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Thirty years of levitation melting calorimetry – a balance

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Abstract

Investigations on liquid high melting metals are frequently tainted with side reactions with the crucible material. Levitation techniques enable the containerless investigation of thermophysical properties over a wide temperature range. An overview is given on enthalpy measurements by levitation drop calorimetry on solid and liquid metals using the electromagnetic levitation technique. The resulting melting enthalpies and melting entropies of the IVb-metals (Ti, Zr, Hf), the Vb-metals (V, Nb, Ta), and the VIb-metals (Cr, Mo, W) show a group dependence where the IVb-metals are characterized by melting entropies in the range 6.8–8 J mol^{-1} K^{-1}, the Vb-metals have a value between 10 and 11 J mol^{-1} K^{-1}, and the VIb-metals show melting entropies in the range 13–14 J mol^{-1} K^{-1}. Another overview is given on mixing enthalpy measurements by levitation alloying calorimetry. The liquid Fe–Ti system is presented as an example for a temperature dependent mixing enthaply where modelling according to the regular associate model enables the calculation of the thermodynamic properties of mixing.

Keywords: Drop calorimetry; Levitation alloying calorimetry; Enthalpy; Melting entropy; Mixing enthalpy

1. Indroduction

On October 31, 1923, a patent was granted to Dipl.-Ing. Otto Muck in Munich with the not exciting title ‘‘Process and arrangement for melting, especially of conductors, etc. by electrical induction currents’’ [1]. The detailed claim shows that a melting technique is aspired to ‘‘use inductive current to heat and melt a sample which is simultaneously levitated by electrodynamic force’’. The patent fell into oblivion in the following decades although the inventor indicated a number of striking advantages of his process, like ‘‘avoiding of all chemical and physical reactions which normally take place between melt and crucible’’. At that time it was difficult to produce the needed field-strength for the required frequencies. On the other hand no profitable field of application was discovered.

In the beginning of the 1950s electromagnetic levitation of solid and liquid metals was proved by Westinghouse Electric Corporation, USA, and presented by two publications [2,3]. Already in 1953 Scheibe [4] in Hanau, Germany, also experimented with this technique and kept metals such as aluminium, brass, copper, iron, titanium, silver, and platinium in levitated condition. For the following 10 years the experimental and theoretical conditions of electromagnetic levitation melting were examined [5–21] until the first metallurgical applications started with gas–metal reactions [22–24] which demonstrated the significant advantages of containerless melting. To

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Table 1
Overview to drop calorimetric investigations until 1975

Year	Authors	Element	Temperature range (K)
1970	Chaudhuri et al. [25]	Cu	1386–1887 (l)
1970	Chaudhuri et al. [25]	Pt	2202–2631 (l)
1970	Treverton and Margrave [26]	Mo	2692–3112 (l)
1971	Berezin et al. [27]	Mo	2890–2925 (l)
1971	Treverton and Margrave [28]	Fe	1804–2142 (l)
1971	Treverton and Margrave [28]	Ti	1969–2313 (l)
1971	Treverton and Margrave [28]	V	2205–2638 (l)
1971	Treverton and Margrave [29]	Co	1774–2345 (l)
1971	Treverton and Margrave [29]	Pd	1846–2334 (l)
1972	Sheindlin et al. [30]	Nb	1650–2707 (s)
1972	Berezin et al. [31]	V	2084–2325 (l)
1974	Stretz and Bautista [32]	Y	1799–2360 (l)
1974	Stephens [33]	Cu	1428–2007 (l)
1974	Stephens [33]	U	1428–2398 (l)
1975	Stretz and Bautista [34]	Nd	1446–2246 (l)
1975	Stretz and Bautista [35]	La	1250–2420 (l)

date levitation melting is a process applied in research; there is indication that production of special materials under space condition can be performed by this technique in the future.

Around 1970 several laboratories combined levitation melting with a drop calorimeter. Most of the metals were measured in the liquid state as it is principally simpler to achieve high temperatures. The pioneers of the first five years in this field are listed in Table 1 [25–35]. In 1973 we started with the planning and performing of our own levitation calorimeter with the thesis of Betz [36]. In spite of numerous improvements in the following years [37,38] the original concept of the arrangement was retained.

2. Containerless melting

Metals have been industrially heated and melted in an electromagnetic field since a long time. In order to pass over to a containerless melting a part of the energy input into the metal has to be used to keep this in a levitated manner whereas the rest of the energy heats the sample. The electromagnetic field is frequently produced by rotation-symmetrical coils. Stable levitation is given when the buoyancy forces equal the weight of the sample. Beside of the horizontal field components also the vertical ones are of influence for stable levitation. Therefore the dimension and testing of special coils for special materials takes a broad field of know-how and experimentating as calculations only give a qualitative picture.

The relation between the efficiency input and the bearing strength of the coil is of special importance for the resulting temperature of the sample. The final temperature of the specimen depends on the electrical (current and frequency) and physical properties (volume, density, electrical resistance, heat losses by radiation and convection). The temperature of the sample can be influenced additionally by cooling gases or gas mixtures. One of the disadvantages of the method is the restriction of the sample weight (1–20 g); high power input is needed as a consequence of the relation between the surface tension and the density of the metallic materials.

3. Levitation drop calorimetry

3.1. Melting chamber and drop calorimeter

The melting chamber consists of a stainless steel recipient with a volume of around 30 l (Fig. 1). A water cooled internal copper jacket protects the recipient from the magnetic field of the coil. The sample can be kept in the induction coil by a quartz manipulator until the current is switched on and the sample is levitated by the electromagnetic field. After this the manipulator is withdrawn and a copper mould is positioned under the levitated sample in order to protect the calorimeter below by an erroneous drop. The wanted drop results from the switch-off of the HF-generator (65 kW with 450 or 900 kHz). Two quartz windows at opposite sides enable the optical temperature measurement with pyrometers and the recording of the sample by a video camera. The front of the cylindric recipient is a totally transparent window which enables all visual observation and manipulation inside the chamber from outside. Gate values connect the vessel with the vacuum system (final pressure 5×10^{-6} mbar) and the drop calorimeter. The inert gas normally used is helium. In order to lower the temperature of the sample a gas cooling system is used, which is quite powerful to examine samples in the undercooled state also.

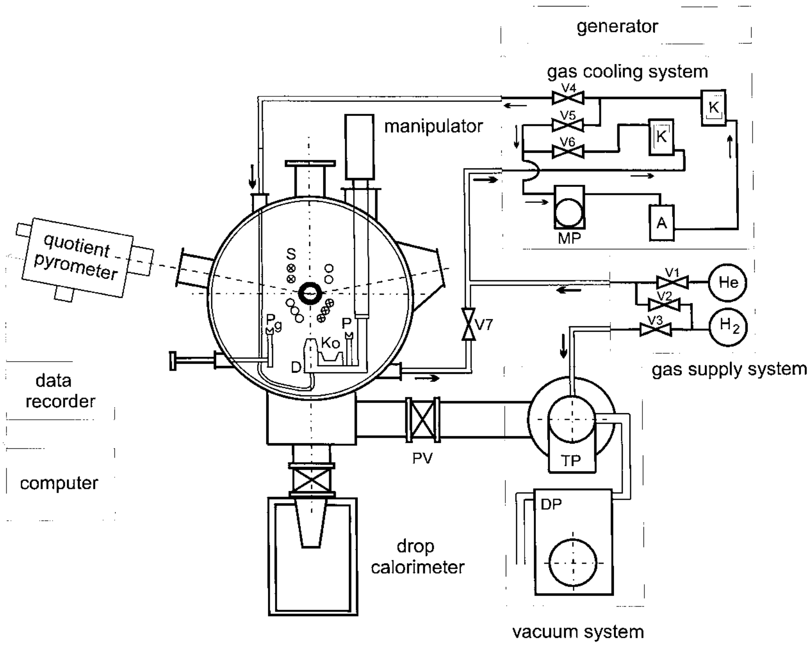

Fig. 1. Levitation drop calorimeter.

The isoparabolic working drop calorimeter consists principally of a metal block (measuring system) and a jacket held at a constant temperature. By drop of a sample into the calorimeter a temperature–time curve is observed comparable to Fig. 2. The evaluation of the ΔT_K values can be performed either by graphical or analytical methods. We used a numerical integral method, suggested by Bonnell [39], and Stretz and Bautista [35]. The enthalpy of the sample at temperature T measured by the calorimeter is given by the following equation:

$$H(T) - H(298) = C_K \cdot \Delta T_K \cdot \frac{M}{m} + Q_t \cdot \frac{M}{m} + [H(T_f) - H(298)]\ [\mathrm{J\,mol^{-1}}], \quad (1)$$

where C_K is the calorimeter constant, ΔT_K the corrected temperature rise of the block, M the molar mass and m the mass of the sample. The heat losses Q_t of the sample during its drop into the calorimeter follow the equations of radiation and convection. They are calculated and run up to 3–5% of the total heat amount. The third term considers the enthalpy correction which is due to the difference between the standard temperature and calorimeter temperature. A detailed description of the calorimeter and the corresponding evaluation is given in [38].

3.2. Results

Fig. 3 shows the enthalpy–temperature curve for the solid and liquid phase = 8 of vanadium. In all our investigations on high melting metals we have observed that for the liquid state of the metals always a straight $H-T$ connection exists, e.g., the heat capacity of the melt is described by a constant value. The undercooled melt follows the same value; this is shown in Fig. 4 for niobium ($\Delta T = 300$ K). From the enthalpy difference between the solid and liquid at the melting point the melting enthalpy ΔH_M is calculated and the melting entropy follows according to $\Delta S_M = \Delta H_M / T_M$. Table 2 shows the melting data of high and highest melting elements determined by us; some of them were formerly unknown. Since a long time it is known that metals show a linear dependence between their melting enthalpy and melting temperature. This general trend was first expressed by G. Tammann (1922) by a nearly constant melting entropy with a mean slope of $9.2\ \mathrm{J\,mol^{-1}\,K^{-1}}$.

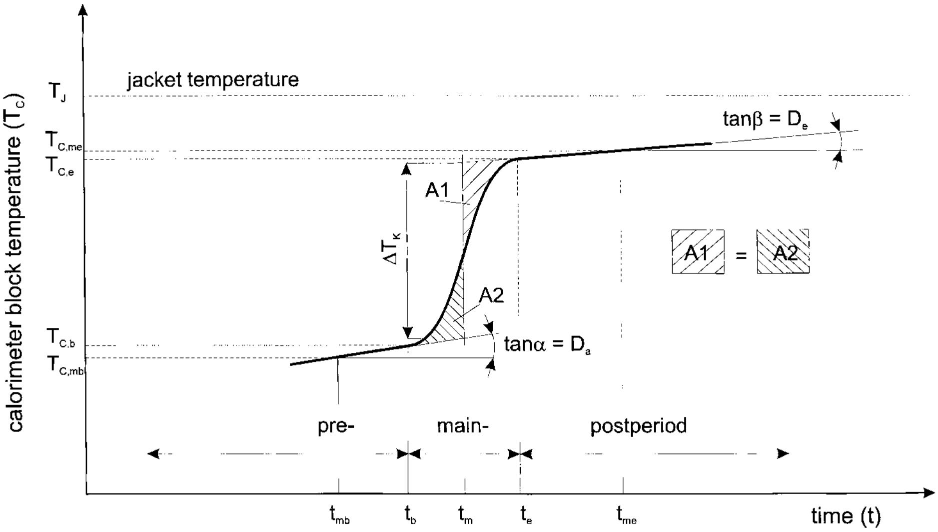

Fig. 2. Schematic temperature–time curve of the calorimeter.

Significant deviations from Tammann's rule are mean while detected. Beyond that we found for the high melting metals a group relationship which was first detected in the $\Delta H_M - T_M$-diagram (Fig. 5). The $\Delta S_M - T_M$-diagram (Fig. 6) shows the melting entropy of the VIb-metals Cr [43], Mo [41] and W [42] with more than 13 J mol^{-1} K^{-1}. The Vb-metals V [46], Nb [44] and Ta [45] have a value between 10 and 11 J mol^{-1} K^{-1} and the IVb-group is characterized by a value in the range of 6.8–8 J mol^{-1}. The group dependence can be explained with the help of the vacancy theory [47].

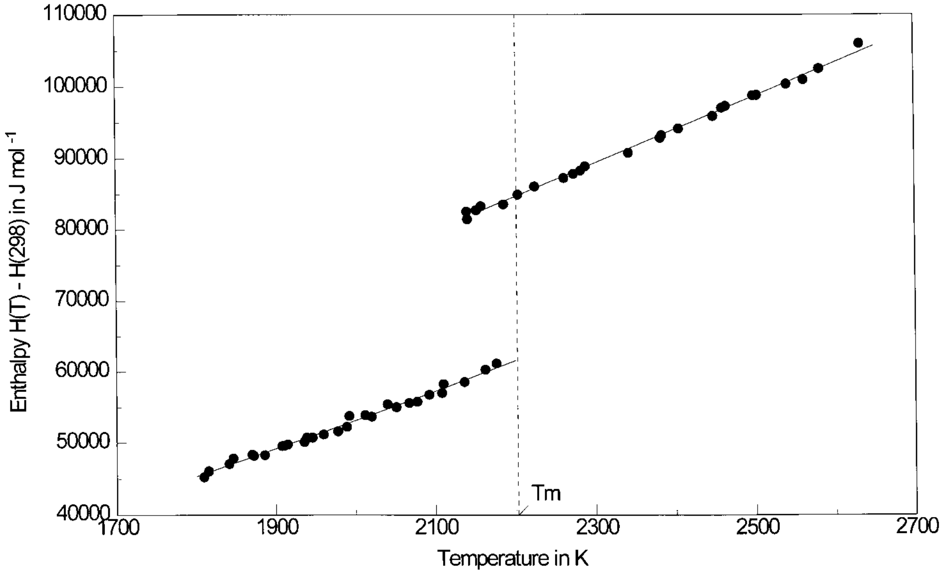

Fig. 3. Temperature dependence of the enthalpy of solid and liquid vanadium [40].

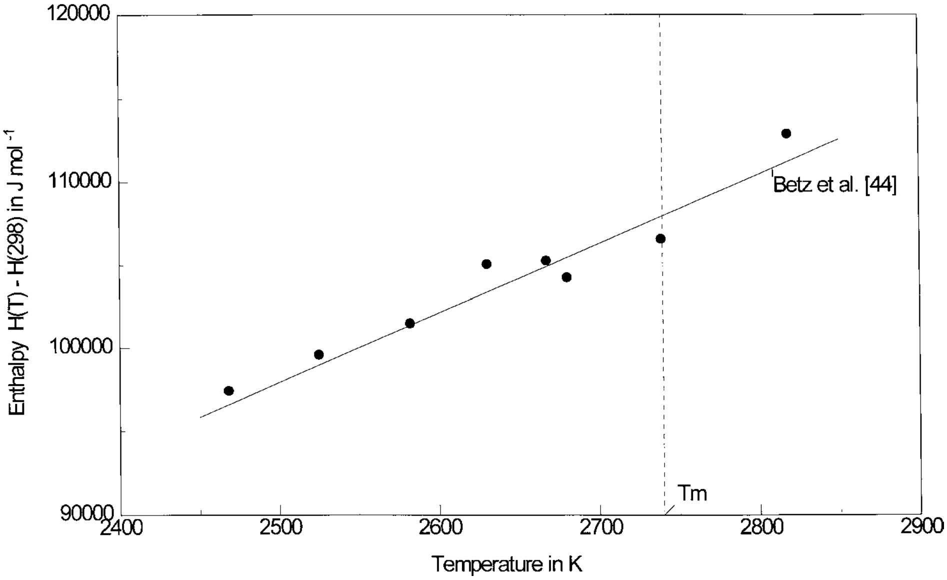

Fig. 4. Temperature dependence of the enthalpy of liquid niobium [38].

4. Levitation alloying calorimetry

4.1. Method and evaluation

In addition to the enthalpies of metals and alloys the metallurgist is especially interested in mixing enthalpies of metallic alloys. This information opens a wide field of applications like the calculation of phase diagrams, chemical activities, theoretical models of the liquid state and heat calculations for technical alloying processes. From the very beginning of levitation melting we were keenly interested to join two metallic samples within a levitation field in order to determine the heat effect of mixing. For exothermic mixing enthalpies we have solved this problem in the following way: small solid samples (0.1 g) are consecutively added from a watercooled revolving magazine (16 chambers) into a levitated liquid base sample of about 2 g (Fig. 7). During the mixing process the temperature–time curve (Fig. 8) is continuously recorded by a quotient pyrometer. From the temperature change ΔT_L the mixing enthalpy can be calcu-

Table 2
Melting enthalpies and entropies of high-melting metals (own investigations)

Year	Author	Element	T_M (K)	ΔH_M (J mol^{-1})	ΔS_M (J mol^{-1} K^{-1})
1979	Betz [36]	Mo	2892	39116	13.53
1979	Betz [36]	Nb	2740	30500	11.13
1984	Arpaci [45]	Ta	3288	33718	10.25
1984	Arpaci [42]	W	3683	50340	13.66
1991	Lin [40]	Cr	2136	29674	13.89
1991	Lin [40]	V	2202	23036	10.46
1991	Lin [40]	Re	3453	34159	9.89
1998	Drewes [47]	Ti	1943	13206	6.80
1998	Drewes [47]	Zr	2125	17127	8.06
1998	Drewes [47]	Hf	2500	19867	7.95

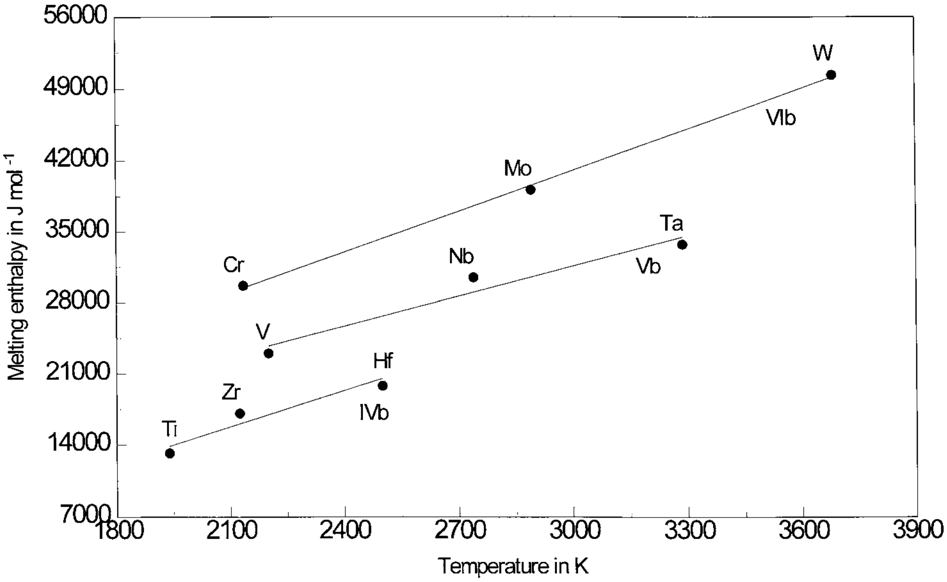

Fig. 5. Melting enthalpy versus melting temperature of the IVb, Vb, and VIb metals.

lated (here for one fusion):

$$\Delta h^{\mathrm{m}} = -\{n_{\mathrm{B}}[H_{\mathrm{B}}(T_{\mathrm{g}}) - H_{\mathrm{B}}(T_{\mathrm{R}})] + (n_{\mathrm{A}} + n_{\mathrm{B}})C_p^{\mathrm{AB}}\Delta T_{\mathrm{L}}\}\ [\mathrm{J\,mol^{-1}}], \quad (2)$$

where n_A and n_B are the mole numbers of the levitated basis sample A and the added secondary sample B. The values of the enthalpy of B at experiment temperature, $H_B(T_g)$, and room temperature, $H_B(T_R)$ can be taken from the literature. C_p^{AB} is the heat capacity of the alloy. As heat capacities of high melting and reactive alloys are normally unknown, the evaluation

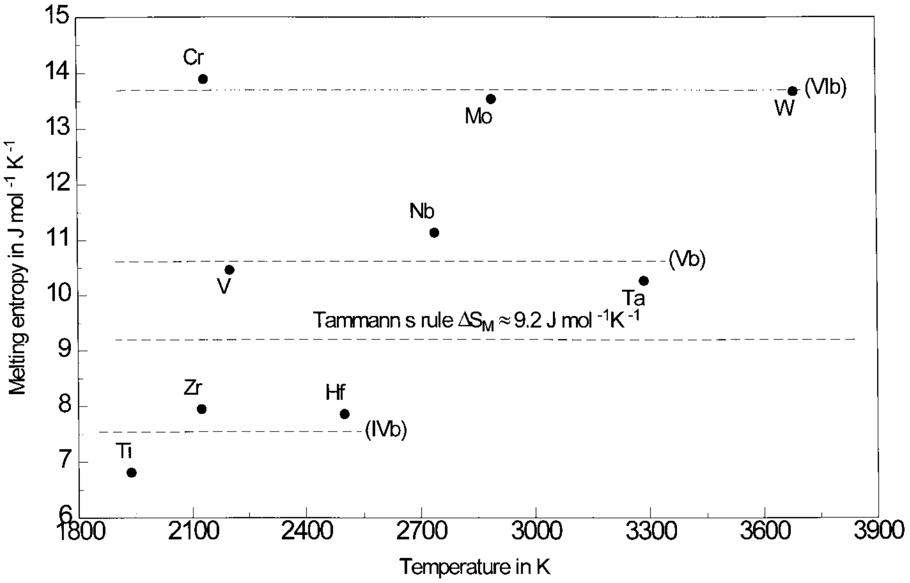

Fig. 6. Melting entropy versus melting temperature of the IVb, Vb, and VIb metals.

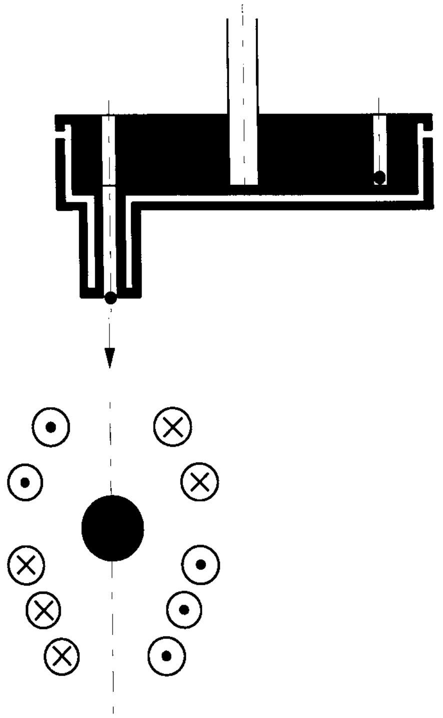

Fig. 7. Levitation coil with revolving magazine for mixing experiments.

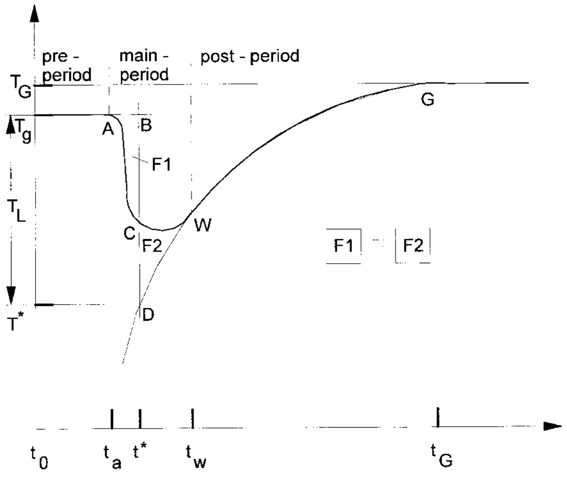

Fig. 8. Schematic temperature–time curve of a single alloying process.

is done in a first approach by using the ideal heat capacity according to the rule of Neuman–Kopp. Today we use a mathematical procedure [48,49] which enables to consider occuring excess heat capacities encountered in high melting and reactive alloys. These results follow from the measured temperature dependence of the mixing enthalpy. Eq. (3) describes the

Table 3
3Maximum value of the mixing enthalpy of high-melting systems (own investigations)

Year	System A–B(–C)	$\Delta H^M_{\max}$ (kJ mol^{-1})	n_B	Reference	T (K)
1980	Fe–Cu	+9	0.50	[50]	1873
1980	Nb–Si	−43 (no max.)	0.30	[50]	3000
1985	Mo–Si	−38	0.48	[51]	3000–3200
1992	V–Si	−58	0.45	[52]	2240
1993	Fe–V	−12	0.50	[53]	2320
1995	Fe–Nb	−14	0.55	[54]	1935
1995	Fe–Zr	−26	0.40	[55]	1923
1995	Fe–Ti	−22	0.55	[56,62]	1950
1996	Ni–V	−17	0.50	[57]	2020
1996	Ni–Nb	−18	0.50	[57]	2000
1996	Ni–Ta	−16	0.50	[57]	2000
1996	Ni–Zr	−45	0.35	[58]	2000
1997	Fe–Nb–V			[59,62]	1910
1998	Fe–Ni–Cr			[60,61]	1900
1998	Ni–Ti	−39	0.40	[62]	1800
1998	Ti–Zr	−6	0.50	[62]	2300
1998	Fe–Zr–Ti			[62]	2152
1998	Fe–Ni–Ti			[62]	1916
1998	Fe–Ni–Zr			[62]	1892

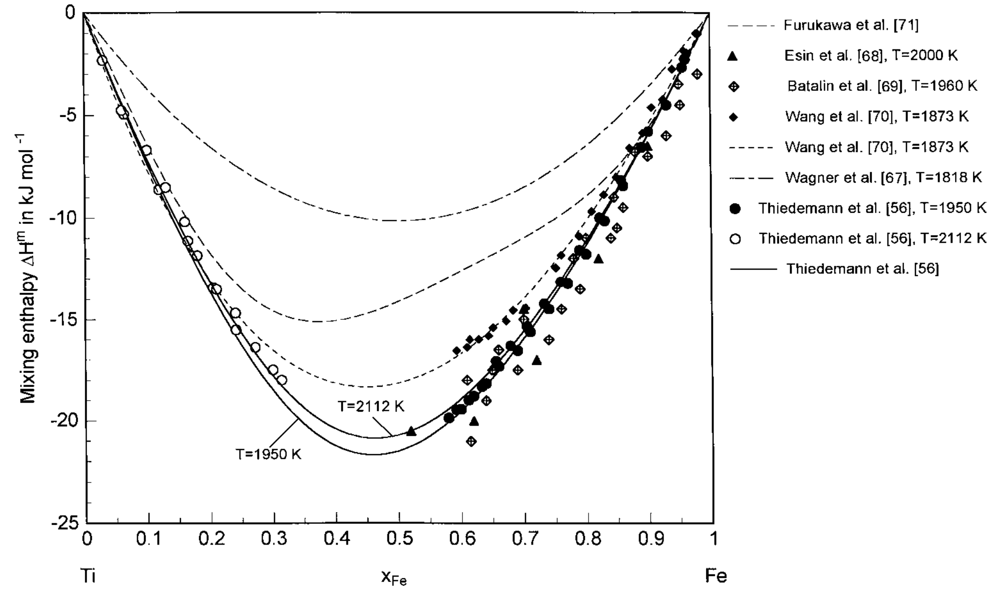

Fig. 9. Mixing enthalpy of liquid iron–titanium alloys.

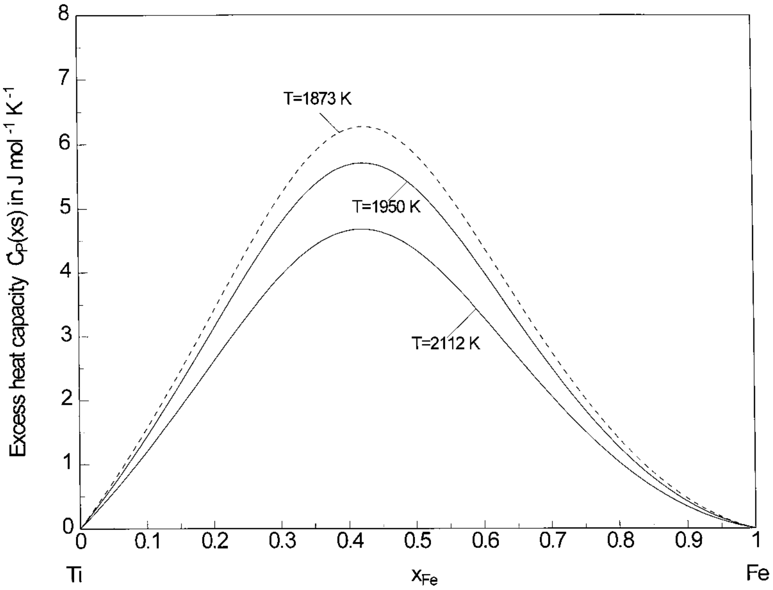

Fig. 10. Excess heat capacity of liquid iron–titanium alloys [56].

molar mixing enthalpy after the *j*th merger:

$$\Delta \overset{m}{\underset{j}{H}} = \Bigg(n_{\mathrm{A}} + \sum_{i=1}^{i=j} n_{\mathrm{B},i} \Bigg)^{-1} \times \Bigg\{ - \sum_{i=1}^{i=J} \big[n_{\mathrm{B},i} [H_{\mathrm{B}}(T_{\mathrm{g}}) - H_{\mathrm{B}}(T_{\mathrm{R}})] + \Bigg(n_{\mathrm{A}} + \sum_{m=1}^{m=i} n_{\mathrm{B},m} \Bigg) (C_{p_i^{\mathrm{AB}}}(\mathrm{id}) \Delta T_{\mathrm{L},i}) + \Bigg(n_{\mathrm{A}} + \sum_{m=1}^{m=i} n_{\mathrm{B},m} \Bigg) (C_{p_i^{\mathrm{AB}}}(\mathrm{xs}) \Delta T_{\mathrm{L},i}) \Bigg) \Bigg\}. \quad (3)$$

A detailed description of the method is given in [49].

4.2. Results

The technique can be checked by adding solid samples of the same material as the liquid solvent. With respect to the enthalpies of the added sample up to experiment temperature no mixing enthalpy should be observed. Table 3 shows the binary and ternary systems measured by us in chronological series. In order to give a qualitative picture for the binding energies the binary systems are characterized by their most exothermic value at the corresponding concentration.

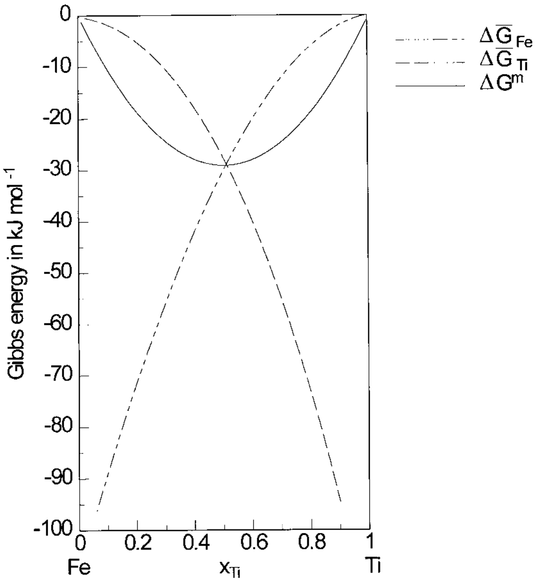

Fig. 11. Calculated integral free enthalpy of mixing and partial free enthalpies of mixing at 1873 K [56].

At the beginning of the decade we performed our measurements at different temperatures; the analysis of the experimental values should be supported in this case by a solution model. From our experience

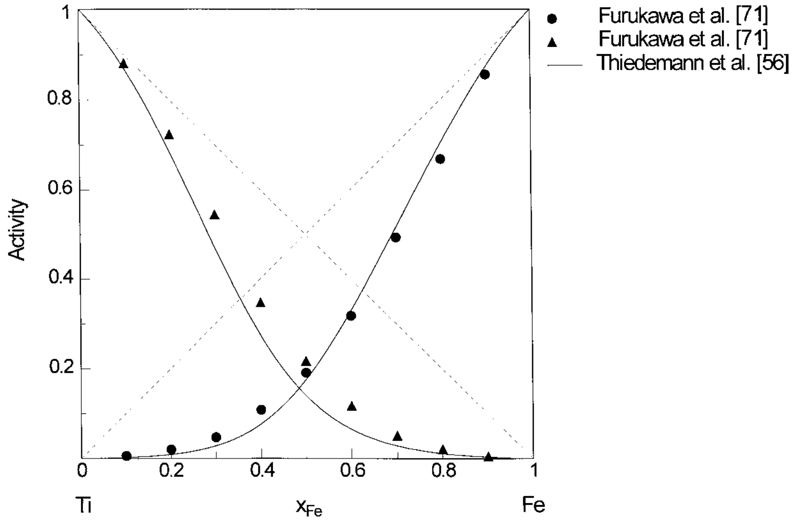

Fig. 12. Activities of iron and titanium in liquid Fe–Ti alloys at 1873 K.

[63–65] we prefer the regular association model [66]. With the mathematical description of the temperature dependence of the mixing enthalpies all mixing properties of the solution are available. Critical comparison with literature data is possible in each case. This shall be demonstrated for the system iron–titanium which is important for steel technology, hydrogen storage and metallic glass-formation.

Until 1995 discrepances existed in the mixing enthalpy of this system (Fig. 9) [67–70]. By our measurements [56] in nearly the entire concentration range at $T = 1950$ and 2112 K and by application of the association model, equations could be derived for the temperature dependence of the mixing enthalpy ΔH^{M} and entropy ΔS^{M}. The derivation of ΔH^{M} as a function of temperature provides the excess heat capacities (Fig. 10). The maxima of the curves indicated the concentrations with highest interaction energy in the melt. By means of the Gibbs–Helmholtz equation, $\Delta G^{\mathrm{M}} = \Delta H^{\mathrm{M}} - T\Delta S^{\mathrm{M}}$, we come to the free enthalpy and its partial derivatives (Fig. 11) and the component activities (Fig. 12) follow according to $\Delta G_i^{\mathrm{M}} = RT \ln a_i$. At that point we can compare our calculated results with activity measurements by Knudson cell combined with mass spectrometry studied 20 years before [71]. The result is satisfactory.

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