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Radiance Temperatures (in the Wavelength Range 521 to 1500 nm) of Rhenium and Iridium at Their Melting Points by a Pulse-Heating Technique[1]

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The melting-point radiance temperatures (at seven wavelengths in the range 521 to 1500 nm) of rhenium and iridium were measured by a pulse-heating technique. The method is based on rapid resistive self-heating of the specimen from room temperature to its melting point in less than 1 s and on simultaneously measuring the specimen radiance temperature every 0.5 ms with two high-speed pyrometers. Melting was manifested by a plateau in the radiance temperature-versus-time function for each wavelength. The melting-point radiance temperatures for a given specimen were determined by averaging the measured temperatures along the plateau at each wavelength. The melting-point radiance temperatures for each metal were determined by averaging results for several specimens at each wavelength. The results are as follows.

Rhenium	Iridium
2989 K at 521 nm	2458 K at 523 nm
2916 K at 614 nm	2402 K at 617 nm
2891 K at 656 nm	2380 K at 656 nm
2853 K at 707 nm	2349 K at 711 nm
2789 K at 807 nm	2297 K at 808 nm
2719 K at 905 nm	2243 K at 906 nm
2324 K at 1500 nm	1944 K at 1500 nm

Based on estimates of the random and systematic errors arising from pyrometry and specimen conditions, the expanded uncertainty (two standard-deviation

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level) in the reported values is ± 8 K.

KEY WORDS: emissivity (normal spectral); high-speed pyrometry; high-temperature fixed points; iridium; melting; pyrometry; radiance temperature; rhenium.

1. INTRODUCTION

High-speed multiwavelength pyrometry has been used in this laboratory over the last two decades to study the reproducibility and wavelength dependence of the radiance temperature[4] at the melting point of selected pure metals in the wavelength range 500 to 1000 nm. During the initial melting period, the radiance temperature at each wavelength is essentially constant and very reproducible for different specimens of a given metal, which suggests that such measurements may provide high-temperature references for secondary calibrations of optical pyrometers [1]. Recent work has extended these measurements to the longer wavelength of 1500 nm [2, 3], and an effort has been made to study as many of the high-melting point pure metals as can be done practically. In the present paper, we extend these measurements to two additional high-melting point metals, rhenium and iridium.

The measurement technique is based on rapid resistive self-heating of a strip-shaped specimen from room temperature to its melting point in less than one second by passing a large electrical current pulse through the specimen. As the specimen heats, its radiance temperature at each of seven wavelengths (nominally between 500 and 1500 nm) is measured by two pyrometers every 0.5 ms. Radiances at each of six wavelengths in the nominal range 500 to 900 nm are measured with a high-speed six-wavelength pyrometer and radiances at two wavelengths, nominally 650 and 1500 nm, are measured with a high-speed two-wavelength pyrometer. The melting-point radiance temperatures for a given metal specimen were determined by averaging the measured radiance temperatures along the melting plateau for each wavelength. All temperatures reported in this paper, except where explicitly noted otherwise, are based on the International Temperature Scale of 1990 (ITS-90) [4].

Details concerning the design and construction of the pulse-heating system [5, 6] and the design, operation, and calibration of the six-

[4] The radiance temperature (sometimes referred to as brightness temperature) of the specimen surface at a given wavelength is the temperature at which a blackbody at the same wavelength has the same radiance as the surface. The wavelength is the effective wavelength of the measuring pyrometer.

wavelength pyrometer [7] are given in the cited publications. The 650-nm channel of the two-wavelength pyrometer is used for calibration of the 1500-nm channel and as a control to compare its results to that of the similar wavelength of the six-wavelength pyrometer. Details concerning the calibration and operation of the two-wavelength pyrometer are given in earlier publications [2, 3].

2. MEASUREMENTS

Measurements of the radiance temperature of rhenium and iridium at their melting points were performed on nine rhenium specimens and eight iridium specimens in the form of strips cut from 50x50-mm metal foils. The purity of the rhenium material was 99.97% by mass and that of iridium was 99.8% by mass. A typical analysis of the rhenium foil, as reported by the manufacturer, yielded the following impurities (ppm, by mass): Fe, 70; N, O, 40; Mo, 25; C, 20; Al, H, 5; Co, Cr, and Si, 3; Cu, Mn, Sn, and Ti, 2; and Ca, B, K, Mg, and Ni, 1. A typical analysis of the iridium foil, as reported by the manufacturer, yielded the following impurities (ppm, by mass): Ta, <300; Mo, 70: Ru, 30; Ba, Cd, Os, and W, <30; Rh and Pt, 10; As, Sb, and Zn, <10; Si, 3; Cu, Fe, 2; Ag, Al, and B, 1; and gases and unreported elements, 1420.

The nominal dimensions of each specimen strip cut from the foil were a length of 50 mm and a width of 3 mm. The thickness of the rhenium strips was 0.25 mm, and that of the iridium strips was 0.127 mm. To remove possible surface contaminants the surfaces of the specimens were mechanically treated with abrasives, and each specimen was subjected to one or two "preheat pulses." Each rhenium specimen was heated to a radiance temperature of approximately 2760 K, and each iridium specimen was heated to a radiance temperature of approximately 2300 K. Each experiment was performed with the specimen strip in an argon gas environment (approximately 0.15 MPa) to minimize contamination of the specimen surface at high temperatures. The duration of the electrical current pulse, used to heat each specimen from room temperature to its melting temperature, ranged from approximately 170 to 550 ms.

Figure 1 shows typical radiance temperature data obtained at three representative wavelengths (656, near 900, and 1500 nm) by the pyrometers during pulse-heating experiments on rhenium and iridium, respectively, near and at their melting points. Melting of the specimen is manifested by a plateau region in the measured radiance temperature results for each wavelength. The flat region along each plateau (indicated by the dashed line) was used to determine the melting radiance temperature at each wavelength. The radiance temperature along this flat region was essentially

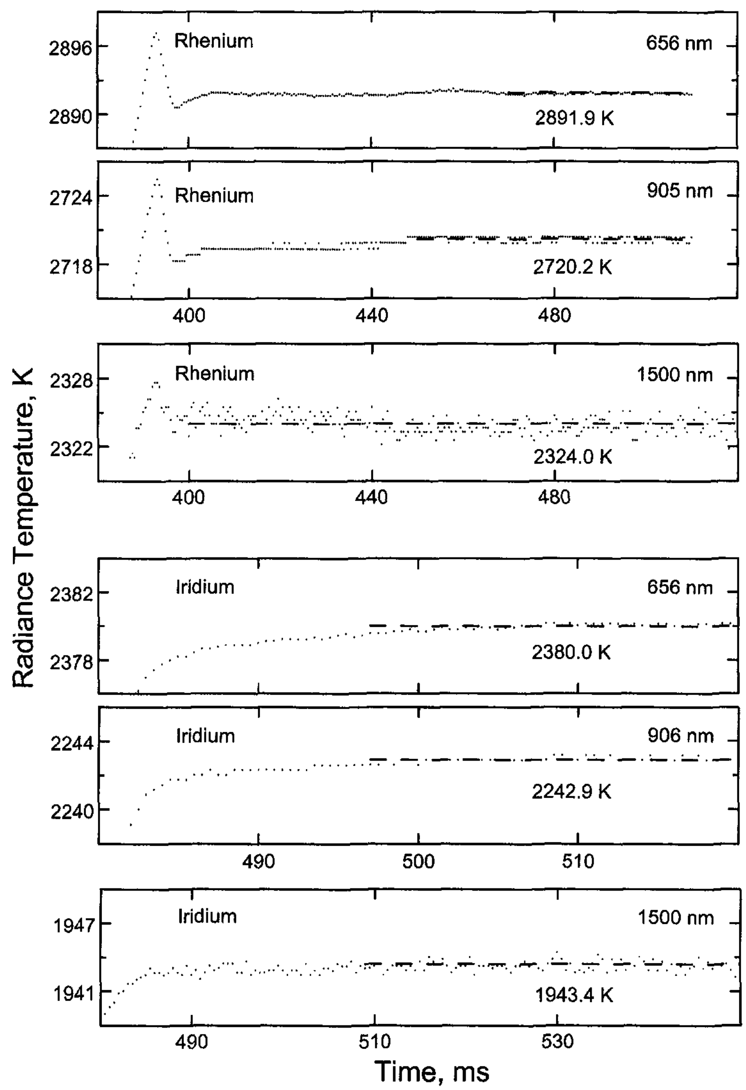

Fig. 1. Variation of the radiance temperatures of rhenium and iridium specimens at three representative wavelengths (656, about 900, and 1500 nm) just before and during melting as measured with the pyrometers during typical pulse-heating experiments. The effective wavelengths, shown for each channel, were determined following the definition given by Kostkowski and Lee [8]. Each labeled temperature indicates the average melting-point radiance temperature for the experiment computed from the plateau data indicated by the dashed line.

constant within a range of less than ± 1 K for all specimens. The effective wavelength for each pyrometer channel was determined at the respective average radiance temperature using the method of Kostkowski and Lee [8].

3. RESULTS

For each specimen, the radiance temperature at the melting point was determined for each wavelength by averaging the measured temperatures along the flat portion of the corresponding plateau (indicated by the dashed lines in Fig. 1).

For the rhenium specimens, the number of temperatures used to obtain an average radiance temperature ranged from 51 to 346, depending upon the heating rate and the behavior of the specimen during melting. The standard deviation of an individual plateau radiance temperature from the average obtained for that specimen was less than 0.3 K in the wavelength range 521 to 905 nm and was equal to 1.1 K at 1500 nm. The trend (or slope) of the radiance temperature along each plateau was determined by fitting a linear function in time to the measured radiance temperatures using the method of least squares. The slope of the linear functions for all experiments on rhenium was in the range -10 to $23\ \mathrm{K \cdot s^{-1}}$. The temperature difference between the beginning and end of the plateau, as determined from this slope, was in the range -0.3 to 0.6 K. Heating rates for each specimen were determined by fitting a linear function in time to the radiance temperatures measured during the premelting period. The heating rates for the rhenium specimens (slopes of the linear functions approximately 25 K below the melting plateau) ranged from 1200 to $3100\ \mathrm{K \cdot s^{-1}}$. An examination of the results on rhenium showed no relationship between heating rate and the measured melting-point radiance temperature.

For the iridium specimens, the number of temperatures used to obtain an average radiance temperature ranged from 36 to 341. The standard deviation of an individual plateau radiance temperature from the average obtained for that specimen was less than 0.4 K in the wavelength range 523 to 906 nm and was 0.8 K at 1500 nm. The slope of the linear functions representing the radiance temperatures of iridium along each plateau was in the range -5 to $44\ \mathrm{K \cdot s^{-1}}$. The temperature difference between the beginning and end of the plateau, as determined from this slope, was in the range -0.3 to 1.1 K. Heating rates for iridium specimens, determined as described above for rhenium, were in the range 744 to $4700\ \mathrm{K \cdot s^{-1}}$. As with rhenium, the results on iridium showed no relationship between heating rate and the measured melting-point radiance temperature.

Table I presents the final experimental results of the radiance temperatures of rhenium and iridium at their melting points in the wavelength

Table I. Average Radiance Temperature and Normal Spectral Emissivity, at Seven Wavelengths, of Rhenium and Iridium at Their Melting Points

Material and melting temperature (K)[a]	Effective wavelength (nm)[b]	Radiance temp. (K)[c]	SD (K)[d]	Normal spectral emissivity[e]
Rhenium (9 specimens),				
$T_m = 3458$ K [9]	521	2989.2	0.7	0.286
	614	2915.8	0.7	0.283
	656	2890.9	0.6	0.288
	707	2853.3	0.7	0.287
	807	2789.4	0.8	0.289
	905	2719.2	0.8	0.285
	1500	2324.5	0.5	0.246
Iridium (8 specimens),				
$T_m = 2719$ K [10]	523	2458.1	0.5	0.342
	617	2401.5	0.4	0.322
	656	2380.3	0.3	0.317
	711	2348.7	0.3	0.309
	808	2296.5	0.4	0.299
	906	2243.2	0.4	0.289
	1500	1943.6	0.7	0.239

[a] Melting temperature based on ITS-90.
[b] Determined at the respective radiance temperature using the definition of effective wavelength given by Kostkowski and Lee [8].
[c] Average of the specimen melting-point radiance temperatures at each effective wavelength.
[d] Standard deviation of an individual specimen melting-point radiance temperature from the average of values obtained from all specimens.
[e] Determined by means of Planck's law from the average plateau radiance temperature and the respective melting temperature for each material.

range 521 to 1500 nm. Depending upon the wavelength, the standard deviation of an individual average melting temperature from the overall average is in the range from 0.5 to 0.8 K for rhenium and 0.3 to 0.7 K for iridium. Also given in Table I are the corresponding values for the normal spectral emissivity of rhenium and iridium at their melting points. Emissivity was calculated by means of Planck's law using the present results for radiance temperature and the value of 3458 K for the melting point of rhenium [9] and 2719 K for the melting point of iridium [10].

4. ESTIMATE OF UNCERTAINTIES

The major sources of uncertainty are (i) the calibration and operation of the pyrometer and (ii) the physical/chemical conditions and melting behavior of each specimen. A detailed analysis of magnitudes of the uncertainties from all sources is given in an earlier publication [11]. Specific items in the error analysis (particularly those related to temperature range) were recomputed whenever the present conditions differed from those in the earlier publication. The expanded uncertainty (two standard-deviation level) in the reported values for melting-point radiance temperatures at each of the seven wavelengths in the range 521 to 1500 nm is estimated to be about ± 8 K.

5. DISCUSSION

A search of the literature found no papers that report the radiance temperature of rhenium or iridium at their melting points. However, two papers that report results for normal spectral emissivity of rhenium at its melting point were found, and these results were used in conjunction with the reported melting temperatures to compute values of radiance temperatures to compare with the present results. These values and the present results for the radiance temperature at the melting point of rhenium are listed in Table II and are plotted as a function of wavelength in Fig. 2. Figure 2 also shows a plot of the radiance temperature results for iridium as a function of wavelength. The present results for normal spectral emissivity of rhenium and those values reported in the literature are also listed in Table II, and these results as well as the present results for the normal spectral emissivity of iridium at its melting point are plotted as functions of wavelength in Fig. 3.

The results reported by Hiernaut et al. [12] for the normal spectral emissivity of rhenium at its melting point are based on measurements performed with specimens of lower purity. This may account for the lower melting point for rhenium determined by Hiernaut et al., which is approximately 130 K below the accepted value. The radiance temperatures determined from their results show a smaller slope than the present results. This behavior is a direct result of the constant value for emissivity that they report for wavelengths in the region 500 to 1000 nm.

The radiance temperature at the melting point of rhenium at 650 nm determined from the normal spectral emissivity reported by Lin and Frohberg [13] is in good agreement with the interpolated value from our results. Their value at 547 nm is about 40 K higher than our interpolated value, and over the short wavelength range, their values show a steeper slope than the present results.

As can be seen in Fig. 3, our results for the normal spectral emissivity of rhenium at its melting point are essentially flat (at a value less than 0.30)

Table II. Radiance Temperatures (T_{rad}) and Normal Spectral Emissivities (ε) at Wavelengths (λ) in the Range 521 to 1500 nm of Rhenium at Its Melting Point as Reported in the Literature

Element	Investigators	Ref. No.	Year	Purity (mass%)	Heating method[a]	λ (nm)	T_{rad} (K)	ε (reported)
Rhenium	Hiernaut et al.	12	1989	99.5	L	500	2951[b]	0.34
						650	2856[b]	0.34
						1000	2661[b]	0.34
	Lin and Frohberg	13	1992	99.99	E	547	3008[c]	0.320
						650	2906[c]	0.296
	Present work			99.97	R	521	2989 ± 8	0.286
						614	2916 ± 8	0.283
						656	2891 ± 8	0.288
						707	2853 ± 8	0.287
						807	2789 ± 8	0.289
						905	2719 ± 8	0.285
						1500	2324 ± 8	0.246

[a] Method used to heat the specimen (specimen geometry in parentheses): L, laser pulse heating (sphere); E, electromagnetic levitation/induction heating (sphere); R, resistive self-heating (strip).

[b] Based on the constant emissivity (0.34 for the wavelength range 500 to 1000 nm) and the melting temperature (3318 K on ITS-90) reported by the investigators [12].

[c] Based on the emissivities (0.320 at 547 nm and 0.296 at 650 nm) measured by the investigators and a melting temperature of 3458 K (ITS-90) as reported by the investigators [13].

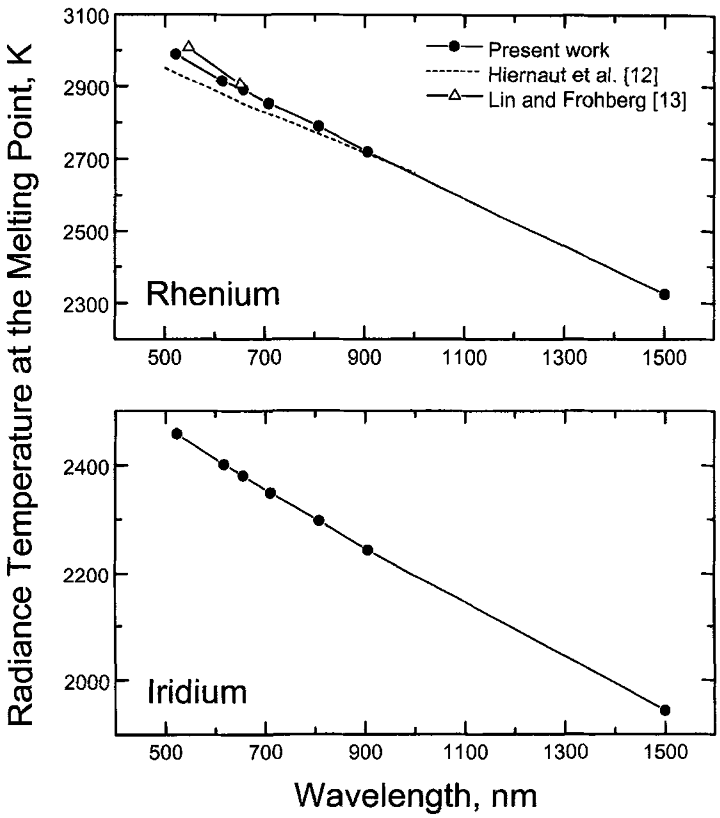

Fig. 2. Comparison (on ITS-90) of literature values and present results for the radiance temperature of rhenium at its melting point as a function of wavelength and the present results for the radiance temperature of iridium at its melting point as a function of wavelength.

in the wavelength region 500 to 800 nm, which is unusual behavior compared to results obtained previously for other high-temperature metals [14]. In our experience, normal spectral emissivity at the melting point of high-temperature metals exhibits a monotonic decrease with increasing wavelength in the region 500 to 1500 nm (similar to the relationship shown by iridium in Fig. 3). The decrease between 500 and 1000 nm is usually 10% or greater, depending upon the material [14]. Because this behavior was so unusual, other samples of rhenium, from two vendors, were used in additional melting-point experiments. Some of these additional experiments were done with the pyrometer before it was modified to increase its sensitivity for lower temperature measurements [15], and many were done

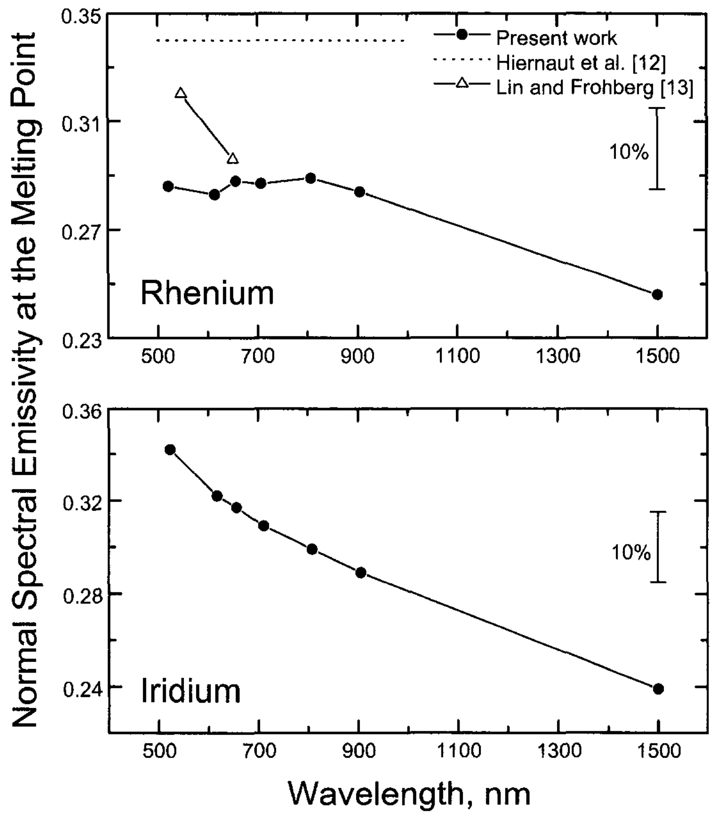

Fig. 3. Variation of the normal spectral emissivity of rhenium and iridium at their melting points as a function of wavelength as reported by different investigators.

after new calibrations of the pyrometer. All additional experiments yielded results similar to those shown in Fig. 3.

In the literature, we found a report which shows the spectral emissivity of rhenium exhibiting the same flat behavior with respect to wavelength as our results, but at temperatures below the melting point. Barnes [16] did an extensive study of the wavelength variation of the optical constants of several refractory metals including rhenium and iridium at temperatures below their melting points. For both materials, he measured the spectral emissivity in the temperature range 1100 to 2500 K and in the wavelength range 400 to 2000 nm. The results for iridium at all temperatures show a decreasing spectral emissivity over the wavelength range 400 to 800 nm, but the results for rhenium show the beginning of a flat region around 400 to 450 nm at 2000 K and a broader flat region from 400 to 600 nm at 2500 K. If this behavior continues to the melting point for rhenium, then the variation of emissivity with wavelength as shown in Fig. 3 could well result.

At approximately 800 nm, our results for normal spectral emissivity of rhenium at its melting point show the typical decrease with increasing wavelength, decreasing about 15% between 800 and 1500 nm. The results for iridium show a decrease of approximately 30% over the entire wavelength range, which is in agreement with past experience with other metals.

As a result of the model used by Hiernaut et al. for the determination of normal spectral emissivity from their measurements, their value for normal spectral emissivity of rhenium at its melting point is a constant independent of the wavelength. This constant value is about 19% higher than our values in the same wavelength range. The results of Lin and Frohberg show a negative slope with a difference of about 7.5% between their reported measurements at 547 and 650 nm.

6. CONCLUSIONS

The present measurements for the melting-point radiance temperatures of rhenium and iridium have yielded highly reproducible results (within ± 1 K) at seven wavelengths in the range 521 to 1500 nm. These results are similar to those found for many other metals [1] and contribute additional data to suggest that the radiance temperature of high-temperature metals at their melting points is essentially constant and reproducible for different specimens of the metal. However, the melting plateaus of both these metals showed less stability and a greater tendency to exhibit positive or negative slopes compared to the melting behavior of niobium, molybdenum, and tungsten. This less than ideal melting behavior and the expense of these two materials make it less likely that either rhenium or iridium would be chosen as suitable candidates for the establishment of secondary temperature references.

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