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Enthalpy of formation of the [In–Sn–Zn] liquid system

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Abstract

With a fully automated high temperature calorimeter, the enthalpy of formation of the ternary liquid system [In–Sn–Zn] has been measured at about T_e=713 K, over a large molar fraction range. From the enthalpy of formation of the binary liquid systems [In–Zn], [Sn–Zn] and [In–Sn] previously published, the ternary enthalpies of formation have been estimated by using several well-known relations. Calculated and experimental results have been compared.

Keywords: In–Sn–Zn system; Enthalpy of formation; High temperature calorimetry; Liquid alloys

1. Introduction

Lead–tin solder alloys are widely used in electronics industry because of their physical properties (low melting point, good corrosion resistance, correct electrical conductivity, ...). But, because of the toxic nature of lead its use is restricted in many applications. So, new lead free solders are required and tin-based multicomponent materials with alloying elements silver, zinc, indium, bismuth, antimony and/or copper are likely to be most promising. The melting point or the melting range of the new material will be one of the most important requirements. So liquidus temperature must be well determined.

Indeed, enthalpy and Gibbs free energy data and thermodynamic calculations are not the unique way to design a solder alloy (electrical conductivity, wettability, mechanical properties, ... must be known). However for n-components alloys (n>3), experimental determinations of the equilibrium phase diagram will be more and more time-consuming and consequently thermodynamic data and calculations will be helpful to guide the choice of a new material.

So, in the present paper, the results of the calorimetric study of the ternary liquid system [In–Sn–Zn] are given; this is a first stage allowing the calculation of the equilibrium phase diagram of this system.

The equilibrium phase diagrams and the thermochemistry of the limiting binary alloys are well known and briefly recalled in the following. For the ternary system, thermodynamic studies based on phase equilibria calculations were made recently [1] but no experimental thermodynamic properties are available.

2. Literature review

Using the Thermo-Calc thermochemical software and the thermodynamic data of the limiting binary alloys, Yoon et al. [1] have calculated the phase diagram of the [In–Sn–Zn] system. Moreover, with a differential scanning calorimeter, several melting

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and phase transition temperatures have been determined by these authors. Near the [Sn–In] system, two ternary peritectic points and a ternary eutectic point have been found. Nevertheless until now, the thermodynamic data of this ternary system seems to be unknown. In the following the main characteristics of the limiting binary systems have been recalled.

2.1. [In–Zn] system

2.1.1. Phase diagram

The phase diagram (Fig. 1) exhibits a eutectic point at T_{eut}=416.65 K and two extremal solid solutions: (In) terminal solid solution (solubility maximum 2.06 at.% Zn at the eutectic temperature) and the

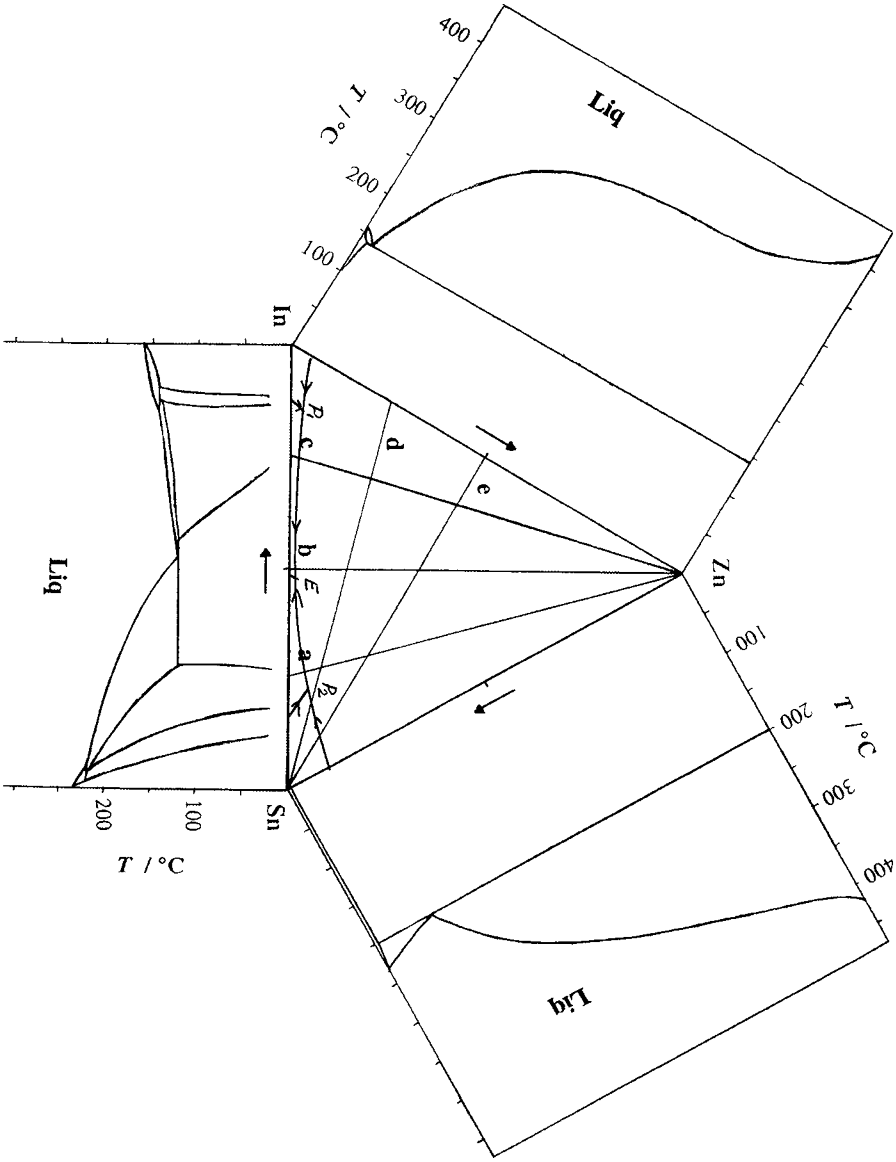

Fig. 1. The limiting equilibrium phase diagrams: [In+Zn] from [2], [Zn+Sn] from [4], [Sn+In] from [5]. The (a, b, c, d, e) lines correspond to the investigated isopleths P_1, P_2 and E are the peritectic and eutectic points of the ternary system (from [1]), respectively.

chp (Zn) solid solution (solubility maximum of approximately 0.12 at.% In).

2.1.2. Thermodynamics

An important review of thermodynamic properties of [In–Zn] alloys is available [2]. Hultgren et al. [3] using most of these data proposed for the enthalpy of formation of the liquid phase the following relation:

$$\Delta_{\mathrm{mix}}H^{\circ}_{\mathrm{m}}/\mathrm{kJ\,mol^{-1}} = 10.83 + 5.60x_{\mathrm{Zn}} - 6.94x_{\mathrm{Zn}^2} + 8.04x_{\mathrm{Zn}^3} \quad (1)$$

Hajra and Sinha [4] have published a critical assessment of this system. Using several models (subregular, exponential, Krupkowski equation, ...) they have recalculated the activity of In and Zn at 714 K. These results are in good agreement with experimental values proposed by Moser et al. [5]. Moreover differences between calculated and experimental temperatures of the liquidus line on a large molar fraction range ($0.1<x_{\mathrm{Zn}}<1$) are also very small.

2.2. [Sn–Zn] system

2.2.1. Phase diagram

As for the [In–Zn] system, the phase diagram (Fig. 1) is a simple eutectic type. The equilibrium phases are the liquid, the tetragonal (Sn) terminal solid solution (solubility maximum 0.6 at.% Zn at the eutectic temperature $T_{\mathrm{eut}}=471.65$ K) and the chp (Zn) solid solution (solubility maximum of approximately 0.039 at.% Sn).

2.2.2. Thermodynamics

Thermodynamic properties of [Sn–Zn] alloys have been well investigated by a lot of methods: emf, vapor pressure, calorimetry, ... A review is available [5].

As for the [In–Zn] system, Hultgren et al. [3] used most of these data to evaluate thermodynamic properties of liquid [Sn–Zn] alloys. They proposed some values for enthalpies of formation of the liquid phase, well represented by the following relation:

$$\Delta_{\mathrm{mix}}H^{\circ}_{\mathrm{m}}/\mathrm{kJ\,mol^{-1}} = 8.06 + 14.98x_{\mathrm{Zn}} - 25.19x_{\mathrm{Zn}^2} + 24.44x_{\mathrm{Zn}^3} \quad (2)$$

Also for this system Hajra and Sinha [4] have compared calculated and experimental curves of liquidus and activity (zinc and tin): both show a good agreement.

In 1993, Srivastawa and Sharma [6] have proposed a critical compilation of the thermodynamic properties of the Sn–Zn system. From this study, it seems that the data compiled by Hultgren et al. can be kept.

2.3. [In–Sn] system

2.3.1. Phase diagram

The phase diagram (Fig. 1) presents a eutectic point and a complex solid field with two intermediate phases β and γ. Two peritectic reactions are observed at 416 and 497 K [7].

2.3.2. Thermodynamics

A lot of authors have studied the thermodynamic properties of [Sn–In] alloys. Between 520 and 720 K the enthalpy of formation of this liquid system has been obtained by calorimetry by several groups. All these data are in very good agreement (±15 J mol^{-1}). As for the [In–Zn] and [Sn–Zn] systems, the equation proposed by Hultgren et al. [3] for enthalpies of formation of the liquid phase:

$$\Delta_{\mathrm{mix}}H^{\circ}_{\mathrm{m}}/\mathrm{kJ\,mol^{-1}} = -0.97 + 0.47x_{\mathrm{Sn}} - 0.08x_{\mathrm{Sn}^2} - 0.24x_{\mathrm{Sn}^3} \quad (3)$$

has been used.

From all these experimental data the equilibrium phase diagram has been calculated by Lee et al. [7]. Calculated and experimental temperatures are in good agreement.

2.4. [In–Sn–Zn] system

DTA measurements were performed along four cross-sections ($x_{\mathrm{Sn}}/x_{\mathrm{Zn}}$: 2:1, 1:1, 1:1 and $x_{\mathrm{In}}=0.10$) by Xie and Mikula [8] in 1998. Moreover on several samples, X-ray diffraction and microprobe analysis were done. More recently, Hertz et al. [9] proposed a description of the enthalpy of formation of this ternary system based on the enthalpy of mixing of the three limiting systems corrected by a ternary term ($-5545x_{\mathrm{In}}x_{\mathrm{Sn}}x_{\mathrm{Zn}}$ in J mol^{-1}).

3. Experimental procedure

The enthalpies of formation of the liquid [In–Sn–Zn] system were measured at two different temperatures (about 713 and 907 K) by direct-reaction calorimetry using the drop method [10,11].

3.1. Calorimeter

Measurements were performed with a high temperature Calvet calorimeter described elsewhere [12] along five cross sections.

Small pieces of zinc (between 50 and 170 mg) were added consecutively into the liquid bath formed by an indium–tin alloy first (isopleths a, b, c (Fig. 1)), or tin in indium–zinc alloy, respectively (isopleths d, e (Fig. 1)).

For example the reactions, along the isopleths a, b, c, can be symbolized as follows:

$$(\mathrm{In}_k + \mathrm{Sn}_{(1-k)})_{\mathrm{liq},} T_\mathrm{e} + a_1\mathrm{Zn}_{\mathrm{sol},} T_\mathrm{o} \rightarrow (\mathrm{In}_{x1}\mathrm{Sn}_{y1}\mathrm{Zn}_{(1-x1-y1)})_{\mathrm{liq},} T_\mathrm{e}$$

$$\dots$$

$$(\mathrm{In}_{xi} + \mathrm{Sn}_{yi}\mathrm{Zn}_{(1-xi-yi)})_{\mathrm{liq},} T_\mathrm{e} + a_i\mathrm{Zn}_{\mathrm{sol},} T_\mathrm{o} \rightarrow (\mathrm{In}_{xi}\mathrm{Sn}_{yi}\mathrm{Zn}_{(1-xi-yi)})_{\mathrm{liq},} T_\mathrm{e}$$

with k the molar fraction of indium in the binary system (In–Sn) and x_i and y_i the indium and tin molar fractions in the ternary system, respectively. T_o (of about 298 K) and T_e (713 or 907 K) are the ambient and the experimental temperatures, respectively.

3.2. Materials

High purity metals, indium (99.999 wt.% In) from Preussag Metals, tin (99.9999 wt.% Sn) and zinc (99.9999 wt.% Zn) from Koch-Light Co. were used. These metals were cut into small pieces, rinsed with acetone of high purity (pro analysis, Merck) and dried in air.

A small flow of high purity argon (Argon N56 from Air Liquide Company) is maintained during the measurements in the experimental chamber and in the cell.

Crucibles made of pure graphite (Carbone Lorraine Co.) were used as experimental cell to synthesize the alloys.

3.3. Calibration and precision

The calorimeter was calibrated at the end of each measurement series by dropping known amounts of α-alumina (purchased from NIST [13]) from ambient temperature into the calorimeter at working temperature T_e. The relative enthalpies of the α-alumina pieces were calculated by using the $C_\mathrm{p}=f(T)$ data published by NIST.

Relative enthalpies of metals were taken from the Scientific Group Thermodata Europe (SGTE) [14].

At 900 K, the vapor pressures of indium and tin are sufficiently low to assume only negligible loss of metal by evaporation ($p_{(\mathrm{In})} = 10^{-5}$ Pa, $p_{(\mathrm{Sn})}=10^{-8}$ Pa). Because the vapor pressure is not so low ($p_{(\mathrm{Zn})}=10^{-2}$ Pa), each alloy was weighed after the end of a measurement series and compared with the sum of the masses of the charged metals: the differences detected were always less than 0.5% of the total mass. The error in the enthalpies of mixing was estimated to be in the same order of magnitude as the scatter (of about 2%) found with the calibration drops of α-alumina. So, uncertainties are evaluated at about 5% for the $\Delta_\mathrm{mix}H^\circ_\mathrm{m}$ values and at less than 1% for the molar fraction x.

The experimental temperatures were obtained with a [Pt/Pt-Rh 10 wt.%] thermocouple located near the crucible with an accuracy of ±2 K, at least.

4. Results

4.1. Molar enthalpies of mixing

Calorimetric experiments were performed at two temperatures (about 713 and 907 K) in order to obtain the enthalpies of mixing over a larger concentration range of the liquid phase and to test the temperature dependence.

Additions of Zn (Fig. 2a):

$x_\mathrm{In}/x_\mathrm{Sn}=0.25/0.75$	at 714 K: $0<x_\mathrm{Zn}<0.56_7$
(isopleth a)	at 907 K: $0<x_\mathrm{Zn}<0.55_0$
$x_\mathrm{In}/x_\mathrm{Sn}=0.50/0.50$	at 713 K: $0<x_\mathrm{Zn}<0.53_5$
(isopleth b)	at 714 K: $0<x_\mathrm{Zn}<0.61_6$
$x_\mathrm{In}/x_\mathrm{Sn}=0.75/0.25$	at 712 K: $0<x_\mathrm{Zn}<0.64_6$
(isopleth c)	at 713 K: $0<x_\mathrm{Zn}<0.59_3$

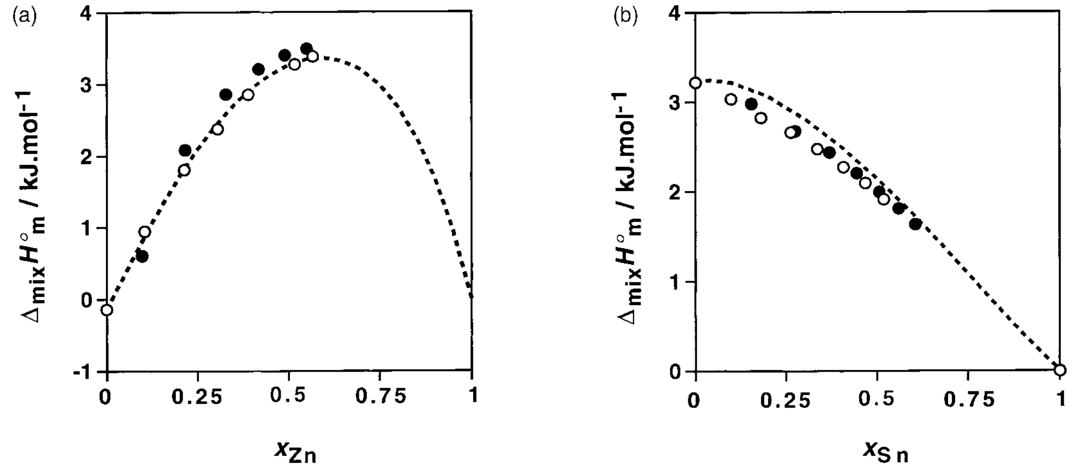

Fig. 2. (a) Experimental values of the enthalpy of formation (kJ mol^{-1}) of the [Zn+Sn+In] liquid system along the isopleth a (x_{In}/x_{Sn}=0.25/0.75) (experimental results at 714 K (○) and 907 K (●) respectively); calculated by Muggianu's equation (- - -). (b) Experimental values of the enthalpy of formation (kJ mol^{-1}) of the [Zn+Sn+In] liquid system along the isopleth e (x_{Zn}/x_{In}=0.50/0.50) at 713 K; calculated by Muggianu's equation (- - -).

Table 1
Experimental results of the enthalpy of formation of the [In–Sn–Zn] liquid system (referred to the liquid state) in kJ mol^{-1}[a]

x_{In}/x_{Sn}=0.25/0.75				x_{In}/x_{Sn}=0.50/0.50				x_{In}/x_{Sn}=0.75/0.25			
T=714 K		T =907 K		T=713 K		T=714 K		T=712 K		T=713 K	
x_{Zn}	$\Delta_{mix}H^{\circ}_{m}$	x_{Zn}	$\Delta_{mix}H^{\circ}_{m}$	x_{Zn}	$\Delta_{mix}H^{\circ}_{m}$	x_{Zn}	$\Delta_{mix}H^{\circ}_{m}$	x_{Zn}	$\Delta_{mix}H^{\circ}_{m}$	x_{Zn}	$\Delta_{mix}H^{\circ}_{m}$
0.0000	−0.14	0.0000	−0.14	0.0000	−0.20	0.0000	−0.20	0.0000	−0.16	0.0000	−0.16
0.1040	0.95	0.0973	0.61	0.1072	0.90	0.1238	1.20	0.1302	1.38	0.1788	1.81
0.2132	1.81	0.2159	2.08	0.1961	1.68	0.2256	2.09	0.2510	2.34	0.3047	2.80
0.3049	2.37	0.3283	2.86	0.2704	2.25	0.3322	2.74	0.3553	2.94	0.4045	3.31
0.3887	2.85	0.4188	3.21	0.3353	2.59	0.4175	3.07	0.4562	3.28	0.4835	3.57
0.5183	3.27	0.4903	3.40	0.4005	2.90	0.4878	3.20	0.5306	3.39	0.5440	3.65
0.5671	3.38	0.5503	3.49	0.4644	3.07	0.5577	3.35	0.5954	3.35	0.5928	3.64
				0.5354	3.23	0.6157	3.30	0.6460	3.16		

[a] Addition of Zn to (In–Sn) liquid alloys at 714 and 907 K.

Table 2
Experimental results of the enthalpy of formation of the [In–Sn–Zn] liquid system (referred to the liquid state) in kJ mol^{-1}[a]

x_{Zn}/x_{In}=0.25/0.75				x_{Zn}/x_{In}=0.50/0.50			
T=713 K		T=907 K		T=713 K (a)		T=713 K (b)	
x_{Sn}	$\Delta_{mix}H^{\circ}_{m}$	x_{Sn}	$\Delta_{mix}H^{\circ}_{m}$	x_{Sn}	$\Delta_{mix}H^{\circ}_{m}$	x_{Sn}	$\Delta_{mix}H^{\circ}_{m}$
0.0000	2.24	0.0000	2.24	0.0000	3.22	0.0000	3.22
0.0792	1.98	0.0748	2.08	0.1538	2.98	0.0972	3.03
0.1580	1.77	0.1712	1.79	0.2751	2.67	0.1804	2.82
0.2294	1.63	0.2531	1.58	0.3707	2.43	0.2629	2.66
0.2961	1.53	0.3360	1.35	0.4445	2.20	0.3362	2.47
0.3554	1.39	0.4237	1.15	0.5064	1.99	0.4084	2.27
0.4160	1.25	0.5006	1.02	0.5597	1.81	0.4681	2.09
0.4808	1.13			0.6051	1.63	0.5184	1.91
0.5340	1.03						

[a] Addition of Sn to (In–Zn) liquid alloys at 714 K.

Additions of Sn (Fig. 2b):

x_{Zn}/x_{In}=0.25/0.75 (isopleth d)	at 713 K: $0<x_{Sn}<0.53_4$ at 907 K: $0<x_{Sn}<0.50_1$
x_{Zn}/x_{In}=0.50/0.50 (isopleth e)	at 713 K (a): $0<x_{Sn}<0.60_5$ at 713 K (b): $0<x_{Sn}<0.51_8$

The experimental results of the enthalpy of formation (given in Tables 1 and 2) are referred to the liquid state and correspond to the following reactions at T_e and $p°$:

$$\text{Isopleths a, b, c}: \quad \Delta_{\text{mix}}H^{\circ}_{\text{m}} = f(x_{\text{Zn}})_{x_{\text{In}}/x_{\text{Sn}}\text{const}}$$

Results Table 1

$$(\text{In}_k + \text{Sn}_{(1-k)})_{\text{liq}}, T_{\text{e}} + a_1\text{Zn}_{\text{liq}}T_{\text{o}} \rightarrow (\text{Sn}_{x1}\text{In}_{y1}\text{Zn}_{(1-x1-y1)})_{\text{liq}}, T_{\text{e}}$$

$$\text{Isopleths d, e}: \quad \Delta_{\text{mix}}H^{\circ}_{\text{m}} = f(x_{\text{Sn}})_{x_{\text{Zn}}/x_{\text{In}}\text{const}}$$

Results Table 2

$$(\text{In}_k + \text{Zn}_{(1-k)})_{\text{liq}}, T_{\text{e}} + a_1\text{Sn}_{\text{liq}}T_{\text{o}} \rightarrow (\text{In}_{x1}\text{Zn}_{y1}\text{Sn}_{(1-x1-y1)})_{\text{liq}}, T_{\text{e}}$$

Fig. 2 reveals an atomic repulsion in the alloys ($\Delta_{\text{max}}H^{\circ}_{\text{m}}$ values are positive). The scatter between all results is less than 4%. The experimental data have been represented by the following polynomial equation:

$$\Delta_{\text{max}}H^{\circ}_{\text{m}}/\text{kJ mol}^{-1} = A + Bx + Cx^2 + Dx^2 \tag{4}$$

with $x=x_{\text{Zn}}$ for each isopleth a, b or c; or $x=x_{\text{Sn}}$ for each isopleth d or e.

The coefficients have been calculated by the least squares method (Table 3).

Table 3
Coefficients of the equation for the enthalpies of formation of the liquid phase for the five isopleths: $\Delta_{\text{mix}}H^{\circ}_{\text{m}}=A+B\cdot x+C\cdot x^2+D\cdot x^3$ in kJ mol^{-1}

$x_{\text{In}}/x_{\text{Sn}}$	*A*	*B*	*C*	*D*
Addition of Zn				
0.75/0.25	−0.161	13.007	−10.606	−2.240
0.50/0.50	−0.196	11.240	−6.419	−4.619
0.25/0.75	−0.143	9.384	0.052	−9.294
$x_{\text{Zn}}/x_{\text{In}}$	*A*	*B*	*C*	*D*
Addition of Sn				
0.50/0.50	3.225	−1.853	−1.052	−0.319
0.25/0.75	2.237	−2.609	0.316	0.056

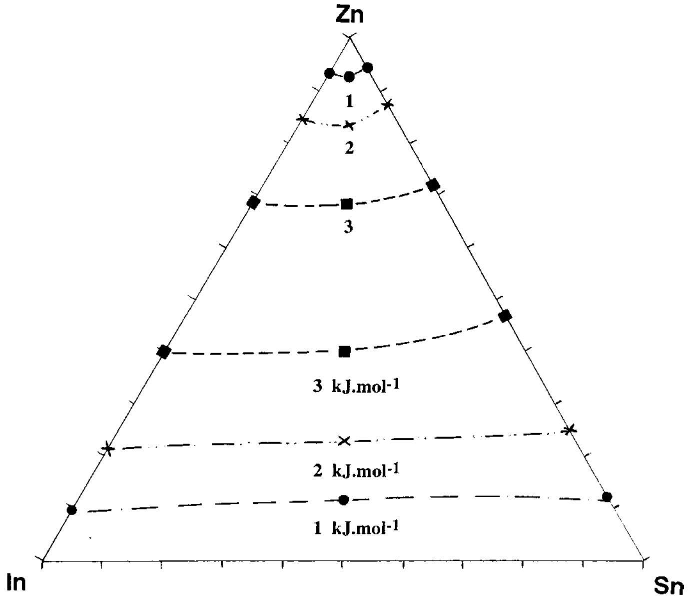

Fig. 3. Isoenthalpic lines of the liquid [Zn+Sn+In] system obtained by interpolation of the experimental results: 1 kJ mol^{-1} (- · -); 2 kJ mol^{-1} (- · · -); 3 kJ mol^{-1} (- - - -).

Table 4
Values of the enthalpy of formation in kJ mol^{-1} at the intersection points: from calorimetric measurements by additions of Zn (column 5), by additions of Sn (column 6) and estimated by using several relations (columns 9–13)[a]

	Molar fraction		$\Delta_{mix}H^{\circ}_{m}$ (experiments)		$\Delta_{mix}H^{\circ}_{m}$ (estimation)				
	X_{sn}	x_{Zn}	Add. of Zn	Add. Of Sn	K	M	T-(In)	T-(Sn)	T-(Zn)
Point 1 (c, d)	0.200	0.200	2.00	1.73	1.82	1.84	1.89	1.98	1.72
Point 2 (b, d)	0.429	0.143	1.27	1.18	1.24	1.30	1.32	1.38	1.16
Point 3 (a, d)	0.692	0.077	0.57	0.60	0.59	0.64	0.63	0.67	0.56
Point 4 (c, e)	0.143	0.429	3.29	2.94	3.26	3.16	3.33	3.40	2.98
Point 5 (b, e)	0.333	0.333	2.67	2.48	2.71	2.71	2.82	2.91	2.51
Point 6 (a, e)	0.600	0.200	1.66	1.67	1.70	1.74	1.78	1.81	1.61

[a] K, M, T-(In), T-(Sn) and T-(Zn) correspond to Kohler equation, Muggianu equation and to the three asymmetrical forms of the Toop equation, respectively.

Taking into account the temperature range investigated and the accuracy of the results, the enthalpy of formation of the liquid phase may be considered independent of temperature.

From these data, the lines of the isoenthalpy values (1, 2 and 3 kJ mol^{-1}) of formation have been deduced (Fig. 3).

Using Eqs. (1)–(3), the enthalpies of formation have been estimated with several well-known relations proposed by Kohler [15], Muggianu [16] and Toop [17]. Generally, experimental and calculated values present a quite good agreement. The best representation of the molar enthalpy of mixing was obtained by the Muggianu's equation with a correction term $x_{In}{\cdot}x_{Sn}{\cdot}x_{Zn}$ $(A'x_{In}+B'x_{Sn}+C'x_{Zn})$ with $A'=-4.594$, $B'=-1.439$ and $C'=3.175$ (in kJ mol^{-1}) calculated by the least squares method.

The isopleths (a, b, c) and (d, e) lead us to six intersection points for which calculated and experimental results can be compared (Table 4).

Table 5
Limiting partial molar enthalpy kJ mol^{-1} of liquid zinc $[\Delta_{mix}H^{\circ}_{m_{(Zn)}}=f\ (x_{Zn})$ with $x_{Zn}\rightarrow 0]$ and liquid tin $[\Delta_{mix}H^{\circ}_{m_{(Sn)}}=f\ (x_{Sn})$ with $x_{Sn}\rightarrow 0]$ in different [In+Sn] and [Zn+In] binary melts respectively (extrapolated values)[a]

x_{In}/x_{Sn}	$\Delta_{mix}H^{\circ}_{m}$ (Zn)	x_{Zn}/x_{In}	$\Delta_{mix}H^{\circ}_{m}$ (Sn)
0.00/1.00	8.06 [3]	0.00/1.00	−0.97 [3]
0.25/0.75	10.62	0.25/0.75	0.23
0.50/0.50	12.14	0.50/0.50	1.78
0.75/0.25	12.19	0.75/0.25	–
1.00/0.00	10.83 [3]	1.00/0.00	22.29 [3]

[a] For the binary systems, the values are from [3].

4.2. Limiting molar partial enthalpies

For each isopleth (a, b, c), the value of the limiting molar partial enthalpy of liquid zinc in the [In–Sn] liquid system $[\Delta_{mix}H^{\circ}_{m_{(zn)}}=f(x_{Zn})$ with $x_{Zn}\rightarrow 0]$ has been determined. These data versus molar fraction ratio as shown in Table 5 and Fig. 4.

In the same way, for each isopleth (d, e), the value of the limiting molar partial enthalpy of liquid tin in the [Zn–In] liquid system has been determined, too (Table 5, Fig. 5).

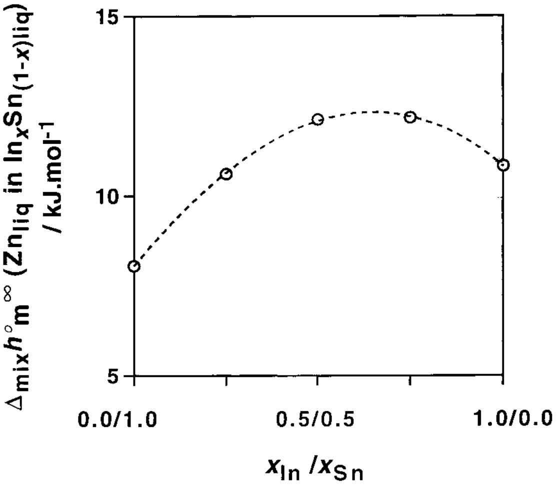

Fig. 4. The limiting partial molar enthalpy of liquid zinc $[\Delta_{mix}H^{\circ}_{m_{(Zn)}}=f\,(x_{Zn})]$ in the [In+Sn] liquid system versus the molar fraction ratio x_{In}/x_{Sn}.

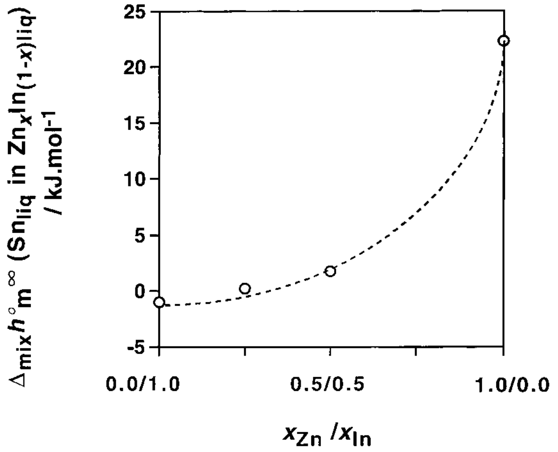

Fig. 5. The limiting partial molar enthalpy of liquid tin $[\Delta_{\text{mix}}H^{\circ}_{\text{m}_{(\text{Sn})}} = f(x_{\text{Sn}})]$ in the [Zn+In] liquid system versus the molar fraction ratio $x_{\text{Zn}}/x_{\text{In}}$.

5. Conclusion

The integral molar enthalpy of formation of the [In–Sn–Zn] liquid alloys has been measured in a large molar fraction range. The $\Delta_{\text{mix}}H^{\circ}_{\text{m}}$ function is positive in the largest molar fraction range. Moreover, the trends of the variation of the limiting partial molar enthalpy of liquid tin and zinc versus the molar fraction ratio $x_{\text{Zn}}/x_{\text{In}}$ and $x_{\text{In}}/x_{\text{Sn}}$ have been deduced.

Using the same relation of interpolation the other thermodynamic properties of this ternary system can be proposed but experimental activity data will be indispensable to ascertain these proposals.

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