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Radiance Temperatures at 1500 nm of Niobium and Molybdenum at Their Melting Points by a Pulse-Heating Technique[1]

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Radiance temperatures at 1500 nm of niobium and molybdenum at their melting points were measured by a pulse-heating technique. The method is based on rapid resistive self-heating of the strip-shaped specimen from room temperature to its melting point in less than 1 s and measuring the specimen radiance temperature every 0.5 ms with a high-speed infrared pyrometer. Melting of the specimen was manifested by a plateau in the radiance temperature-versus-time function. The melting-point radiance temperature for a given specimen was determined by averaging the measured values along the plateau. A total of 12 to 13 experiments was performed for each metal under investigation. The melting-point radiance temperatures for each metal were determined by averaging the results of the individual specimens. The results for radiance temperatures at 1500 nm are as follows: 1983 K for niobium and 2050 K for molybdenum. Based on the estimates of the uncertainties arising from the use of pyrometry and specimen conditions, the combined uncertainty (two standard-deviation level) in the reported values is ± 8 K.

KEY WORDS: emissivity (normal spectral); high-speed pyrometry; high-temperature reference points; melting; molybdenum; niobium; radiance temperature.

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1. INTRODUCTION

Melting-point radiance temperatures,[5] and related normal spectral emissivities, of selected pure metals have been suggested as possible candidates for high temperature references for use in relation to optical temperature measurements [1]. The successful utilization of this scheme requires an accurate knowledge of the wavelength dependence of the radiance temperatures, and normal spectral emissivities, of the selected metals. In the past two decades, extended measurements in the wavelength range 500–1000 nm have been made at the National Institute of Standards and Technology (NIST) in the United States and at the Istituto di Metrologia "G. Colonnetti" (IMGC) in Italy [1].

The objective of the present paper is to report extension of the radiance temperature measurement capabilities to longer wavelengths, 1500 nm. In order to achieve this, a high-speed infrared pyrometer was constructed. The pyrometer incorporates an InGaAs photodiode as the detector. It has been shown [2] that this type of detector has a satisfactory level of linearity and stability. In the present work, radiance temperatures at 1500 nm of niobium and molybdenum at their respective melting points were measured.

2. MEASUREMENT SYSTEM

A functional diagram of the high-speed system that is used for performing measurements at high temperatures is shown in Fig. 1. The specimen was heated from room temperature to its melting point in less than 1 s by the passage of a high-current pulse through it. The heating rate of the specimen was adjusted by adjusting the voltage of the battery bank and the Inconel resistor in series with the specimen. The switch and the electronic equipment were controlled by time-delay pulse generators. Details concerning the construction and operation of the pulse heating system are given in earlier publications [3, 4].

The high-speed radiance temperature measurements were made with a two-wavelength pyrometer, operating at 1500 and 651 nm. The 651-nm channel was used for calibration and control purposes. The pyrometer's achromatic lens system focuses the radiance from a circular target area (0.2-mm diameter) on the specimen onto the head of a fiber-optic bundle, which is randomly bifurcated in order to distribute nearly equal fractions

[5] 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.

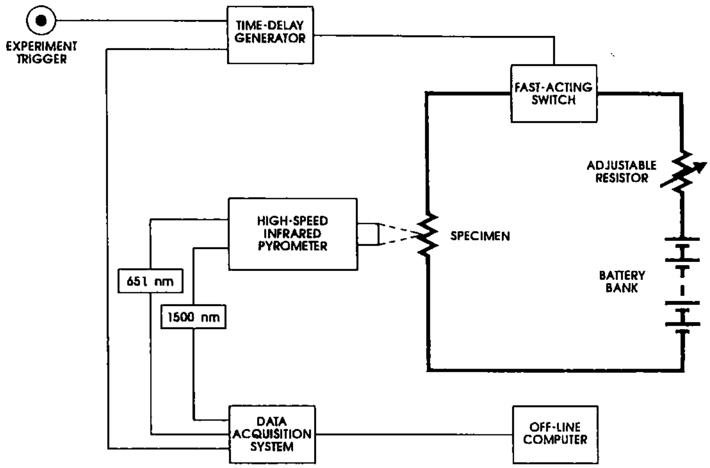

Fig. 1. Functional diagram of the high-speed thermophysical measurement system.

of target radiance between the two channels. In each channel, the radiance is directed through an interference filter onto a detector. The interference filters are 1500 nm, 21-nm bandwidth, and 651 nm, 34-nm bandwidth. The detectors are InGaAs photodiode for the 1500-nm channel and Si photodiode for the 651-nm channel. The photocurrents of both detectors are converted to proportional voltages and amplified by high-stability amplifiers. The amplified signals are recorded simultaneously every 0.5 ms with a high-speed digital data acquisition system.

3. MEASUREMENTS

The measurements were performed on strip-shape specimens having the following nominal dimensions: length, 50 mm; width, 3.2 mm; and thickness 0.13 mm. Before the experiments, the surfaces of the specimens were abraded in order to remove possible surface contaminants. The resulting surface roughness was about 0.2 μm (rms). All the experiments were performed with the specimen in an argon gas environment at slightly above atmospheric pressure.

The measurements on niobium were performed on four specimens. Three of the specimens were heated to melting several times, which was possible when the pulse current was stopped during melting before the collapse of the specimen. A total of 13 experiments was made. The purity of niobium was 99.9+ %. The manufacturer's typical analysis indicated the presence of the following impurities (ppm by mass): Zr, 100; Fe, Hf, O, and Si, 50 each; C, Cu, and Ti, 40 each; N, 26; Al, Cr, Ca, Mg, Mn, Mo, Ni, Pb, and V, 20 each; Co, Sn, and W, 10 each; H, Cd, 5 each; and B, 1.

The measurements on molybdenum were performed on six specimens. Three of the specimens were heated to melting several times. A total of 12 experiments was made. The purity of molybdenum was 99.97%. The manufacturer's typical analysis indicated the presence of the following impurities (ppm by mass): W, 150; Cr, Fe, Ni, Si, and Sn, 50 each; Al, Ca, Cu, Pb, Mg, and Mn, 10 each.

The specimens were heated from room temperature to melting in 350–800 ms, which corresponds to heating rates in the range 3000–10,000 $K \cdot s^{-1}$. Variation of the radiance temperature (at 1500 nm) of niobium and molybdenum as a function of time near and at their melting points is shown in Fig. 2. Melting of the specimen is manifested by a plateau in the radiance temperature-versus-time function. It may be seen that the radiance temperature is essentially constant with a temperature difference between the beginning and the end of the plateau of no more than 1 K.

Prior to the pulse-heating experiments, the infrared pyrometer was calibrated in two steps. First, the 651-nm channel was calibrated with a secondary

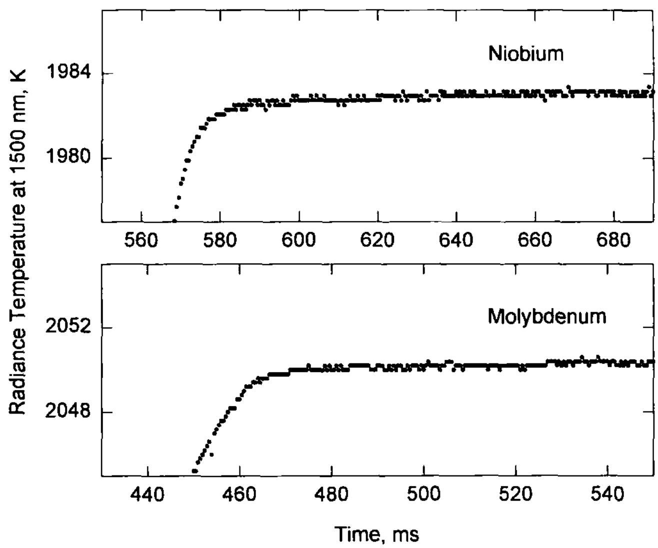

Fig. 2. Experimental data on radiance temperature at 1500 nm as a function of time near and at the melting point of niobium and molybdenum for typical experiments.

standard (tungsten-filament lamp), which, in turn, had been calibrated by the Radiometric Physics Division at NIST. Then the temperature calibration of the 651-nm channel was used to calibrate the 1500-nm channel by performing (steady-state) measurements on a graphite-tube blackbody furnace. All temperatures reported in this paper, except where explicitly noted otherwise, are based on the International Temperature Scale of 1990 (ITS-90) [5, 6].

Table I. Summary of Measurements of the Radiance Temperature at 1500 nm of Niobium and Molybdenum at Their Respective Melting Points

Experiment No.	Number of temperatures[a]	Radiance temperature at melting (K)[b]	SD (K)[c]
	Niobium		
1	100	1983.3	0.1
2	250	1983.3	0.3
3	250	1983.3	0.2
4	250	1983.3	0.2
5	240	1983.3	0.2
6	150	1982.4	0.3
7	160	1982.6	0.3
8	140	1982.7	0.2
9	140	1982.9	0.2
10	130	1983.6	0.1
11	140	1983.6	0.1
12	140	1983.3	0.1
13	160	1983.3	0.2
	Molybdenum		
1	80	2049.5	0.3
2	100	2048.5	0.1
3	80	2053.3	0.1
4	120	2053.2	0.1
5	60	2051.6	0.1
6	80	2049.8	0.3
7	140	2051.4	0.1
8	90	2050.7	0.1
9	80	2049.8	0.1
10	160	2048.8	0.2
11	160	2049.2	0.1
12	120	2049.4	0.1

[a] Number of temperatures used in averaging the results at the plateau to yield an average for the radiance temperature at the melting point of the specimen.
[b] The average value (for an experiment) of the measured radiance temperature at the plateau.
[c] Standard deviation of the measured temperatures from the average value of the plateau in an individual experiment.

4. RESULTS

For a given experiment on a specimen, the plateau radiance temperature at each wavelength (1500 and 651 nm) was determined by averaging the measured temperatures along the flat portion of the corresponding plateau. For the individual experiments on niobium and molybdenum the average radiance temperature, as well as the number of temperatures used for averaging, and the standard deviation of an individual temperature from the average are given in Table I. It may be seen that the number of temperatures used for averaging ranged from 60 to 250, which depended on the heating rate of the specimen and the behavior of the specimen during melting. The standard deviation of an individual temperature from the average is in the range 0.1 to 0.3 K.

The final result of the radiance temperature at the melting point of a metal is obtained by averaging the results of the individual experiments for that metal. The final results for niobium and molybdenum are presented in Table II. The normal spectral emissivity (at 1500 nm) at the melting point is determined, using Planck's law, from the measured radiance temperature and the value of the melting temperature for the three metals also given in Table II.

The radiance temperature measurements at 651 nm were used for control and checking purposes. The present results on niobium and molybdenum are 2423 and 2530 K, respectively. Both niobium and molybdenum show an excellent agreement (within 1 K) with those reported earlier [9, 10].

Table II. Final Results for the Average Radiance Temperature and Normal Spectral Emissivity at 1500 nm of Niobium and Molybdenum at their Respective Melting Points

Metal investigated	Radiance temperature (K)[a]	SD (K)[b]	Normal spectral emissivity[c]
Niobium	1983.1	0.4	0.254
Molybdenum	2050.4	1.6	0.249

[a] Average of the plateau temperatures for all the experiments for a given metal.

[b] Standard deviation of an individual plateau radiance temperature from the average plateau radiance temperature.

[c] Determined by means of Planck's law from the average plateau radiance temperature and the melting point of the metal: 2749 K for Nb [7] and 2893 K for Mo [8]. The melting points are given on ITS-90.

Table III. Coefficients of Eq. (1) for Radiance Temperature (T_r) and Normal Spectral Emissivity ($\varepsilon_{N,\lambda}$) for Niobium and Molybdenum in the Wavelength Range 500 to 1500 nm

	Niobium		Molybdenum	
Item	T_r (K)	$\varepsilon_{N,\lambda}$	T_r (K)	$\varepsilon_{N,\lambda}$
a	2817.6	0.4725	2959.7	0.4570
b	−0.64100	-2.436×10^{-4}	−0.69586	-2.180×10^{-4}
c	5.6394×10^{-5}	6.532×10^{-8}	5.9719×10^{-5}	5.292×10^{-8}
SD[a]	0.9	0.0012	1.2	0.0011

[a] Standard deviation of an individual point from the function represented by Eq. (1).

5. ESTIMATE OF UNCERTAINTIES

A detailed analysis of sources and magnitudes of standard uncertainties[6] in temperature measurements with an optical pyrometer similar in operation to the one used in this work is given elsewhere [11]. Standard uncertainties arise mainly from calibration and operation of the pyrometer, as well as from condition and melting behavior of each specimen. The combined (using the root-sum-of-squares method) standard uncertainties yield ± 8 K for expanded uncertainty[7] for the measured value of radiance temperature at 1500 nm and at the melting point. Combined standard uncertainties of radiance temperature and melting temperature yield an extended uncertainty in normal spectral emissivity at the melting point of about $\pm 3\%$ for each metal.

6. DISCUSSION

During the last two decades, significant progress has been made in accurate measurements, utilizing pulse-heating methods, of the radiance temperature and normal spectral emissivity of several high-melting-point metals at their melting points [1]. All these measurements were performed in the wavelength range from about 500 to 1000 nm. The results, in most

[6] Uncertainties in measured quantities that contribute to the uncertainty of a measured result are reported as a standard uncertainty, which is an estimated standard deviation evaluated either by statistical methods or by other means as explained in the cited publication.

[7] Expanded uncertainty is determined by multiplying the combined standard uncertainty (root-sum-of-squares of the individual standard uncertainties) by a coverage factor which, by convention, is taken to be 2.

cases, have shown that both radiance temperature and normal spectral emissivity decrease with increasing wavelength. One of the objectives of the present work was to extend the measurements to longer wavelengths with the intent to investigate the trend of radiance temperature and normal spectral emissivity at wavelengths beyond 1000 nm, up to 1500 nm. Figure 3 presents the radiance temperature results of the present work for niobium and molybdenum at 1500 nm, in addition to the results of the earlier measurements performed in our laboratory [9, 10] in the nominal wavelength range from 500 to 900 nm. A similar presentation for normal spectral emissivity is shown in Fig. 4. It may be seen that, for the investigated metals, both radiance temperature and normal spectral emissivity decrease with increasing wavelength in the range 500–1500 nm.

The dashed curves in Figs. 3 and 4 represent polynomial functions fitted to the data using the least-squares method. The functions are of the form

$$y = a + bx + cx^2 \tag{1}$$

where y is either radiance temperature (K) or normal spectral emissivity and x is wavelength (nm).

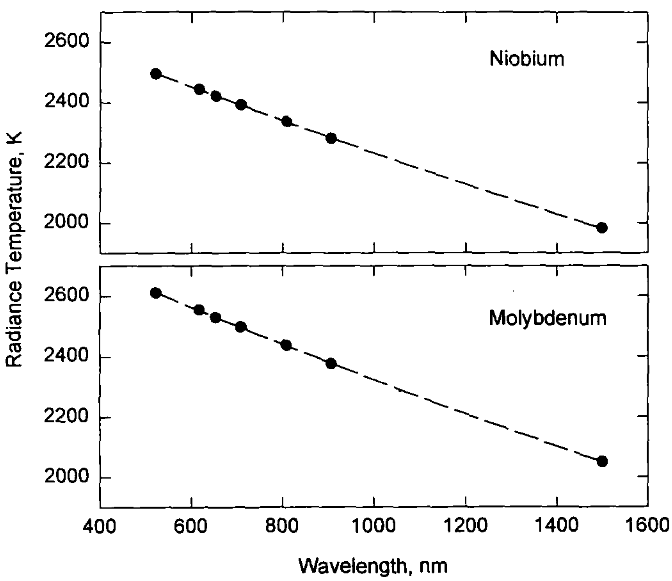

Fig. 3. Variation of radiance temperature at the melting point as a function of wavelength for niobium and molybdenum.

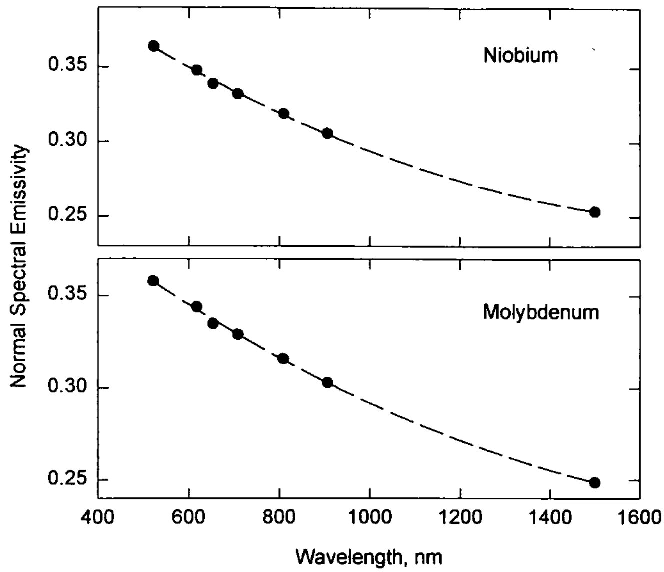

Fig. 4. Variation of normal spectral emissivity at the melting point as a function of wavelength for niobium and molybdenum.

Comparisons of the earlier measurements in the wavelength range 500–900 nm with those reported in the literature have been given in the individual papers on the investigated metals: niobium [9] and molybdenum [10]; therefore they are not repeated here. No data on radiance temperature or normal spectral emissivity at 1500 nm for these metals at their melting points were found in the literature.

To substantiate further the trend in both radiance temperature and normal spectral emissivity at melting point with respect to wavelength, measurements similar to those in the present work have to be performed on other metals on which data at shorter wavelengths exist.

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