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THERMODYNAMIC PROPERTIES OF THE Sb_2Te_3 COMPOUND

F.R.Aliyev¹, E.N.Orujlu², D.M.Babanly³

¹Azerbaijan State Oil and Industry University

²Institute Catalysis and Inorganic Chemistry, NAS of Azerbaijan

³French–Azerbaijani University, UFAZ

fariz_ar@hotmail.com

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Thermodynamic properties of the Sb_2Te_3 compound were studied by measuring electromotive force (EMF) with a liquid electrolyte in the temperature range of 300–450 K. The partial molar functions of antimony in alloys and the corresponding standard integral thermodynamic functions of the Sb_2Te_3 compound were calculated for the first time based on the EMF measurements under standard conditions. Comparative analysis of obtained results with literature data was carried out.

Keywords: Sb_2Te_3 , EMF method, partial molar functions, thermodynamic functions, tetradymite-like structure.

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Introduction

Antimony and bismuth tellurides with a tetradymite-like layered structure and phases based on them have been in the center of attention of researchers since the last century due to their unique properties. These materials exhibit thermoelectric properties and are used in mini-power generation systems, micro-coolers, temperature control devices, etc. [1–4]. Discovery of a new quantum state of matter – a topological insulator (TI) [5, 6] has sharply increased interest in layered compounds. It was found that layered phases with TIs properties are considered extremely promising for various applications including spintronic, medicine, quantum computers, lasers, security systems, etc. [7–11].

The thermodynamic functions of compounds are their fundamental characteristics. These properties in conjunction with phase diagrams provide a basis for crystal growth, doping, and optimization of preparation conditions for the new similar materials [12, 13].

Despite the increased interest to antimony tellurides, sufficiently reliable data of phase equilibria in the Sb–Te system has not been obtained yet, and their thermodynamic properties have not been studied in detail [14–16]. Analysis of the literature shows that available thermodynamics data refers mainly to the Sb_2Te_3 compound.

Various studies have been performed on

the phase equilibria of Sb–Te system [17–21]. The system is characterized by the formation of the congruently melting Sb_2Te_3 compound at 891 K. This compound has a rhombohedral lattice (sp. gr. $R\bar{3}m$) of the tetradymite type with the following lattice parameters in the hexagonal configuration: $a=4.264$ Å, $c=30.458$ Å. Mixed-layer compounds of the $n\text{Sb}_2\cdot m\text{Sb}_2\text{Te}_3$ [21, 22] homologous series which have been studied very poorly, due to the difficulty of obtaining high-quality samples, especially monocrystals, also exist in this system. Hence, a detailed investigation of the mutual interaction in the Sb–Te system is needed for more accurate results.

In this contribution, we have presented results of the thermodynamic study of Sb_2Te_3 compound using EMF method. This method is widely used to investigate thermodynamic properties of binary and complex metal chalcogenides [23–29]. In the study of solid metal chalcogenide phases, it is advisable to carry out the measurements at temperatures below the solidus. With this goal, glycerol solutions of alkali metal salts [23–27] and ionic liquids [28, 30] are successfully used.

Experimental part

To study the thermodynamic properties of the Sb_2Te_3 system by the EMF method, the concentration cells of the type

(–)Sb(solid)|glycerol Sb^{3+} |(Sb₂Te₃+Te)(solid)(+) (1) were assembled and their EMF in the temperature range 300–450 K were measured.

Elemental antimony was used as the left electrode and alloys from the Sb₂Te₃+Te region of the Sb–Te system (65 and 75 at.% Te) as right electrodes (Figure 1). These alloys were synthesized by fusion of high purity initial elemental components (purchased from Alfa Aesar) in evacuated ($\sim 10^{-2}$ Pa) quartz ampoules at 1000 K. After fusion, samples were annealed at 750 K for 200 h in order to reach the equilibrium state. The phase composition of the obtained alloys was confirmed by PXRD method which was performed at room temperature on a Bruker D2 PHASER diffractometer using CuK α radiation within $2\theta=5^{\circ}$ – 70° .

As an electrolyte, we used a glycerol solution of KCl with the addition of 0.1 wt.% of SbCl₃. Due to the presence of moisture or oxygen in the electrolyte, glycerol was thoroughly dehydrated and degassed at ~ 450 K under a dynamic vacuum and anhydrous chemically pure salts were used.

EMF measurements were performed using the Keithley 2100 6 1/2 Digit Multimeter.

The first equilibrium values of the EMF were recorded after maintaining the electrochemical cell at ~ 350 K for 40–60 hours, and subsequent values were obtained every 3–4 hours after a specific temperature was established. The system was considered to be in equilibrium if the EMF values were constant or their variations did not differ from each other upon repeated measurements at a given temperature by more than 0.2 mV, regardless of the direction of the temperature change.

Results and discussion

Results of EMF measurements of the electrochemical cells of type (1) showed that the EMF values linearly depend on temperature (Figure 2). This allows us to use EMF data for thermodynamic calculations. A linear relationship between EMF and temperature made it possible to treat EMF measurement results using the least-squares method via computer software. Experimentally obtained data for temperature (T_i), EMF (E_i) and data associated with the calculation steps for Sb₂Te₃ + Te of the Sb–Te system are listed in Table 1.

Fig. 1. PXRD pattern of the alloy of composition 75 at. % Te of the Sb–Te system.

Fig. 2. Temperature dependences of EMF of the concentration cells type (1) for $\text{Sb}_2\text{Te}_3 + \text{Te}$ phase region of the Sb-Te system.

Table 1. Experimentally obtained data for temperature (T_i), EMF (E_i) and data related to the stages of calculation for the $\text{Sb}_2\text{Te}_3 + \text{Te}$ phase region of the Sb-Te system

T_i, K	E_i, mV	$T_i - \bar{T}$	$E_i(T_i - \bar{T})$	$(T_i - \bar{T})^2$	$\bar{E}$	$E_i - \bar{E}$	$(E_i - \bar{E})^2$
299.7	106.05	-51.69	-5481.37	2671.51	106.19	-0.14	0.02
301.8	106.11	-49.59	-5261.64	2458.84	106.22	0.11	0.01
305.6	105.15	-45.79	-4814.47	2096.42	106.27	-1.12	1.26
308.5	106.99	-42.89	-4588.44	1839.27	106.31	0.68	0.46
311.7	105.73	-39.69	-4196.07	1575.03	106.36	-0.63	0.39
315.6	106.28	-35.79	-3803.41	1280.69	106.41	-0.13	0.02
319.4	107.23	-31.99	-3429.93	1023.15	106.46	0.77	0.59
323.3	107.01	-28.09	-3005.55	788.86	106.52	0.49	0.24
329.5	106.02	-21.89	-2320.42	479.03	106.60	-0.58	0.34
334.7	106.96	-16.69	-1784.81	278.44	106.68	0.28	0.08
337.3	105.78	-14.09	-1490.09	198.43	106.71	-0.93	0.87
341.8	107.32	-9.59	-1028.84	91.90	106.78	0.54	0.30
345.9	106.45	-5.49	-584.06	30.10	106.83	-0.38	0.15
348.1	107.25	-3.29	-352.49	10.80	106.86	0.39	0.15
350	107.98	-1.39	-149.73	1.92	106.89	1.09	1.19
353.2	106.41	1.81	192.96	3.29	106.93	-0.52	0.27
356.5	107.63	5.11	550.35	26.15	106.98	0.65	0.42
359.5	107.86	8.11	875.10	65.83	107.02	0.84	0.70
362	107.23	10.61	1138.07	112.64	107.06	0.17	0.03
367.3	107.88	15.91	1716.73	253.23	107.13	0.75	0.56
371.4	106.65	20.01	2134.42	400.53	107.19	-0.54	0.29
374.3	106.12	22.91	2431.56	525.02	107.23	-1.11	1.22
379.7	107.35	28.31	3039.44	801.64	107.30	0.05	0.00
382.9	107.98	31.51	3402.81	993.09	107.35	0.63	0.40
386.1	107.22	34.71	3721.96	1205.02	107.39	-0.17	0.03
389.4	107.69	38.01	4093.66	1445.01	107.44	0.25	0.06
391.1	106.88	39.71	4244.56	1577.15	107.46	-0.58	0.34
395.7	107.66	44.31	4770.77	1963.67	107.52	0.14	0.02
398.8	106.75	47.41	5061.37	2248.02	107.57	-0.82	0.67
400.8	107.63	49.41	5318.36	2441.68	107.59	0.04	0.00
$\bar{T} = 351.4$	$\bar{E} = 106.908$		$\Sigma = 400.79$	$\Sigma = 28886.37$			$\Sigma = 11.09$

We used the method of processing the results of EMF measurements described in [23, 31]. Obtained linear equation of the type (2) are listed in the literature recommended form:

$$E = a + bT \pm t[(S_E^2/n) + S_b^2 \cdot (T - \bar{T})^2]^{1/2} \quad (2)$$

where n – is the number of pairs of values of E and T ; S_E and S_b – are the variances of individual measurements of EMF and coefficient b , respectively; $\bar{T}$ – average absolute temperature, Student's t -test. At the confidence level of 95% and $n=30$, the Student's test is $t \leq 2$.

$$E_{mv} = 102.03 + 0.0139 \pm 2 \left[\frac{0.37}{30} + 1.3 \cdot 10^{-5} \times (T - 351.39)^2 \right]^{1/2} \quad (3)$$

Using the obtained equations of type (3) and thermodynamic expressions:

$$\Delta \bar{G}_{Sb} = -zFE \quad (4)$$

$$\Delta \bar{H}_{Sb} = -z \left[E - T \left(\frac{\partial E}{\partial T} \right)_p \right] = -zFa \quad (5)$$

$$\Delta \bar{S}_{Sb} = -zF \left(\frac{\partial E}{\partial T} \right)_p = zFb \quad (6)$$

The partial molar functions of antimony in the alloys at 298 K were calculated.

$$\Delta \bar{G}_{Sb} = -30.73 \pm 0.09 \text{ kJ/mole}$$

$$\Delta \bar{H}_{Sb} = -29.53 \pm 0.49 \text{ kJ/mole}$$

$$\Delta \bar{S}_{Sb} = 4.02 \pm 1.38 \text{ J/(K} \cdot \text{mole)}$$

According to the phase diagram of the Sb–Te system, these quantities are thermodynamic functions of the following virtual-cell reaction.

According to the reaction equation (7), the standard thermodynamic functions of formation Sb_2Te_3 compound were calculated using the following expressions:

$$\Delta_f G_{\text{Sb}_2\text{Te}_3}^\circ = 2\Delta \bar{G}_{Sb} \quad (8)$$

$$\Delta_f H_{\text{Sb}_2\text{Te}_3}^\circ = 2\Delta \bar{H}_{Sb} \quad (9)$$

$$\Delta_f S_{\text{Sb}_2\text{Te}_3}^\circ = 2\Delta \bar{S}_{Sb} \quad (10)$$

The standard entropy was calculated as:

$$S_{\text{Sb}_2\text{Te}_3}^\circ = 2\Delta \bar{S}_{Sb} + 2S_{Sb}^\circ + 3S_{Te}^\circ$$

The values obtained in this work for the standard integral thermodynamic functions of the Sb_2Te_3 compound with comparison of the results of several original works and the data recommended in modern handbooks and digital databases are given in Table 2.

Table 2. Standard integral thermodynamic functions of Sb_2Te_3

$-\Delta_f G^0$ (298K)	$-\Delta_f H^0$ (298K)	$\Delta_f S^0$ (298K)	S^0 (298K)	References, method, temperature
kJ·mole ⁻¹		J·mole ⁻¹ ·K ⁻¹		
61.46 ±0.18	59.06 ±0.98	8.04 ±2.76	247.5 ±4.4	This work, EMF, 300-450 K
*62.3 ± 1.2	*91.6 ± 1.2	−43.4		[32] EMF 645-712 K
59.7 ±2.1	59.3 ± 10.5			[33] EMF 653-693 K
62.3 ± 1.3	58.6 ± 14.6	11.7 ± 26.8		[34] EMF 530-560 K
	60.74 ± 1.3			[35] DSC
	56.6 ± 0.2			[36] Calorimetry
	54.9			[37] calculation
	60.7			[38] calculation
*66.9 ± 2.7				[39] calculation
	56.6 ± 0.2			[40] recommended
58.5	56.5		246.44	[41] recommended
	56.5 ± 1.3		246.4 ± 3.3	[42] recommended
56.9 ± 2.2	56.5 ± 1.3	1.4± 4.2	241.5 ±2.9	[43] recommended

* at 673 K

As can be seen, a comparison of results shows that values given in fundamental databases and reference books [40–43] are a little differs from our values. For $\Delta_f G^0$ function, difference is ~5–7%, while ~4% for $\Delta_f H^0$, and only ~1–2% for S^0 .

The standard Gibbs free energy for formation of Sb_2Te_3 is precisely determined in this work. Our results differ only up to ~3% from the data obtained by high-temperature EMF measurements [32, 34]. Comparison of values for $\Delta_f H^0$ shows that our results are very close to the data reported in [33–36] and differ from [32] by ~50%.

As a result of this study, comparative analysis of obtained results with literature data was performed and a new mutually consistent set of standard thermodynamic functions for the Sb_2Te_3 compound was obtained.

Conclusions

In the paper, we report the results of a thermodynamic study of Sb_2Te_3 using EMF method with a glycerol electrolyte in the 300–450 K temperature range. According to the EMF measurements, the partial molar functions of antimony in alloys, the standard Gibbs free energy, the enthalpy of formation, and the standard entropies of the Sb_2Te_3 compound were experimentally calculated for the first time under standard conditions. Obtained results supplement and clarify the previously obtained thermodynamic data for antimony telluride.

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Sb_2Te_3 BİRLƏŞMƏSİNİN TERMODİNAMİKİ XASSƏLƏRİ**F.R.Əliyev, E.N.Oruclu, D.M.Babanlı**

Sb_2Te_3 birləşməsinin termodinamiki xassələri, 300–450 K temperatur intervalında maye elektrolit ilə elektrik hərəkət qüvvəsi (EHQ) usulundan istifadə etməklə öyrənilmişdir. 298 K-də sürmənin ərintilərdə parsial molar funksiyaları və Sb_2Te_3 birləşməsinin müvafiq inteqral termodinamiki funksiyaları EHQ ölçmələri əsasında hesablanmışdır. Əldə edilən nəticələr ilə ədəbiyyat məlumatlarının müqayisəli təhlili aparılmışdır.

Açar sözlər: Sb_2Te_3 , EHQ üsulu, parsial molar funksiyalar, termodinamiki funksiyalar, tetradimitəbənzər laylı quruluşlar.

ТЕРМОДИНАМИЧЕСКИЕ СВОЙСТВА СОЕДИНЕНИЯ Sb_2Te_3 **Ф.Р.Алиев, Э.Н. Оруджлу, Д.М.Бабанлы**

Термодинамические свойства соединения Sb_2Te_3 исследованы путем измерения электродвижущей силы (ЭДС) с жидким электролитом в температурном интервале 300–450 К. Парциальные молярные функции сурьмы в сплавах при 298 К и соответствующие стандартные интегральные термодинамические функции соединения Sb_2Te_3 были рассчитаны на основе измерений ЭДС. Проведен сравнительный анализ полученных результатов с литературными данными.

Ключевые слова: Sb_2Te_3 , метод ЭДС, парциально молярные функции, термодинамические функции, тетрадимитоподобные слоистые структуры.