Normal Spectral Emissivity at 684.5 nm of the Liquid Binary System Fe–Ni

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Received September 12, 2004; accepted December 3, 2004 Published online November 14, 2005 © Springer-Verlag 2005

Summary. Normal spectral emissivity at 684.5 nm of the liquid binary system Fe–Ni was measured by means of a fast ohmic pulse heating technique combined with fast ellipsometry. The experimental values observed of all liquid alloys are dependent on composition and temperature in the entire range of mixing. Spectral emissivity was measured for pure iron, Fe90Ni10, Fe80Ni20, Fe64Ni36, Fe50Ni50, Fe40Ni60, Fe30Ni70, Fe20Ni80, and pure nickel. Emissivity data as a function of temperature (at melting and in the liquid phase up to 2300 K) and as a function of the mixing ratio are presented and compared to the few existing literature-values.

Keywords. Calorimetry; Phase transition; Thermodynamics; Liquid metal; Pulse heating.

Introduction

Metal working industry and especially the foundry industry need accurate thermophysical data of liquid alloys as input parameters for numerical simulations to optimize casting processes. This paper presents a systematic investigation of the dependence of emissivity of binary alloys on the concentration of the constituent elements. The obtained results can improve the accuracy of relevant optical temperature measurements.

In previous work [1], the behavior of electrical resistivity of the binary Fe–Ni system in the solid and in the liquid phase was determined by using a fast pulse heating technique. The aim of those investigations was the measurement of the dependence of the electrical resistivity on mass fraction of the alloyed elements. Investigations on the dependence of normal spectral emissivity on the concentration of the alloy are performed within this paper.

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Results and Discussion

Figure 1 shows measured values of 11 experiments of normal spectral emissivity at a wavelength of 684.5 nm for the alloy Fe30Ni70 as a function of temperature. In contrast to the liquid phase, the values obtained in the solid phase are strongly

Fig. 1. Normal spectral emissivity at 684.5 nm versus temperature for Fe30–Ni70-alloy; \blacksquare – measured datapoints; solid lines represent linear fit of the measured data in the liquid; vertical dotted line: liquidus temperature

Fig. 2. Normal spectral emissivity at 684.5 nm versus temperature for Fe–Ni-alloys; solid lines represent least square fits of the measured data; \circ - liquidus temperature

dependent on treatment of the wire before the experiment. At liquidus temperature, which is indicated by a vertical dotted line, emissivity is extrapolated to a value of 0.24 ± 0.03 .

Data for all other alloys and pure materials were processed in the same way and are presented in Fig. 2. The corresponding least-squares fits are listed in Table 1. The emissivities at the liquidus temperature of pure iron and nickel are higher than all values of the measured alloys. The lowest value of normal spectral emissivity is obtained for Fe30Ni70 with 0.24. All trend lines, except the one of alloy Fe90Ni10,

Table 1. Normal spectral emissivity at a wavelength of 684.5 nm and liquidus temperature T_l [2] of the Fe–Ni-alloys

| No. | $Mass\%$ -Fe $\%$ | Emissivity at liquidus $\varepsilon(T_l)$ | T_I/K | Emissivity in the liquid phase (range T_l to 2300 K) |
|----------------|----------------------|--|---------|---|
| $\overline{1}$ | Ni 99.9 | 0.365 | 1726 | $\varepsilon = 0.345 + 1.145 \cdot 10^{-5} \cdot T_l$ |
| 2 | 18.6 | 0.288 | 1715 | $\varepsilon = 0.156 + 7.701 \cdot 10^{-5} \cdot T_I$ |
| 3 | 28.2 | 0.238 | 1712 | $\varepsilon = 0.143 + 5.419 \cdot 10^{-5} \cdot T_1$ |
| $\overline{4}$ | 41.7 | 0.280 | 1710 | $\varepsilon = 0.227 + 3.082 \cdot 10^{-5} \cdot T_I$ |
| -5 | 48.5 | 0.295 | 1715 | $\varepsilon = 0.212 + 4.860 \cdot 10^{-5} \cdot T_I$ |
| 6 | 62.7 | 0.295 | 1727 | $\varepsilon = 0.156 + 8.031 \cdot 10^{-5} \cdot T_l$ |
| 7 | 79.9 | 0.316 | 1755 | $\varepsilon = 0.253 + 3.594 \cdot 10^{-5} \cdot T_I$ |
| 8 | 89.0 | 0.344 | 1777 | $\varepsilon = 0.359 - 8.457 \cdot 10^{-6} \cdot T_l$ |
| 9 | Fe 99.5 | 0.362 | 1811 | $\varepsilon = 0.307 + 3.037 \cdot 10^{-5} \cdot T_l$ |

Fig. 3. Literature values for emissivity of iron and nickel versus temperature; O - measurements from this work for nickel; \diamondsuit – values from *Krishnan et al.* [3]; \star – value from *Kaschnitz et al.* [4]; \Box – values from *Lange* and *Schenck* [5]; \triangle – values from *Krishnan* and *Nordine* [6]; \bigtriangledown , \Box – values from Bonnell et al. [7]. \bullet – data from this work for iron; \star – value from *Kaschnitz et al.* [8]

Fig. 4. Composition dependence of normal spectral emissivity at 684.5 of iron–nickel alloys at liquidus temperature; \blacksquare - measured data from this work; \odot - literature data from *Ratanapupech* et al. [9]

show an increase of emissivity with temperature. No explanation was found for this behavior by now.

Figure 3 presents the obtained data of pure iron and nickel which are in good agreement with literature values within the stated uncertainties. It should be noted, that the literature values shown in this graph have been corrected from the individual wavelength published to the wavelength 684.5 nm using data from Refs. [4, 8].

To illustrate the dependence of normal spectral emissivity at the liquidus temperature from the mixing ratio of iron and nickel, data of this work and literature values of Ratanapupech and Bautista [9] are plotted in Fig. 4. It can be seen that the agreement between the present results and this published data is excellent. The behavior of the iron–nickel-alloy shows clearly that emissivity can not be described in form of a linear dependence between the values of the pure metals which was often assumed for optical temperature measurement.

Uncertainties

An estimation of uncertainties is evaluated by determining the signal-to-noise ratio of different individual measurements and by analyzing the reproducibility of different measurements, which are the main contributions to the uncertainty budget. For investigations of materials with an emissivity value at the melting point of $\epsilon > 0.25$ the signal-to-noise ratio of a single experiment does not exceed $\pm 6\%$. With reference to GUM (Guide to the Expression of Uncertainty in Measurement) [10], the uncertainties are expanded with a coverage factor of $k = 2$. By adopting this concept for the present uncertainty estimation, one

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Conclusions

Normal spectral emissivity at a wavelength of 684.5 nm of the Fe–Ni alloy system in the liquid phase was measured using a fast pulse heating technique and fast ellipsometry. The results verify previous measurements at liquidus temperature of Ratanapupech and Bautista [9] and show the non-linearity of normal spectral emissivity between the two pure metals iron and nickel. Further investigations on emissivity as a function of composition for other binary alloy systems are necessary to draw a clearer picture of the general behaviour of binary alloys.

Experimental

Rod-shaped bars were sand-cast by alloying high purity iron and nickel, supplied from Fa. Matthey Johnson, UK, after a careful desoxidation of the melt. From the center part of the castings, cylindrical specimens of 9 mm in diameter were machined and subsequently drawn to wires of 0.5 mm in diameter and then cut to pieces of 50 mm length. The composition of the Fe–Ni wire specimens has been analyzed by means of energy dispersive x-ray spectroscopy (EDAX), the values are shown in Table 1. The alloy Fe20Ni80 was supplied by Ref. [11].

Fast pulse-heating techniques allow the measurement of thermophysical properties of metals and alloys at temperatures that are inaccessible to most other techniques. This setup involves high heating rates up to 10^8 K/s which result in short measurement times (μ s). This minimizes chemical interactions between the sample and its environment during the experiment. We perform pulse-heating experiments on elements and alloys, where wire-shaped samples of 0.5 mm diameter and of 50 mm length are resistively volume-heated as part of a fast capacitor discharge circuit. Time-resolved measurements with sub- μ s resolution of current through the specimen are performed with a Pearson–probe, the voltage drop across the specimen is measured with knife-edge contacts and ohmic voltage dividers, the radiance temperature of the sample with a pyrometer. These measurements allow the determination of heat of fusion, as well as heat capacity and electrical resistivity as a function of temperature in the melting region and in the liquid phase. See Ref. [12] for more details on the experiment and on the data reduction. Within this paper emissivity measurements at a wavelength of 684.5 nm are reported. The emissivity measurements are done by combining a laser polarimetry technique $(\mu s - \text{Division of})$ Amplitude Photopolarimeter $[\mu s-DOAP]$) with our established method for performing high speed measurements on liquid metal samples at high temperatures during microsecond pulse-heating experiments. The μ s–DOAP detects the change of the polarization of laser light reflected from the liquid sample surface during pulse heating from which the normal spectral emissivity is computed. For more details of the DOAP see Ref. [13].

The normal spectral emissivity was measured at a wavelength of 684.5 nm from melting up to 2300 K in the liquid phase on wires from pure iron, Fe90Ni10, Fe80Ni20, Fe64Ni36, Fe50Ni50, Fe40Ni60, Fe30Ni70, Fe20Ni80, and pure nickel (see Table 1 for exact mass fractions of iron, other constituent nickel; determined by means of EDAX [14] and chemical analysis [11]; all other impurities $(P, S, Si, C, etc.)$ are less than 0.6%).

Acknowledgements

This work was supported by the Fonds zur Förderung der wissenschaftlichen Forschung (FWF), Vienna, Austria under contract No. P15055.

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Verleger: Springer-Verlag GmbH, Sachsenplatz 4–6, 1201 Wien, Austria. – Herausgeber: Österreichische Akademie der Wissenschaften, Dr.-Ignaz-Seipel-Platz 2, 1010 Wien, und Gesellschaft Österreichischer Chemiker, Eschenbachgasse 9, 1010 Wien, Austria. – Redaktion: Getreidemarkt 9/163-OC, 1060 Wien, Austria. – Satz und Umbruch: Thomson Press Ltd., Chennai, India. – Offsetdruck: Manz Crossmedia, 1051 Wien, Austria. – Verlagsort: Wien. – Herstellungsort: Wien. – Printed in Austria.