂ Te ₃	359.54±0.35	364.32±1.49	-16.03±3.94
TbTe	257.28±0.18	263.50±0.75	-20.88±1.99

$$\Delta \overline{G}_{TbTe}(TbTe_{1+x}) = \Delta \overline{G}_{Tb} (TbTe_{1+x}) - \Delta \overline{G}_{Tb}(TbTe). (7)$$

and

 $\Delta \overline{G}_{\text{Tb}}(\text{TbTe}_{1+x}) = \Delta \overline{G}_{\text{TbTe}}(\text{TbTe}_{1+x}) + \Delta \overline{G}_{\text{Tb}}(\text{TbTe}).(8)$

The partial molar functions of terbium in TbTe_{1+x} alloys (right electrodes of type (2) cell) were calculated using the relation (8) and are presented in Table 3. 3.

The phase compositions of alloys (Table 3) of indicated two-phase region show that the partial molar functions of terbium in them are the thermodynamic characteristics of the following potential formation reactions (the state of substances is crystalline):

 $Tb + 3Te = TbTe_{3}$ $Tb + 2TbTe_{3} = 3TbTe_{2}$ $Tb + 3TbTe_{2} = 2Tb_{2}Te_{3}$ $Tb + Tb_{2}Te_{3} = 3TbTe$

Therefore, the standard thermodynamic functions of the formation of terbium tellurides can be calculated using the relations

$$\Delta Z^{0} (\text{TbTe}_{3}) = \Delta \overline{Z}_{\text{Tb}},$$

$$\Delta Z^{0} (\text{TbTe}_{2}) = \frac{1}{3} \Delta \overline{Z}_{\text{Tb}} + \frac{2}{3} \Delta Z^{0} (\text{TbTe}_{3}),$$

$$\Delta Z^{0} (\text{Tb}_{2}\text{Te}_{3}) = 0.5 \Delta \overline{Z}_{\text{Tb}} + 1.5 \Delta Z^{0} (\text{TbTe}_{2}),$$

$$\Delta Z^{0} (\text{TbTe}) = \frac{1}{3} \Delta \overline{Z}_{\text{Tb}} + \frac{1}{3} \Delta Z^{0} (\text{Tb}_{2}\text{Te}_{3}),$$

where $Z \equiv G, H, S$, while the standard entropies were calculated as

$$S^{0} (TbTe_{3}) = [\Delta \overline{S}_{Tb} + S^{0} (Tb)] + 3S^{0} (Te),$$

$$S^{0} (TbTe_{2}) = \frac{1}{3} [\Delta \overline{S}_{Tb} + S^{0} (Tb)] + \frac{2}{3} S^{0} (TbTe_{3}),$$

$$S^{0} (Tb_{2}Te_{3}) = 0.5 [\Delta \overline{S}_{Tb} + S^{0} (Tb)] + 1.5S^{0} (TbTe_{2}),$$

$$S^{0} (TbTe) = \frac{1}{3} [\Delta \overline{S}_{Tb} + S^{0} (Tb)] + \frac{1}{3} S^{0} (Tb_{2}Te_{3}).$$

For the thermodynamic calculations, in addition to our experimental data (Table 3), we used the literature data [28] on the standard entropies of elemental terbium (73.51 \pm 0.42 kJ/mol) and tellurium (49.50 \pm 0.21 kJ/mol). The results are presented in Table 4. In all cases, the standard uncertainties were calculated by accumulation of errors method.

Table 4 shows the estimated data for the TbTe and Tb_2Te_3 compounds given in [17, 18].

4. Conclusions

We have presented the results of a comprehensive study of solid-phase equilibria in the Tb-Te system and the thermodynamic properties of terbium tellurides by EMF and XRD methods. The compounds TbTe, Tb_2Te_3 , $TbTe_2$, and $TbTe_3$ were revealed in the system based on the experimental data. The partial thermodynamic functions of TbTe and Tb in alloys have been determined based on EMF measurements of types (1) and (2) concentration cells in the 300–450 K temperature range. The standard thermodynamic functions of the standard entropies of the

Compound	$-\Delta_f G^0$ (298 K)	$-\Delta_f H^0$ (298 K)	ΔS^0 (298 K)	<i>S</i> ⁰ (298 К)
r r r r	кЈ/	mol	J/(mol·K)	
TbTe ₃	385.1±0.3	386.6±1.4	-5.0 ± 3.7	194.9±4.8
TbTe ₂	380.8±0.4	382.9±1.5	-7.0±3.8	165.6±4.6
	750.98±0.7	756.5±3.0	-18.5±7.8	277.0±9.3
Tb ₂ Te ₃	_	795±125 [17]		264±21 [17]
	803.5	818 [19]		247.6 [19]
ThTo	336.1±0.3	340.0±1.3	-13.13.3	109.9±3.9
ible		314±63 [17]		97±10 [17]

Table 4. Standard integral thermodynamic functions of terbium tellurides

TbTe, Tb_2Te_3 , $TbTe_2$, and $TbTe_3$ compounds were calculated by the combination of these data.

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Conflict of interests

The authors declare that they have no known competing financial interests or personal relationships that could have influenced the work reported in this paper.

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