PHYSICAL PROPERTIES AND NORMAL SPECTRAL EMISSIVITY OF IRIDIUM UP TO 3500 K [1]

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[1] To be presented at the 16th Symposium on Thermophysical Properties, Boulder, CO, USA, July 30 – August 4, 2006

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ABSTRACT

Only 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 successfully been used by different institutions around the globe to obtain temperature dependent thermophysical data. At the Institute of Experimental Physics, Graz University of Technology, Austria, a constantly improved pulse-heating setup has been used for more than two decades to determine thermophysical properties of metals and alloys in the solid and the liquid states.

Furthermore, a division-of-amplitude photopolarimeter has been added to the setup with different perspectives: (i) to improve the accuracy of the optical (pyrometric) temperature measurement, (ii) to fulfill the growing need of industry for optical material information, and (iii) for scientific reasons, as, in the majority of cases, few emittance data can only be found for molten materials in literature.
The before mentioned techniques, often combined with additional DCS experiments, helped to systematically study a wide variety of pure, commercially available metals throughout the periodic system, to create a database on material information. Due to various reasons such as availability, purity, or price, some metals such as iridium have been long time excluded from investigations.

This work is dedicated to report our recent results on iridium for both, thermophysical data in the solid and the molten states, as well as normal spectral emittance obtained at a wavelength of 684.5 nm up to 3500 K. The newly obtained values are presented, discussed, and compared with available literature data.

KEYWORDS: Ellipsometry; Iridium; Normal Spectral Emissivity; Pulse-Heating; Thermal Conductivity; Thermal Diffusivity; Thermophysical Properties.

1. INTRODUCTION

Iridium was discovered by Smithson Tennant (England) in 1803. Iridium is named after the Latin word “iris” meaning rainbow because of the colourful nature of iridium compounds, as its salts are highly coloured. 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, and an alloy of 90% platinum and 10% iridium was used in 1889 to construct the standard metre bar and kilogram mass, kept by the International Bureau of Weights and Measures near Paris. The metre 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 nor by aqua regia, but is attacked by molten salts, such as NaCl and NaCN. Iridium is rare on Earth (much more rare than gold and platinum), but relatively common in meteorites.

The following uses for iridium are stated: making crucibles and equipment for use at high temperatures, thermocouples such as Pt-Pt/Ir, used for electrical contacts and Pt/Ir sparkplugs, hardening agent for platinum and further iridium forms an alloy with osmium used for tipping pens and compass bearings.

The measured density of iridum is only slightly lower than that of osmium, which is therefore often listed as the heaviest element known. However, calculations of density from the space lattice may produce more reliable data for these elements than actual measurements and give a density of 22650 kg/m³ for iridium versus 22610 kg/m³ for osmium. Definitive selection between the two is therefore not possible at this time. Information summarized from CRC Handbook [1].

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 within a recent project.

2. EXPERIMENT AND DATA REDUCTION

Determination of thermophysical properties of liquid metals, as performed at the ‘Subsecond Thermophysics Group’ at Graz University of Technology with a long tradition, is still relevant and of permanent interest for the metal working industry as well as for science and academia. Accurate data at the melting transition and in the liquid state are often rare, but essentially needed 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 of 0.5 mm diameter, of 60 mm length, and a purity 99.9% were purchased from Alfa Aesar (reference 19587) and subsequent resistively pulse heated as part of a capacitor driven discharge circuit in nitrogen atmosphere at ambient pressures.

The directly obtained quantities current, voltage drop, surface radiance, expansion and normal spectral emissivity allow the further determination of thermophysical properties such as temperature of the sample, specific enthalpy, heat of fusion, isobaric heat capacity, electrical resistivity (at initial geometry and under consideration of thermal expansion), thermal conductivity, and thermal diffusivity. The accessible range of measurement extends from room temperature up to superheated liquid states. Experimental details have already been described extensively elsewhere e.g. Gallob et al. [2] and Wilthan et al. [3].

To enable accurate and unambiguous temperature determination over such a vast range, pyrometric temperature detection based on Planck's law on radiation is used. Furthermore, normal spectral emissivity data are determined by an ellipsometric device (µs-DOAP) to compensate uncertainties arising from the unknown emissivity and its behavior throughout the temperature range of the measurement. For more details on the µs-DOAP see e. g. Seifter et al [4] and Cagran et al. [5].

3. RESULTS

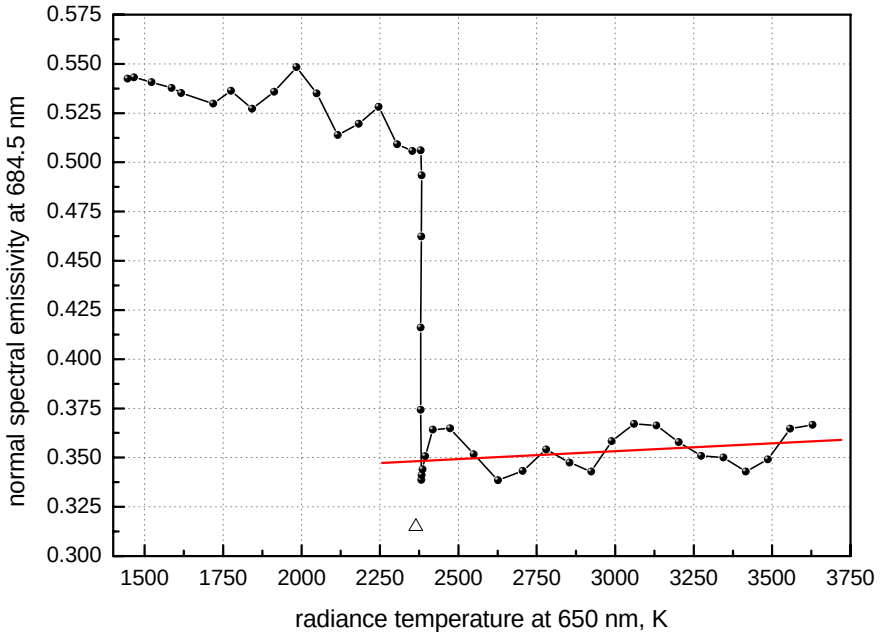

Fig. 1: Normal spectral emissivity at 684.5 nm (average of 9 individual measurements) of iridium versus radiance temperature at 650 nm. Full line with circles: measurement data from the present study, full line: linear least-squares fit to the liquid state, open up-triangle: literature reference value taken from [8] interpolated for 684.5 nm.

In Fig. 1 normal spectral emissivity of iridium at a wavelength of 684.5 nm versus radiance temperature T_{rad} at 650 nm is plotted and compared to literature. At the end of the solid phase emissivity values decrease smoothly to values in the range of about 0.506. These values observed in the solid phase strongly depend on the surface preparation, here with abrasive paper grade 1200 or 4000. As the surface smoothens

during melting[1] a strong decrease in the measured emissivity values is observed. The melting temperature of Iridium, T_m, is 2719 K as reported from Bedford et al. [6] and T_m = (2719 ± 6) K as reported by Arblaster [7], while for the radiance temperature at melting at a wavelength of 656 nm 2380 K is given by McClure et al. [6]. Within this work an emissivity of 0.348 at 684.5 nm was obtained for the end of melting, thus resulting in a radiation temperature of 2365 K at melting. The emissivity value of McClure [8] of 0,315 at melting (interpolated for 684,5 nm) is depicted in Fig.1. An average of 9 measurements in the liquid phase deliver the linear fit for normal spectral emissivity in the radiance temperature range up to 3750 K. The fit is presented in Table 1. For convenience and easier data comparison, all corresponding least-squares results are summarized in Table 1. For liquid iridium a slight increase of normal spectral emissivity at 684.5 nm up to 3755 K is observed, which is similar to the behavior of platinum (Wilthan et al. [3]).

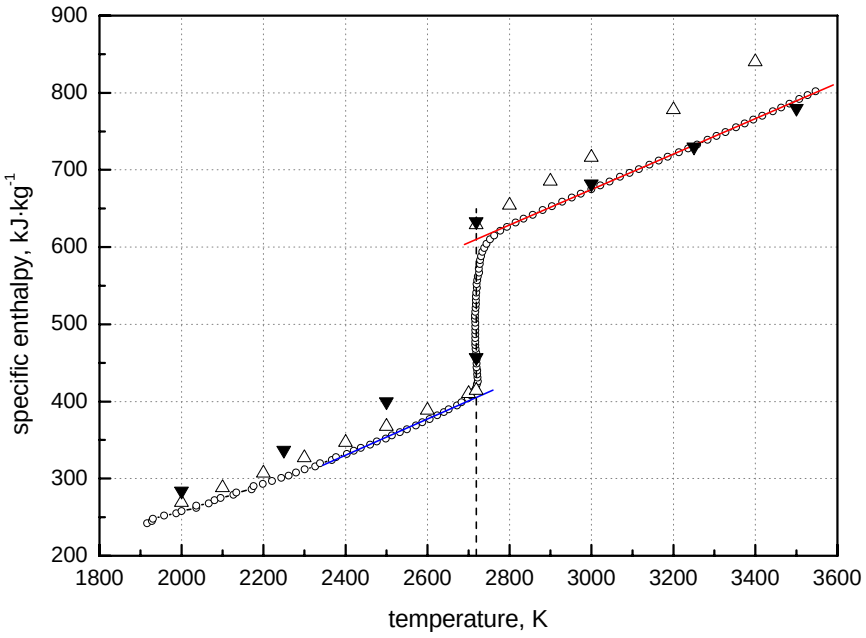

Fig. 2: Specific enthalpy of iridium in the solid and the liquid states as a function of temperature. Open circles: averaged measurement data from the present study, solid lines: linear-least squares fits, dashed line: melting temperature (2719 K), open up-triangles: literature reference values taken from [7], solid down-triangles: literature data taken from [11].

Fig. 2 presents specific enthalpy H versus true temperature T, wherein enthalpy at room temperature was assigned as zero. All temperature dependent results reported within the following figures (Figs. 2-5) cover a range from 1800 K to about 3600 K. An average of 9 measurements in the liquid phase delivers the linear fits for the solid and for the liquid states, listed in Table 1. Talking specifically about enthalpy, we acquire (factor b in Table 1) a constant c_p value of 230 $J \cdot kg^{-1} \cdot K^{-1}$ for the end of the solid state as well as for the liquid state, which is quite unusual, as for most other materials measured in our lab so far, the c_p values in the solid and in the liquid phase differ, sometimes even quite

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

much. Hultgren et al. [9] report 217 $J \cdot kg^{-1} \cdot K^{-1}$ for the liquid state. Arblaster [7] reports enthalpy measurements by Kats [10], which lead to a constant c_p value of 309 $J \cdot kg^{-1} \cdot K^{-1}$ for liquid iridium up to 5000 K. The conversion factor from mol to kg used within this work is 5.2024534, using the currently accepted atomic weight of 192.217 as reported by Arblaster [7]. Data of Gathers et al. [11] and recommended values from Arblaster [7] are also depicted in Fig. 2.
At melting, which is indicated by a vertical dotted line, the specific enthalpy changes from Hs(Tm) = 405.2 kJ·kg-1 (index s: solid) to Hl(Tm) = 610.0 kJ·kg-1 (index l: liquid) yielding ΔH = 204.8 kJ·kg-1 for the latent heat of fusion. Thus the entropy of fusion is ΔS = 75.3 J·kg-1K-1. Gathers et al. [11] report for Hs(Tm) = 457 kJ·kg-1 and for Hl(Tm) = 633 kJ·kg-1 yielding ΔH = 176 kJ·kg-1 for the latent heat of fusion at an ambient 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 at ambient pressures [11]. Martynyuk and Tsapkov [12] report for $H_s(T_m)$ = 406 $kJ \cdot kg^{-1}$ and for $H_l(T_m)$ = 572 $kJ \cdot kg^{-1}$ yielding ΔH = 166 $kJ \cdot kg^{-1}$ for the latent heat of fusion. Lebedev et al. [13] report for ΔH =200 $kJ \cdot kg^{-1}$. As reported by Arblaster [7] enthalpy measurements by Kats [10] lead to a value of ΔH (215 ± 59) $kJ \cdot kg^{-1}$. Dinsdale [14] reports ΔH =214 $kJ \cdot kg^{-1}$ Hultgren et al. [9] report for $H_s(T_m)$ = 418 $kJ \cdot kg^{-1}$ and for $H_l(T_m)$ = 553 $kJ \cdot kg^{-1}$ yielding ΔH = 135 $kJ \cdot kg^{-1}$

Fig. 3 presents electrical resistivity as a function of temperature. At the onset of melting, indicated by a vertical dotted line, we obtain for resistivity with initial geometry ρ_{IG} (no correction for thermal expansion, index IG) a value of 0.645 µΩ·m and at the end of melting a value of 0.849 µΩ·m, thus an increase of $\Delta\rho$ = 0.204 µΩ·m at melting is observed. For electrical resistivity that is compensated for thermal volume expansion (index VE), ρ_{VE} we considered literature values of the thermal expansion for iridium from Toulukian [15] and converted density data from Ishikawa [16][2]. The change in diameter results in a shift to higher resistivity values, as the actual cross-section of the wire, which also influences electrical resistivity, is increased. For the liquid state above 3000 K, no expansion data are given in [16] and a linear extrapolation was used instead to estimate thermal expansion up to 3600 K.

At the onset of melting, we now obtain a value of 0.695 µΩ·m and at the end of melting a value of 0.985 µΩ·m for volume compensated electrical resistivity. Thus an increase of $\Delta\rho$ = 0.290 µΩ·m at melting is observed. The values from Gathers [11] and Martynyuk [12] are also shown in Fig. 3. Gathers et al. [11] report for the onset of melting 0.737 µΩ·m and for the end of melting 1.00 µΩ·m delivering an increase $\Delta\rho$ = 0.263 µΩ·m at melting Martynyuk [12] report for the onset of melting 0.659 µΩ·m and for the end of melting 0.855 µΩ·m delivering an increase $\Delta\rho$ = 0.196 µΩ·m at melting. Zinovyev [18] reports for iridium at 2000 K a value of 0.50 µΩ·m.
The four corresponding least squares polynomials for resistivities versus temperature obtained within this work can once again be found in Tab. 1.

[2] Ishikawa et al. published two different papers presenting density data for liquid iridium within the period of a couple months [16, 17] in which the values contradict mutually. Contacting the authors directly, they informed us that the values from [16] represent their best results to date.

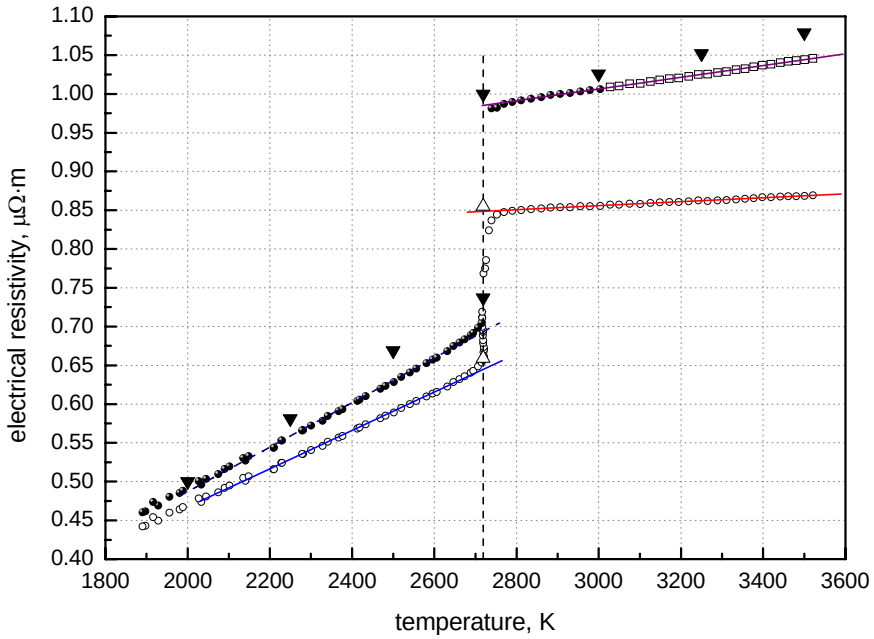

Fig. 3: Electrical resistivity under initial geometry (IG) and considering thermal volume-expansion (VE) of iridium for the solid and the liquid states as a function of temperature. Open circles: averaged measurement data (IG) from the present study, solid circles: resistivity data from the present work considering volume expansion taken from [15, 16], open squares: resistivity data from this work (VE) using extrapolated volume expansion data from [16], solid lines: linear-least squares fits, dashed line: melting temperature (2719 K) [6], open up-triangles: reference values at the melting transition taken from [12], solid down-triangles: literature data taken from [11].

Fig. 4 presents thermal conductivity versus temperature. To estimate thermal conductivity via the Wiedeman – Franz law (see Wilthan et al. [3]) the electrical resitivity considering the thermal volume expansion of the sample material (ρ_{VE}), as introduced within the last paragraph, must be used. At the onset of melting we obtain a value of 94.9 $W \cdot m^{-1} \cdot K^{-1}$ and at the end of melting for the beginning of the liquid phase a value of 67.9 $W \cdot m^{-1} \cdot K^{-1}$. For the end of the solid phase the extrapolated value from Filippov [19] is 105 $W \cdot m^{-1} \cdot K^{-1}$ and the extrapolated value from Geld et at. [20] is 98 $W \cdot m^{-1} \cdot K^{-1}$. Zinovyev [18] reports for iridium at 2000 K a value of 103 $W \cdot m^{-1} \cdot K^{-1}$. Mills et al. [21] report for the end of the solid and the begin of the liquid phases values of 95 $W \cdot m^{-1} \cdot K^{-1}$ and 76 $W \cdot m^{-1} \cdot K^{-1}$, respectively. These values originally reported by Vlasov [22] are also given as a recommendation in [21] due to a lack of more data for the liquid state. The corresponding least squares polynomials obtained within this work for thermal conductivity a limited solid range and the liquid phase are once again listed in Tab. 1.

As the last property in our list, thermal diffusivity a , can be estimated from thermal conductivity (Wilthan et al. [3]). A graph showing thermal diffusivity versus temperature is omitted, as all relevant information about a can be extracted from the linear fits, given in Tab.1. At the onset of melting we obtain a value for a of 1.82×10^{-5} $m^2 \cdot s^{-1}$ and at the end of melting for the beginning of the liquid phase a value of 1.30×10^{-5} $m^2 \cdot s^{-1}$.

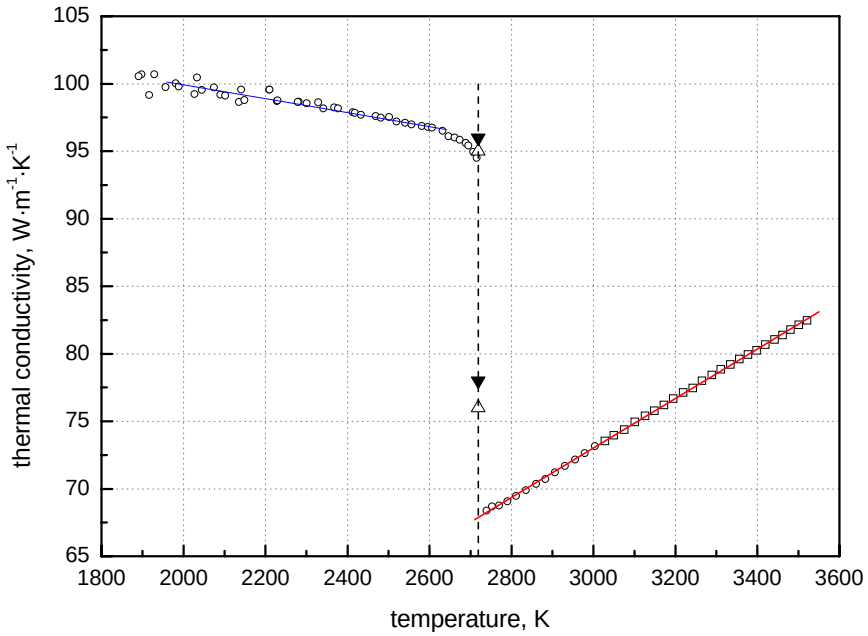

Fig. 4: Thermal conductivity of iridium for the solid and the liquid states as a function of temperature calculated using WFL. Open circles: averaged measurement data from the present study, open squares: conductivity data from this work using extrapolated volume expansion data from [16], solid line: linear-least squares fit to the liquid state, dashed line: melting temperature (2719 K), open up-triangles: reference values at the melting transition taken from [21], solid down-triangles: literature data taken from [22].

Tabular results

Table 1: Summarized results of the recent study given as linear least-squares fits (where applicable) including the temperature range of applicability in K. Fits are given in the form a + b·T, where a & b are the fit parameters and T is temperature. H: enthalpy, ρ_{IG}: electrical resistivity with initial geometry, ρ_{VE}: electrical resistivity considering thermal volume expansion, λ: thermal conductivity, a: thermal diffusivity, ε: normal spectral emittance at 684.5 nm as a function of radiance temperature at 650 nm.

	solid state			**liquid state**		
	a	**b**	**range**	**a**	**b**	**range**
H [kJ·kg^{-1}]	-222.305	0.230	2300 < T < 2719	-14.824	0.2298	2719 < T < 3550
$\rho(IG)$ [μΩ·m]	-0.0274	2.472×10^{-4}	2000 < T < 2719	0.7803	2.520×10^{-5}	2719 < T < 3550
$\rho(VE)$ [μΩ·m]	-0.0861	2.867×10^{-4}	2000 < T < 2719	0.7793	7.570×10^{-5}	2719 < T < 3550
λ [W·m^{-1}·K^{-1}]	110.197	5.14×10^{-3}	2000 < T < 2620	18.0033	1.834×10^{-2}	2719 < T < 3550
$a \times 10^{-5}$ [m^2·s^{-1}]	2.112	9.776×10^{-5}	2000 < T < 2620	0.3459	3.524×10^{-4}	2719 < T < 3550
ε	-	-	-	0.3293	7.988×10^{-6}	2365 < T_r < 3650

Table 2: Values of thermophysical properties of iridium in the solid and liquid states. T: temperature, H: enthalphy, ρ_{IG}: electrical resistivity with initial geometry, ρ_{VE}: electrical resistivity considering thermal volume expansion, λ: thermal conductivity, a: thermal diffusivity, T_r: radiance temperature at 650 nm, ε: normal spectral emittance at 684.5 nm as a function of radiance temperature. NOTE: radiance temperatures are given in the same steps as the thermodynamic temperature but, with the exception of the the liquidus value, don't relate to the thermodynamic temperature.

T [K]	H [kJ·kg^{-1}]	ρ_{IG} [μΩ·m]	ρ_{VE} [μΩ·m]	λ [W·m^{-1}·K^{-1}]	$a \times 10^{-5}$ [m^2·s^{-1}]	T_r [K]	ε@684.5nm
2000	258.02	0.467	0.487	99.92	1.92	-	-
2100	275.56	0.492	0.516	99.40	1.91	-	-
2200	293.24	0.516	0.545	98.89	1.90	-	-
2300	306.7	0.541	0.573	98.38	1.89	-	-
2400	329.7	0.566	0.602	97.86	1.88	-	-
2500	352.7	0.591	0.631	97.35	1.87	-	-
2600	375.7	0.615	0.659	96.83	1.86	-	-
2700	398.7	0.640	0.688	95.42	1.83	-	-
2719 (s)	**403.1**	**0.645**	**0.693**	**94.90**	**1.82**	**-**	**-**
2719 (l)	**610.0**	**0.849**	**0.985**	**67.87**	**1.30**	**2365 (l)**	**0.348**
2800	628.6	0.851	0.991	69.36	1.33	2700	0.351
2900	651.6	0.853	0.999	71.19	1.37	2800	0.352
3000	674.6	0.856	1.006	73.02	1.40	2900	0.352
3100	697.6	0.858	1.014	74.86	1.44	3000	0.353
3200	720.5	0.861	1.022	76.69	1.47	3100	0.354
3300	743.5	0.863	1.029	78.53	1.51	3200	0.355
3400	766.5	0.866	1.037	80.36	1.54	3300	0.356
3500	789.5	0.869	1.044	82.19	1.58	3400	0.356

4. DISCUSSION

There are several literature data of liquid Iridium available, but only data for liquid emissivities are very rare. The values of normal spectral emissivity ε of iridium at a wavelength of 684.5 nm at the end of melting for the liquid reported here give a reasonable agreement to the one reported from McClure et al. [8], albeit our value is somewhat higher.

For enthalpy versus temperature the recommended values of Arblaster [7] give a good agreement to our values in the end of the solid state, but do not match so well in the liquid. The pulse heating values of Gathers et al. [11] match our values in the liquid excellent, but at the end of the solid phase they tend to higher values, which results in a lower value of heat of fusion reported by them.

The comparison of the latent heat of fusion obtained here gives a wide variation of values. We obtain a value of ΔH =204.8 kJ·kg^{-1}, Kats [10] ΔH =215 kJ·kg^{-1} cited by [7] (deviation from value of this work + 5%), Dinsdale [14] ΔH =214 kJ·kg^{-1} (+ 4.5%), Lebedev [13] ΔH =200 kJ·kg^{-1} (- 2.5 %), Gathers [11] ΔH =176 kJ·kg^{-1} (- 14 %) and

Martynyuk [12] ΔH =166 kJ·kg^{-1} (- 20 %) Hultgren et al. [9] ΔH = 135 kJ·kg^{-1} (- 34 %). Here it is to notice, that other pulse heating experiments [11, 12] delivered quite lower values, while all other values lie within a 5 % deviation interval. The quite old recommended value of [9] seems to be very low, therefore newer recommendations such as those from Arblaster [7] should be to be considered.

At the time of these measurements on iridium our high speed system [23] for monitoring sample expansion was not in service, meanwhile it has been improved and is operable again. Therefore the relation density versus temperature for Iridium had to be taken from Toulukian [15] and Ishikawa [16] to consider the volume change during our resistivity measurements. Our resistivity values without volume correction on melting match the values of Martynyuk [12] excellent, while the values of Gathers at al. [11] mach our volume corrected values excellent. The value of Zinovyev [18] at 2000 K also fits the data of this work quite well.

For thermal conductivity this work reports at the onset of melting, 94.9 W·m^{-1}·K^{-1}, Vlasov [22] reports 95 W·m^{-1}·K^{-1}, the extrapolated value from Filippov [19] is 105 W·m^{-1}·K^{-1} and the extrapolated value from Geld et at. [20] is 98 W·m^{-1}·K^{-1}, all these values are in good agreement to the theoretical values of the electron thermal conductivity calculated using Mott´s model as shown in Zinovyev [18]. Only the comparison of thermal conductivity at the end of melting to literature values delivers strong differences. This work obtains a value of 67.9 W·m^{-1}·K^{-1}, whereas the only available literature value from Vlasov [22] is 76 W·m^{-1}·K^{-1}. Generally values of thermal conductivity at the end of melting at the begin of the liquid phase obtained by pulse heating lie sometimes lower than comparable literature values, up to now no explanation for this behavior could be found.

5. UNCERTAINTIES

According to the guide to the expression of uncertainty in measurement GUM 1999 [24] 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 emissivity, ε, 6 %; enthalpy in the solid, H_s, 4%; enthalpy in the liquid, H_l, 2.5%; heat of fusion, ΔH, 8%; isobaric heat capacity, c_p, 8%; specific electrical resistivity with initial geometry in the solid , $\rho_{IG,S}$, 4%; specific electrical resistivity with initial geometry in the liquid , $\rho_{IG,L}$, 4%; resistivity considering volume expansion, ρ_{VE}, 6%; thermal conductivity, λ, 12%; and finally thermal diffusivity, a, 16 %.

6. CONCLUSION

Ruthenium, rhodium, palladium, osmium, iridium, and platinum together make up a group of elements referred to as the platinum group metals. Measurements on rhodium are scheduled; osmium and ruthenium are not available in wire shape. Platinum and palladium have already been investigated and show a slightly increase of normal

spectral emissivity at 684.5 nm in the liquid phase. Furthermore for liquid Iridium a set of thermophysical data such as enthalpy, isobaric heat capacity, electrical resistivity, thermal conductivity and thermal diffusivity as a function of temperature is given. As temperature measurement is combined with simultaneous emissivity measurements, there is no ambiguity in the temperature dependent data reported here.

6. ACKNOWLEDGEMENTS

This work has been supported by the *FWF, "Fonds zur Foerderung der wissenschaftlichen Forschung"*, Grant P-15055.
The authors are also deeply grateful for the help of B. Wilthan in calculating the uncertainties for these data.

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