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A Multiwavelength Reflectometric Technique for Normal Spectral Emissivity Measurements by a Pulse-Heating Method[1]

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A new technique has been developed for the direct measurement of the normal spectral emissivity at several wavelengths in pulse-heating conditions, adding some novel features to previous versions of this type of apparatus. Pulse-heating experiments were performed on niobium strip specimens, taking the specimen from room temperature to the melting point using rapid resistive self-heating. The normal spectral emissivity was measured at three wavelengths by a multi-wavelength reflectometric technique. At the same time, the radiance temperature was measured at the same wavelengths by a high-speed pyrometer from approximately 1100 K to the melting point. Details of the method, the measurement apparatus, and the calibration technique are described. Preliminary results for the normal spectral emissivity of niobium at 633, 750, and 900 nm over a wide temperature range are presented.

KEY WORDS: high-speed multi-wavelength pyrometer; multi-wavelength reflectometric technique; niobium; normal spectral emissivity; pulse-heating method.

1. INTRODUCTION

The pulse-heating technique is considered a very accurate method for the measurement of several thermophysical properties (heat capacity, enthalpy, heat of fusion, electrical resistivity, hemispherical total emissivity, thermal expansion, etc.) at high temperatures, because it overcomes limitations of

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steady-state experiments [1–3]. The most accurate measurements of the mentioned thermophysical properties using the pulse-heating technique are performed on tubular specimens with a blackbody hole. In certain cases these specimens are difficult to obtain because the material does not exist in tubular form or because of machining difficulties. When blackbody cavities are not available, the best measurement solution is the simultaneous determination under pulse-heating conditions of the radiance temperature and of the normal spectral emissivity of the material, performed in the same experimental apparatus using samples that are simple to machine. Two techniques have been proposed recently to perform direct measurements of thermophysical properties on simple specimens.

The method developed at the National Institute of Standards and Technology (NIST, U.S.A.) makes use of rod specimens and can be used for direct measurements of the normal spectral emissivity using laser polarimetry [4]. The technique developed at the CNR Istituto di Metrologia "G. Colonnetti" (IMGC, Italy) makes use of specimens in the form of strips, and the normal spectral emissivity is measured by integrating sphere reflectometry in pulse-heating conditions [5, 6]. A millisecond pulse-heating apparatus has been developed at the Harbin Institute of Technology (HIT, People's Republic of China). The design is based on systems previously developed both at NIST and IMGC for the measurement of several thermophysical properties with millisecond time resolution. Details regarding the experimental apparatus may be found in an earlier publication [7]. Recently a new technique has been developed at HIT for the direct measurement of the normal spectral emissivity at several wavelengths in pulse-heating conditions, adding some novel features to the apparatus. This paper describes the new technique and presents some preliminary experimental measurements of the normal spectral emissivity of niobium strip specimens at different wavelengths over a wide temperature range obtained recently at HIT.

2. METHOD AND APPARATUS

The new technique developed at HIT combines multi-wavelength pyrometry with a novel multi-wavelength reflectometric technique. Measurements are performed on a strip specimen placed in the typical environmental chamber of the pulse-heating method. The radiance temperatures are measured on one side of the strip at several wavelengths by the multi-wavelength pyrometer. The other side of the strip faces the porthole of a small integrating sphere placed inside the environmental chamber (details of the arrangement are presented in Fig. 1). The side of the strip facing the sphere receives a collimated beam generated by a laser diode and

modulated by a mechanical chopper. The reflected beam is collected by the integrating sphere, taken out with an optical fiber, and measured by a silicon detector placed outside the experimental chamber.

The reflectivity is measured in relation to the known reflectivity of a barium sulfate ($BaSO_4$) reference specimen. The measured quantity is the spectral directional-hemispherical reflectivity ρ of the specimen at the wavelength of the interference filter placed in front of the silicon detector. According to Kirchhoff's law for opaque materials, this quantity is the complement to the normal spectral emissivity ε of the specimen ($\varepsilon = 1 - \rho$). Using this apparatus, the normal spectral emissivity of the specimen at one wavelength can be measured in one experiment over a wide temperature range. The normal spectral emissivity at other wavelengths can be measured in another experiment by changing the laser diode and the interference filter in front of the sphere detector to another wavelength and making use of the corresponding pyrometric channels. A digital lock-in technique is used to discriminate between the reflected modulated beam and the continuous radiation emitted by the specimen at high temperatures.

The radiance temperature of the specimen during a pulse experiment is measured with a high-speed eight-wavelength pyrometer designed and constructed specifically for this apparatus. The instrument is capable of temperature evaluation at a rate of up to 10^4 measurements per second and operates at a fixed distance (300 mm) from its target. This distance was chosen to maximize the radiation collected by the front objective in relation to the various constraints of the apparatus (window size, location of the

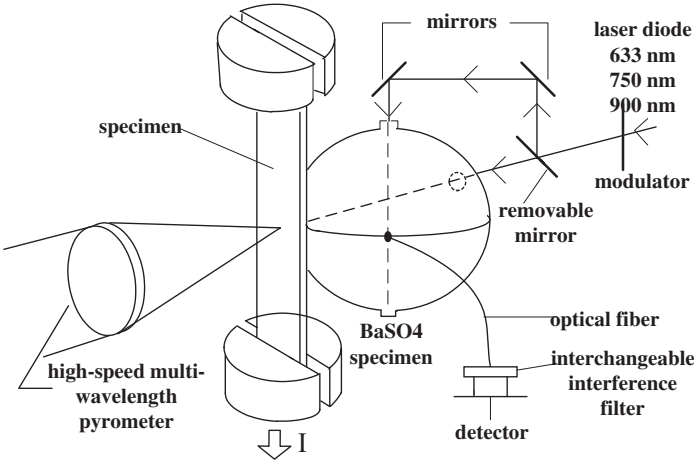

Fig. 1. Schematic representation of the multi-wavelength pulse-heating reflectometric technique.

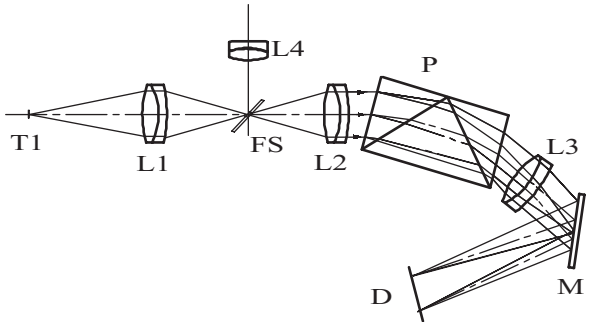

Fig. 2. Optical diagram of the high-speed eight-wavelength pyrometer.

specimen, physical dimensions, etc.). The optical scheme of the pyrometer is shown in Fig. 2. The radiation emitted by the specimen T1 is focused by the objective L1 on a rectangular field stop FS, which is located in the focal plane of the collimating lens L2. The parallel beam generated by the collimator is dispersed by the prism P. A telescopic lens L3 focuses the field stop in its focal plane, one image for each wavelength. An array of rectangular photodiodes D is used as a detector, operating in the wavelength range 500 to 1100 nm. In the version of the instrument described in this paper, the normal spectral emissivity measurements are performed in three wavelength bands. Three operating channels of the pyrometer have been selected to measure the radiance temperature at 633, 750, and 900 nm. Interference filters at the same peak wavelengths have been placed in front of the silicon detector collecting radiation from the integrating sphere, and three laser diodes operated at the same wavelengths have been used. Additional technical details on the system characteristics can be found in an earlier publication [7].

3. MEASUREMENTS AND CALIBRATIONS

Measurements were performed on niobium strip specimens with the following nominal dimensions: length, 80 mm; width, 10 mm; and thickness, 1 mm. All experiments were performed in high vacuum (better than 10^{-3} Pa). Typical operational conditions to cover the range from room temperature to the melting point were: current pulse duration, 2.5 s; heating rate, 1000 $K \cdot s^{-1}$. The lock-in reference frequency during all experiments was in the range 780 to 800 Hz. The normal spectral emissivity is strongly dependent on surface conditions, and all specimens exhibited large changes in normal spectral emissivity during the initial experiments.

A stable surface condition was reached only after the specimens had been taken to their melting point a few times, interrupting the current pulse before the destruction of the specimen. After several experiments to the melting point, the specimen surface became shiny and exhibited very large grains.

The measurement technique is not absolute and therefore requires an appropriate reflectivity reference standard to take into account different factors (including the geometry of the integrating sphere and possible imperfections in its internal coating). This is done in a separate calibration experiment using mirrors placed in an appropriate holder to direct the collimated beam to a $BaSO_4$ specimen (Fig. 1) and measuring the silicon detector output for all the temperatures of interest in these experiments. In the calibration experiment, the removable mirror is set at an appropriate angle, so that the collimated beam is directed toward the reference specimen. This calibration can be repeated as many times as necessary by simply moving the reflectors to the appropriate position. A typical measured signal when the collimated beam is directed toward the $BaSO_4$ specimen is shown in Fig. 3. Theoretically, a constant value should be observed because the $BaSO_4$ specimen remains at room temperature during the entire experiment, but a variation of about 2% is observed, with the signal during the heating and cooling phases following slightly different paths. On account of this phenomenon, all experimental measurements on niobium strips are related to $BaSO_4$ reference measurements performed exactly in the same operational conditions.

In the direct experiment the mirror is removed to send the collimated beam toward the niobium specimen. Additional corrections are necessary as a result of the following causes:

(i) for safety reasons the hot strip specimen must be placed at an appropriate distance from the sphere (generally about 2 mm), with partial loss of the reflected beam;

(ii) specimens taken to their melting point exhibit large grain growth and become strongly specular. This condition increases the radiation lost from the beam entrance hole after a single reflection;

(iii) the $BaSO_4$ calibration experiment requires a slightly different optical path with three additional mirrors; and

(iv) the $BaSO_4$ and Nb experiments are performed at different times.

An estimate of the total effect of these corrections is obtained with measurements at the melting point, where the normal spectral emissivity is a well known literature value. All the measurements presented in this paper

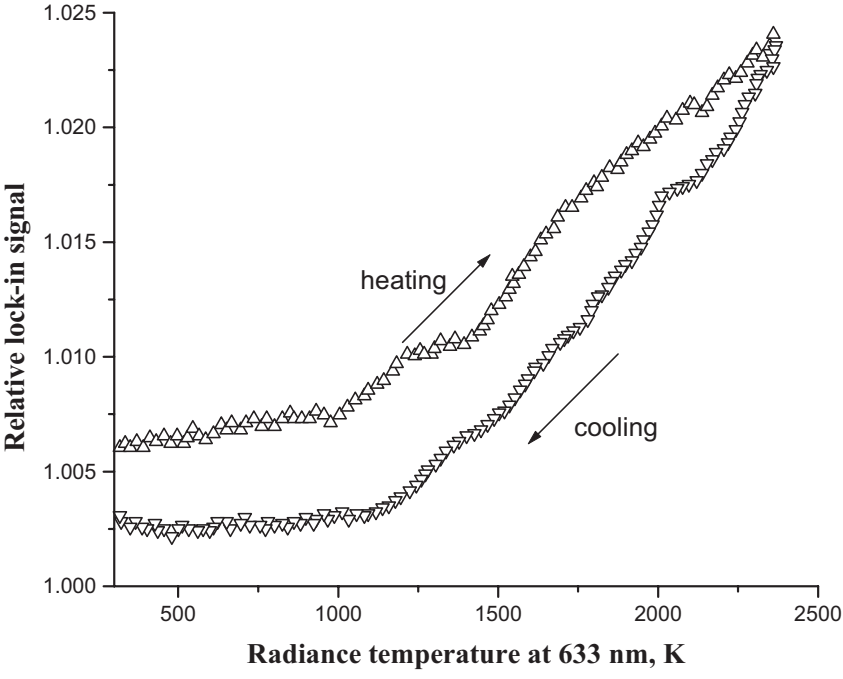

Fig. 3. Typical digital lock-in signal as a function of radiance temperature during an experiment on a $BaSO_4$ specimen (triangles pointing upward refer to heating; triangles pointing downward refer to cooling).

have been obtained by adopting the values 0.3489, 0.3280, and 0.3087 for the normal spectral emissivity (at 633, 750, and 900 nm) of niobium at its melting point, as measured earlier at NIST and IMGC [8].

4. EXPERIMENTAL RESULTS

The strip specimens were machined at the HIT from commercial niobium foil of mass purity 99.9%. Experiments were performed on several niobium strips, but final results are presented for a typical specimen that was pulse-heated to its melting point eight times. The typical shape of the normal spectral emissivity at 633, 750, and 900 nm versus temperature curves during the heating period is presented in Fig. 4. The normal spectral emissivity measured during heating shows a moderate decrease from 1000 to 1700–2000 K at all wavelengths. This is probably due to the evaporation of some surface oxide layer during heating and is consistent

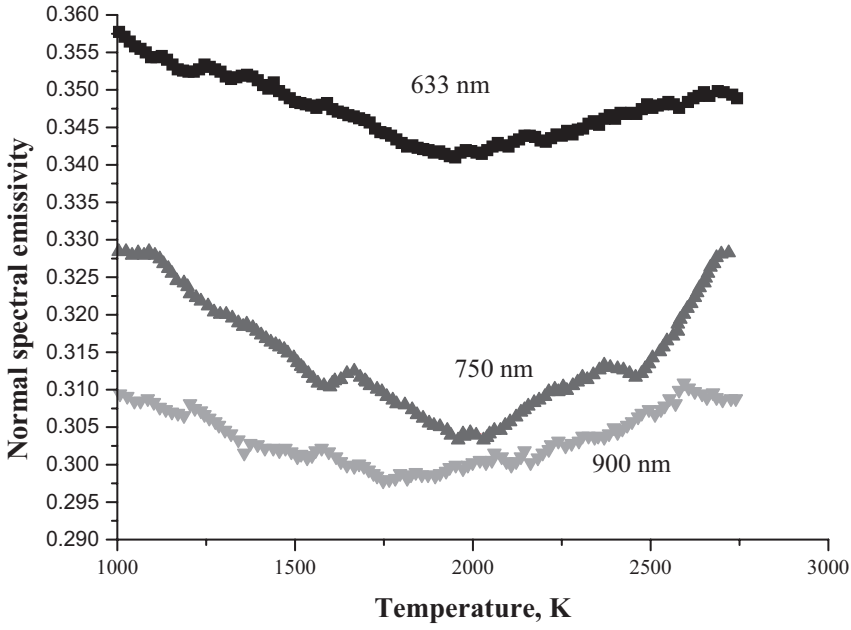

Fig. 4. Experimental results for the normal spectral emissivity of niobium at 633, 750, and 900 nm as a function of temperature during the heating period.

with similar results obtained at IMGC at 900 nm [5]. After reaching a minimum, the normal spectral emissivity increases with temperature up to the melting point. The typical shapes of the normal spectral emissivity of niobium at 633 nm versus temperature curves during both heating and cooling are presented in Fig. 5. The experimental data below 1000 K are experimental measurements of the normal spectral emissivity and temperatures computed by interpolation during heating and by extrapolation during cooling. It may be clearly seen that the normal spectral emissivity of the strip specimen has a different temperature behavior between the heating and cooling curves, and this trend is clearly visible in all experiments. The oxide layer that is evaporated during heating is probably reformed during cooling. Since the system is operated under high vacuum, this process will take some time, and therefore the normal spectral emissivity during cooling will follow a different temperature trend with respect to the heating period. It is also clearly seen that the emissivity, when the specimen cools freely back to room temperature is the same as the emissivity when the experiment started. This result demonstrates that a stable specimen surface has been reached.

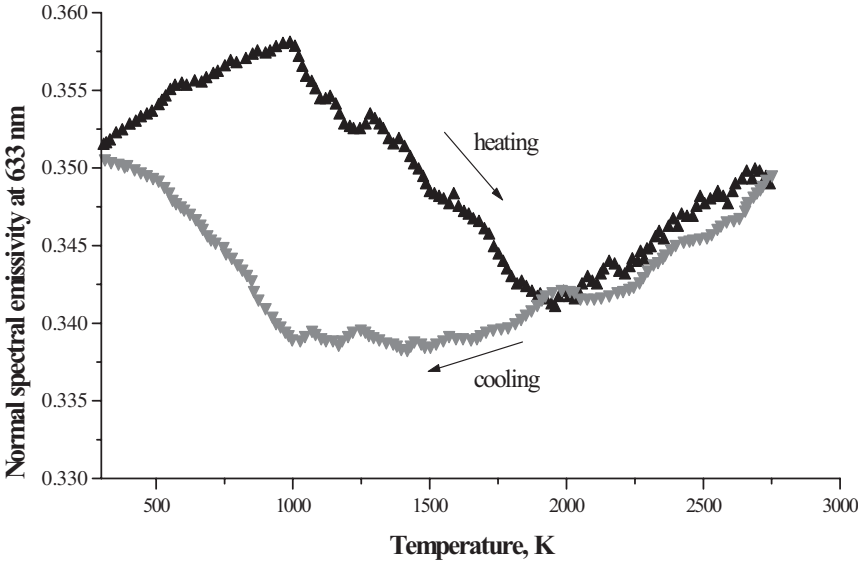

Fig. 5. Experimental results for the normal spectral emissivity at 633 nm as a function of temperature for a niobium strip specimen (triangles pointing upward refer to heating; triangles pointing downward refer to cooling).

5. CONCLUSIONS

Preliminary results for the normal spectral emissivity of niobium at 633, 750, and 900 nm over a wide temperature range are presented. The measurements were performed with a new pulse-heating technique developed at HIT, which is a high-speed version of a novel multi-wavelength reflectometric technique associated with multi-wavelength pyrometry. The dependence of the normal spectral emissivity on surface conditions has been confirmed, indicating the presence of a thin oxide layer that evaporates during the heating phase of the experiment but is formed again during cooling at lower temperature. Consequently, the normal spectral emissivity of the niobium strip does not follow the same trend during the heating and cooling periods.

The work in this research area will continue with additional experiments on other materials and with an estimate of uncertainties in other measurement situations.

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