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Thermophysical Properties and Normal Spectral Emittance of Iridium up to 3500 K

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An ohmic pulse-heating experiment together with radiometry and μ s-photopolarimetry is deployed at the Institute of Experimental Physics, Graz University of Technology, to obtain temperature-dependent thermophysical properties of conducting samples in the solid and molten states. This experimental setup has been used within the present work to gather data for solid and liquid iridium. Results for both thermophysical properties, as well as the normal spectral emittance obtained at a wavelength of 684.5 nm up to 3500 K are reported. The newly obtained values for iridium are presented in graphical and tabular form and compared to available literature data. The uncertainties for all reported properties are stated and it follows that, considering these expanded uncertainties, the recent data are in very good agreement with literature sources. Mutually motivated by these good results and by the scarce (if any) data available for the liquid state, the thermal conductivity and thermal diffusivity of liquid iridium are estimated by means of the Wiedemann– Franz law.

KEY WORDS: ellipsometry; iridium; normal spectral emittance; pulse-heating; thermal conductivity; thermal diffusivity; thermophysical properties.

1. INTRODUCTION

Only a few techniques have been established to perform measurements on physical properties of metals and alloys up to temperatures in the liquid state. One of these techniques is the so-called ohmic pulse-heating, which has been successfully used by different institutions around the world to obtain temperature-dependent thermophysical property data. At

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the Institute of Experimental Physics, Graz University of Technology, Austria, a constantly improving pulse-heating setup has been used for more than two decades to determine thermophysical properties of metals and alloys in the solid and liquid states.

Furthermore, a division-of-amplitude photopolarimeter has been added to the setup with several objectives: (i) to improve the accuracy of the optical (pyrometric) temperature measurement, (ii) to fulfill the growing need of industry for optical material properties, and (iii) for scientific reasons, as, in the majority of cases, only scarce emittance data can be found for molten materials in the literature.

The before-mentioned techniques, often combined with additional DSC experiments, have helped to systematically study a wide variety of pure, commercially available metals throughout the periodic system, in order to create a database on material properties. Due to various reasons, such as availability, purity, or price, some metals, such as iridium, have been excluded from investigations for a long time.

Iridium was first discovered by Smithson Tennant (England) in 1803. Iridium is named after the Greek word "Iris," originally in Greek mythology a "messenger of the gods" personified as the rainbow, because of the colorful nature of iridium compounds, as its salts are highly colored. Iridium is white, similar to platinum, but with a slight yellowish cast. It is very hard and brittle, making it very hard to machine, form, or work. It is the most corrosion-resistant metal known. An alloy of 90% platinum and 10% iridium was used in 1889 to construct the standard meter bar and kilogram mass, maintained by the International Bureau of Weights and Measures near Paris. The meter bar was replaced by the definition of the fundamental unit of length in 1960, but the kilogram prototype is still the international standard of mass.

Iridium is not attacked by any of the acids or by aqua regia, but is attacked by molten salts, such as NaCl and NaCN. Iridium is rare on earth (much rarer than gold and platinum), but relatively common in meteorites.

The following applications of iridium are reported: it is used to make crucibles and high-temperature equipment, thermocouples such as Pt-Pt/Ir, electrical contacts, and Pt/Ir spark plugs, and as a hardening agent for platinum. Furthermore, iridium forms an alloy with osmium that is used for tipping pens and compass bearings.

The measured density of iridium is only slightly lower than that of osmium, which is therefore often listed as the heaviest element known [1]. However, calculations of density from the space lattice produced more reliable data for these elements than actual measurements and gave a density of 22,560 kg·m−³ for iridium vs. 22,590 kg·m−³ for osmium [2], hence,

making osmium definitely the densest metal known [3]. Albeit other densities are more commonly reported in the literature, the cited density value of 22,560 kg·m−³ for iridium at 20◦C was carefully studied and will be used within this work for calculations, if applicable.

We have chosen iridium for our measurements because of general scientific interest, as data for the liquid phase are sparse, and because of a systematic investigation of the platinum group metals that we have undertaken within a recent project.

2. EXPERIMENT AND DATA REDUCTION

Determination of thermophysical properties of liquid metals, as performed by the "Subsecond Thermophysics Group" at the Graz University of Technology with a long tradition, is still relevant and of paramount importance for the metal working-industry, as well as for scientific and academic interests. Accurate data at the melting transition and in the liquid state are often rare, but essential as input data for many different kinds of computer simulations.

Based on a pulse-heating system, thermophysical properties of conducting materials are accessible from the solid state up to the end of the stable liquid state. Therefore, wire-shaped annealed iridium samples with a diameter of 0.5 mm, a length of 60 mm, and a purity of 99.9% were purchased from Alfa Aesar (reference 19587) and subsequently resistively pulse-heated as part of a capacitor-driven discharge circuit in a nitrogen atmosphere at ambient pressure.

The measured quantities for current, voltage drop, surface radiance, radial expansion, and normal spectral emittance, $\bar{3}$ allow the determination of thermophysical properties such as the specific enthalpy, isobaric heat capacity, electrical resistivity (assuming initial geometry, as well as compensated for thermal expansion), thermal conductivity, and thermal diffusivity of the sample as functions of true temperature, as well as its heat of fusion. The accessible range of measurement extends from room temperature up to superheated liquid states. Experimental details have already been described extensively elsewhere, e.g., by Gallob et al. [4] and Wilthan et al. [5].

To enable accurate and unambiguous temperature determination over such a vast range, pyrometric temperature detection based on Planck's law of blackbody radiation is used. In addition, the normal spectral emittance is measured with an ellipsometric device $(\mu s\text{-DOAP})$, greatly

³ IUPAC recommends the term 'emittance' instead of the widely used 'emissivity' because the latter term is often reserved for radiant power divided by volume and by solid angle.

Fig. 1. Normal spectral emittance at 684.5 nm (average of nine individual measurements) of iridium vs. radiance temperature at 650 nm. Solid line with circles: measurement data from the present study, solid line: linear least-squares fit to the liquid-state data, open up-triangle: McClure et al. [10] (interpolated for 684.5 nm).

reducing uncertainties arising from the unknown emittance and its behavior throughout the temperature range of the measurements. For more details on the μ s-DOAP, see Seifter et al. [6] and Cagran et al. [7].

3. RESULTS

In Fig. 1 the normal spectral emittance of iridium at a wavelength of 684.5 nm is plotted vs. the radiance temperature T_{rad} at 650 nm and compared to literature results. At the end of the solid phase, the emittance decreases smoothly to a value in the range of about 0.506. This value in the solid phase strongly depends on the surface preparation of the sample, here with abrasive paper grade 1200 or 4000. As the surface becomes

thermal diffusivity, and ε: normal spectral emittance at 684.5 nm as a function of radiance temperature,

thermal diffusivity, and ε : normal spectral emittance at 684.5 nm as a function of radiance temperature, T_r, at 650 nm.

*T*r, at 650 nm.

smoother during melting⁴ a strong decrease in the measured emittance is observed. The melting temperature of iridium, T_m , is 2719 K as reported by Bedford et al. [8] and $T_m = (2719 \pm 6)$ K as reported by Arblaster [9], while the radiance temperature at melting at a wavelength of 656 nm is 2380 K as given by McClure et al. [10]. In the present work an emittance of 0.348 at 684.5 nm was obtained at the end of melting, yielding a radiation temperature of 2392 K at melting. The emittance value of McClure [10] of 0.315 at melting (interpolated by means of a 2nd order polynomial⁵ for 684.5 nm) is also depicted in Fig. 1. For maximum readability, Fig. 1 also shows a double x-axis with radiance temperature, *T*r, at 650 nm and (absolute) temperature, T . The latter is calculated from T_r and the measured emittance results, neglecting the small wavelength difference between the radiance temperature at 650 nm and emittance measurement at 684.5 nm. An average of nine measurements in the liquid phase gives a linear fit for the normal spectral emittance in the radiance temperature range up to 3650 K. The fit is presented in Table I. For convenience and easier data comparison, all corresponding least-squares results are summarized in Table I. For liquid iridium a slight increase of the normal spectral emittance at 684.5 nm with temperature up to 3755 K is observed, which is similar to the behavior of platinum (Wilthan et al. [5]).

Figure 2 presents the specific enthalpy H vs. true temperature T , wherein the enthalpy at room temperature was assigned as zero. All temperature-dependent results reported within the following figures (Figs. 2–4) cover a range from 1800 K to about 3600 K. Nine measurements were averaged to give linear fits for the solid and liquid states, as listed in Table I. Considering specifically the enthalpy, we acquire (coefficient b in Table I) a constant c_p value of 232 J·kg⁻¹·K⁻¹ for the end of the solid state and 233 J·kg⁻¹·K⁻¹ for the liquid state. These almost identical numbers for solid and liquid states are quite unusual, as for most other materials measured in our lab to date, the c_p values in the solid and liquid phases differ, sometimes even considerably. Hultgren et al. [11] report a value of 217 J·kg⁻¹·K⁻¹ for c_p in the liquid state. Arblaster [9] reports enthalpy measurements by Kats [12], which yield a constant c_p value of 309 J·kg⁻¹·K⁻¹ for liquid iridium up to 5000 K. The conversion

⁴ As the surface smoothens, reflectivity of the sample rises which can, in turn, be related to a decrease of emittance as follows from Kirchhoff's law for opaque materials.

⁵ Due to better data accuracy, a second-order polynomial spanning all reported wavelengths was chosen instead of a direct linear interpolation considering the two nearest wavelengths. However, comparing the two approaches it can be seen that the differences in emittance are small (ε = 0.315 by polynomial vs. ε = 0.313 by linear interpolation) and, if converted back to radiance temperatures, this interpolation difference is about four times smaller than the stated uncertainty in Ref. 10

Fig. 2. Specific enthalpy of iridium in the solid and liquid states as a function of temperature. Open circles: averaged measurement data from the present study, solid lines: linear leastsquares fits, dashed line: melting temperature (2719 K), open up-triangles: Arblaster [9], solid down-triangles: Gathers et al. [13].

factor from mole to kilogram used within this work is 5.2024534 mol·kg⁻¹, using the currently accepted relative atomic mass of 192.217 as reported by Arblaster [9]. Data of Gathers et al. [13] and recommended values from Arblaster [9] are also depicted in Fig. 2.

At melting, which is indicated by a vertical dashed line in Figs. 2–4, the specific enthalpy changes from $H_s(T_m) = 404.3 \text{ kJ} \cdot \text{kg}^{-1}$ (subscript s: solid) to $H_1(T_m) = 608.8 \text{ kJ} \cdot \text{kg}^{-1}$ (subscript l: liquid) yielding $\Delta H = 204.5 \text{ kJ} \cdot \text{kg}^{-1}$ for the latent heat of fusion. Thus, the entropy of fusion is $\Delta S = 75.2 \text{ J} \cdot \text{kg}^{-1} \text{K}^{-1}$. Gathers et al. [13] report $H_s(T_m) =$ 457 kJ·kg^{-1} and $H_1(T_m) = 633 \text{ kJ·kg}^{-1}$ yielding $\Delta H = 176 \text{ kJ·kg}^{-1}$ for the latent heat of fusion at a pressure of 0.3 GPa. Due to the enthalpy of pressurization, the enthalpy values of Gathers should be decreased by 2.5% to be comparable to values measured at ambient pressure [13]. Martynyuk and Tsapkov [14] report $H_s(T_m) = 406 \text{ kJ} \cdot \text{kg}^{-1}$ and $H_1(T_m) = 572 \text{ kJ} \cdot \text{kg}^{-1}$ yielding a value of $\Delta H = 166 \text{ kJ} \cdot \text{kg}^{-1}$ for the latent heat of fusion. Lebedev et al. [15] report $\Delta H = 200 \text{ kJ·kg}^{-1}$. As reported by Arblaster [9], enthalpy measurements by Kats [12] lead to a value of $\Delta H = (215 \pm 10^2)$

Fig. 3. Electrical resistivity assuming initial sample geometry (IG) and considering for thermal volume expansion (VE) of iridium in the solid and liquid states as a function of temperature. Open circles: averaged measurement data (IG) from the present study, solid circles: resistivity data from the present work considering volume expansion with density data from Refs. 17 and 18, open squares: resistivity data from this work (VE) using extrapolated volume expansion data from Ref. 18, solid lines: linear least-squares-fits, dashed line: melting temperature (2719 K) [8], open up-triangles: reference values at the melting transition taken from Martynyuk et al. [14], solid down-triangles: data taken from Gathers et al. [13].

59) kJ·kg⁻¹. Dinsdale [16] reports $\Delta H = 214 \text{ kJ·kg}^{-1}$. Hultgren et al. [11] report $H_s(T_m) = 418 \text{ kJ·kg}^{-1}$ and $H_1(T_m) = 553 \text{ kJ·kg}^{-1}$ yielding $\Delta H =$ $135 \mathrm{kJ·kg}^{-1}$.

Figure 3 presents the electrical resistivity as a function of temperature. At the onset of melting, indicated by a vertical dashed line, we obtain a value of $0.644 \mu\Omega$ ·m for the resistivity assuming initial sample geometry ρ_{IG} (no compensation for thermal expansion, index IG). At the end of melting our measured value is 0.847 $\mu\Omega$ ·m, yielding an increase of $\Delta \rho = 0.203 \,\mu\Omega$ ·m upon melting. For electrical resistivity that is compensated for thermal volume expansion (index VE), ρ_{VE} , we used literature values for the thermal expansion of iridium from Wimber [17] and den-

sity data from Ishikawa et al. $[18]^{6}$. The increase in diameter, due to thermal expansion, results in a shift to higher resistivity values, as the actual cross section of the wire, which affects the measured electrical resistance of the samples, is increased. For the liquid state above 3000 K, no expansion data are given in Ref. 18 and a linear extrapolation was used to estimate the thermal expansion up to 3600 K.

At the onset and at the end of melting, we obtain values of $0.698 \mu \Omega \cdot m$ and $0.980 \mu \Omega \cdot m$, respectively, for the volume-compensated electrical resistivity. Thus, an increase of $0.282 \mu \Omega \cdot m$ is observed during melting. The values from Gathers et al. [13] and Martynyuk et al. [14] are also shown in Fig. 3. Gathers et al. [13] report a value of $0.737 \mu \Omega$ ·m for the onset of melting and of $1.00 \mu \Omega$ ·m for the end of melting, yielding an increase of $0.263 \mu \Omega$ ·m during melting. Martynyuk et al. [14] report $0.659 \mu \Omega$ ·m for the onset of melting and $0.855 \mu \Omega$ ·m for the end of melting yielding an increase of $0.196 \mu \Omega$ ·m upon melting. Zinovyev [20] reports a value of $0.50 \mu \Omega$ ·m for iridium at 2000 K. The four corresponding least-squares polynomials for resistivity vs. temperature obtained within this work are summarized in Table I.

Figure 4 presents the thermal conductivity vs. temperature. To estimate the thermal conductivity via the Wiedemann–Franz law, WFL, (see Wilthan et al. [5]) we used the volume-compensated electrical resistivity values (ρ_{VE}), as reported above. At the onset of melting, we obtain a value of 94.3 W·m⁻¹·K⁻¹, and at the end of melting (beginning of the liquid phase), a value of $68.2 W·m^{-1}·K^{-1}$. At the end of the solid phase, the extrapolated value of Filippov [21] is $105 \,\mathrm{W}\cdot\mathrm{m}^{-1}\cdot\mathrm{K}^{-1}$ and the extrapolated value from Geld et al. [22] is 98 W·m⁻¹·K⁻¹. Zinovyev [20] reports a value of $103 \text{ W} \cdot \text{m}^{-1} \cdot \text{K}^{-1}$ at 2000 K . Mills et al. [23] report for the end of the solid and the beginning of the liquid phase values of 95 and 76 W·m−1·K−1, respectively. These values, originally reported by Vlasov et al. [24] are also given as a recommendation in Ref. 23 due to the lack of additional data for the liquid state. The corresponding least-squares polynomials obtained within this work for the thermal conductivity in the solid (up to a temperature 100 K below the m.p.), and in the liquid phase are once again listed in Table I.

Finally, the thermal diffusivity, *a*, can be estimated from the thermal conductivity (Wilthan et al. [5]). We refrained from showing a graph of thermal diffusivity vs. temperature, as all relevant information about *a* can

⁶ Ishikawa et al. published two different papers presenting density data for liquid iridium within a period of a couple of months [18, 19] in which the values disagree considerably. Contacting the authors directly, they informed us that the values in Ref. 18 represent their best results to date.

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*T*r: radiance temperature at 650 nm, ε normal spectral emittance

ume expansion, λ : thermal conductivity, *a*: thermal diffusivity, T_f : radiance temperature at 650 nm, *s* normal spectral emittance at 684.5 nm as a function of radiance temperature, T_f .

ume expansion, λ: thermal conductivity, *a*: thermal diffusivity,

at 684.5 nm as a function of radiance temperature,

be extracted from the linear fits, given in Table I. At the onset of melting, we obtain a value of 1.944×10^{-5} m²·s⁻¹ and at the end of melting (beginning of the liquid phase) a value of 1.500×10^{-5} m²·s⁻¹.

4. DISCUSSION

There are plenty of data on liquid iridium available in the literature, but data for liquid emissivities are very rare. The values of the normal spectral emittance ε of iridium at a wavelength of 684.5 nm at the end of melting for the liquid reported here are in reasonable agreement with the values reported by McClure et al. [10], albeit our value is somewhat higher.

For the enthalpy vs. temperature the recommended values of Arblaster [9] are in good agreement with our values at the end of the solid state, but do not match so well in the liquid. The pulse-heating values of Gathers et al. [13] are in excellent agreement with our values in the liquid, but they are higher than ours at the end of the solid phase, which explains the lower value for the heat of fusion reported by them.

A comparison of the latent heat of fusion obtained here gives a wide variation of values. We obtained a value of $\Delta H = 204.5 \,\mathrm{kJ \cdot kg^{-1}}$, Kats [12] reported $\Delta H = 215 \text{ kJ·kg}^{-1}$ as cited in Ref. 9 (deviation from our value of +5%), Dinsdale [16] reported $\Delta H = 214 \text{ kJ} \cdot \text{kg}^{-1}$ (+4.5%), Lebedev [15] reported 200 kJ·kg⁻¹ (−2.5%), Gathers [13] reported 176 kJ·kg⁻¹ (−14%), Martynyuk [14] reported 166 kJ·kg−¹ (−20%), and Hultgren et al. [11] reported 135 kJ·kg^{-1} (-34%). It is noteworthy, that while pulse-heating experiments [13, 14] delivered values quite lower than ours, all other literature values agree with ours within 5%. The quite old recommended value of Ref. 11 seems to be very low. Therefore, newer recommendations such as those from Arblaster [9] should be considered.

At the time of these measurements on iridium, our high-speed system [25] for monitoring sample expansion was out of order. Therefore, densityversus-temperature data of Wimber [17] and Ishikawa [18] were used to compensate our resistivity measurements for thermal expansion. Without this volume compensation, our resistivity values agree very well with the values of Martynyuk [14], which are also reported at initial geometry. Our volume-compensated results show excellent agreement with the values of Gathers et al. [13] who also included volume expansion information within his results. The value of Zinovyev [20] at 2000 K also fits our data quite well.

For thermal conductivity, this work reports $94.3 \text{ W} \cdot \text{m}^{-1} \cdot \text{K}^{-1}$ at the onset of melting, Vlasov [24] reports $95 \text{W} \cdot \text{m}^{-1} \cdot \text{K}^{-1}$, the extrapolated value from Filippov [21] is $105 \,\mathrm{W} \cdot \mathrm{m}^{-1} \cdot \mathrm{K}^{-1}$, and the extrapolated value

Fig. 4. Thermal conductivity of iridium in the solid and liquid states as a function of temperature calculated using WFL. Open circles: averaged measurement data from the present study, open squares: conductivity data from this work using extrapolated volume expansion data from Ref. 18, solid line: linear least-squares fit to the liquid-state data, dashed line: melting temperature (2719 K) , open up-triangles: reference values at the melting transition taken from Mills et al. [23], solid down-triangles: data from Vlasov et al. [24].

from Geld et al. [22] is $98 \text{W} \cdot \text{m}^{-1} \cdot \text{K}^{-1}$. All these values are in good agreement with the theoretical values of the electron thermal conductivity calculated using Mott's model, as shown by Zinovyev [20]. Only the comparison of thermal conductivity at the end of melting to literature values delivers large, considerable differences. This work obtains a value of 68.2 W·m⁻¹·K⁻¹, whereas the only available literature value from Vlasov [24] is 76 W·m⁻¹·K⁻¹. Generally, values of thermal conductivity at the end of melting obtained by pulse-heating are lower than comparable literature values. No explanation for this behavior has been found.

5. UNCERTAINTIES

In accordance with to the guide to the expression of uncertainty in measurement (GUM 1999 [26]), uncertainties reported here are expanded relative uncertainties with a coverage factor of $k = 2$. For the calculated

thermophysical properties the following uncertainties have been obtained and should be applied: temperatures below 2400 K , T , 4% ; temperatures above 2400 K, *T*, 1.7%; normal spectral emittance, ε , 6%; enthalpy in the solid, H_s , 4%; enthalpy in the liquid, H_1 , 2.5%; heat of fusion, ΔH , 13%; isobaric heat capacity in the solid, *c*p,s, 3.4%; isobaric heat capacity in the liquid, *c*p,l, 5.9%; specific electrical resistivity with initial geometry in the solid, $\rho_{IG, S}$, 4%; electrical resistivity with initial geometry in the liquid, $\rho_{IG, L}$, 2.5%; and finally thermal diffusivity in the solid and liquid, a_s and *a*l, 6.5 and 7.7%. Exact details of the evaluation of the stated uncertainties have been carried out within the Ph.D. thesis of Wilthan [27] and are planned to be published elsewhere. For the remaining properties of resistivity considering volume expansion, ρ_{VE} and for thermal conductivity, λ , temperature-dependent density data from literature sources (Wimber [17] for the solid and Ishikawa [18] for the liquid state) were used for calculations. As both sources do not report any uncertainty budget, no final uncertainty statement for ρ_{VF} and λ can be provided within this manuscript.

6. CONCLUSION

Ruthenium, rhodium, palladium, osmium, iridium, and platinum are members of a group of elements referred to as the platinum group-metals. Measurements on rhodium are planned; osmium and ruthenium are not available in the form of a wire. Platinum and palladium have already been investigated and show a slight increase of the normal spectral emittance at 684.5 nm in the liquid phase. Furthermore, for liquid iridium a set of thermophysical property data such as enthalpy, isobaric heat capacity, electrical resistivity, thermal conductivity, and thermal diffusivity as a function of temperature is given. As radiance temperature measurements are combined with simultaneous emittance measurements, there is no ambiguity in the temperature dependent-data reported here.

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