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THERMODYNAMIC STUDY OF THE CdSb COMPOUND BY THE ELECTROMOTIVE FORCE MEASUREMENTS METHOD

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Abstract. *Thermodynamic properties of CdSb binary compound were determined using an electromotive force measurement (e.m.f.) method with a liquid electrolyte in the ~300-450K temperatures interval. Equilibrium alloys from the CdSb+Sb phase region of the Cd-Sb system was analyzed by means of the e.m.f. measurements. Phase compositions of all samples were controlled using powder X-rays diffraction method. The partial molar functions of cadmium in alloys, the standard thermodynamic functions of formation, as well as the standard entropy of the CdSb compound were calculated. A comparative analysis of obtained results with literature data was performed.*
Keywords: *cadmium antimonide, CdSb, e.m.f. measurements, thermodynamic properties, glycerol electrolyte.*

Introduction

Metal pnictides and phases based on them have been at the center of attention of researchers since last century due to their appealing functional properties. Some of them exhibit promising electro-physical properties such as low thermal conductivity, magnetic ordering, significant Seebeck coefficient, giant magnetoresistivity, etc. and used in various industrial processes, including producing electronic products, photomultiplier devices, semiconductors, optic materials, batteries, glasses, fireworks, cooling and power generation devices, high electron mobility transistors, and so on [1-4].

Among them, cadmium antimonide CdSb - a narrow band gap semiconductor with strong anisotropy, thermoelectric, optical, and photovoltaic properties have found practical applications due to its intriguing physical properties and excellent thermoelectric capacity at low temperatures. Promising thermoelectric characteristics and the specific topology of the forbidden band gap of CdSb make it a noteworthy material for thermoelectric devices and anisotropic thermoelectric sensors, whether utilized as a fundamental material or as an alloy with other compounds [5-11].

The phase diagram of the Cd–Sb binary system is characterized by formation of an intermediate compound of the CdSb composition, melting with an open maximum at a temperature of ~730 K [12]. CdSb is in eutectic equilibrium with elemental cadmium (7 at.% Sb and 563K) and antimony (57 at.% Sb and 718 K). It crystallizes in an orthorhombic cell with the following lattice constants: a= 6.471Å, b= 8.263 Å, c= 8.526 Å (D_{2k}^{15} - *P/bca* space group) [13].

Thermodynamic properties of applied materials are considered fundamental characteristics and are necessary to predict their thermal behavior. Researchers delve into enthalpy, entropy, and Gibbs free energy to unravel CdSb's response to varying temperature and pressure conditions. Its specific heat capacity, phase transition behavior, and thermal conductivity further contribute to understanding its thermodynamic intricacies [14-17]. As scientists explore CdSb for potential applications in electronic and optoelectronic devices, a precise comprehension of its thermodynamic properties becomes imperative, paving the way for optimized performance and leveraging its capabilities in technological advancements.

Various modifications of the *e.m.f.* method, depending on the measurement temperature, nature of constituent phases, etc. are applied for thermodynamic investigations in inorganic systems. Different types of liquid, ionic liquid, and solid electrolytes can be used for the *e.m.f.* measurements [18-23].

Considering above, present contribution is devoted to the thermodynamic study of CdSb compound using the *e.m.f.* method with liquid electrolyte in the temperature range of ~300-450 K.

Experimental part

To study the thermodynamic properties of the CdSb binary compound, several alloys have been selected from the CdSb + Sb heterogeneous phase region of the T-x phase diagram [12]. High purity (99.999 % pure) elemental components: cadmium – Sigma-Aldrich; *CAS number - 7439-89-6 and* antimony – Alfa-Aesar; *CAS number - 7440-36-0* have been used for sample preparation. Stoichiometric amounts of the elemental Cd and Sb for the total mass of 1 gram for all samples were computed and weighed on an analytical balance before being put into quartz tubes. Quartz ampoules have been pumped to a pressure of 10^{-2} Pa and heated to the temperature of ~1100 K inside a muffle furnace. Synthesis was carried out around 3-5 hours, followed by a long-term (~800 hours) homogenization through thermal treatment below the relevant solidus temperature.

The powder X-ray diffraction (PXRD) technique was used to identify the phase compositions of the prepared samples. PXRD measurements were performed at room temperature using the D2 Phaser diffractometer with CuK_{α} emission. Alloys were scanned between ~ 5 and 75°. Topaz 4.2 software program was used to analyze the diffraction patterns obtained.

The obtained powder diffraction patterns of two samples are given in Figure 1. As can be seen, the PXRD spectrums of samples are composed of diffraction peaks of the CdSb compound and an elementary antimony.

Following electrochemical cells have been assembled to measure the electromotive force of alloys:

$$(-)\ Cd(s) / glycerol + KCl + CdCl_2 / Cd\ in\ alloy\ (+) \qquad (1)$$

Cadmium was used as a left electrode of the cell, while equilibrium alloys of the Cd-Sb system were used as right electrodes. Samples were powdered, pressed into tablets (5-6 mm in diameter), and attached to the molybdenum rod to prepare the right electrodes. Prepared electrode tablets were covered with glass coatings to prevent possibility of the contact between them inside an electrolyte solution.

A liquid electrolyte of the constructed cell was a glycerol solution of KCl (Sigma Aldrich, 99.999%) with a small addition of anhydrous $CdCl_2$ (Sigma Aldrich, 99.999%). Due to moisture and oxygen in the electrolyte, glycerol was thoroughly dehydrated and degassed at ~350K under a dynamic vacuum. Preparation of the electrolyte and electrodes, as well as the assembly technique of the electrochemical cell (1), was put into practice as described in [14-16, 23].

The digital multimeter Keithley 2100 6 ½, having 1014 Ω input resistance and ±0.1 mV accuracy, was used to collect *e.m.f.* data in the ~300-450 K temperature interval. A chrome-alumni thermocouple and a mercury thermometer were used for temperature measurements. After keeping the cell at ~350K for 50–60 hours, the first equilibrium values of the potential difference were obtained. Then, subsequent measurements were taken every 3-4 h when a particular temperature was established. The *e.m.f.* values differed between repeated measurements not exceeding 0.2 mV at the established temperature were accepted as equilibrium data.

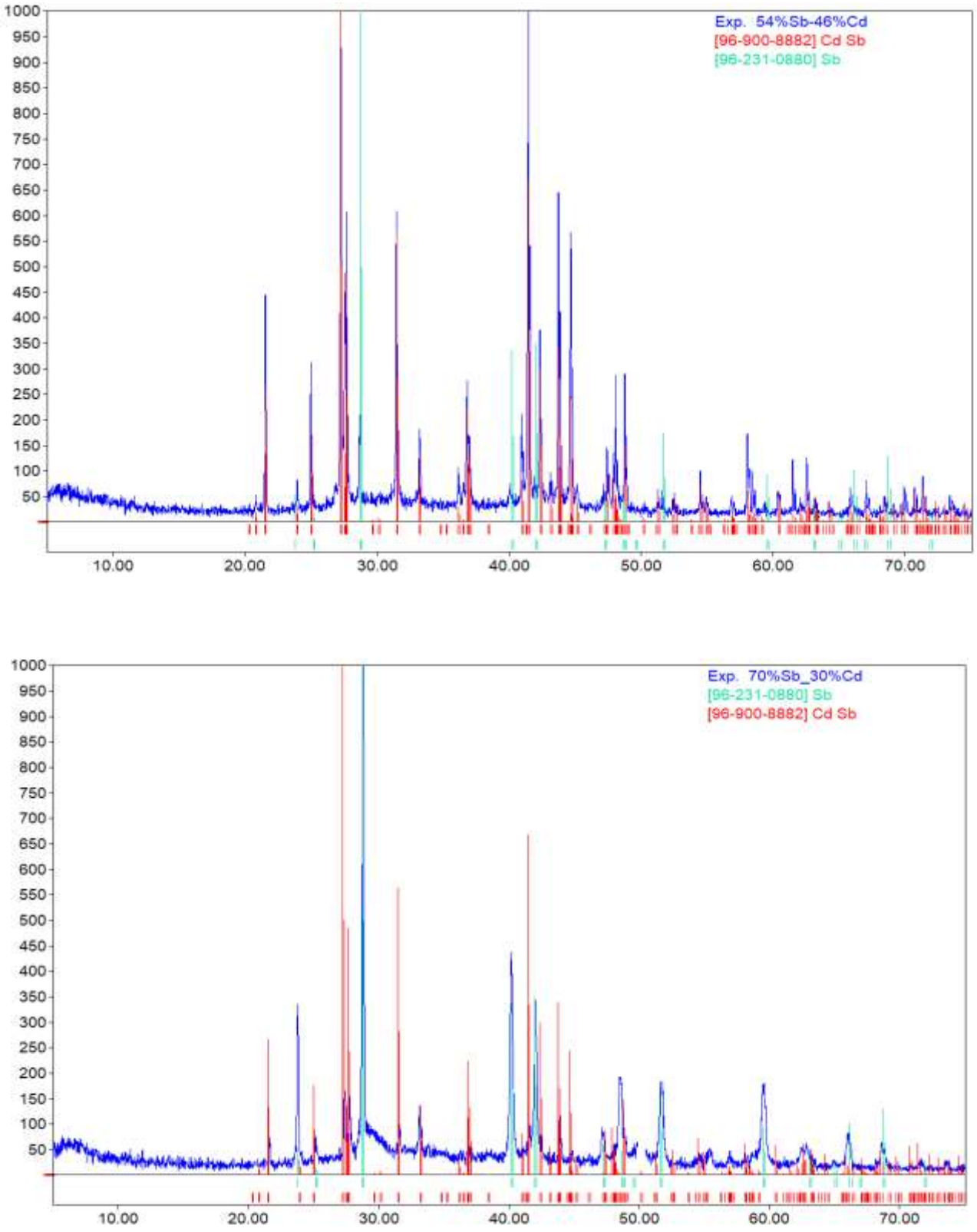

Fig. 1. PXRD spectrums of samples along the Cd-Sb binary system:
a) 46 at. % Cd and b) 30 at. % Cd

Results and discussion

The *e.m.f.* values of all alloys were constant within the CdSb + Sb two-phase field, independent of the overall composition of the electrode alloy. Linear dependence of the *e.m.f.* data on temperature (Fig.2) allowed them to be used for thermodynamic calculations. To do so, collected

temperature and *e.m.f.* data were treated using the least-squares method via the Microsoft Office Excel computer software to yield linear equations of the $E = a + bT$ form. Table 1 represents experimentally obtained data for temperature (T_i,), *e.m.f.* (E_i) and the data associated with the calculation steps of the applied method for the sample with 46 at. % Cd composition.

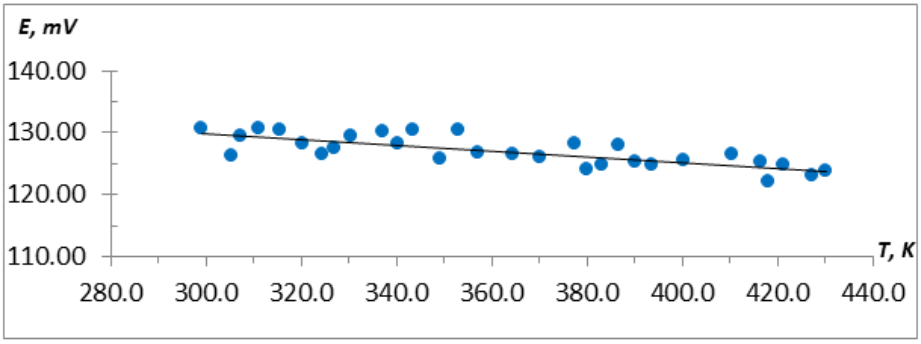

Fig. 2. Temperature dependences of the *e.m.f.* data for the concentration cell of the type (1)

The method of processing the *e.m.f.* data was carried out as described in [23-25], and the obtained linear equation is given in the form of the expression (2):

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

Where n- is the number of experimental *e.m.f.* (E_i) and temperature (T_i) values; S_E and S_b- are the variances of individual *e.m.f.* measurements and b constant, respectively; $\bar{T}$ - average absolute temperature, *t*-Student's test. At the confidence level of 95% and experimental points n=30, the student's test is t≤2.

Obtained linear equation for the CdSb+Sb phase area of the Cd-Sb system is given below:

$$E, mV = 143.85 - 0.0468T \pm 2\,[\frac{2.42}{30} + 5.06 \cdot 10^{-5}\,(T - 363.09)^2]^{1/2} \qquad (3)$$

Partial molar functions of cadmium in the alloys at 298 K have been calculated using this equation and the thermodynamic expressions (4)-(6):

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

$$\Delta\bar{H}_{Cd} = -\,z\,[E - T\left(\frac{\partial E}{\partial T}\right)_P] = -\,zFa \qquad (5)$$

$$\Delta\bar{S}_{Cd} = -\,zF\left(\frac{\partial E}{\partial T}\right)_P = zFb \qquad (6)$$

Calculated partial molar functions of cadmium are given below:

$$\Delta\bar{G}_{Cd} = -\,25.07 \pm 0.10\ kJ/mole$$
$$\Delta\bar{H}_{Cd} = -\,27.76 \pm 0.50\ kJ/mole$$
$$\Delta\bar{S}_{Cd} = -\,9.03 \pm 1.37\ J/(K \cdot mole)$$

Table 1. Computer-processed *e.m.f* measurements for the sample of the 46 at. % Cd composition

T_i, K	E_i, mV	$T_i - \bar{T}$	$E_i\,(T_i - \bar{T})$	$(T_i - \bar{T})^2$	$\breve{E}$	$E_i - \breve{E}$	$(E_i - \breve{E})^2$
299,1	130,63	-63,99	-8359,01	4094,72	129,86	0,77	0,60
305,3	126,10	-57,79	-7287,32	3339,68	129,57	-3,47	12,03

307,2	129,41	-55,89	-7232,72	3123,69	129,48	-0,07	0,00
311,1	130,70	-51,99	-6795,09	2702,96	129,30	1,40	1,97
315,6	130,34	-47,49	-6189,85	2255,30	129,09	1,25	1,57
320,4	128,12	-42,69	-5469,44	1822,44	128,86	-0,74	0,55
324,5	126,33	-38,59	-4875,07	1489,19	128,67	-2,34	5,48
327,1	127,40	-35,99	-4585,13	1295,28	128,55	-1,15	1,32
330,6	129,25	-32,49	-4199,33	1055,60	128,38	0,87	0,75
337,2	130,11	-25,89	-3368,55	670,29	128,08	2,03	4,14
340,3	128,20	-22,79	-2921,68	519,38	127,93	0,27	0,07
343,7	130,22	-19,39	-2524,97	375,97	127,77	2,45	5,99
349,4	125,66	-13,69	-1720,29	187,42	127,51	-1,85	3,41
353,1	130,22	-9,99	-1300,90	99,80	127,33	2,89	8,34
357,2	126,64	-5,89	-745,91	34,69	127,14	-0,50	0,25
364,5	126,30	1,41	178,08	1,99	126,80	-0,50	0,25
370,2	125,83	7,11	894,65	50,55	126,53	-0,70	0,49
377,6	128,10	14,51	1858,73	210,54	126,19	1,91	3,66
380,1	123,88	17,01	2107,20	289,34	126,07	-2,19	4,79
383,3	124,64	20,21	2518,97	408,44	125,92	-1,28	1,64
386,7	127,80	23,61	3017,36	557,43	125,76	2,04	4,16
390,2	125,11	27,11	3391,73	734,95	125,60	-0,49	0,24
393,8	124,70	30,71	3829,54	943,10	125,43	-0,73	0,53
400,4	125,51	37,31	4682,78	1392,04	125,12	0,39	0,15
410,5	126,30	47,41	5987,88	2247,71	124,65	1,65	2,73
416,6	125,26	53,51	6702,66	2863,32	124,36	0,90	0,81
418,2	121,93	55,11	6719,56	3037,11	124,29	-2,36	5,56
421,3	124,65	58,21	7255,88	3388,40	124,14	0,51	0,26
427,4	122,91	64,31	7904,34	4135,78	123,86	-0,95	0,90
430,1	123,70	67,01	8289,14	4490,34	123,73	-0,03	0,00
$\overline{T}$ =363,09	**$\overline{E}$ =126,865**		**$\sum$ = -2236,75**	**$\sum$= 47817,47**			**$\sum$ = 72,62**

According to the T-x phase diagram of the Cd-Sb system, these quantities are thermodynamic functions of the following virtual-cell reaction:

$$Cd\ (s) + Sb\ (s) = CdSb\ (s) \qquad (7)$$

Therefore, calculated partial molar functions of cadmium in the CdSb + Sb two-phase region are the standard thermodynamic functions for the formation of the CdSb compound from its elemental constituents.

The standard entropy was calculated as:

$$S^{\circ}_{CdSb} = \Delta_f S^{\circ}(CdSb) + S^{\circ}_{Cd} + S^{\circ}_{Sb} = 88.42 \pm 2.12\ J/(K \cdot mole)$$

Errors were calculated using the error accumulation method. Absolute entropies of the elementary cadmium and antimony used for calculations were taken from [26] : $S^{0}_{Cd} = 51.756 \pm 0.125$ J/(mole $\cdot$ K); $S^{0}_{Sb} = 45.689 \pm 0.627$ J/(mole$\cdot K$). Calculated standard integral thermodynamic functions of the CdSb compound are given in Table 2 together with available literature data.

Table 2. Standard integral thermodynamic functions of the CdSb compound

$-\Delta_f G^0$ (298K)	$-\Delta_f H^0$ (298K)	$\Delta_f S^0$ (298K)	S^0 (298K)	**Method, year, temperature**	**Reference**
kJ·mole^{-1}		J·mole^{-1}·K^{-1}			
25.07± 0.10	27.76 ± 0.50	- 9.03 ± 1.37	88.42± 2.12	*e.m.f.*, this work 300-450 K	-
	13.6	- 2.8		*e.m.f.*, 1935 530-560 K	[27]
	15.6	- 6.2		*e.m.f.*, 1935 513-563K	[28]
	15.0±1.2	- 4.7±1.8		*e.m.f.*, 1979 643-708K	[29]
	27.9			vap.pres., 1960 550-721K	[30]
	13.7±1.0	- 2.5±2.1		vap.pres., 1961 549-652K	[31]
	13.3±1.0	1.7±2.0		evalutation, 1980	[32]
	13.3±1.3		95.6±0.8	recommendation, 1993	[33]
24.7	25.52		94.56	recommendation, 2008	[34]
24.7	25.52		94.56±4.18	recommendation, 2006	[26]

As can be seen from the Table 2, the high temperature *e.m.f* measurements of the CdSb compound were carried out at ~500-750K temperature intervals in different years. Ansara I. and Bernard C. presented the optimal quantities for CdSb compound taking into account the results of these works. Data obtained by the vapor pressure measurement method in [31] are close to these quantities. However, Silvestri V.J. [30] obtained nearly two times greater values by vapor pressure measurements at approximately the same temperature.

It should be noted that, the available data on the enthalpy of formation of this compound recommended in modern databases are considerably different from each other. So that, the reference book [33] gives the values recommended by [32], while the quantities recommended in [26] and [34] are very close to the results obtained by Silvestri V.J. [30].

The *e.m.f.* method in the present work has been applied with high accuracy and thermodynamic properties have been calculated at lower temperature. As can be seen from Table 2, our results are close to the ones given in fundamental databases [26] and [34].

It is not easy to explain the significantly lower values of *e.m.f* during high-temperature measurements. Perhaps this is due to the additional processes taking place in an electrochemical cell at high temperatures.

Thereby, 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 CdSb compound was obtained.

Conclusion

In the present paper, we report new mutually consistent thermodynamic data for the CdSb binary compound obtained by means of the *e.m.f.* method with a glycerol electrolyte in the 300-450 K temperatures range. According to the *e.m.f.* measurements, the partial molar functions of cadmiun in alloys, the standard thermodynamic functions of the formation and the standard entropy of the

CdSb compound were experimentally calculated for the first time under standart conditions. Obtained results supplement and clarify the previously obtained thermodynamic data for cadmium antimonide.

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ELEKTRİK HƏRƏKƏT QÜVVƏSİNİN ÖLÇÜLMƏSİ ÜSULU İLƏ CdSb BİRLƏŞMƏSİNİN TERMODİNAMİK TƏDQİQİ

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Xülasə: CdSb ikili birləşməsinin termodinamik xassələri ~300-450K temperatur intervalında maye elektrolitli elektrik hərəkət qüvvəsi (e.h.q.) ölçmələri ilə təyin edilmişdir. Bu məqsədlə Cd-Sb binar sisteminin CdSb+Sb faza sahəsindən seçilmiş tarazlıq nümunələri e.h.q. ölçmələri ilə analiz olunmuşdur. Bütün nümunələrin faza tərkibinə toz rentgenoqrafiyası üsulu ilə nəzarət edilmişdir. Ərintilərdə kadmiumun parsial molyar funksiyaları, həmçinin CdSb birləşməsinin standart əmələgəlmə termodinamik funksiyaları və standart entropiyası hesablanmışdır. Əldə edilmiş nəticələrin ədəbiyyat məlumatları ilə müqayisəli təhlili həyata keçirilmişdir.
Açar sözlər: kadmium stibnie, CdSb, e.h.q. ölçmələri, termodinamik xassələr, qliserin elektroliti

ТЕРМОДИНАМИЧЕСКОЕ ИССЛЕДОВАНИЕ СОЕДИНЕНИЯ CdSb МЕТОДОМ ИЗМЕРЕНИЯ ЭЛЕКТРОДВИЖУЩЕЙ СИЛЫ

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Резюме: Термодинамические свойства бинарного соединения CdSb определены методом измерения электродвижущей силы (э.д.с.) с жидким электролитом в интервале температур ~300-450K. Равновесные сплавы из фазовой области CdSb+Sb системы Cd-Sb были анализированы измерением э.д.с. Фазовый состав всех образцов контролировали методом порошковой дифракции рентгеновских лучей. Рассчитаны парциальные молярные функции кадмия в сплавах, стандартные термодинамические функции образования, а также стандартная энтропия соединения CdSb. Проведен сравнительный анализ полученных результатов с литературными данными.
Ключевые слова: стибнит кадмия, CdSb, измерения э.д.с., термодинамические свойства, глицериновый электролит