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Radiance Temperatures (in the Wavelength Range 525 to 906 nm) of Vanadium at Its Melting Point by a Pulse-Heating Technique

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The radiance temperatures (at six wavelengths in the range 525 to 906 nm) of vanadium at its melting point 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-vs-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 vanadium as determined by averaging the results at each wavelength for 16 specimens (standard deviation in the range 0.3 to 0.4 K, depending on the wavelength) are 2030 K at 525 nm, 1998 K at 622 nm, 1988 K at 652 nm, 1968 K at 714 nm, 1935 K at 809 nm, and 1900 K at 906 nm. Based on estimates of the random and systematic errors that arise from pyrometry and specimen conditions, the resultant uncertainty (2 SD level) in the reported values is about ± 7 K at each wavelength.

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

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1. INTRODUCTION

A high-speed multiwavelength pyrometer [1], capable of simultaneously measuring radiance temperatures[3] at six wavelengths (nominally in the range 500 to 900 nm), has been used to study the wavelength dependence of the radiance temperature at the melting points of selected refractory metals: niobium [2], molybdenum [3], tungsten [4], and tantalum [5]. From these measurements it was found that, during the initial melting period, the radiance temperature at each wavelength was essentially constant and was very reproducible for different specimens of a given metal. These findings suggested that melting-point radiance temperatures of selected metals may provide easily realizable high-temperature references for secondary calibrations of optical pyrometers and for *in situ* temperature checks on complicated measurement systems [6]. In an effort to determine the suitability of other metals for these purposes, particularly those with lower melting temperatures, the six-wavelength pyrometer was modified to increase its sensitivity and was used in melting-point radiance temperature measurements on the Group IVB metals, titanium, zirconium, and hafnium [7]. The purpose of this paper is to describe similar measurements on vanadium.

The measurement method is based on rapid resistive self-heating of the specimen from room temperature to its melting point in less than 1 s by passing a large electrical current pulse through the specimen. As the specimen heats, its radiance temperature at each of six wavelengths (between 525 and 906 nm) is measured simultaneously every 0.5 ms. The melting-point radiance temperatures for a given specimen are determined by averaging the measured temperatures along the melting plateau in the radiance temperature-versus-time function for each wavelength. Details concerning the design and construction of the pulse-heating system [8, 9] as well as the design, operation, and calibration of the six-wavelength pyrometer [1] are given in the earlier publications. All temperatures reported in this paper, except where explicitly noted otherwise, are based on the International Temperature Scale of 1990 (ITS-90) [10].

2. MEASUREMENTS

Measurements of the radiance temperature of vanadium at its melting point were performed on 16 specimens in the form of strips cut from

[3] 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.

50×50-mm sheets of 99.7 + % vanadium. As reported by the manufacturer, a typical analysis of the material yielded the following impurities in ppm (by mass): Si, 440; Mo, 410; Al, 340; Fe, 210; O, 250; Nb, 190; C, Cr, Sn, and Ta, each less than 100; Cu, Hf, N, Ni, P, S, Ti, W, and Zr, each less than 50; B and H, each less than 5; and U, less than 1. The nominal dimensions of each specimen strip were 50-mm length, 3-mm width and 0.25-mm thickness. The surfaces of most of the specimens were mechanically abraded with sandpaper in order to remove possible surface contaminants. Three grades of abrasive were used, yielding surface roughnesses (rms) of approximately 0.15, 0.2, and 0.4 μm. Also, experiments were performed on two specimens with "as-received" surface conditions (approximately 0.1 μm in rms roughness). Each experiment was performed with the vanadium strip in an argon gas environment (approximately 0.15 MPa) to minimize contamination of the specimen surface at high temperatures. Duration of the electrical current pulse, used to heat each specimen from room temperature to its melting temperature, ranged from approximately 290 to 680 ms.

Typical data obtained during a pulse-heating experiment are presented in Fig. 1, which shows the variation of radiance temperature as measured by the six-wavelength pyrometer as a function of time near and at the melting point of vanadium. The melting of the specimen is manifested by the plateau region in the radiance-versus-time results for each wavelength. As shown in Fig. 1, the radiance temperature during melting exhibits a small extended overshoot before settling down into a flat region (indicated by the dashed line for each wavelength). The radiance temperature along this flat region is essentially constant at each wavelength, with a temperature difference between beginning and end of the region of approximately 1 K or less. The effective wavelength (as defined by Kostkowski and Lee [11]) for each pyrometer channel was determined at the respective average radiance temperature and is shown in Fig. 1.

3. RESULTS

For each specimen, the plateau radiance temperature at each effective wavelength was determined by averaging the measured temperatures along the flat portion of the corresponding plateau (indicated by the dashed line in Fig. 1). The number of temperatures used for averaging ranged from 21 to 91, depending on the heating rate and the behavior of the specimen during melting. The standard deviation of an individual temperature from the average radiance temperature is in the range 0.1 to 0.4 K for all 16 experiments.

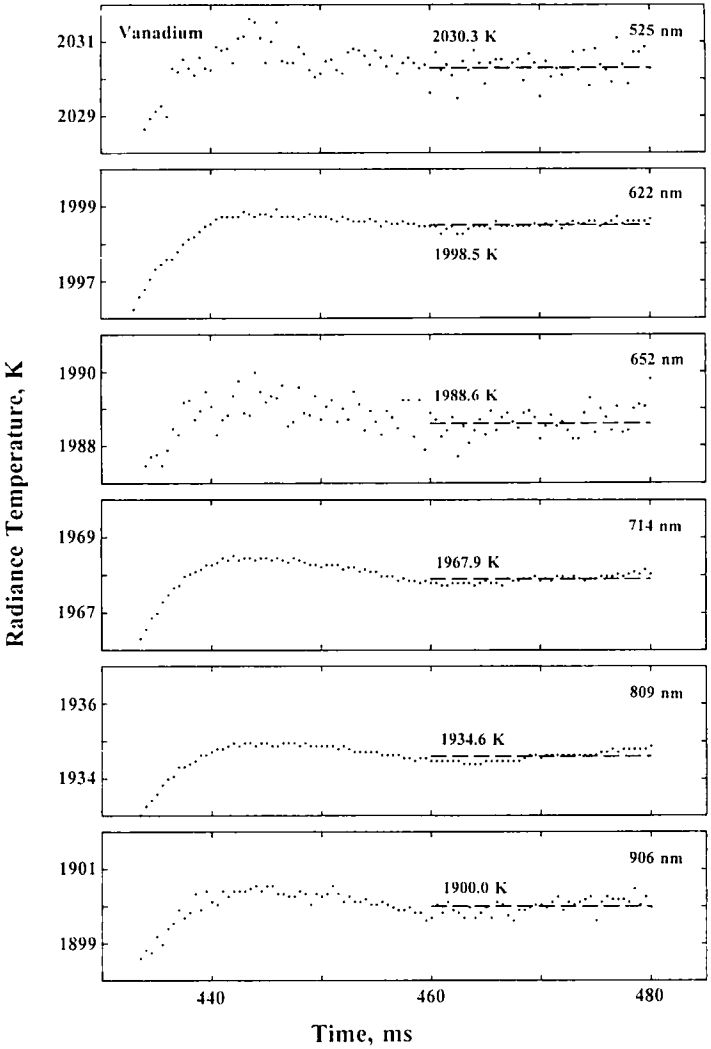

Fig. 1. Variation of the radiance temperatures just before and during melting as functions of time as measured by the six-wavelength pyrometer during a typical experiment. The effective wavelengths, shown for each channel, were determined following the definition given by Kostkowski and Lee [11]. Each labeled temperature indicates the average melting-point radiance temperature for this experiment computed from the plateau data indicated by the dashed line.

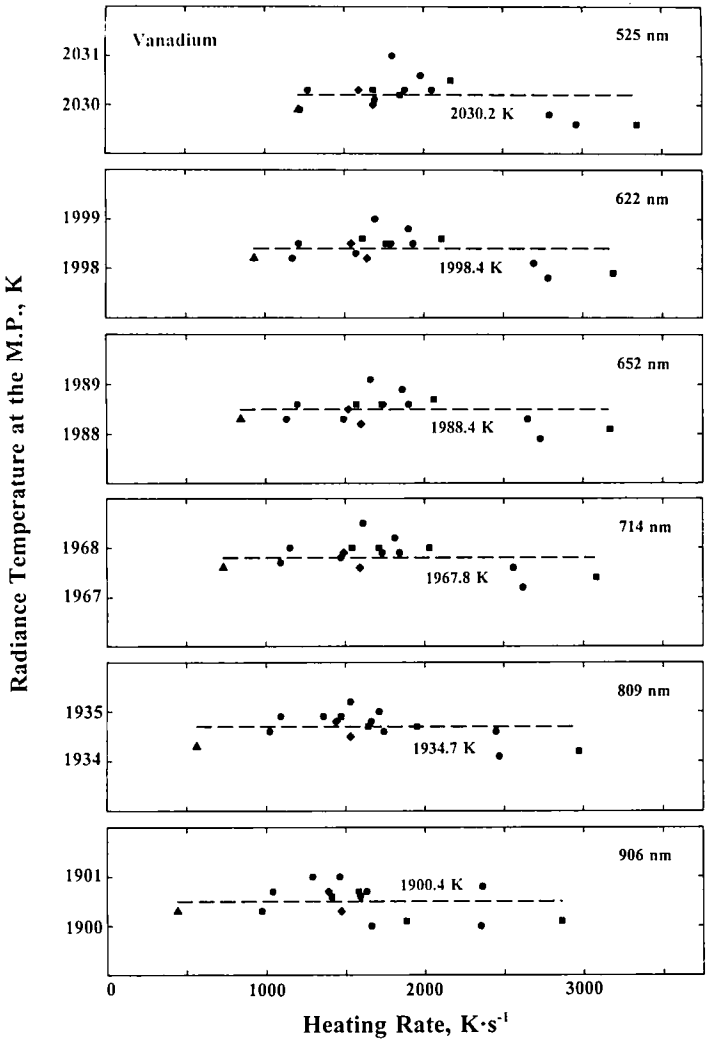

Fig. 2. Melting-point radiance temperatures as a function of the heating rate for sixteen experiments on vanadium. Each dashed line and labeled temperature indicates the average melting-point radiance temperature at a given wavelength for 16 vanadium experiments. The symbols refer to the approximate initial surface roughness (rms) of the specimens prior to melting as follows: ◆, 0.1 μm; ●, 1.5 μm; ▲, 0.2 μm; ■, 0.4 μm.

The trend (or slope) of radiance temperature along each plateau was determined by fitting a linear function (in time) to the measured radiance temperatures using the least-squares method. The slope of the linear function for all the experiments was in the range from -20.1 to $43.8\ K \cdot s^{-1}$. The temperature difference between the beginning and the end of the plateau, as determined from this slope, was in the range -0.6 to $1.0\ K$. 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 (slopes of the linear functions approximately 20 K below the melting plateaus) ranged from 440 to $3340\ K \cdot s^{-1}$.

The melting-point radiance temperatures for 16 experiments at each effective wavelength are plotted against heating rate in Fig. 2. As can be seen, variation of the plateau radiance temperature is less than 1 K for the range of heating rates used in this study. Figure 2 also shows that the results for radiance temperature do not depend on the initial roughness of the specimen surface. Table I presents the final experimental results of the radiance temperature of vanadium at its melting point at each effective wavelength. The standard deviation of an individual plateau radiance temperature for a specimen from the average obtained for 16 specimens is in the range from 0.3 to 0.4 K, depending upon the wavelength, and the maximum absolute deviation is 0.8 K. Also given in Table I are the corresponding values for the normal spectral emissivity of vanadium at its melting

Table I. Final Results for the Average Radiance Temperatures and Normal Spectral Emissivities, at Six Wavelengths, of Vanadium at Its Melting Point

Effective wavelength (nm)[a]	Radiance temp. (K)[b]	SD (K)[c]	Max. abs. dev. (K)	Normal spectral emissivity[d]
525	2030.2	0.4	0.8	0.351
622	1998.4	0.3	0.6	0.345
652	1988.4	0.3	0.6	0.343
714	1967.8	0.3	0.7	0.338
809	1934.7	0.3	0.6	0.329
906	1900.4	0.3	0.5	0.319

[a] Determined at the respective radiance temperature following the definition of effective wavelength given by Kostkowski and Lee [11].

[b] Average of the plateau radiance temperatures at each effective wavelength for 16 vanadium experiments.

[c] Standard deviation of an individual plateau radiance temperature for a specimen from the average of values obtained for 16 specimens.

[d] Determined by means of Planck's law from the average plateau radiance temperature and the value 2201 K (on ITS-90) for the melting point of vanadium [12].

point that were calculated by means of Planck's law using the present results for radiance temperature and the value 2201 K for the melting point of vanadium [12].

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 [2]. Specific items in the uncertainty were recomputed whenever the present conditions differed from those in the earlier publication. The resultant uncertainty in the reported values for melting-point radiance temperatures at each of the six wavelengths is estimated to be ± 7 K (2 SD level).

5. DISCUSSION

Radiance temperatures at the melting point of vanadium as reported in the literature along with the present results are listed in Table II. To provide a common basis for comparison, the reported values for the radiance temperature at the melting point were adjusted to ITS-90. The results are plotted as a function of wavelength in Fig. 3. Also listed in Table II are the reported values for normal spectral emissivity. Because the values for normal spectral emissivity depend upon the choice of the melting temperature, the reported values for emissivity were adjusted to a common melting temperature (2201 K on ITS-90 [12]) and are plotted as a function of wavelength in Fig. 4.

The earliest experiments performed to determine the normal spectral emissivity of vanadium at its melting point used electromagnetic levitation/induction heating methods and conventional disappearing-filament optical pyrometry at wavelengths near 650 nm. The results of Treverton and Margrave [13] and those of Berezin et al. [15] are 6 K higher and 1 K lower, respectively, than the present results near 650 nm. Both results are well within the combined uncertainties of the respective investigations. The results of Bonnell et al. [14] at 645 nm are among the lowest at this wavelength, 16 K lower than the present results. The more recent results of Lin and Frohberg [18], using heating and measurement techniques similar to those in these earlier investigations, are 30 K higher (at 650 nm) than the present results, a result much greater than the combined uncertainties of the two investigations. Their result for normal spectral emissivity at 650 nm is from 14% to almost 30% higher than that reported by other

Table II. Radiance Temperatures and Normal Spectral Emissivities at Wavelengths (λ) in the Range 500 to 1000 nm of Vanadium at Its Melting Point as Reported in the Literature

Investigator(s)	Ref. No.	Year	Purity (mass. %)	Heating method[a]	λ (nm)	Radiance temperature (K)		Normal spectral emissivity	
						As reported	On ITS-90	As reported	Adjusted for a common MP[b]
Treverton & Margrave	13	1971	99.7	E	650	1995	1994	0.400	0.352
Bonnell et al.	14	1972	99.9	E	645	1973	1972	0.343	0.308
Berezin et al.	15	1976		E	650	1988 ± 4	1987 ± 4	0.358	0.339
Cezairliyan et al.	16	1979	99.9+	R	653	1992 ± 7	1991 ± 7	0.363	0.348
					993	1875 ± 7	1874 ± 7	0.325	0.317
Hiernaut et al.	17	1989	99.5	L	500		2018[c]	0.31	0.306
					650		1970[c]	0.31	0.307
					1000		1865[c]	0.31	0.308
Lin & Frohberg	18	1991	99.9	E	547	2049	2048	0.410	0.410
					650	2019	2018	0.402	0.402
Present work			99.7+	R	525		2030 ± 7		0.351
					622		1998 ± 7		0.345
					652		1988 ± 7		0.343
					714		1968 ± 7		0.338
					809		1935 ± 7		0.329
					906		1900 ± 7		0.319

[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] Adjusted values for emissivity are based on the reported melting-point radiance temperatures (on ITS-90) and the melting temperature of 2201 K (on ITS-90) for vanadium [12].

[c] Based on the constant emissivity (0.31 for the wavelength range 500 to 1000 nm) and the melting temperature (2199 K on ITS-90) reported by the investigators [17].

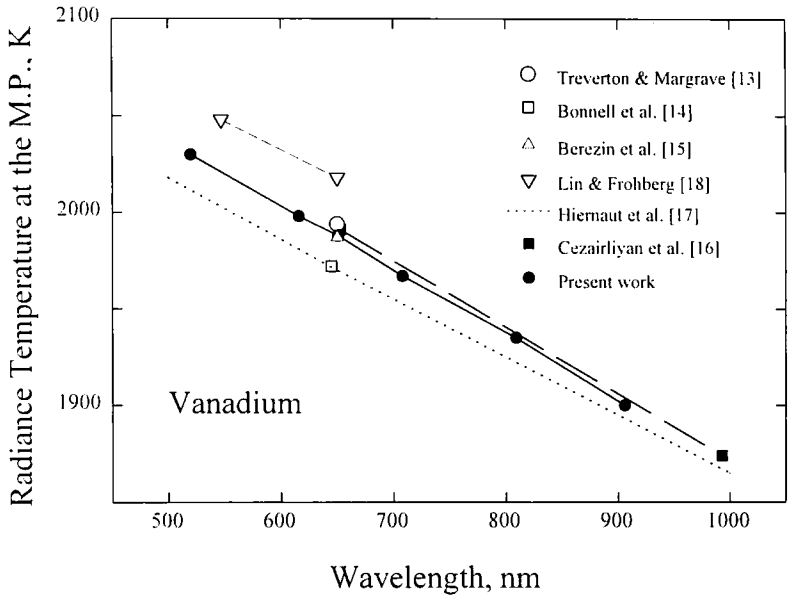

Fig. 3. Comparison (on ITS-90) of literature values and present results for the radiance temperature of vanadium at its melting point. The symbol types refer to methods used for specimen heating, as follows: filled symbols, resistive self-heating; open symbols, electromagnetic levitation induction heating;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

investigators, suggesting the possibility that the surface of their samples were contaminated.

Pulse-heating techniques and high-speed pyrometry were used earlier by Cezairliyan et al. [16] to measure the melting-point radiance temperature of vanadium strips. The measurements, performed at the National Institute of Standards and Technology (NIST) and at the Istituto di Metrologia "G. Colonnetti" (IMGC), used high-speed pyrometers operating at single wavelengths of 653 nm (NIST) and 993 nm (IMGC). As shown in Fig. 3, the earlier results are in good agreement with the present results. The earlier value at 653 nm is about 3 K higher than the interpolated value at 653 nm based on the present results and the earlier value at 993 nm is only 2 K higher than the extrapolated value at 993 nm based on the present results. The good agreement between these two sets of results (performed about 15 years apart with different pyrometers) and the excellent reproducibility of each set to within ± 1 K, suggest that mechanical abrasion of the specimen surface and utilization of rapid heating techniques ensure reliability of radiance temperature measurements.

The investigation of the normal spectral emissivity of vanadium by Hiernaut et al. [17] does not report results on the radiance temperature at

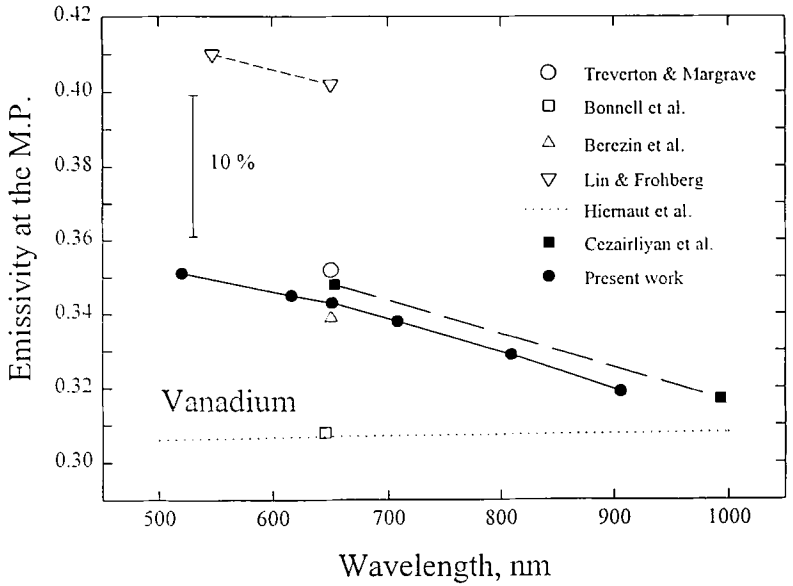

Fig. 4. Variation of the normal spectral emissivity of vanadium at its melting point as a function of wavelength. The symbol types have the same meaning as given in the legend to Fig. 3. The plotted data correspond to emissivity values adjusted for a common melting temperature of 2201 K (on ITS-90) [12].

the melting point. The values shown in Table II were computed from their reported values of normal spectral emissivity at the melting point and their measured melting temperature. Their technique, which has been applied to several other metals with high melting temperatures (including Nb, Mo, Ta, and W), involves melting a small spherical specimen with a laser pulse and measuring its radiance temperatures with a six-wavelength pyrometer as it cools and resolidifies. From an assumed relationship between emissivity and wavelength, the measured radiance data are used to determine values for the true melting temperature of the specimen and its normal spectral emissivities at the six wavelengths. As a result of their model, these investigators found that the emissivities at the melting point (during resolidification) of not only vanadium, but of all the refractory metals studied, are independent of wavelength over the wavelength range 500 to 1000 nm. This finding appears to be in significant disagreement with a recent review of the literature data by Cezairliyan et al. [19], which shows that normal spectral emissivity of high-melting-temperature metals at their melting points decreases monotonically with increasing wavelength. As shown in Fig. 4, the present results for the emissivity of vanadium exhibit a change of almost 10% between the wavelengths 525 and 906 nm. This

difference cannot be attributed realistically to temperature measurement error since, for example, an uncertainty of 7 K in radiance temperature corresponds to a relative uncertainty of less than 4% in emissivity. Figure 4 also shows that the earlier results of Cezairliyan et al. [16] show the same wavelength dependence as the present results and the results of Lin and Frohberg [18] at two wavelengths exhibit a slope very similar to the present results.

Ronchi et al. [20] has suggested that this disagreement may be explained by differences they observe in emissivity behavior between melting of the solid and the freezing of the liquid. Their data for melting of the solid indicate an emissivity change over the 500- to 1000-nm range of about 5%, which may suggest that there could be changes in the morphology of the specimen surface during the solid–liquid transition that depend on the heating method used. However, as Fig. 4 shows, emissivity results obtained from both rapid pulse-heating techniques, in which data were taken only during initial melting of the solid, and electromagnetic levitation/induction heat techniques, in which data were taken as the solid melts into its liquid state, show the same strong dependence on wavelength.

Additional insight into this problem could be obtained in a number of ways. If the actual radiance temperature data from the resolidification experiments on laser-heated specimens were published, it would have been possible to compare them directly with the results obtained from pulse-heating and electromagnetic levitation/induction heating experiments. It should also be possible to cycle between melting of the solid and freezing of the liquid during electromagnetic levitation/induction heating experiments and to monitor the radiance temperature for differences between the two processes.

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