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Hemispherical Total Emissivity of Niobium, Molybdenum, and Tungsten at High Temperatures Using a Combined Transient and Brief Steady-State Technique[1]

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The hemispherical total emissivity of three refractory metals, niobium, molybdenum, and tungsten, was measured with a new method using a combined transient and brief steady-state technique. The technique is based on rapid resistive self-heating of a solid cylindrical specimen in vacuum up to a preset high temperature in a short time (about 200 ms) and then keeping the specimen at that temperature under steady-state conditions for a brief period (about 500 ms) before switching off the current through the specimen. Hemispherical total emissivity is determined at the temperature plateau from the data on current through the specimen, the voltage drop across the middle portion of the specimen, and the specimen temperature using the steady-state heat balance equation based on the Stefan–Boltzmann law. Temperature of the specimen is determined from the measured surface radiance temperature and the normal spectral emissivity; the latter is obtained from laser polarimetric measurements. Experimental results on the hemispherical total emissivity of niobium (2000 to 2600 K), molybdenum (2000 to 2700 K), and tungsten (2000 to 3400 K) are reported.

KEY WORDS: hemispherical total emissivity; high temperature; molybdenum; niobium; normal spectral emissivity; pulse heating; tungsten.

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1. INTRODUCTION

Recently, a new method for measuring the hemispherical total emissivity of electrically conducting materials at high temperatures using a feedback-controlled pulse-heating technique was developed and applied to tantalum [1]. The technique is based on rapid resistive self-heating of a solid cylindrical specimen in vacuum up to a preset high temperature in a short time (about 200 ms) and then keeping the specimen at that temperature under steady-state conditions for a brief period (about 500 ms) before switching off the current through the specimen. Hemispherical total emissivity is determined at the temperature plateau from the steady-state heat balance equation based on the Stefan–Boltzmann law.

In the present study, hemispherical total emissivity of three refractory metals, niobium, molybdenum, and tungsten were measured with the new technique in the temperature range from 2000 K to near the melting point of each metal.

2. MEASUREMENT TECHNIQUE

In this combined transient and brief steady-state method, a rod-shaped specimen is kept at a preset high temperature using a feedback control technique after rapid heating from room temperature. The principle of measuring hemispherical total emissivity is based on the heat balance on the specimen in the brief steady-state period, which is referred to as the "plateau." Under steady-state conditions, electrical power imparted to the specimen is equal to the power loss from the specimen. At temperatures above about 1500 K, heat loss from the "effective" (middle portion) specimen at the plateau is primarily by thermal radiation. This is due to the fact that heat loss by axial conduction to the ends of the specimen is very small in comparison to heat loss by radiation during the brief steady-state period. Based on the Stefan–Boltzmann law, the power balance on the effective specimen yields the hemispherical total emissivity,

$$\varepsilon_{\mathrm{ht}} = EI/\{A_{\mathrm{s}}\sigma(T_{\mathrm{s}}^4 - T_{\mathrm{e}}^4)\} \tag{1}$$

where E is the voltage drop across the effective specimen, I is the current through the specimen, T_{s} is the specimen temperature at the plateau, T_{e} is the temperature of the environment surrounding the specimen, A_{s} is the surface area of the effective specimen, and σ is the Stefan–Boltzmann constant. Hemispherical total emissivity is computed for each data set of E, I, and T_{s}. A final value for the hemispherical total emissivity of a specimen at a given temperature is then obtained by averaging the individual results at the plateau.

3. MEASUREMENT SYSTEM

The measurement system, with the exception of the feedback control system, the pyrometer, and the data acquisition system, is essentially the same as that described in earlier publications [2, 3]. The recently developed feedback control system consists of a solid-state switch, a personal computer, and a high-speed pyrometer. The details of the feedback control system are given in a recent publication [1].

A high-speed optical pyrometer operating at 651 nm was used to measure the surface radiance temperature of the specimen. Data on radiance temperature were used for both the feedback control system and the calculation of hemispherical total emissivity. The target of the pyrometer was a circular area 0.2 mm in diameter. The temperature range of the pyrometer was approximately from 1300 to 2000 K. Up to three neutral density filters were used to extend the measurements to higher temperatures. The true temperature of the specimen was determined from the measured surface radiance temperature and the normal spectral emissivity based on Planck's law. The normal spectral emissivity was obtained from laser polarimetric measurements [4]. The pyrometer was calibrated against a secondary standard (tungsten-filament lamp) which, in turn, was calibrated by the Radiometric Physics Division at NIST.

Data on voltages across the standard resistor in the main circuit and the probes attached to the specimen, as well as those of the pyrometer and the polarimeter, were recorded with a digital data acquisition system (16-bit resolution) at the rate of 2 kHz for each experimental quantity.

4. MEASUREMENTS

4.1. Specimens

One experiment each were performed on rod-shaped specimens of niobium, molybdenum, and tungsten. A pair of fine grooves, 25 mm apart, was fabricated at the middle portion of the specimen for the voltage probes. The surface of each specimen was polished with No. 600 abrasive paper. Before the experiments, the specimens were preheated near the upper limit of the temperature range of each metal in an argon gas (99.999% pure) environment at slightly above atmospheric pressure. After preheating, the experiments were conducted with the specimens in a vacuum environment at about 3×10^{-3} Pa (2×10^{-5} Torr). The characteristics of the specimens are listed in Table I.

Table I. Characteristics of the Specimens

Metal	Dimensions (nm)	Purity (mass%)	Preheating
Nb	ϕ 1.953 × 72.99	99.9	1 s at 2550 K, 5 times
Mo	ϕ 2.371 × 76.23	99.95	1 s at 2800 K, 5 times
W	ϕ 2.032 × 73.33	99.95	1 s at 3420 K, 5 times

4.2. Normal Spectral Emissivity

In the present study, normal spectral emissivity, $\varepsilon_{n\lambda}$, was measured simultaneously with the other experimental quantities with a high-speed laser polarimeter described in detail elsewhere [4]. Measurements of $\varepsilon_{n\lambda}$ were at 633 nm, the wavelength of the helium–neon laser of the polarimeter. The measured values of $\varepsilon_{n\lambda}$ were converted to correspond to 651 nm, the pyrometer wavelength, by interpolating the results from auxiliary measurements in the bracketing wavelengths of 624 and 651 nm. A single value for emissivity was obtained at the plateau by averaging the individual values over a finite period on the plateau (typically, about 400 data points over 200 ms).

4.3. Hemispherical Total Emissivity

In a typical experiment for the determination of hemispherical total emissivity, the specimen was heated from room temperature to a preset high temperature in about 200 ms; then it was maintained at a constant temperature for about 500 ms utilizing the feedback control. This procedure was repeated at several preset temperatures. The hemispherical total emissivity for each point at the plateau was computed using Eq. (1) based on the room-temperature dimensions of the specimen. A single value for emissivity was obtained at the plateau by averaging the individual emissivity values over a finite period on the plateau (typically, about 200 data points over 100 ms).

Before the experiments, optimum pulse heating periods were determined for each specimen through test experiments in order to minimize the uncertainty in hemispherical total emissivity due to axial heat conduction across the effective specimen [1]. In the test experiments, measurements of hemispherical total emissivity were repeated on the same specimen at a given plateau temperature by varying the pulse heating period in the range between 100 and 600 ms. The optimum value of the pulse heating period

was selected where the deviation due to the axial heat conduction converged within 0.5% of the constant hemisperical total emissivity measured with short heating periods less than 150 ms. The pulse heating periods in the final measurements were always less than the optimum pulse heating period (typically about 200 ms).

5. RESULTS

5.1. Normal Spectral Emissivity

The results of the normal spectral emissivity (at 633 nm) of niobium, molybdenum, and tungsten are shown in Fig. 1. The solid lines represent linear functions fitted to the results using the least-squares method. The function for the three metals is

$$\varepsilon_{n\lambda} = a_1 + a_2 T \tag{2}$$

where T is in K. The values of the coefficients a_1 and a_2, and standard deviation from the fitted function are given in Table II. The values of $\varepsilon_{n\lambda}$ at 100 K intervals are presented in Table III.

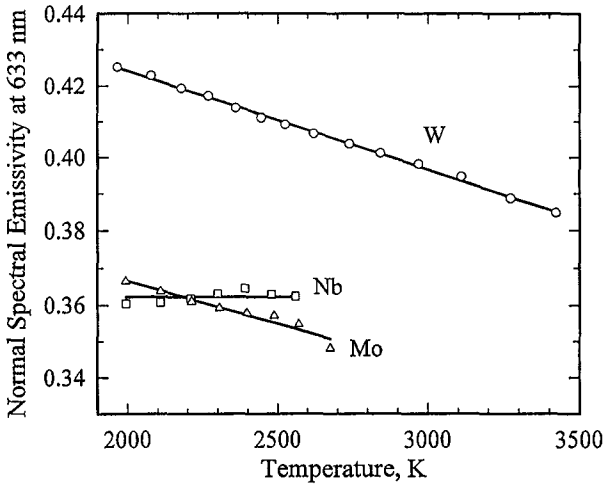

Fig. 1. Normal spectral emissivity (at 633 nm) of niobium, molybdenum, and tungsten measured with the millisecond-resolution laser polarimeter. The solid lines represent linear functions [Eq. (2)] fitted to the results of each metal.

Table II. Coefficients and Standard Deviations of Eqs. (2) and (3) for the Normal Spectral Emissivity and Hemispherical Total Emissivity of Niobium, Molybdenum, and Tungsten

	Normal spectral emissivity at 633 nm [Eq. (2)]			Hemispherical total emissivity [Eq. (3)]			
Metal (temp. range; K)	a_1	$10^5 a_2$ (K^{-1})	SD (%)	b_1	$10^4 b_2$ (K^{-1})	$10^8 b_3$ (K^{-2})	SD (%)
Nb (2000–2600)	0.3622	0	0.4	−0.1185	2.305	−2.936	0.1
Mo (2000–2700)	0.4134	−2.342	0.4	−0.1657	2.662	−3.735	0.3
W (2000–3400)	0.4792	−2.753	0.2	−0.1342	2.573	−3.481	0.4

5.2. Hemispherical Total Emissivity

The results of the hemispherical total emissivity of niobium, molybdenum, and tungsten as a function of temperature are shown in Fig. 2. The solid curves represent quadratic functions fitted to the results using the least-squares method. The function for the three metals is

$$\varepsilon_{ht} = b_1 + b_2 T + b_3 T^2 \tag{3}$$

Table III. Normal Spectral Emissivity and Hemispherical Total Emissivity of Niobium, Molybdenum, and Tungsten Based on Eqs. (2) and (3)

Temperature (K)	Normal spectral emissivity at 633 nm			Hemispherical total emissivity		
	Nb	Mo	W	Nb	Mo	W
2000	0.362	0.367	0.424	0.225	0.217	0.241
2100	0.362	0.364	0.421	0.236	0.229	0.253
2200	0.362	0.362	0.419	0.246	0.239	0.263
2300	0.362	0.360	0.416	0.256	0.249	0.273
2400	0.362	0.357	0.413	0.266	0.258	0.283
2500	0.362	0.355	0.410	0.274	0.266	0.291
2600	0.362	0.353	0.408	0.282	0.274	0.299
2700		0.350	0.405		0.281	0.307
2800			0.402			0.313
2900			0.399			0.319
3000			0.397			0.324
3100			0.394			0.329
3200			0.391			0.333
3300			0.388			0.336
3400			0.386			0.338

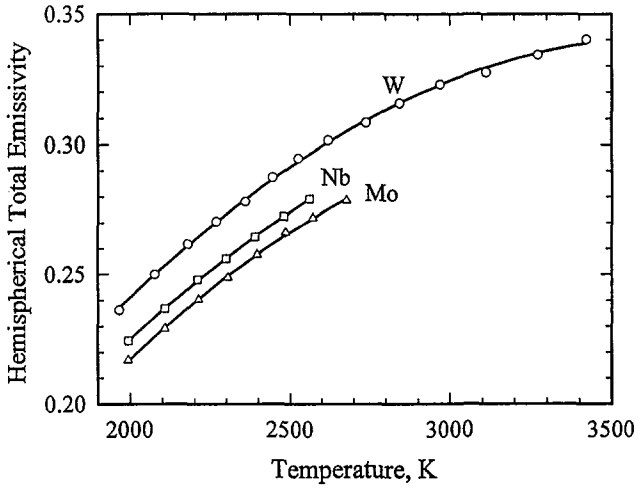

Fig. 2. Hemispherical total emissivity of niobium, molybdenum, and tungsten measured using the feedback control technique. The solid curves represent quadratic functions [Eq. (3)] fitted to the results of each metal.

where T is in K. The values of the coefficients b_1, b_2, and b_3 and the standard deviation from the fitted function are given in Table II. Values of ε_{ht} at 100-K intervals are presented in Table III. The uncertainty (at the 2 SD level) in the hemispherical total emissivity is estimated to be not more than $\pm 2\%$. The details of the estimates are given in an earlier publication on similar measurements on tantalum [1].

6. DISCUSSION AND CONCLUSION

Figures 3, 4, and 5 show hemispherical total emissivities of niobium, molybdenum, and tungsten, respectively, reported in the literature [2, 5–30]. The final fits to the present results are also shown by the thick solid curves. Since the optical properties of metals are very sensitive to surface conditions, such as surface roughness and contamination, the results given in the literature show a large scatter of up to 50%. The present results are in reasonably good agreement, within $\pm 10\%$, with most of the values given in the literature.

In conclusion, the results reported in this paper, in addition to those reported earlier [1], support the validity of the new combined transient and brief steady-state technique for measuring hemispherical total emissivity of electrically conducting materials at high temperatures.

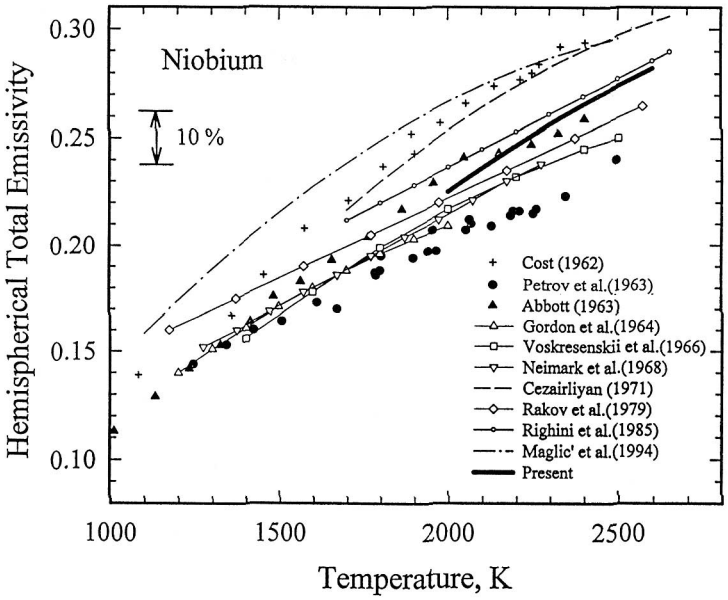

Fig. 3. Hemispherical total emissivity of niobium given in the literature [11, 15, 16, 18, 19, 21, 23, 26, 28, 30].

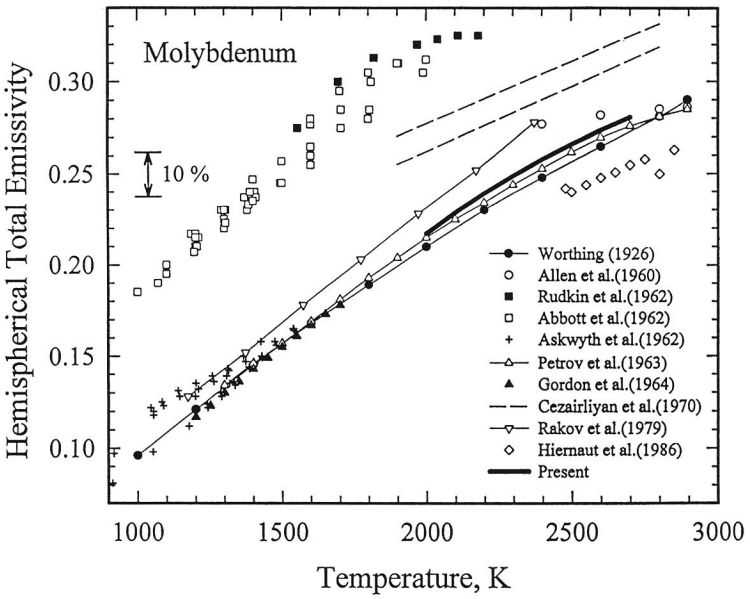

Fig. 4. Hemispherical total emissivity of molybdenum given in the literature [2, 6, 9, 10, 12–14, 17, 26, 29].

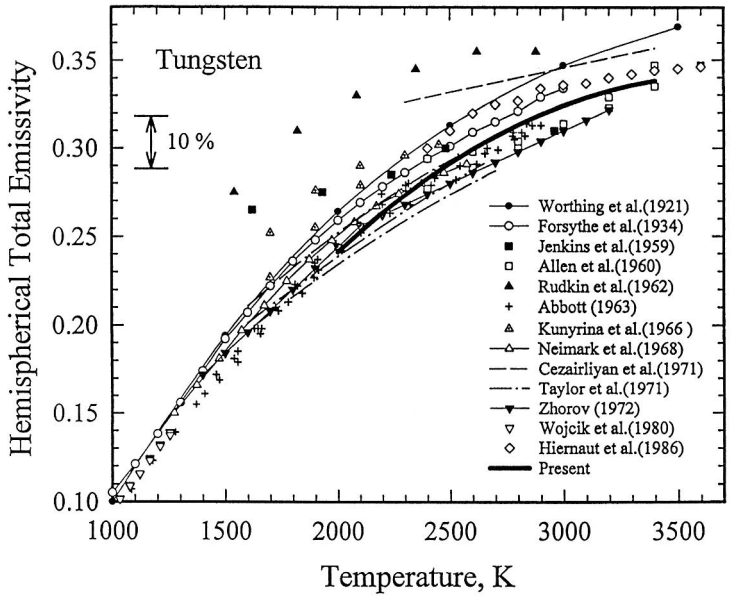

Fig. 5. Hemispherical total emissivity of tungsten given in the literature [5, 7–10, 16, 20–22, 24, 25, 27, 29].

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