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Enthalpies and heat capacities of liquid Fe–Cr–Ni alloys with the focus on pure liquid chromium

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Abstract

Investigations on measuring the activities and the mixing enthalpies of the Fe–Ni, Ni–Cr and Fe–Cr liquid binary systems show only weak negative deviations from ideal behaviour. Therefore, only small atomic interactions occur between the atoms in these melts. Extending this argument to the liquid Fe–Cr–Ni alloy system where, especially in the iron-rich corner, the activities are characterized only by small deviations from the Roault's law, it is predicted that the heat capacities of the ternary alloys follow ideal behaviour and can be calculated as a result of the weighted sum of the individual contributions of each pure component. The determination of the heat capacity as a derivation of enthalpy with respect to temperature, from heat-content measurements by levitation drop calorimetry for three liquid iron-rich ternary Fe–Cr–Ni alloys, thus enabled calculations of the heat capacity for pure chromium based on the literature values for iron and nickel. The heat capacities for the liquid chromium indirectly obtained using these thermodynamic correlations are: (36.56 ± 7.53), (38.91 ± 7.84), and (40.73 ± 6.71) J mol^{-1} K^{-1}. The values confirm the frequently cited standard of 39.33 J mol^{-1} K^{-1}, as proposed by Hultgren et al., but differ clearly from the estimate of 50 ± 8 J $mol^{-1}K^{-1}$ proposed by Gurvich et al. and used in the SGTE database. © 1998 Elsevier Science B.V.

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1. Introduction

In order to critically evaluate the results of related calculations on thermodynamic equilibrium, a crucial first step is establishing a common base line data set for use by all research groups. Unified adoption of a common standard for thermophysical material properties is accomplished by disseminating a comprehensive summary in the open literature to encourage use, with the prospect for future updates as the fidelity of the data is improved with time. To this end, the Scientific Group Thermodata Europe (SGTE) database was proposed by Dinsdale [1] to assure traceability in the assessment of predictions of phase equilibrium for multicomponent systems based on interconsistency between the data sets for pure elements worldwide.

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The SGTE database, in turn, is currently accessed by the computational program Thermo-Calc (Royal Institute of Technology, Stockholm, Sweden) which has been successfully used to calculate the ternary diagram and phase equilibrium in the commercially important Fe–Cr–Ni system [2]. Due to the reactive nature of this system, containerless processing using an electromagnetic levitation system has been employed to investigate rapid solidification of undercooled liquid melts. From a theoretical standpoint, experimental studies on phase selection [3,4] and metastable phase growth [5,6] require accurate thermophysical properties to access the implications of solidification data in relation to model predictions. Comparison of the results from different research groups has been complicated due to differences in thermodynamic data sets. These results are of significant industrial interest for the application of product microstructural control in steel–alloy strip casting [7].

Of particular interest in the Fe–Cr–Ni system is the behaviour of chromium, which due to its high vapor pressure and reactive nature, has been difficult to characterize in the molten state. Conventional calorimeter-measurement techniques suffer from significant contamination by the crucible and, to date, only one experimental determination of the liquid-phase heat capacity has been performed by Lin and Frohberg [8]. In this investigation, the enthalpy of the liquid phase was measured with a levitation drop calorimeter in the 2100–2395 K range and the heat capacity was obtained as the derivation of enthalpy by temperature with a result of (50.71±2.47) J mol^{-1} K^{-1}. Discussing this result with respect to the small temperature range of the investigation and the high evaporation rate for chromium, which caused problems during temperature measurement [9], this result should tend to a lower value. Moreover, a possible temperature dependence of the spectral emissivity was neglected and corrections for the heat capacity could be obtained necessarily by considering the high sensitivity of temperature derivations of materials properties on the associated true temperatures, obtained from the apparent temperature of the pyrometric measurement [10].

The conventional value cited for the heat capacity of liquid chromium of 39.33 J mol^{-1} K^{-1}, as proposed in Hultgren et al. [11] and accepted in many recent data tabulations [12–14] was derived by analogy with other monatomic metals. This value is in contrast to the estimate of (50±8) J mol^{-1} K^{-1} proposed by Gurvich et al. [15] and used in the SGTE database [16]. The value was estimated while assuming values for the heat capacity of solid chromium to be near the melting point, but there was no comparative discussion with respect to the value given by Hultgren et al. [11] in Refs. [15,16].

It is the purpose of this paper to present an experimental verification of the heat capacity of molten chromium to resolve this discrepancy. Keeping this aim in view, we have measured the enthalpy of the liquid phases of three iron-rich alloys, namely, Fe70 Cr15 Ni15, Fe70 Cr16 Ni14, and Fe70.2 Cr18.5 Ni11.3 (in wt%) to obtain their heat capacities as temperature derivatives of the enthalpies and in the next step, to focus on the individual contribution of chromium to these values.

2. Concept

The heat capacity of a ternary alloy is generally given as follows:

$$C_{\mathrm{p}}^{\mathrm{ABC}} = C_{\mathrm{p}}^{\mathrm{ABC}}(\mathrm{id}) + C_{\mathrm{p}}^{\mathrm{ABC}}(\mathrm{xs}) \qquad (1)$$

with the Neumann–Kopp rule for the ideal heat capacity

$$C_{\mathrm{p}}^{\mathrm{ABC}}(\mathrm{id}) = x_{\mathrm{A}}C_{\mathrm{p}}^{\mathrm{A}} + x_{\mathrm{B}}C_{\mathrm{p}}^{\mathrm{B}} + x_{\mathrm{C}}C_{\mathrm{p}}^{\mathrm{C}} \qquad (2)$$

where C^{A}, C^{B}, and C^{C} are the heat capacities of the pure metals and x_{A}, x_{B}, and x_{C} are their mole fractions. The excess heat capacity, $C_{\mathrm{p}}^{\mathrm{ABC}}(\mathrm{xs})$ depends on concentration, x, and temperature, T, and is generally given

$$C_{\mathrm{p}}^{\mathrm{xs}}(x,T) = \frac{\partial \Delta H^{\mathrm{m}}(x,T)}{\partial T} \qquad (3)$$

where $\Delta H^{\mathrm{m}}(x,T)$ is the mixing enthalpy of the melt. It can be seen from Eq. (3) that no excess part has to be considered if the mixing enthalpy is temperature independent. This is valid for ideal solutions where $\Delta H^{\mathrm{m}}=0$, and also for regular [17] and subregular solutions [18]. More detailed information on different types of liquid solutions and their representation by thermodynamic modelling are given in Ref. [19].

Looking into the literature for the three liquid binary systems Fe–Cr, Ni–Cr, Fe–Ni and the liquid ternary Fe–Ni–Cr one finds that the Fe–Cr system described in Refs. [20,21] is a nearly-ideal solution with a slight negative deviation as a result of mass-spectrometric determination of activities at 1873 K. This behaviour was confirmed in Ref. [22] in the 1976–2156 K range. The Ni–Cr system was also characterized as nearly ideal by activity determination from electromotive force measurements performed in Ref. [23] at 1873 K. Although, the mixing enthalpy of liquid Fe–Ni alloys shows a negative deviation from ideal behaviour [24] but the system was also described as nearly ideal by investigations on the activities [25] and thermodynamic modelling [26]. Finally, the activities in liquid ternary Fe–Cr–Ni alloys have been intensively investigated (see Refs. [21,27,28]), wherein the authors arrived at the corresponding result that the system shows only a slight negative deviation from ideal behaviour with negligible interatomic interactions for iron-rich alloys (>60% Fe), but with stronger interactions in the nickel-rich corner as proved by the stronger negative deviations of the activities of the three components.

Summarizing the results of our previous work [29] on the mixing enthalpies of the named systems we found Fe–Cr alloys to be ideal, Ni–Cr alloys are regular with a symmetrical slight exothermic behaviour and a maximum of -4 kJ mol^{-1}, and Fe–Ni alloys are characterized by an asymmetrical exothermic behaviour of ca. -5 kJ mol^{-1} at $x_{\mathrm{Ni}}=0.6$, which can be attributed to stronger atomic interactions occurring with increasing nickel concentration [30]. Fig. 1 shows the isoenthalpy diagram of the liquid ternary Fe–Cr–Ni system. The diagram was calculated based on the results for the binaries using the 'thermodynamic adapted power series' formalism by Tomiska et al. [31–33]. It can be seen from Fig. 1 that in accordance with the activity investigations in Refs. [21,27,28] iron- and chromium-rich alloys are also characterized by an almost ideal behaviour whereas stronger interactions of up to -5 kJ mol^{-1} occur in the nickel-rich corner.

Taking the appropriate step from the thermodynamic situation presented above Eq. (2) is valid for the iron-rich corner of the liquid ternary system and can be used to calculate the heat capacity of these alloys. Conversely, measuring the heat capacity of an alloy yields the possibility to compare the experimental result with respect to Eq. (2) and, therefore, with respect to the individual contributions of each component.

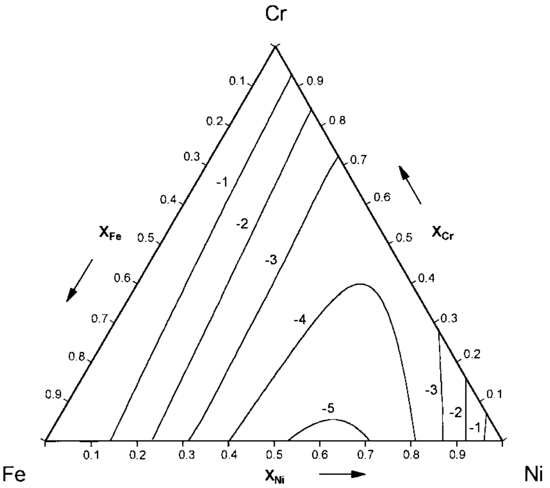

Fig. 1. Mixing enthalpy of liquid Fe–Cr–Ni alloys in kJ mol^{-1}.

3. Levitation drop calorimetric enthalpy measurements

The purity specification of the material used was 99.9%. Analysis of the oxygen content yields a value of ca. 150 ppm. The samples were processed under 95% He with 5% H_2 and the detected oxygen content after levitation decreased to <15 ppm. The samples were overheated 100 K above the melting point, T_{m}, and, subsequently, adjusted to a stable plateau 30 K above T_{m}. From that plateau, the samples were heated or cooled to the experimental temperature. The average time to reach thermal equilibrium was 2 min. Under these conditions, the maximum undercooling to be reached was in the 120–150 K range. A detailed description of the levitation drop calorimeter and the corresponding data evaluation has appeared in Ref. [34].

Temperature measurement was performed with an infrared pyrometer which was calibrated at the melting points of the alloys. Due to the relative small temperature range of the measurements of 200–250 K the uncertainty of the temperatures measured was assumed to be ±10 K. Evaporation losses have been tracked by measuring the sample mass change over the experiment duration. Microprobe investigations on different samples showed no significant change in the concentration of the alloys.

The specific error, $\delta\Delta h$(FeCrNi)=30 J, of the drop calorimeter was conservatively calculated for Fe70 Cr15 Ni15 samples with 8 mm diameter and a reference experimental temperature of 1803 K. As the result of the complete analysis we have obtained an absolute error for the molar enthalpies of $\delta(\Delta H)_{\mathrm{abs}}$=896 J mol^{-1} and for the heat capacity an absolute error of $\delta(C_{\mathrm{p}})_{\mathrm{abs}}$= 10.80 J mol^{-1} K^{-1}. It can be seen from the general equation for $\delta(C_{\mathrm{p}})_{\mathrm{abs}}$

$$\delta(C_{\mathrm{p}})_{\mathrm{abs}} = \frac{1}{\Delta T}\delta\Delta(\Delta H) + \frac{\Delta(\Delta H)}{\Delta T^2}\delta\Delta T \qquad (4)$$

that the major contribution to this error is attributed to the small temperature range, ΔT, of the investigations. On the other hand, the extension of the measurements to higher temperatures results in a deleterious increase in chromium losses caused by evaporation. Moreover, Eq. (4) neglects the number of points measured. Considering the absolute error for the molar enthalpies and the heat capacity, we have investigated three alloys with only a small difference in composition to confirm the reproducibility of the results and to underline their interpretation. A detailed discussion on the accuracy limits is given in the Section 4.

4. Results and discussion

The enthalpy measurements have been performed in the following temperature ranges: Alloy 70/15/15 between 1607 and 1857 K, alloy 70/16/14 between 1672 and 1875 K, and alloy 70.2/18.5/11.3 between 1648 and 1857 K. The experimental results on the molar enthalpy, $H_{\mathrm{T}}-H_{298}$, are given in Table 1. The temperature dependent enthalpy can be formally described by a linear least-squares fit:

$$\Delta H_i(T,\mathrm{liq}) = \Delta H(T_{\mathrm{m}},\mathrm{liq}) + C_{\mathrm{p_{liq}}}(T-T_{\mathrm{m}}) \qquad (5)$$

with the melting temperature T_{m}, the enthalpy of the liquid alloy at the melting point $\Delta H(T_{\mathrm{m}}$,liq), and the heat capacity of the liquid state $C_{\mathrm{p_{liq}}}$. The fit to the data yields:

$$\text{Alloy } 70/15/15: \quad \Delta H = 62849 + 44.16(T - 1736) \qquad (6)$$

$$\text{Alloy } 70/16/14: \quad \Delta H = 62602 + 43.46(T - 1731) \qquad (7)$$

$$\text{Alloy } 70.2/18.5/11.3: \quad \Delta H = 62570 + 43.86(T - 1728) \qquad (8)$$

Fig. 2 exemplarily presents the values for the 70/15/15 alloy, wherein the first calculated accuracy limit of $\delta(\Delta H)_{\mathrm{abs}}$=896 J mol^{-1} and the calculated function

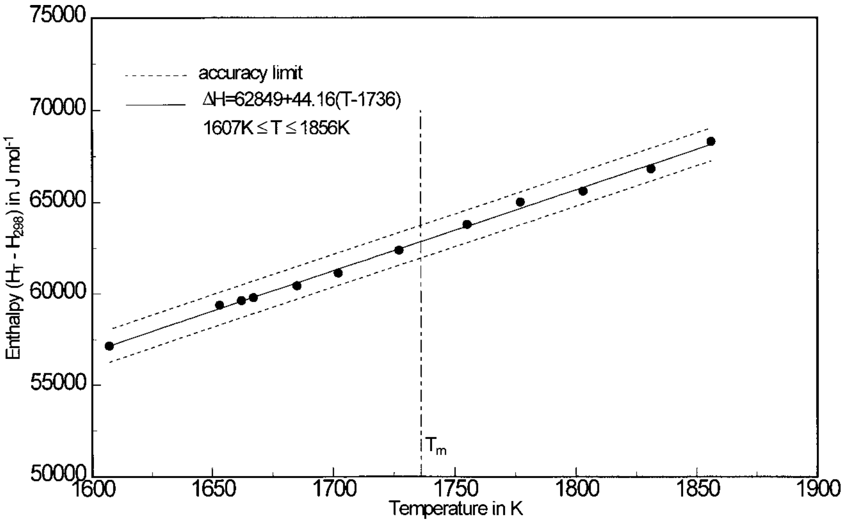

Fig. 2. Temperature dependence of the enthalpy of liquid Fe70 Cr15 Ni15 alloy.

Table 1
Molar enthalpies of the liquid Fe–Cr–Ni alloys investigated. Reference state is the solid phase at 298 K

Alloy, T_m	Temperature in K	$H_T - H_{298}$, exp. in J mol^{-1}	$H_T - H_{298}$, calc. in J mol^{-1}	D_H in %
FeCrNi 70/15/15, 1736 K	1607	57150	57152	−0.01
	1653	59377	59184	0.33
	1662	59627	59581	0.08
	1667	59797	59802	−0.01
	1685	60434	60597	−0.27
	1702	61135	61348	−0.35
	1727	62381	62452	−0.11
	1755	63794	63688	0.17
	1777	65013	64660	0.55
	1803	65609	65808	−0.30
	1831	66829	67044	−0.32
	1856	68314	68148	0.24
FeCrNi 70/16/14, 1731 K	1672	60404	60039	−0.60
	1698	60903	61169	0.44
	1707	61174	61560	0.63
	1725	62514	62342	−0.27
	1750	63503	63429	−0.12
	1770	64487	64298	−0.29
	1785	64765	64950	0.29
	1793	65362	65298	−0.10
	1811	66109	66080	−0.04
	1826	66936	66819	−0.18
	1846	67336	67601	0.39
	1854	67750	67949	0.29
	1875	69163	68861	−0.44
FeCrNi 70.2/18.5/11.3, 1728 K	1648	58929	59061	−0.22
	1670	60057	60026	0.05
	1701	61464	61386	0.13
	1723	61863	62351	−0.78
	1732	62950	62745	0.33
	1748	63767	63447	0.50
	1751	63208	63579	−0.58
	1771	64931	64456	0.74
	1792	65568	65377	0.29
	1815	66542	66386	0.24
	1837	67290	67351	−0.09
	1857	67870	68228	−0.52

(Eq. (6)) are also given. It can be seen, that in reality the scattering of the experimental values is much better as indicated by our calculated error. Table 1 shows the calculated values, according to Eqs. (6)–(8), and the deviation, D_H, of the experimental results from the calculated functions. Making the point that the physical consequences from the ideal behaviour of the alloys are a constant heat capacity and the linearity of the enthalpy values, these deviations can be used to calculate the absolute error of the molar enthalpies as the mean of the deviations between experimental value and calculated linear function. The following results have been obtained: 70/15/15, $\delta(\Delta H)_{abs}=144$ J mol^{-1}; 70/16/14, $\delta(\Delta H)_{abs}=201$ J mol^{-1}; 70.2/18.5/11.3, $\delta(\Delta H)_{abs}=239$ J mol^{-1}.

The use of Eq. (4) now yields: 70/15/15, $\delta(C_p)_{abs}=4.71$ J mol^{-1} K^{-1}; 70/16/14, $\delta(C_p)_{abs}=5.53$ J mol^{-1} K^{-1}; 70.2/18.5/11.3, $\delta(C_p)_{abs}=5.84$ J mol^{-1} K^{-1}.

In order to calculate the heat capacity of liquid chromium, according to Eq. (2), it is necessary to consider the reported heat capacities of iron and nickel. The generally accepted value for iron of 46.024 J mol^{-1} K^{-1} was proposed by Orr and Chipman ([35], and cited in Refs. [11,12]) considering three individual investigations: Heat capacity measurements from Vollmer et al. [36] with an accuracy limit of ±3%, enthalpy measurements from Ferrier and Olette [37], and Morris et al. [38]. Orr and Chipman did not give an accuracy limit for their proposal, only the advice that all data are adequately represented by the adopted value. This value was confirmed by Hixson et al. [39] with 45.44 J mol^{-1} K^{-1} and Beutl et al. [40] with 46.07 J mol^{-1} K^{-1} and an accuracy limit of ±7%. Both investigations used pulse-heating technique experiments. Summarizing the situation found in the literature, we assume the uncertainty of the 46.024 value with ±1 J mol^{-1} K^{-1}.

Looking at nickel one finds a similar situation. The chosen value of 38.911 J mol^{-1} K^{-1} was adopted by JANAF [12], based on the study of Vollmer et al. [36], wherein the original value of 39 J mol^{-1} K^{-1} was given with an accuracy limit of ±3%. The earliest investigation on the heat capacity of liquid nickel was performed by Umino [41] with a result of 38.49 J mol^{-1} K^{-1} and an error of ±1.5–2%. In agreement with these results a value of 39.3 J mol^{-1} K^{-1} was published by Margrave [42]. In contrast a higher value of 43.095 J mol^{-1} K^{-1} was obtained by Geoffray et al. ([43], cited in Ref. [11]). It can be stated that the low values are generally accepted today. Due to the good agreement of the investigations and with respect to the given accuracy limits we assume the uncertainty of the 38.911 value with ±1 J mol^{-1} K^{-1}.

Table 2 summarizes the results from calculations for the heat capacity of the three alloys according to Eq. (2) with the heat capacities for the liquid iron and nickel and the two citations for the heat capacity of liquid chromium, 39.33 [11] and 50.00 J mol^{-1} K^{-1}. Line 3 represents our experimental result according to Eqs. (6)–(8). It can be seen that the values obtained are in a very good agreement with the calculations in line 1 where the chromium heat capacity of 39.33 J mol^{-1} K^{-1} was used.

Table 3 presents the calculation, according to Eq. (2), for the heat capacity of liquid chromium using the experimental results from line 3 in Table 2 and the two chosen values for the heat capacity of liquid iron and nickel. The heat capacities obtained confirm the estimate of 39.33 J mol^{-1} K^{-1} by Hultgren et al. [11], but they differ clearly from the estimate of 50 J mol^{-1} K^{-1} proposed by Gurvich et al. [15] and used in the SGTE database [16] and from the experimental value of 50.71 J mol^{-1} K^{-1} given by Lin et al. [8] as well.

Table 3
Derived heat capacities of liquid chromium. All values are given in J mol^{-1} K^{-1}

70/15/15	70/16/14	70.2/18.5/11.3
40.73±6.71	36.56±7.53	38.91±7.84

5. Conclusions

The thermodynamic relations between ideal mixing properties and heat capacities enable calculations of the heat capacity of Fe–Cr–Ni alloys based on the weighted sum of the individual contributions of pure components. Conversely, determining the heat capacity of an alloy yields the possibility of comparing the experimental result with respect to the heat capacities of the pure components. The estimated literature value for the heat capacity of liquid chromium of 39.33 J mol^{-1} K^{-1} was thus confirmed by enthalpy measurements on three iron-rich Fe–Cr–Ni alloys.

Table 2
Calculated heat capacities, C_p, of the three FeCrNi alloys according to Eq. (2), and experimental result according to Eqs. (6)–(8). All values are given in J mol^{-1} K^{-1}

Line	Condition	70/15/15	70/16/14	70.2/18.5/11.3
1	$C_{p_{Cr}} = 39.33$	43.94	43.94	43.95
2	$C_{p_{Cr}} = 50.00$	45.65	45.76	46.05
3	Cp according to Eqs. (6)–(8)	44.16±4.71	43.46±5.53	43.86±5.84

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