

Radiance Temperatures (in the Wavelength Range 523–907 nm) of Group IVB Transition Metals Titanium, Zirconium, and Hafnium at Their Melting Points by a Pulse-Heating Technique

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The melting-point radiance temperatures (at six wavelengths in the range 523–907 nm) of the Group IVB transition metals titanium, zirconium, and hafnium 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 temperatures every 0.5 ms with a high-speed six-wavelength pyrometer. 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 as follows:

Titanium	Zirconium	Hafnium
1827 K at 527 nm	1982 K at 526 nm	2292 K at 523 nm
1803 K at 624 nm	1951 K at 622 nm	2254 K at 619 nm
1796 K at 652 nm	1941 K at 652 nm	2241 K at 651 nm
1780 K at 717 nm	1921 K at 715 nm	2216 K at 711 nm
1755 K at 810 nm	1889 K at 810 nm	2175 K at 808 nm
1730 K at 907 nm	1857 K at 906 nm	2134 K at 906 nm

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Based on estimates of the random and systematic errors arising from pyrometry and specimen conditions, the combined uncertainty (95% confidence level) in the reported values is about ± 8 K at each wavelength.

KEY WORDS: emissivity; hafnium; high-speed pyrometry; high-temperature fixed points; melting; multiwavelength pyrometry; radiance temperatures; titanium; zirconium.

1. INTRODUCTION

Earlier measurements [1] performed more than a decade ago at the National Institute of Standards and Technology (NIST) and at the Instituto di Metrologia "G. Colonnetti" (IMGC) showed that, for the refractory metals studied, melting-point radiance temperatures⁴ are essentially constant during the initial melting period and are highly reproducible for different specimens of the same metal, usually within a few degrees kelvin. However, these measurements were performed only at wavelengths near 650 nm (NIST) and 1000 nm (IMGC), and on a limited number of metals.

In an effort to study the wavelength dependence of radiance temperatures of the refractory metals in more detail, a high-speed pyrometer capable of making simultaneous measurements at multiple wavelengths was constructed at NIST [2] and was subsequently used to measure the melting-point radiance temperatures at six wavelengths (nominally in the range 500–900 nm) of niobium [3], molybdenum [4], tungsten [5], and tantalum [6]. It was found that the melting-point radiance temperatures at each wavelength for these metals also exhibited constancy during the initial melting period as well as a high degree of reproducibility, as in the earlier work.

In the present paper, we describe similar measurements of radiance temperature performed on the Group IVB transition metals titanium, zirconium, and hafnium at their melting points. The measurement technique is based on passing a large electrical current pulse through the specimen, causing it to undergo rapid resistive self-heating from room temperature to its melting point in less than 1 s, and measuring simultaneously the specimen radiance temperatures at six wavelengths (between 500 and 900 nm) every 0.5 ms. The melting-point radiance temperatures for a given specimen were determined by averaging the measured temperatures along the plateau in the radiance temperature versus time function for each wavelength. The melting-point radiance temperatures for titanium, zirconium,

⁴ Radiance temperature (sometimes referred to as brightness temperature) of the specimen surface is the temperature at which a blackbody has the same radiance as the surface, corresponding to the effective wavelength of the measuring pyrometer.

and hafnium were determined by averaging results for 16, 17, and 8 specimens, respectively. Details concerning the design and construction of the pulse-heating system [7, 8] as well as the design, operation, and calibration of the multiwavelength pyrometer [2] are given in the cited publications. All temperatures reported in this paper, except where explicitly noted otherwise, are based on the International Temperature Scale of 1990 (ITS-90) [9].

Table I. Impurities (ppm by Weight) in the Metals as Supplied by Each Manufacturer

Impurity	Ti	Zr	Hf
Al	0.6	50	<20
As	<0.1		
B	<0.1	<0.4	
C		120	<50
Ca	0.5	<30	
Cd		0.4	
Cl	0.4	<20	
Co	<0.1	<15	
Cr	1	100	
Cu	1	25	35
Fe	3	600	275
Hf		65	
K	2		
Li	0.1	<1	
Mg	0.5	<10	
Mn	2	<15	
Mo	1	<25	<30
Na	1	<10	
Nb	0.1		75
Ni	0.5	<40	
P	<0.1	<25	
Pb	0.3	<20	
Si	0.4	<25	
Sn		<100	
Ta			<100
Ti		25	<20
U		<3.5	<1
V	0.1	<30	
W	0.6	<30	<10
Zn	0.5	15	
Zr	0.1		3×10^4
Rare earths		<5	
H	72	25	4
N	197	35	25
O	546	1000	275
Purity	99.9%	99.8%	96.9%

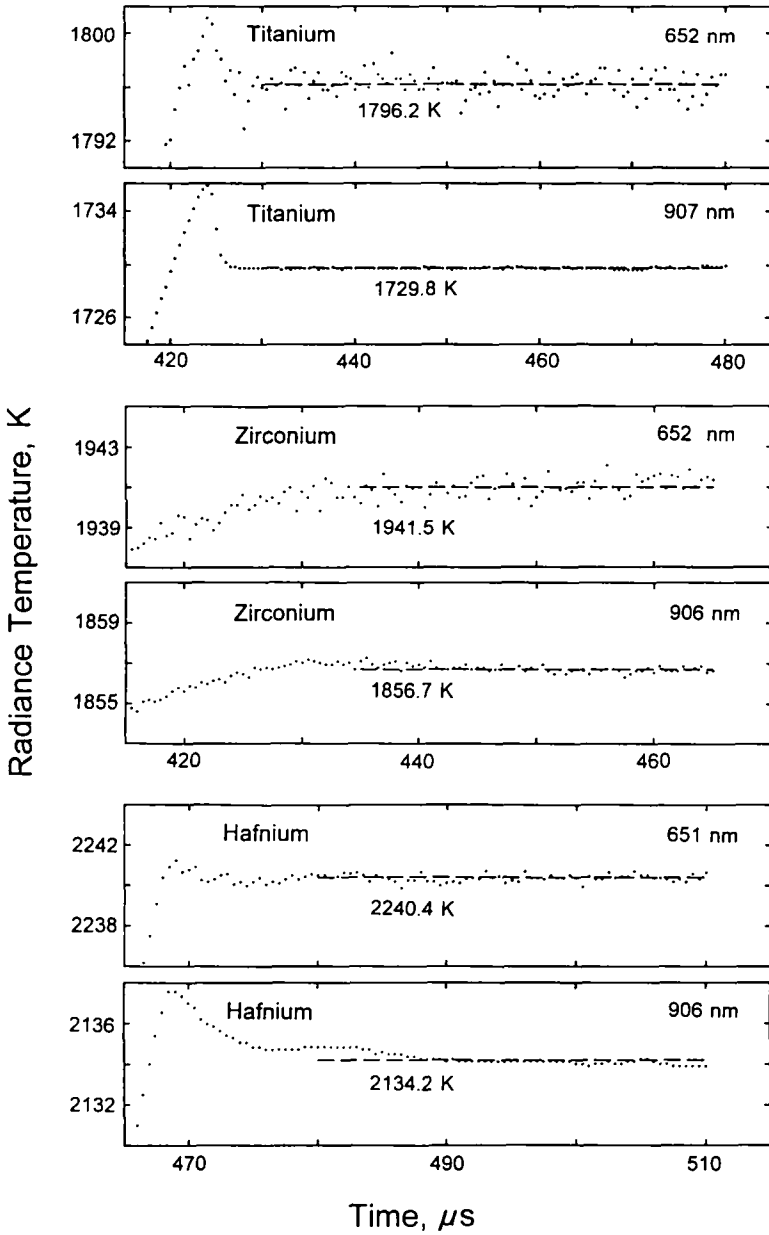


Fig. 1. Variation of the radiance temperatures of typical titanium, zirconium, and hafnium strip specimens at two representative wavelengths (near 650 and 900 nm) just before and during melting as measured by the six-wavelength pyrometer. The effective wavelengths shown on each graph were determined following the definition given by Kostkowski and Lee [10]. Each labeled temperature indicates the melting-point radiance temperature at the given wavelength.

2. MEASUREMENTS

Measurements of radiance temperature of the Group IVB metals at their respective melting points were performed on specimens in the form of strips fabricated from thin sheets (0.25-mm thickness) of 99.9% pure Ti and 99.8% pure Zr and a sheet (1.6-mm thickness) of 96.9% pure Hf. The specimen strips had nominal dimensions of 50-mm length \times 4-mm width. Table I lists the impurities in the source sheets supplied by the manufacturers, based on their analyses of typical stock material. To remove possible surface contaminants, the surfaces of each specimen strip were mechanically treated with abrasives, yielding surface roughnesses of approximately 0.15–0.2 μm (rms) for Ti, 0.15–0.5 μm (rms) for Zr, and 0.4 μm (rms) for Hf.

Each pulse-heating experiment was performed with the specimen strip in an argon gas environment (nominally at 0.2 MPa) to minimize evaporation and/or contamination of the specimen at high temperatures. The duration of the electrical current pulse, used to heat each specimen rapidly from room temperature to its melting point, ranged from approximately 210 to 600 ms for Ti, 180 to 700 ms for Zr, and 400 to 520 ms for Hf.

Figure 1 presents data obtained at two representative wavelengths (nominally 650 and 900 nm) by the six-wavelength pyrometer during pulse-heating experiments on titanium, zirconium, and hafnium near and at their respective melting points. The melting of a given specimen was manifested by a plateau in the radiance versus time function at each of the six wavelengths (nominally 500, 600, 650, 700, 800, and 900 nm). At each wavelength, the measured radiance temperature along the plateau was essentially constant, with the temperature difference between beginning and end of the plateau approximately in the range -1 to 2 K. The effective wavelength for each pyrometer channel was determined at the respective plateau radiance temperature following the definition of Kostkowski and Lee [10].

3. RESULTS

In our measurements on the Group IVB transition metals, well-formed melting plateaus were observed in only 16 of 29 melting experiments for titanium, 17 of 20 for zirconium, and only 8 of 19 for hafnium. For a given "successful" experiment, the plateau radiance temperature at each effective wavelength was determined by averaging the measured temperatures along the flat portion of the corresponding plateau. The trend (or slope) of radiance temperature along the flat portion was determined by fitting the measured temperatures by a linear function of time with the least-squares method. The specimen heating rates were determined by fitting linear functions of time to the radiance temperatures measured during the premelting

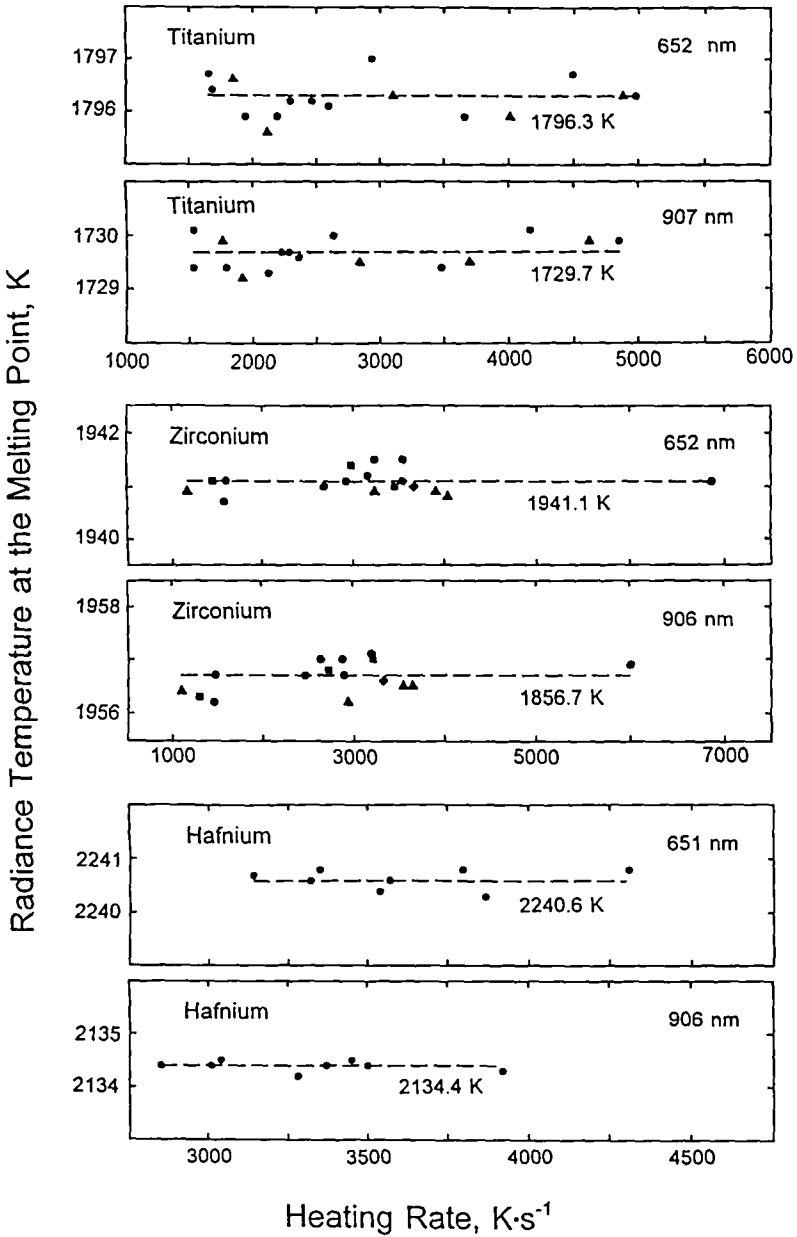


Fig. 2. Melting-point radiance temperatures as a function of the heating rate at two representative wavelengths (near 650 and 900 nm) for experiments performed on titanium, zirconium, and hafnium. Each dashed line and labeled temperature indicates the average melting-point radiance temperature at the given wavelength. The different symbols indicate results of experiments on specimens with different surface roughnesses.

period. Plateau radiance temperatures at two representative wavelengths are plotted against heating rate in Fig. 2. As can be seen, the plateau radiance temperatures do not depend on heating rate.

3.1. Titanium

The number of temperatures along the plateau used for averaging ranged from 41 to 161, depending on the heating rate and the behavior of the specimen during melting in a given experiment. The standard deviation of an individual temperature from the average is in the range 0.1 to 1.3 K. The slope of the plateau takes values between -20 and $46 \text{ K} \cdot \text{s}^{-1}$. The temperature difference between the beginning and end of the plateau, as determined from this slope, is in the range -0.6 to 1.3 K . The heating rates (slopes of the linear functions approximately 20 K below the melting plateaus) range from 1530 to $5500 \text{ K} \cdot \text{s}^{-1}$.

3.2. Zirconium

The plateau radiance temperatures were obtained by averaging from 21 to 121 temperatures along the plateau, depending on the experiment. The standard deviation of an individual temperature from the average is in the range 0.1 to 0.7 K. The slope of the plateau takes values between -21.4 and $115 \text{ K} \cdot \text{s}^{-1}$, corresponding to a temperature difference between the beginning and end of the plateau in the range -0.4 to 1.9 K . The heating rates (determined approximately 25 K below the melting plateaus) range from 1100 to $6800 \text{ K} \cdot \text{s}^{-1}$.

3.3. Hafnium

Plateau radiance temperatures were obtained by averaging from 31 to 91 temperatures along the plateau, depending on the experiment. The standard deviation of an individual temperature from the average is in the range 0.1 to 0.4 K. The slope of the plateau takes values between -35.8 and $22 \text{ K} \cdot \text{s}^{-1}$, corresponding to a temperature difference between the beginning and end of the plateau in the range -0.8 to 1.0 K . The heating rates (slopes of the linear functions approximately 15 K below the melting plateaus) range from 2850 to $4540 \text{ K} \cdot \text{s}^{-1}$.

The final results for radiance temperature of the Group IVB metals at their respective melting points, as presented in Table II, were obtained by averaging the values of plateau radiance temperature at each effective wavelength for the different specimens of a given metal. Depending on the wavelength, the standard deviation of an individual plateau radiance temperature from the average is in the range 0.3–0.4 K for titanium, 0.2–0.4 K

for zirconium, and 0.1–0.3 K for hafnium; the maximum absolute deviation from the average is in the range 0.5–0.7 K, 0.4–0.6 K, and 0.2–0.5 K, respectively. Also given in Table II are the corresponding values for normal spectral emissivity of these metals at their respective melting points that were calculated by means of Planck's law, on the basis of the present results for radiance temperature and literature values for the melting temperatures (T_m) of titanium [11], zirconium [12], and hafnium [13].

Table II. Final Results for the Average Radiance Temperature and Normal Spectral Emissivity (at Six Wavelengths) of Titanium, Zirconium, and Hafnium at Their Melting Points

Material and melting temp. ^a	Effective wavelength (nm) ^b	Radiance temp. (K) ^c	SD (K) ^d	Max. abs. dev. (K)	Norm. spectral emissivity (K) ^e
Titanium (16 specimens), $T_m = 1945$ K [11]					
	527	1826.7	0.3	0.5	0.403
	624	1803.1	0.3	0.5	0.393
	652	1796.3	0.4	0.7	0.391
	717	1779.8	0.3	0.5	0.384
	810	1755.1	0.3	0.5	0.372
	907	1729.7	0.3	0.5	0.362
Zirconium (17 specimens), $T_m = 2127$ K [12]					
	526	1981.5	0.3	0.5	0.389
	622	1950.6	0.2	0.5	0.374
	652	1941.1	0.2	0.4	0.370
	715	1920.8	0.3	0.5	0.362
	810	1889.1	0.4	0.6	0.349
	906	1856.7	0.3	0.5	0.337
Hafnium (8 specimens), $T_m = 2471$ K [13]					
	523	2292.1	0.3	0.5	0.421
	619	2253.7	0.3	0.4	0.405
	651	2240.6	0.2	0.3	0.400
	711	2216.1	0.2	0.3	0.391
	808	2175.1	0.2	0.3	0.376
	906	2134.4	0.1	0.2	0.364

^a Melting temperature based on ITS-90.

^b Determined at the respective radiance temperature following the definition of effective wavelength given by Kostkowski and Lee [10].

^c Average of the plateau radiance temperatures at each effective wavelength for each material.

^d Standard deviation of an individual plateau radiance temperature from the average of values obtained for the different specimens.

^e Determined by means of Planck's law from the average plateau radiance temperature and the respective melting temperature for each material.

4. ESTIMATE OF UNCERTAINTIES

The major error sources arise from (i) the calibration and operation of the pyrometer and (ii) the physical/chemical conditions and melting behavior of each specimen. Detailed analyses of the sources and magnitudes of random and systematic errors and the corresponding uncertainties in the measurement of melting-point radiance temperatures with the six-wavelength pyrometer are given elsewhere [2, 3]. Specific items in the error analysis were recomputed whenever the present conditions differed from those in the earlier publications. The resultant combined uncertainty in the reported values for melting-point radiance temperatures at each of the six wavelengths is estimated to be ± 8 K (at the 95% confidence level).

5. DISCUSSION

During the early to mid-1970s, Bonnell et al. [14] and Berezin et al. [15] used electromagnetic levitation/induction heating methods and conventional disappearing-filament optical pyrometry to measure the melting-point radiance temperatures of Ti and Zr near 650 nm. At about the same time, Cezairliyan, Righini, and their coinvestigators utilized subsecond pulse-heating techniques and high-speed photoelectric pyrometry to obtain melting-point radiance temperature data on Ti [16], Zr [12], and Hf [13] at wavelengths near 650 nm (performed at NIST) and on Ti [16] near 1000 nm (performed at IMG). These investigators also reported values for normal spectral emissivity, which were obtained from the measured surface radiance temperatures on the basis of Planck's law and an independent knowledge of the true melting temperatures.

Several years ago, Hiernaut et al. [17] performed measurements on laser-pulse heated specimens with a high-speed six-wavelength pyrometer in order to obtain emissivity data for Zr and Hf and several other refractory metals (including V, Nb, Mo, Ta, and W) during rapid cooling of the specimens from temperatures above their melting points. Using a model for the evaluation of temperature and spectral emissivities based on an assumed relationship between emissivity and wavelength, the investigators determined that, for the wavelength range 500–1000 nm, these metals are effectively "gray" at their melting point T_m , i.e., their emissivities $\epsilon_{N\lambda}$ are independent of λ ($d\epsilon_{N\lambda}/d\lambda = 0$); also, they determined that, in all cases, the slope $d\epsilon_{N\lambda}/d\lambda$ changed sign at (or very near) the melting temperature, i.e., their emissivities are characterized by $d\epsilon_{N\lambda}/d\lambda > 0$ at $T > T_m$. Their model yielded values of T_m for Zr and Hf that are higher than the literature values in Table II by about 10 and 40 K, respectively.

Table III. Radiance Temperatures and Normal Spectral Emissivities at Wavelengths (λ) in the Range 500–1000 nm of Titanium, Zirconium, and Hafnium at Their Melting Points as Reported in the Literature

Element	Investigator	Ref. No.	Year	Purity (wt %)	Heating method ^a	λ (nm)	Radiance temperature (K)			Normal spectral emissivity	
							As reported	On ITS-90	As reported ^b	As reported ^b	As reported ^b
Titanium	Bonnell et al.	14	1972	99.95	E	645	1814 ± 7	1814 ± 7		0.434	
	Berezin et al.	15	1976	99.91	E	650	1801 ± 4	1801 ± 4		0.412	
	Righini et al.	16	1977	99.9	R	653	1800 ± 6	1800 ± 6		0.401	
						997	1711 ± 6	1711 ± 6		0.362	
	Krishnan et al.	18	1991	99.95	E	632.8		1768 ^c		0.310 ^d	
	Present work			99.9	R	527		1827 ± 8		0.403	
					624		1803 ± 8		0.393		
					652		1796 ± 8		0.391		
					717		1780 ± 8		0.384		
					810		1755 ± 8		0.372		
					907		1730 ± 8		0.362		
Zirconium	Bonnell et al.	14	1972	99.9	E	645	1918 ± 7	1917 ± 7		0.318	
	Cezairliyan and Righini	12	1975	99.98	R	650	1940 ± 8	1939 ± 8		0.367	
	Hiernaut et al.	17	1989	99.8	L	500		1992 ^e		0.37	
						650		1952 ^e		0.37	
					1000		1864 ^e		0.37		

Krishnan et al. Present work	19	1993	98.9 ^a 99.8	E R	633 526 622 652 715 810 906	1933 ^c 1933 ^c	1933 ^c 1981 ± 8 1951 ± 8 1941 ± 8 1921 ± 8 1889 ± 8 1857 ± 8	0.345 ^d 0.389 0.374 0.370 0.362 0.349 0.337
Hafnium Cezairiyani and McClure Hiernaut et al.	13 17	1976 1989	96.97 ^e 99.9 ^b	R L	653 500 650 1000	2236 ± 10 2320 ^f 2269 ^f 2157 ^f	2235 ± 10 22320 ^f 2269 ^f 2157 ^f 2292 ± 8 2254 ± 8 2241 ± 8 2216 ± 8 2175 ± 8 2134 ± 8	0.39 0.39 0.39 0.39 0.421 0.405 0.400 0.391 0.376 0.364
Present work			96.97	R	523 619 651 711 808 906			

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

^b Values for emissivity are given as reported by the investigators.

^c Based on the emissivity (0.310) measured by the investigators and a melting temperature of 1945 K (on ITS-90) for titanium [11].

^d Emissivity of the liquid measured directly by laser polarimetry.

^e Based on the constant emissivity (0.37 for the wavelength range 500–1000 nm) and the melting temperature (2139 K on ITS-90) measured by the investigators.

^f Based on the emissivity (0.345) and a melting temperature of (2125 K on ITS-90) measured by the investigators.

^g Includes 3% wt zirconium.

^h Zirconium content not reported by the investigators.

ⁱ Based on the constant emissivity (0.39 for the wavelength range 500–1000 nm) and the melting temperature (2511 K on ITS-90) measured by the investigators.

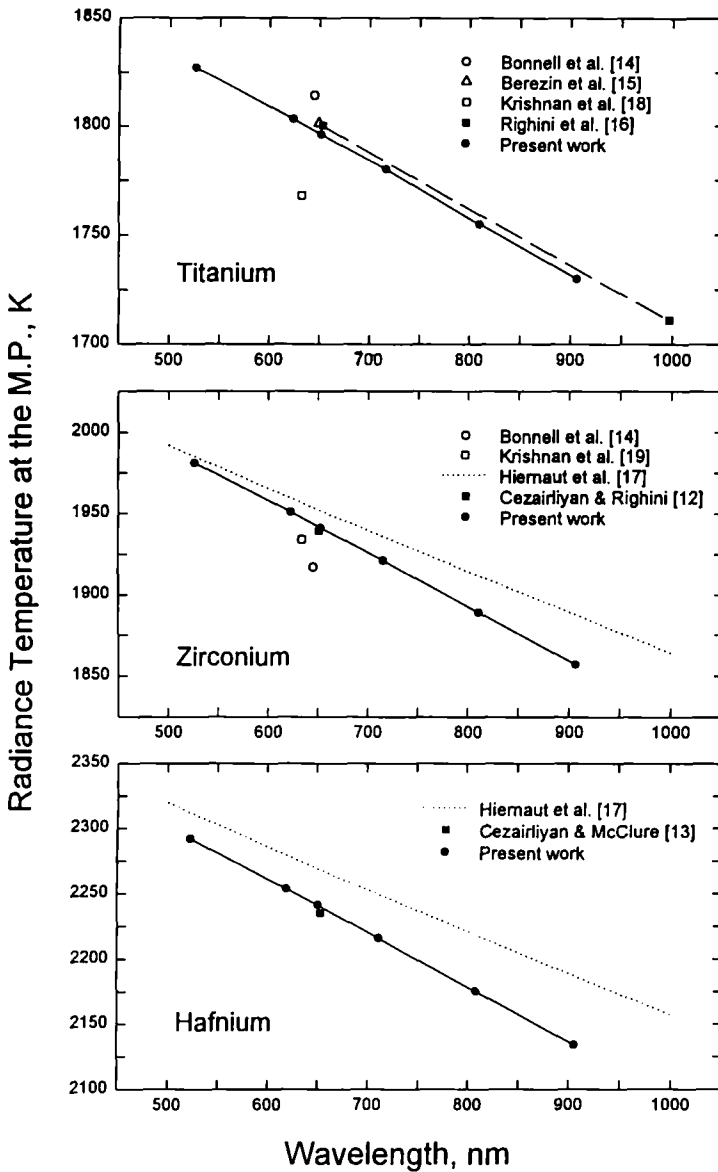


Fig. 3. Comparison (on ITS-90) of literature values and present results for the radiance temperature of titanium, zirconium, and hafnium at their melting points. The symbol types refer to methods used for specimen heating, as follows: filled symbols, resistive self-heating; open symbols, electromagnetic levitation/induction heating; dotted curve, laser pulse heating.

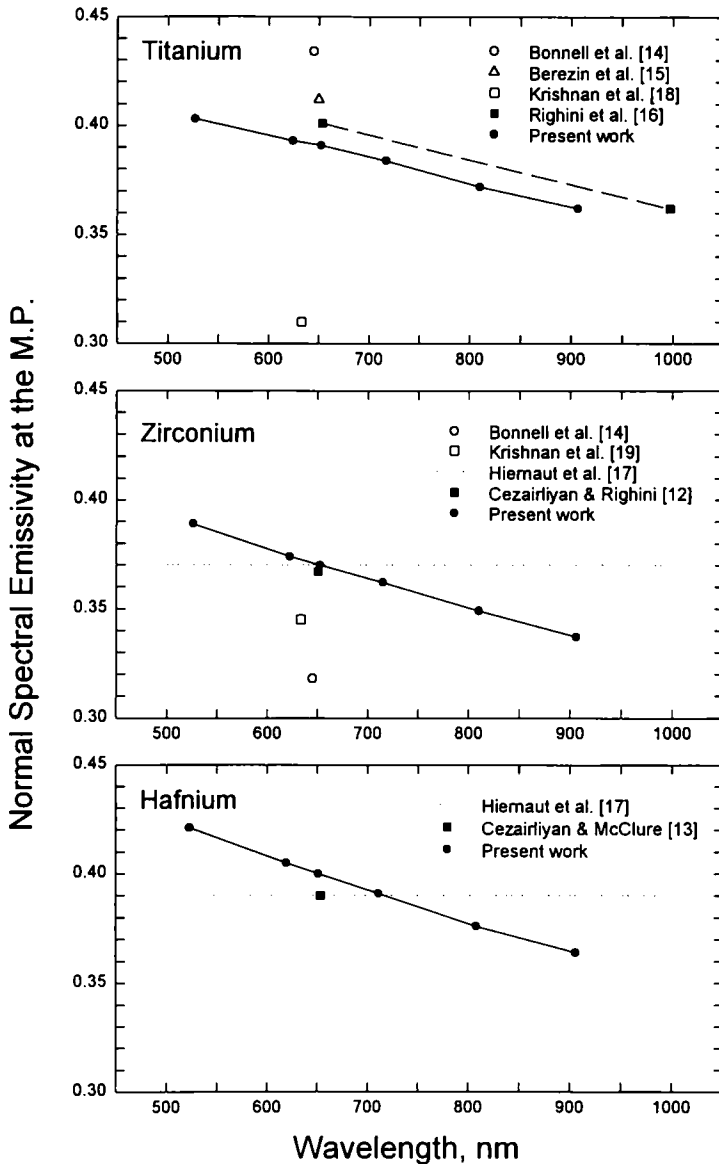


Fig. 4. Variation of the normal spectral emissivity of titanium, zirconium, and hafnium at their melting points, as reported by different investigators, as a function of wavelength. The symbol designations are the same as those given in the legend to Fig. 3.

Recently, Krishnan and coinvestigators measured normal spectral emissivities (at 632.8 nm) of liquid Ti [18] and Zr [19] using a newly developed laser-polarimetry technique in experiments which utilized electromagnetic levitation/heating supplemented by CO₂ laser beam heating. Also, on the basis of their emissivity data and measurements of radiance temperature with an automatic optical pyrometer, they obtained (via Planck's law) a value for T_m of Zr that is 2 K lower than the literature value given in Table II.

A comparison of the reported values for radiance temperature and normal spectral emissivity of Ti, Zr, and Hf at their melting points with the present results is given in Table III. Figures 3 and 4 present these data (on ITS-90) as a function of wavelength. As can be seen, the present results indicate that the melting-point emissivities of titanium, zirconium, and hafnium change by approximately 15% in the wavelength interval 500–900 nm. Recent measurements in our laboratory on melting-point radiance temperatures of Nb [3], Mo [4], W [5], and Ta [6] also show a similar strong wavelength dependence for their normal spectral emissivities. Specific comments on the comparison of results from different investigations follow for each of the Group IVB metals.

5.1. Titanium

The radiance temperature data obtained by Berezin et al. [15] and by Righini et al. [16] are in good agreement with the present results, whereas the melting-point radiance temperature measured by Bonnell et al. [14] is 16 K higher, which is approximately the same magnitude as the combined experimental uncertainties. The radiance temperature derived from the data on emissivity reported by Krishnan et al. [18] and a literature value for T_m [11] is 28 K lower than the present data.

5.2. Zirconium

The radiance temperature data obtained by Cezairliyan and Righini [2] are in excellent agreement with the present results, whereas the melting-point radiance temperature measured by Bonnell et al. [14] is 25 K lower, which is somewhat larger than the combined experimental uncertainties. The radiance temperature derived from the data on emissivity and T_m reported by Krishnan et al. [19] is 15 K lower than the present data, a difference that is within the combined experimental uncertainties. The radiance temperatures based on the constant emissivity ($\epsilon_{N_z} = 0.37$) and T_m measured by Hiernaut et al. [7] show a different trend with changing wavelength than the present results. Their finding that $de_{N_z}/d\lambda = 0$ at

$T = T_m$ for Zr (as well as for the other metals studied) appears to be in disagreement with a recent review [20] of literature data which indicates that, at T_m , the normal spectral emissivities of high-temperature metals have a negative slope ($d\epsilon_{N\lambda}/d\lambda < 0$). The present results on emissivity of Zr yield a value $d\epsilon_{N\lambda}/d\lambda = -1.4 \times 10^{-4} \text{ nm}^{-1}$ at $T_m = 2127 \text{ K}$, which is in good agreement with data on liquid Zr obtained recently by Krishnan et al. [21] using pulsed dye laser polarimetry. Their results for the nominal wavelength region 400–900 nm yield a value $d\epsilon_{N\lambda}/d\lambda = -1.6 \times 10^{-4} \text{ nm}^{-1}$ at $T = 2335 \text{ K}$. This indicates that, at least in the case of Zr, the slope of the emissivity function ($d\epsilon_{N\lambda}/d\lambda$) is negative not only in the solid phase but also at T_m and in the near liquid phase.

5.3. Hafnium

The radiance temperature data obtained near 650 nm by Cezairliyan and McClure [13] is approximately 5 K lower than the present results, a difference that is within the experimental uncertainty. As in the case of Zr, radiance temperatures based on the wavelength-independent emissivity ($\epsilon_{N\lambda} = 0.39$) and T_m obtained by Hiernaut et al. [17] for Hf show a different trend with changing wavelength than the present results.

Possible sources for at least some of the large differences in results obtained by different investigators are (i) changes in the specimen surface morphology during the solid–liquid transition that may depend on heating method and/or (ii) formation of contaminants (oxides, nitrides, etc.) on the specimen surface, particularly during quasi-steady-state experiments in which highly reactive Group IVB metals are exposed to elevated temperatures for long periods of time (minutes to hours). The above-mentioned review of the literature data [20] on high-temperature metals shows that a similar strong wavelength dependence for emissivity is obtained from measurements using both rapid pulse-heating techniques, in which data were taken only during initial solid melting, and electromagnetic levitation/induction heating techniques, in which specimens were heated into their liquid state. In the present work, highly reproducible results (within $\pm 2 \text{ K}$) were obtained from measurements on specimens with surfaces that had been mechanically abraded to different roughnesses, and also at different heating rates. This high degree of reproducibility suggests that preexisting surface contaminants on the specimens were largely removed by the mechanical abrasion and that, during the subsecond-duration experiments, there was minimal contamination of the specimen surface by residual impurities in the argon gas environment.

6. CONCLUSIONS

As in earlier pulse experiments performed recently in our laboratory on strip specimens of niobium [3], molybdenum [4], tungsten [5], and tantalum [6], highly reproducible results (within ± 2 K) have been obtained for melting-point radiance temperatures of the Group IVB transition metals at six wavelengths in the nominal range 500–900 nm. For the first three metals, similar pulse-heating experiments performed with different measurement and calibration systems at NIST and IMGIC over a span of nearly 20 years have also yielded results in very good agreement, usually within a few degrees kelvin. However, the agreement between results obtained in different investigations performed at NIST and IMGIC on the Group IVB transition metals (and tantalum) is not as good, differing by several degrees kelvin. This less-than-favorable agreement was manifested by difficulties in obtaining well-formed melting plateaus during the pulse-heating experiments. This could be due, at least in part, to the high reactivity of tantalum and the Group IVB transition metals at elevated temperatures.

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