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Thermodynamic Properties of the $GdTe_3$ Compound

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The alloys of the Gd-Te system in the range of compositions > 75 at% Te were studied by the methods of X-ray diffraction (XRD) and electromotive forces (EMF). From the EMF measurements of the concentration cells relative to the GdTe electrode in the 300-450 K temperature range, the partial thermodynamic functions of GdTe in alloys were determined. By combining these data with the corresponding functions of Gd in GdTe, the partial molar functions of gadolinium in $GdTe_3$+Te alloys, and standard thermodynamic functions of formation and standard entropy of the $GdTe_3$ compound were calculated. The obtained results were compared with the literature data.

Keywords: gadolinium tellurides, EMF method, concentration cells, partial molar functions, thermodynamic functions.

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Introduction

The rare-earth chalcogenides are a very interesting class of materials due to their unusual electronic [1-3], thermoelectric [4, 5], magnetic [6-8], optical [9, 10]. In particular, [11, 12] showed the promise for use of REE tritellurides in magnetic sensors, memory devices and spintronics.

Development of methods and optimization of conditions for directed synthesis of new materials based on REE chalcogenides is based on reliable data on phase equilibria and thermodynamic data of the corresponding systems [13-16].

The Gd-Te system [17-20] is characterized by the formation of tellurides with the compositions GdTe, Gd_3Te_4, Gd_2Te_3, Gd_4Te_7, $GdTe_2$, Gd_2Te_5, and $GdTe_3$. The GdTe compound melts congruently at 2098 K, the rest are formed by peritectic reactions. The GdTe and Gd_2Te_3 compounds have a cubic structure, while the rest crystallize in lower symmetry structures.

The thermodynamic properties of gadolinium tellurides have been studied extremely insufficiently. There are no thermodynamic data on these compounds in thermodynamic databases [21, 22]. The most studied compound is Gd_2Te_3. The thermodynamic data of this compound, given in various original papers [23-25], are very different. The standard enthalpy of formation, determined by the method of direct synthesis from components in a calorimetric bomb [23, 24] is equal to -926 ± 22 kJ/mol while the value obtained by the electromotive forces method (EMF) [25] is significantly lower (-689 ± 31 kJ/mol). At the same time, the estimated value by Mills [26] for the standard enthalpy of formation is equal to -794 kJ/mol. Authors of recently published work [20] presented the optimized thermodynamic data for all of the above gadolinium tellurides obtained by modeling the phase diagram taking into account the data of [25]. On the other hand, raises doubts about the reliability of the initial data [25] which were used by the authors [20].

Taking into account the above contradictory data, we undertook a repeated thermodynamic study of the Gd-Te system. This paper presents the results of a study of the thermodynamic properties of gadolinium tritelluride by the EMF method with a glycerol electrolyte. Various modifications of the EMF method with liquid and solid electrolytes are successfully used in thermodynamic studies of a number of binary and ternary chalcogenide systems [27-35].

I. Experiments and results

Studies were carried out by powder X-ray diffraction analysis (Bruker D8 diffractometer with $CuK\alpha_1$ radiation) and electromotive forces (EMF) measurements of the

$$(-)\ GdTe\ (s.)\ |\ glycerol + KCl + \\ + GdCl_3\ |\ (GdTe_3 + Te)\ (s.)\ (+) \qquad (1)$$

concentration cell in the 300 - 450 K temperature range.

In (1) type cell, the electrolyte was a glycerol solution of KCl with the $GdCl_3$ addition. Special attention was paid to the preparation of anhydrous $GdCl_3$, which is extremely hygroscopic. Dehydration was performed under vacuum along with slowly increasing heating for five days. Then the dehydrated salt was melted and transferred into a quartz beaker preheated to 900 K. To remove the oxychloride, the molten salt was treated with dry hydrochloride gas (HCl).

Before use, glycerol was dehydrated and degassed by pumping at a temperature of ~ 450 K. Then anhydrous salts KCl and $GdCl_3$ were added to glycerol in a box with an inert gas. The electrolyte prepared in this way was stored in a sealed ampoule and used as required. Upon the introduction of the electrolyte in a cell, contact with air was minimal.

Taking into account the results of our previous works [34, 35], in the cell as a reference electrode, we used not elementary gadolinium, but gadolinium monotelluride with a small excess of tellurium (composition $GdTe_{1.01}$). Equilibrium alloys with compositions of 80 and 90 at.% Te of the Gd-Te system were used as anodes.

Alloys – anodes were synthesized by a ceramic method from high-purity elemental Gd and Te in evacuated (10^{-2}Pa) quartz ampoules at 1000 K. To prevent the interaction of gadolinium with the inner walls of quartz, syntheses were carried out in graphitized ampoules. The ampoules were graphitized by thermal decomposition of toluene. After the synthesis, samples were ground into powder, thoroughly mixed, pressed into tablets, and annealed at 550 K (1000 h) and then at 450 K (300 h). The phase composition was controlled by XRD. Fig. 1 presents a powder diffraction pattern of an alloy with 90 at% Te composition. As can be seen, the alloy consists of a two-phase mixture $GdTe_3$+Te [36, 37]. This is in good agreement with the phase diagram of the Gd-Te system [18-20] and indirectly indicates the equilibrium of the synthesized alloys.

The assembling of an electrochemical cell is described in detail in [38-40].

The EMF measurements were carried out using a Keihtley Model 193 digital voltmeter with an input resistance of 10^{14} Ohm and an accuracy of ± 0.1 mV. The EMF measurements step was no more than 10 K. The temperature in the concentration cell was measured with chromel-alumel thermocouples and a mercury thermometer with an accuracy of 0.5 K. Elemental gallium (302.9 K), sulfur (386 K), indium (429.7 K), and tin (505 K) were used as standards.

The first equilibrium EMF values were obtained after keeping the cell at ~350 K for 40 - 60 h, and the subsequent ones every 3 - 4 h after a certain temperature was established The EMF values that did not differ from each other during repeated measurements at a given temperature by more than 0.2 mV, regardless of the direction of temperature change, were recorded as equilibrium values. The reversibility of the assembled concentration cell was controlled by the constancy of the masses and phase compositions of the electrodes. Within the experiment, the EMF of each sample was measured 3 times at two constant temperatures. For example, for an alloy with a composition 80 at%Te at 321.2 K, EMF values were: 349.78, 350.13, and 349.88. Table 1 presents the average value of 349.93.

Analysis of the EMF measurements data for alloys 80 and 90 at%Te shows that they coincide and are characterized by linear temperature dependence (Fig. 2). This indirectly indicates the constancy of the coexisting phases in the temperature interval of EMF measurements and allows them to be used for thermodynamic calculations [39, 40].

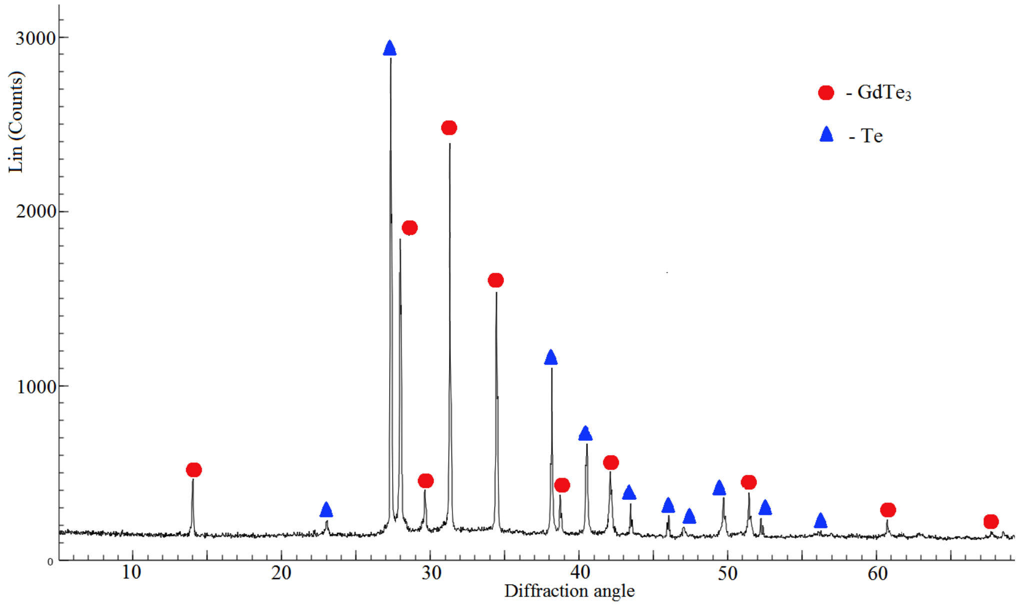

Fig. 1. Powder diffraction pattern of an alloy with 90 at% Te composition of the Gd-Te system.

Table 1

Experimentally obtained data for temperature and EMF and data associated with the calculation steps for the alloy with 80 at%Te composition the Gd-Te system

T_i, K	E_i, mV	$T_i-\bar{T}$	$E_i(T_i-\bar{T})$	$(T_i-\bar{T})^2$	$\tilde{E}$	$E_i-\tilde{E}$	$(E_i-\tilde{E})^2$
301.3	347.75	-73.50	-25558.47	5401.76	348.53	-0.78	0.601
303.3	349.12	-71.50	-24960.92	5111.77	348.58	0.54	0.2939
307.7	347.86	-67.10	-23340.25	4501.96	348.69	-0.83	0.6955
314.4	349.13	-60.40	-21086.29	3647.76	348.87	0.26	0.0672
319.3	348.13	-55.50	-19320.05	3079.88	349.00	-0.87	0.7570
321.2	349.93	-53.60	-18755.08	2872.60	349.05	0.88	0.7741
329.5	349.73	-45.30	-15841.60	2051.79	349.27	0.46	0.2124
333.4	350.87	-41.40	-14524.85	1713.68	349.37	1.50	2.2438
340.6	349.04	-34.20	-11936.00	1169.41	349.56	-0.52	0.2725
346.7	348.79	-28.10	-9799.84	789.42	349.72	-0.93	0.8705
354.4	349.95	-20.40	-7137.81	416.02	349.93	0.02	0.0006
357.9	351.11	-16.90	-5932.59	285.50	350.02	1.09	1.1913
360.9	349.12	-13.90	-4851.60	193.12	350.10	-0.98	0.9558
366.9	349.66	-7.90	-2761.15	62.36	350.26	-0.60	0.3552
370.2	350.68	-4.60	-1611.96	21.13	350.34	0.34	0.1135
377.7	351.82	2.90	1021.45	8.43	350.54	1.28	1.6360
384.1	349.86	9.30	3254.86	86.55	350.71	-0.85	0.7222
390.9	350.13	16.10	5638.26	259.32	350.89	-0.76	0.5764
394.6	351.48	19.80	6960.48	392.17	350.99	0.49	0.2432
400.5	352.22	25.70	9053.23	660.66	351.14	1.08	1.1610
405.4	351.89	30.60	10769.01	936.56	351.27	0.62	0.3822
410.5	350.45	35.70	12512.23	1274.73	351.41	-0.96	0.9146
415.2	352.22	40.40	14230.86	1632.43	351.53	0.69	0.4756
420.9	351.79	46.10	16218.69	2125.52	351.68	0.11	0.0119
424.2	350.05	49.40	17293.64	2440.69	351.77	-1.72	2.9510
428.3	351.21	53.50	18790.91	2862.61	351.88	-0.67	0.4436
433.8	352.92	59.00	20823.46	3481.39	352.02	0.90	0.8079
437.7	352.99	62.90	22204.25	3956.83	352.12	0.87	0.7499
444.2	351.15	69.40	24370.98	4816.82	352.30	-1.15	1.3123
448.2	352.88	73.40	25902.57	5388.05	352.40	0.48	0.2293
$\bar{T}$ = 374.8	$\bar{E}$ = 350.46						

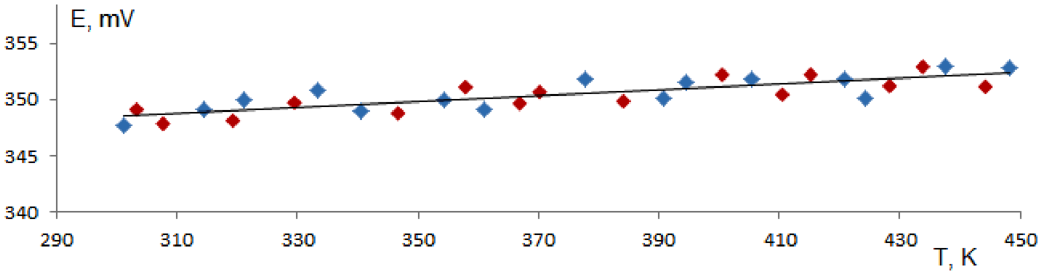

Fig. 2. Temperature dependence of the EMF of the cell of type (1) for alloys from the $GdTe_3$+Te region of the Gd-Te system. Red diamonds – are alloy with composition 80 at% Te, and blue diamonds – are alloy with composition 90 at% Te.

The obtained experimental data were processed using the computer program "Microsoft Office Excel 2003" by the least-squares method. The calculation steps are shown in Table 1.

The obtained equation is presented in the form of

Table 2

Standard integral thermodynamic functions of gadolinium tritelluride at 298 K

$-\Delta_f G^0(298K)$	$-\Delta_f H^0(298K)$	S^0_{298}, kJ·mol⁻¹·K⁻¹	Ref.
кJ·mol⁻¹			
380.2 ± 0.5	385.9 ± 2.4	197.3 ± 7.8	this work
332.6 ± 4.2	347.3 ± 17.2	166.5 ± 2.9	[25]
	344.27	168.0	[20]

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

recommended by a standard textbook [39]. In equation (2), a and b are coefficients, n is the number of data pairs E and T; $\bar{T}$ - the average temperature in K, t is Student's t test, S_E^2 is the variances of individual EMF values. With the number of experimental points n = 30, and the confidence level equal to 95%, the Student's test is t ≤ 2.

From equation

$$E, mV = 340.582 + 0{,}0264T \pm 2\left[\frac{0.79}{30} + 1.28\cdot 10^{-5}(T-374.8)^2\right]^{1/2} \qquad (3)$$

by using the relations of [38, 39]:

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

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

$$\Delta\bar{H}_i = -zF\left[E - T\left(\frac{\partial E}{\partial T}\right)_P\right] = -zFa, \qquad (6)$$

the partial molar Gibbs free energy, enthalpy, and entropy of gadolinium telluride in the $GdTe_3$+Te phase area were calculated.

$$\overline{\Delta G}_{GdTe} = -100.86 \pm 0.18 \text{ kJ·mol}^{-1}$$

$$\overline{\Delta H}_{GdTe} = -98.58 \pm 0.78 \text{ kJ·mol}^{-1}$$

$$\overline{\Delta S}_{GdTe} = 7.64 \pm 2.07 \text{ J·mol}^{-1}\text{·K}^{-1}$$

These quantities are the difference of the corresponding partial molar functions of gadolinium for the left and right electrodes of the cells type (1)

$$\Delta\bar{G}_{GdTe}(GdTe_3) = \Delta\bar{G}_{Gd}(GdTe_3) - \Delta\bar{G}_{Gd}(GdTe)$$

Therefore

$$\Delta\bar{Z}_{Gd}(GdTe_3) = \Delta\bar{Z}_{Gd}(GdTe) + \Delta\bar{Z}_{GdTe}(GdTe_3) \qquad (7)$$

The values of the partial thermodynamic functions of Gd in $GdTe_3$ obtained using Eq. (7) are given below

$$\overline{\Delta G}_{Gd} = -380.16 \pm 0.48 \text{ kJ·mol}^{-1}$$

$$\overline{\Delta H}_{Gd} = -385.88 \pm 2.38 \text{ kJ·mol}^{-1}$$

$$\overline{\Delta S}_{Gd} = -19.18 \pm 6.37 \text{ J·mol}^{-1}\text{·K}^{-1}$$

In the calculations, the data of work [35] were used also.

According to the phase diagram of the Gd-Te system, these partial molar functions of gadolinium are thermodynamic characteristics of the following virtual cell reaction (the state of substances is crystalline):

$$Gd + 3Te = GdTe_3.$$

Hence, the standard thermodynamic functions of $GdTe_3$ formation can be calculated from the relation:

$$\Delta Z^0(GdTe_3) = \Delta\bar{Z}_{Gd},$$

here $Z = G, H, S$, while the standard entropy, from the relation $S^0(GdTe_3) = \Delta\bar{S}_{Gd} + S^0(Gd) + 3S^0(Te)$

The data obtained are shown in Table 2.

When determining the standard entropy of $GdTe_3$, in addition to own experimental results, the literature data [22] on the standard entropies of elemental gadolinium (67.95± 0.63 kJ·mol⁻¹) and tellurium (49.5 ± 0.21 kJ·mol⁻¹) were used. The errors were determined by the method of uncertainty propagation.

As can be seen from Table 2, our data are about 10% higher than the results of [25]. It should be noted that the data [20] are not experimental but were obtained by optimizing the data [25]. Analysis of the results of [25] shows that the character of the EMF dependences on the composition does not satisfy the conditions of thermodynamic equilibrium. For example, the EMF value at 400 K for the $GdTe_3$ + Te two-phase region is E = 1133 mV, and for the region Gd_2Te_3 + Gd_4Te_7 with a lower tellurium composition, it is higher (E = 1157 mV). According to the well-known thermodynamic requirements [38, 39], this is impossible. On the other hand, in our previous works [34, 35] it was shown that the use of cells relative to metallic lanthanide does not give reproducible results: EMF values significantly decrease over time. Taking this into account, we believe that the temperature dependences of the EMF presented in [20] do not reflect the equilibrium state and the obtained values of the thermodynamic functions are underestimated.

Conclusions

By combining data of the EMF measurements of the concentration cell of (–) GdTe (s.) | glycerol + KCl + $GdCl_3$ | ($GdTe_3$+Te) (s.) (+) type with literature data [35], the partial molar functions of GdTe and Gd in the $GdTe_3$+Te phase region of the Gd-Te system were determined for the first time. The data obtained were used to calculate the standard integral thermodynamic functions and the standard entropy of the $GdTe_3$ compound, a promising material for use in magnetic devices and spintronics.

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Термодинамічні властивості сполуки $GdTe_3$

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Сплави системи Gd-Te у діапазоні складів > 75 ат.% Te досліджено методами дифракції рентгенівських променів (XRD) та електрорушійної сили (ЕРС). Із вимірювань ЕРС комірок концентрації відносно електроду GdTe у діапазоні температур 300 - 450 K визначено часткові термодинамічні функції GdTe у сплавах. Поєднуючи ці дані з відповідними функціями Gd в GdTe, розраховано часткові молярні функції гадолінію в сплавах $GdTe_3$ + Te, а також стандартні термодинамічні функції утворення і стандартна ентропія сполуки $GdTe_3$. Отримані результати порівнювали із літературними даними.

Ключові слова: телуриди гадолінію, метод ЕРС, комірки концентрації, часткові молярні функції, термодинамічні функції.