Critical evaluation of the thermodynamic properties of the cadmium-antimony system

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INSTITUT NATIONAL POLYTECHNIQUE DE GRENOBLE

LABORATOIRE DE THERMODYNAMIQUE ET PHYSICO-CHIMIE METALLURGIQUES

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1 → CRITICAL EVALUATION OF THE THERMODYNAMIC PROPERTIES
OF THE CADMIUM-ANTIMONY SYSTEM

by

C. BERNARD and I. ANSARA

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CRITICAL EVALUATION OF THE THERMODYNAMIC PROPERTIES
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PHASE DIAGRAM :

The phase diagram reported by HANSEN⁽¹⁾, ELLIOTT⁽²⁾ and SHUNK⁽³⁾ indicate a stable liquidus with CdSb phase formation exhibiting a small range of non-stoichiometry, and metastable compounds Cd_3Sb_2 and Cd_4Sb_3 . The existence of these metastable compounds has recently been confirmed by HAYER⁽⁴⁾ who redetermined the phase diagram by differential thermal analysis.

The following lists presents the different thermodynamic measurements made on the CdSb compound and the liquid alloys.

CdSb compound

SELTZ ⁽⁵⁾	: e.m.f.	668-713 K	$0.57 < x_{\text{Cd}} < 0.63^*$
OLANDER ⁽⁶⁾	: e.m.f.	573-563 K	$0.135 < x_{\text{Cd}} < 0.84$
BORG ⁽⁷⁾	: vap. pres.	549,3-652,3 K	$x_{\text{Cd}} \approx 0.59$
EVSEEVA ⁽⁸⁾	: vap. pres.	543-650 K	$0.3 < x_{\text{Cd}} < 0.8$
SILVESTRI ⁽⁹⁾	: vap. pres.	550-721 K	$x_{\text{Cd}} = 0.5$
SCHICK ⁽¹⁰⁾	: calorimetry	513-729 K	$x_{\text{Cd}} = 0.5$
KUBASCHEWSKI ⁽¹¹⁾	: calorimetry	549-676 K	$x_{\text{Cd}} = 0.5$
BILTZ ⁽¹²⁾	: calorimetry	298 K	$\begin{cases} x_{\text{Cd}} \approx 0.6 (\text{Cd}_4\text{Sb}_3) \\ x_{\text{Cd}} = 0.5 \end{cases}$

* The symbols are described in appendix I.

FIGURE 1

Liquid alloysHeats of mixing

KAWAKAMI ⁽¹³⁾	: calorimetry	1073 K	$0.256 < x_{Cd} < 0.81$
WITTIG ⁽¹⁴⁾	: calorimetry	913 K	$0.08 < x_{Cd} < 0.9017$
SCHEIL ⁽¹⁵⁾	: calorimetry	738 K	$0.419 < x_{Cd} < 0.907$
CASTANET ⁽¹⁶⁾	: calorimetry	705-740 K	$0.325 < x_{Cd} < 0.975$

Heat content

KUBASCHEWSKI ⁽¹¹⁾	: calorimetry	753-875 K	$x_{Cd} = 0.5$
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Specific heat

SCHICK ⁽¹⁰⁾	: calorimetry	575-911 K	$0 \leq x_{Cd} \leq 1$
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Partial free energies

GEFFKEN ⁽¹⁷⁾	: e.m.f.	693-893 K	$0.136 < x_{Cd} < 0.9$
MORACHEWSKI ⁽¹⁸⁾	: e.m.f.	693-1023 K	$0.1 < x_{Cd} < 0.9$
ELLIOTT ⁽¹⁹⁾	: e.m.f.	648-873 K	$0.144 < x_{Cd} < 0.9481$
SELTZ ⁽²⁰⁾	: e.m.f.	643-773 K	$0.3444 < x_{Cd} < 0.896$
SCHEIL ⁽²¹⁾	: vap. pres.	733 K	$0.32 < x_{Cd} < 0.8$
BAKER ⁽²²⁾	: vap. pres.	1135-1458 K	$0.15 < x_{Cd} < 0.9$

CRITICAL EVALUATION :A) LIQUID ALLOYS :1 - Partial free energies of mixing :

Figure 1 presents the experimental results of $\Delta \bar{G}_{Cd}^{xs}$ obtained at 753 and for 773 K by SELTZ, GEFFKEN, ELLIOTT, MORACHEWSKI. These different measurements agree very well ; Other measurements by SCHEIL which are about 350 cal/mol* less negative in the composition range 0.4-0.6 (values taken from a graphical plot) have not been plotted. The selected values agree within ± 50 cal/mol.

*1 mole refers to $Cd_x Sb_{1-x}$

FIGURE 2

FIGURE 3

2 - Specific heats :

The specific heats of liquid alloys for different compositions have been measured by SCHICK. A numerical analysis has been achieved to present the results by step of 0.05 of x_{Cd} . The description of the analysis and the comparison between calculated and experimental values are given respectively in appendix II and table I. As an excellent agreement is obtained, the calculated values have been used in the assesment.

3 - Heats of mixing :

The heats of mixing measured at different temperatures and by different techniques are plotted in figure 2. In order to compare them, a correction using the C_p values was made and the calculated values at 773 K are shown in figure 3. The selected values, which are listed in table II, agree within + 75 cal/mol. compared to those of GEFFKEN, MORACHEWSKI, and WITTIG. Deviations of about 50 to 200 cal. in the composition range $0.35 < x_{Cd} < 0.8$ observed with SELTZ, SCHEIL and CASTANET's values. BAKER's measurements are less negative by 300 cal. than the average calorimetric values whereas KAMAKAMI's measurements are more negative by 300 cal. ($\Delta H = -781$ and -829 cal/mol respectively for $x_{Cd} = 0.35$ and 0.533) than the average calorimetric measurements. These measurements have been disregarded in the present comparison.

4 - Solid alloys :

The selected values for the thermodynamic properties of formation of the solid compound $Cd_{0.5}Sb_{0.5}$ referred to solid cadmium and antimony are compared in the following table :

	T(K)	ΔH^T (cal/mol)	ΔS^T (cal/mol.K)	
SELTZ	687	- 1625	- 0.335	
OLANDER	538	- 1860	- 0.74	
BORG	298	- 1641 $\pm$ 125	- 0.3 $\pm$ 0.25	
	600	- 1581 $\pm$ 125	- 0.16 $\pm$ 0.25	
SILVESTRI	594.18	- 3337	- 3.09	
EVSEEVA	594.18	385	3.74	
BILTZ	298	- 1500	-	
<u>SELECTED VALUES</u>	729	- 1630	- 0.28	
	298	- 1594	- 0.20	$\Delta C_p \approx 0$ (cal/mol.K)

$$C_p \text{ Cd}_{0.5} \text{ Sb}_{0.5} = 5.32 \times 2.32 \cdot 10^{-3} \cdot T \text{ (cal/mol.K)}$$

FIGURE 4

The selected values agree well with SELTZ and BORG's measurements in the considered temperature range.

5 - Estimation of the heat of fusion of CdSb:

The heat of fusion of $\text{Cd}_{0.5}\text{Sb}_{0.5}$ has been measured by KUBASCHEWSKI who reports a value of 3830 cal/mol, and by SELTZ who derived a value of 4135 from his e.m.f. measurements. From the selected values and considering the following equation :

$$[L_{\langle\text{CdSb}\rangle} = \Delta H_{(\text{CdSb})} - \Delta H_{\langle\text{CdSb}\rangle} + 0.5 L_{\text{Cd}} + 0.5 L_{\text{Sb}}]_{T_{\text{CdSb}}}$$

a value of 4160 cal/mol is obtained.

Another alternative is to calculate the heat of fusion from the following equation⁽²³⁾ :

$$L_{\text{CdSb}} = - RT_{\text{CdSb}}^2 \frac{d \ln a_{(\text{Sb})}}{dx_{\text{Sb}}} / \left(0.5 \frac{d^2 T}{dx_{\text{Sb}}^2} \right)$$

where $d^2T/dx_{(\text{Sb})}^2$ is the second derivative of the equation representing the liquidus line in the vicinity of the composition of the compound. The calculated value is equal to 4255 cal/mol.

The proposed value for the latent heat of fusion is 4160 ± 150 cal/mol.

From the selected values of the thermodynamic properties for the liquids alloys and the intermetallic compound, the equilibrium phase diagram has been calculated, according to the equation described in appendix III. The calculated values are in excellent agreement with the diagram published in HANSEN's compilation as shown in figure 4. It can be observed that a very similar phase diagram is obtained if the specific heat contribution is neglected.

APPENDIX I

$\langle i \rangle$ solid element or "stoichiometric" compound i

(i) liquid element i

$\langle\langle i \rangle\rangle$ compound i in the solid alloy

$\langle\langle i \rangle\rangle$ compound i in the liquid alloy

T_i melting point of element or compound i

L_i latent heat of fusion of element or compound i

$\bar{C}_{p(i)}$ partial specific heat of component i in the liquid phase

$C_{p\langle i \rangle}^\circ$ or $C_{p(i)}^\circ$ specific heat of pure component i in the corresponding state

$$\Delta \bar{C}_{p(i)} = \bar{C}_{p(i)} - C_{p(i)}^\circ$$

C_p^1 specific heat of the liquid alloy

$$\Delta C_p^1 = C_p^1 - x_{\langle\langle i \rangle\rangle} \bar{C}_{p\langle\langle i \rangle\rangle} - (1 - x_i) \bar{C}_{p\langle\langle j \rangle\rangle}$$

$\Delta \bar{H}_{\langle\langle i \rangle\rangle}$, $\Delta \bar{S}_{\langle\langle i \rangle\rangle}$, partial heat and entropy of mixing of compound i referred to the same state

$\Delta H_{\langle i \rangle}$, $\Delta S_{\langle i \rangle}$ heat and entropy of formation of solid i from the solid elements

T equilibrium temperature.

APPENDIX II

Calculation of the partial specific heats of Cd and Sb :

From the experimental values of C_p measured by SCHICK presented in the form of $(C_p^1 = aT + b)_{x_{Cd}}$, $(\bar{C}_{p((Cd))})_{T, x_{Cd}}$ was obtained by a graphical method.

Using SCHICK's value of $C_p^0(Cd)$ for pure liquid cadmium, $(\Delta \bar{C}_{p((Cd))})_{T, x_{Cd}}$ was calculated. The next steps were to calculate

$(\Delta \bar{C}_{p((Cd))} = a'T + b')$ the coefficients a' and b' being obtained by a least square fit. Integration of the GIBBS-DUHEM equation leads to $(\Delta \bar{C}_{p((Sb))})_{x_{Cd}}$.

From these sets of values ΔC_p^1 , $\bar{C}_{p((Cd))}$, $\bar{C}_{p((Sb))}$ and C_p^1 were calculated.

For different temperatures, the calculated values are compared with the experimental ones in table I. As an excellent agreement is obtained, the calculated values are used in the assesment. Table III presents the equations of $\bar{C}_{p((Cd))}$, $\bar{C}_{p((Sb))}$ and C_p^1 .

APPENDIX III

Calculation of the equilibrium phase diagram :

The liquidus representing the equilibrium between the liquid solution and a pure element i is given by the following equation :

$$\left[T = (\Delta \bar{H}_{((i))} + L_i) / (\Delta \bar{S}_{((i))} + L_i / T_i) \right]_{T, x_i}$$

whereas, the liquidus representing the equilibrium between the liquid solution and the intermetallic compound CdSb is given by :

$$\left[T = \frac{x_{((Cd))} \Delta \bar{H}_{((Cd))} + x_{((Sb))} \Delta \bar{H}_{((Sb))} - \Delta H_{<CdSb>} + x_{((Cd))} L_{Cd} + x_{((Sb))} L_{Sb}}{x_{((Cd))} \Delta \bar{S}_{((Cd))} + x_{((Sb))} \Delta \bar{S}_{((Sb))} - \Delta S_{<CdSb>} + x_{((Cd))} L_{Cd}/T_{Cd} + x_{((Sb))} L_{Sb}/T_{Sb}} \right]_{T, x_i}$$

where

$$[\Delta \bar{H}_{((i))}]^T_{, x_i} = [\Delta \bar{H}_{((i))}^{773K}] + \int_{773}^T \Delta \bar{C}_{P((i))} dT_{, x_i} ; [\Delta \bar{S}_{((i))}]^T_{, x_i} = [\Delta \bar{S}_{((i))}^{773K}] + \int_{773}^T (\Delta \bar{C}_{P((i))} / T) dT_{, x_i}$$

$$\Delta H_{<CdSb>}^T = \Delta H_{<CdSb>}^{T_{CdSb}} + \int_{T_{CdSb}}^T C_{P<CdSb>} dT ; \Delta S_{<CdSb>}^T = \Delta S_{<CdSb>}^{T_{CdSb}} + \int_{T_{CdSb}}^T (C_{P<CdSb>} / T) dT ;$$

$$L_i^T = L_i^{T_i} + \int_{T_i}^T C_{P< i >} dT - \int_{T_i}^T C_{P(i)} dT.$$

The liquidus has been calculated using the data listed in tables II. The specific heats of the undercoded liquids have been obtained by use of the equations given in table III.

TABLE I
COMPARISON BETWEEN EXPERIMENTAL AND CALCULATED VALUES

OF THE SPECIFIC HEATS FOR Cd-Sb LIQUID ALLOYS

x_{Cd}	T = 813 K		T = 853 K		T = 903 K		T = 948 K	
	EXP.	CALC.	EXP.	CALC.	EXP.	CALC.	EXP.	CALC.
0.00	8.21	8.21	8.11	8.11	7.99	7.99	7.88	7.88
0.05	8.15	8.21	8.05	8.10	7.93	7.97	7.80	7.85
0.10	8.10	8.09	8.00	7.99	7.87	7.87	7.75	7.76
0.15	8.05	8.10	7.95	8.00	7.85	7.87	7.73	7.76
0.20	7.95	8.06	7.87	7.96	7.80	7.84	7.70	7.73
0.25	7.90	7.93	7.85	7.85	7.75	7.75	7.65	7.66
0.30	7.90	7.83	7.82	7.76	7.75	7.67	7.64	7.60
0.35	7.95	7.92	7.85	7.83	7.80	7.73	7.65	7.63
0.40	8.22	8.28	8.05	8.14	7.90	7.97	7.80	7.81
0.45	8.50	8.85	8.50	8.63	8.15	8.35	7.95	8.10
0.50	9.81	9.43	9.40	9.13	8.92	8.75	8.58	8.41
0.55	9.85	9.78	9.52	9.42	9.02	8.98	8.61	8.58
0.60	9.68	9.71	9.35	9.37	8.90	8.93	8.51	8.53
0.65	9.15	9.21	8.85	8.93	8.55	8.56	8.25	8.25
0.70	8.40	8.42	8.20	8.24	7.99	8.01	7.80	7.80
0.75	7.50	7.65	7.45	7.56	7.55	7.45	7.40	7.36
0.80	7.20	7.15	7.12	7.13	7.15	7.10	7.08	7.07
0.85	7.15	7.05	7.05	7.03	7.06	7.01	7.02	6.99
0.90	7.10	7.12	7.05	7.09	7.04	7.05	7.01	7.02
0.95	7.08	7.15	7.05	7.11	7.02	7.07	7.00	7.03
1.00	7.08	7.07	7.05	7.05	7.02	7.02	6.99	6.99

TABLE II

THERMODYNAMIC PROPERTIES FOR LIQUID ALLOYS AT 773 K
REFERRED TO THE PURE LIQUID ELEMENTS

x_{Sb}	$\Delta \overline{H}_{Sb}$ (cal/mol)	$\Delta \overline{S}_{Sb}^{xs}$ (cal/mol.K)	$\Delta \overline{H}_{Cd}$ (cal/mol)	$\Delta \overline{S}_{Cd}^{xs}$ (cal/mol.K)
0.00	1224.1416	5.1645	0.0	0.0000
0.05	185.1425	3.6000	26.0	0.0393
0.10	- 638.8568	2.2863	92.0	0.1450
0.15	- 1260.8566	1.2136	180.0	0.2972
0.20	- 1666.4815	0.4090	265.0	0.4666
0.25	- 1894.1060	- 0.1573	330.0	0.6295
0.30	- 1897.7740	- 0.4367	330.0	0.7335
0.35	- 1693.5210	- 0.4719	230.0	0.7480
0.40	- 1131.1303	- 0.1534	- 110.0	0.5523
0.45	- 404.2504	0.4364	- 650.0	0.1164
0.50	- 18.4490	0.6408	- 1000.0	- 0.0625
0.55	144.5735	0.5969	- 1180.0	- 0.0116
0.60	196.7341	0.4723	- 1250.0	0.1578
0.65	167.3693	0.3539	- 1200.0	0.3555
0.70§	105.3852	0.2236	- 1070.0	0.6274
0.75	45.1844	0.1078	- 910.0	0.9314
0.80	16.4358	0.0496	- 810.0	1.1291
0.85	4.9389	0.0165	- 755.0	1.2843
0.90	1.4679	0.0009	- 730.0	1.3910
0.95	0.3254	- 0.0014	- 715.0	1.4137
1.00	0.000	0.0000	- 700.0	1.3475

§ : phase boundary. For $x_{Sb} > 0.65$, the data correspond to supercooled alloys.

Melting point : (K) $T_{Cd} = 594.18$ $T_{Sb} = 904$ (24)

Heat of fusion : (cal/mol) $L_{Cd} = 1480 \pm 20$ $L_{Cd} = 4700 \pm 150$ (24)

$[\Delta \overline{H}_{Cd} / (1-x_{Cd})^2]_{x_{Cd} \rightarrow 1} = 11400.0$

$[\Delta \overline{S}_{Cd}^{xs} / (1-x_{Cd})^2]_{x_{Cd} \rightarrow 1} = 17.0$

x_{Cd}	$C_{\text{Cd}} = a_1 + b_1$	$C_{\text{Sb}} = a_1 + b_1$	$C_{\text{Pb}} = a_1 + b_1$	$C_{\text{Sn}} = a_1 + b_1$	$C_{\text{In}} = a_1 + b_1$	range	
	$a \cdot 10^3$	b	$a' \cdot 10^3$	b'	$a'' \cdot 10^3$	b''	
0.00	-0.65526	11.37703	-2.50000	9.56000	-2.50000	9.56000	903.5-910
0.05	5.41558	1.95720	-3.10055	10.06000	-2.67474	9.65486	887-903
0.10	-3.07776	9.27624	-2.37012	9.42779	-2.44088	9.41263	869-903
0.15	-3.26298	9.90966	-2.41378	9.39630	-2.54116	9.47330	850-903
0.20	2.42711	5.27124	-3.61691	10.37907	-2.40810	9.35750	829-903
0.25	4.86587	3.16813	-4.28777	10.95967	-2.04436	9.01178	808-903
0.30	-2.44533	8.37542	-1.72503	8.92900	-1.94112	8.76292	785-833
	-0.63200	7.41900	-2.50000	9.56000	-1.94000	8.92200	833-903
0.35	-13.73276	18.25690	4.08735	4.14817	-2.14970	9.08622	760-836
	-0.63200	7.41900	-2.50000	9.56000	-1.84600	8.62300	836-903
0.40	-24.76938	27.50445	10.67695	-1.37412	-3.50158	10.17731	717-838
	-0.63200	7.41900	-2.50000	9.56000	-1.75300	8.70700	838-903
0.45	-29.66838	31.63221	14.20168	-4.34477	-5.53985	11.84487	724-838
	-0.63200	7.41900	-2.50000	9.56000	-1.65900	8.60000	838-903
0.50	-25.65938	28.34069	10.44204	-1.25621	-7.60867	13.54224	727-838
	-0.63200	7.41900	-2.50000	9.56000	-1.56600	8.49250	838-903
0.55	-14.65567	19.17382	-11.82262	8.96235	-13.38079	14.57866	724-860
	-0.63200	7.41900	-2.50000	9.56000	-1.47260	8.38500	860-903
0.60	-1.58279	8.22472	-19.45178	23.73414	-8.73038	14.42849	714-877
	-0.63200	7.41900	-2.50000	9.56000	-1.37900	8.27800	877-903
0.65	8.16422	0.01567	-36.05757	37.63447	-7.31340	13.18225	695-880
	-0.63200	7.41900	-2.50000	9.56000	-1.28600	8.17050	880-903
0.70	11.32622	-2.69294	-41.96374	42.78438	-4.66077	10.95025	674-881
	-0.63200	7.41900	-2.50000	9.56000	-1.19200	8.06300	881-903
0.75	8.27929	-0.17759	-33.65473	35.91961	-2.20421	8.84671	651-903
0.80	2.62552	4.56787	-14.04296	19.45518	-6.70817	7.54533	628-903
0.85	-1.19552	7.81852	3.50950	4.51197	-0.48977	7.32254	603-903
0.90	-1.26574	7.93349	3.56274	4.07438	-0.78289	7.54758	575-903
0.95	-0.84881	7.59844	-1.77477	8.38283	-0.89511	7.63766	572-903
1.00	-0.63200	7.41900	-10.62020	15.71867	-0.63200	7.41900	594-903

TABLE III

Partial and integral specific heats for liquid alloys.

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