COMPARISON OF DIRECT AND INDIRECT METHODS OF SPECTRAL INFRARED EMITTANCE MEASUREMENT

Leonard Hanssen,[1] Alexander Prokhorov,[1] Vladimir Khromchenko,[1] and Sergey Mekhontsev[1]

[1]National Institute for Standards and Technology, Gaithersburg, MD, USA

ABSTRACT

The recent development of a system for direct spectral emittance of samples in the infrared at NIST has enabled a comparison and validation of measurement methods. Indirect emittance measurements are performed using a diffuse gold integrating sphere system coupled to a Fourier transform infrared spectrometer (FTIR). The absolute spectral reflectance of both specular and diffuse (directional-hemispherical reflectance) samples can be examined with the sphere system for sample temperatures ranging from room temperature to 475 K. The direct emittance measurement system uses a second FTIR and associated optics that compare the relative spectral radiance of samples up to 1300 K to a set of variable temperature blackbody sources. For the lower temperatures required for comparison (≤ 500 K) the direct system measurements include a correction for the ambient reflected light. The application of two independent methods to the measurement of any optical property is a powerful strategy for accurately determining the property (such as emittance) value and obtaining a true estimate of the associated measurement uncertainties. The emittance of a SiC sample at 473 K is obtained using both methods and shows good agreement, indicating initial levels of measurement accuracy.

1. INTRODUCTION

Over the past 1-1/2 decades, NIST has developed a Fourier Transform Infrared Spectrophotometry Laboratory for the support of characterization of the optical properties of materials. The purpose of the laboratory is to develop physical standards, calibration services, and improved measurement methods for characterization of transmittance, reflectance, absorptance, emittance and refractive index of solid materials and optical components. Until recently, the emittance measurement capability has been limited to the indirect method of the measurement of reflectance (for opaque materials) and also transmittance (for transparent materials). The temperature range is 10 K to 570 K for specular samples, and was limited to Room Temperature for diffuse samples. Recently we have expanded the temperature range for the indirect method for diffuse materials up to 475 K.

Over the past 4 years considerable effort has been expended on the design and construction of a direct emittance measurement system for samples at temperatures up to 1370 K.[1] The system uses a Fourier transform infrared spectrometer (FTIR) and filter radiometers to compare the spectral radiance of a heated sample with that from one of a set of reference blackbody sources that cover a temperature range of 250 K to 1370 K. The initial phase is focused on temperatures above 500 K, for which the background reflected flux is small and correctable. For lower temperatures a second phase is planned to employ a controlled background enclosure and a "two temperature" method. Several papers describing the system and characterization of critical elements are available for more detailed descriptions of the hardware and methodologies employed.

2. INDIRECT EMITTANCE MEASUREMENT SYSTEMS

Several setups are available for the measurement of infrared spectral reflectance and transmittance, each with overlapping, but not duplicative capabilities. Two setups are coupled to FTIR spectrometers; a third setup designed for near infrared measurements of specular samples uses a monochromator. The near infrared system uses a vacuum chamber to enable measurement of samples susceptible to low levels of oxygen or water. This system is described in a companion paper.[2] A goniometer system with FTIR source input enables the direct measurement of absolute variable angle reflectance and transmittance for specular samples, as demonstrated by a study of an undoped silicon sample as a proposed standard for the 2 μm to 5 μm spectral range.[3] A custom cryostat can control sample temperatures between 10 K and 600 K, and has been used to measure the emittance (indirectly via reflectance and transmittance) of coated and uncoated sapphire window samples.[4]

The instrument that is used as a "reference" instrument (i.e. one that provides highest accuracy absolute values and consequently is used for scale derivation) is a custom integrating sphere. The sphere employs a unique measurement scheme to provide absolute reflectance and transmittance for both specular and diffuse samples.[5,6,7,8] Recently this sphere has been adapted to enable sample temperature control between 290 K and 475 K. The integrating sphere uses an HgCdTe detector and has a spectral range of 1 μm to 18 μm. when used with appropriate combinations of the FTIR beam splitter and internal source. (Another recent improvement is the use of an InSb detector that should provide more accurate and faster data at short wavelengths).

In Figure 1, a schematic of the integrating sphere sample heater/mount for transparent samples is shown. It includes a ring heater, heat exchanger, thermal insulation, sample rest, outer case, and mounting holes to attach to the integrating sphere. There are four interchangeable internal heat exchangers. Two use electrical heating for higher temperatures to 473 K: one solid holder for opaque samples, and one ring holder for transparent samples – as shown in the Figure 1. Two similar heaters use liquid from a temperature-controlled bath recirculator unit for temperatures between 270 K and 350 K. The ring shape of two of the heaters does not obscure the central region of the sample, allowing light to pass through for a transmittance measurement, as well as allowing the transmitted component to be diverted and absorbed in a reflectance measurement. For opaque samples, the solid heaters are used to obtain the best temperature uniformity. As is the case for any heated sample, accurate determination of the sample temperature is important and thermal gradients between the sample center and the temperature sensor need to be evaluated for poorly conducting samples. This can be addressed in a number of ways including modeling and calibration samples with sensors attached to their surfaces.

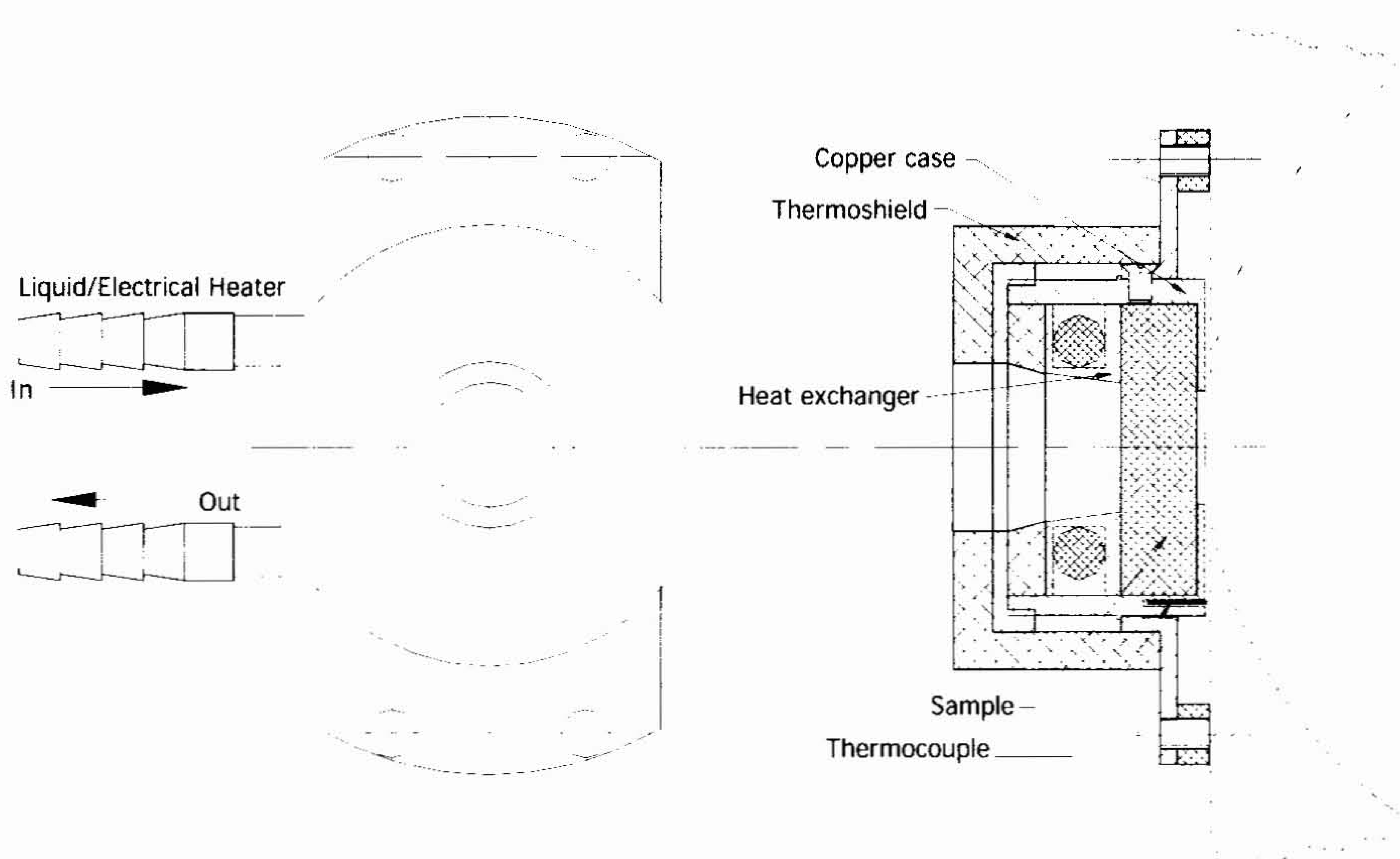

Figure 1. Schematic of sample heater design for the indirect measurement of spectral emittance using an integrating sphere.

An example measurement result is shown in Figure 2 of a 1 mm thick undoped double-side polished silicon wafer. The sample was measured at 295 K, 365 K, and 475 K. The plot has 9 curves corresponding to the reflectance, transmittance, and derived emittance for each temperature. The plot highlights the region near 1 μm where the silicon absorption edge (transition from opaque to transparent behavior) lies. An expanded version of the plot reveals additional small changes (less than 0.01) with increasing temperature: increased reflectance, decreased transmittance and increased emittance. The expanded uncertainty (k = 2) for the curves in Fig. 2 is 0.3 % of the values for R and T, and 0.4 % for ε.

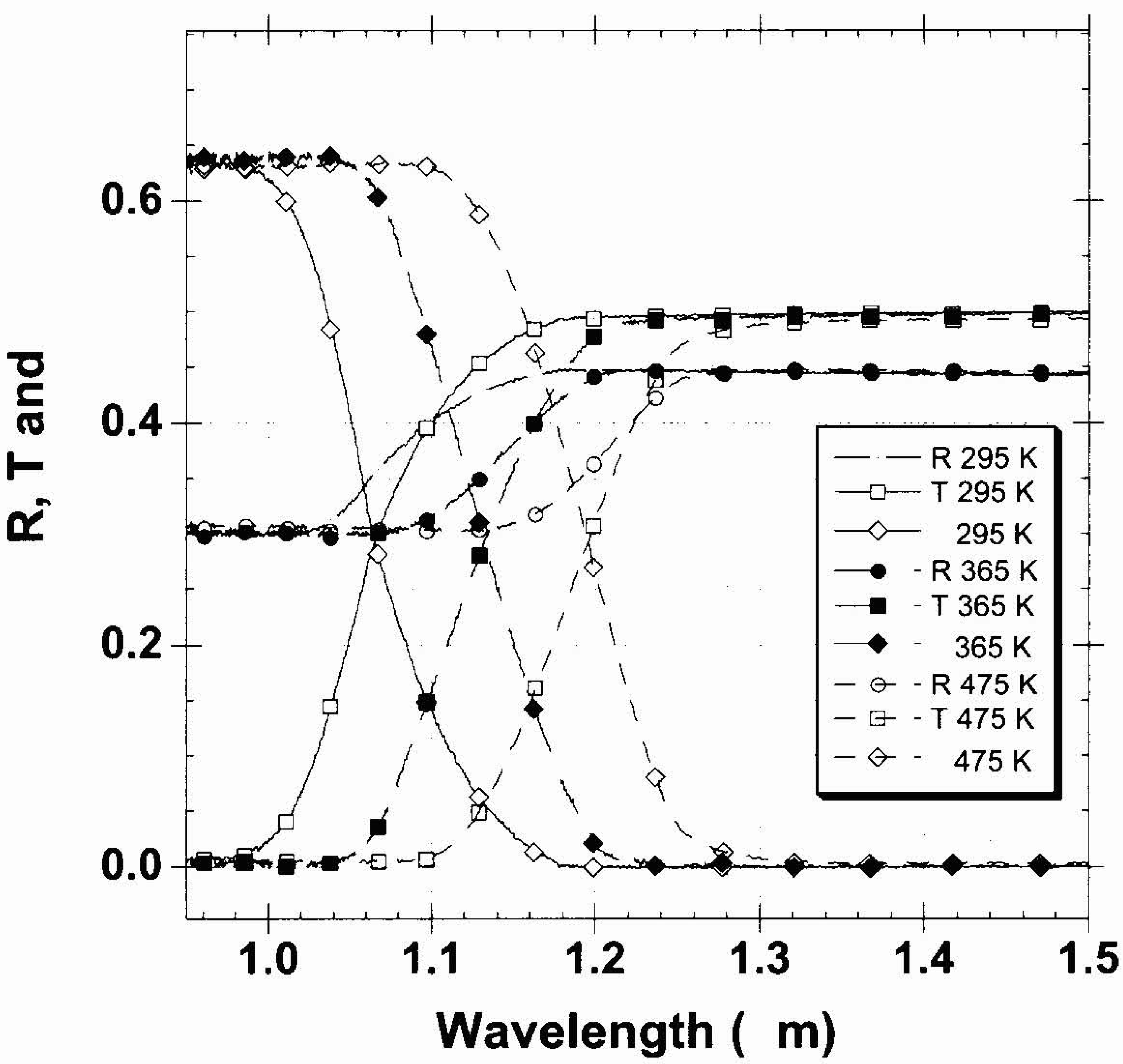

Figure 2. Measured reflectance, transmittance and derived emittance for a silicon wafer at temperatures of 295 K, 365 K, and 475 K.

3. DIRECT EMITTANCE MEASUREMENT SYSTEM

A separate facility in the FTIR Laboratory has been developed for the measurement of spectral emittance using the direct method of radiance comparison of the sample with a blackbody (BB) reference source. Detailed descriptions of the facility, its components and methods can be found elsewhere.[1,9] Here we include a brief overview.

The facility consists of: (1) a set of reference blackbody sources mounted on a motorized stage for selection; (2) interchangeable sample heater/mounts on motorized translation and rotation stages; (3) a removable visible/near-infrared integrating sphere for measuring the sample temperature above 500 K; (4) low scatter interface optics to image the 3 to 5 mm central region of the sample or BB source onto a water cooled field stop; the field stop is re-imaged onto either (5) a Fourier transform spectrophotometer equipped with beamsplitters and detectors to cover a spectral range from the visible through the far infrared or (6) a set of filter radiometers mounted on a motorized translation stage for temperature scale transfer between the BB sources and for sample temperature determination (together with the integrating sphere (3)); (7) a sectored purge enclosure for the entire beam path; (8) electrical supply, signal, purge gas (Ar, or N_2), and cooling water subsystems; (9) control of system elements and data processing via several PC computers using LabView[10] software programs.

For determination of the sample emittance using the direct spectral radiance comparison method, one needs accurate measurement of: (1) the reference blackbody temperature; (2) the reference blackbody emissivity; (3) the sample temperature; and (4) the spectral distribution of the emitted light using the FTIR. Each of these elements are addressed in the facility design.

Each BB contains calibrated platinum resistance thermometer (PRT) or thermocouple (TC) temperature sensors. These are used to control and monitor changes in the BB temperature. For absolute temperature determination, two fixed point BB furnaces with interchangeable crucibles (containing Ag, Al, Zn, Sn and In) are used. Filter radiometers (with filters at 650 nm, 900 nm, 1550 nm, and 2400 nm) are used to transfer the scale from the fixed points to 4 variable temperature BB's covering a temperature range of 250 K to 1400 K.

The spectral emissivities of the BB's have been calculated using a Monte Carlo ray tracing algorithm with input of the measured spectral reflectance of the cavity wall materials and coatings.

For temperatures below 500 K the sample temperature is determined from PRT or TC sensors embedded in the sample mount, or in the sample itself. Temperature gradients can exist between the sensor and the sample surface and, as discussed before, must be taken into account. This problem is typically worse at elevated temperatures. To overcome this problem, for temperatures above 500 K, the sample front surface temperature is determined by a non-contact method employing the vis-NIR integrating sphere and filter radiometers. The first step is a measurement of the sample's hemispherical-directional reflectance at the measurement temperature and at a single wavelength matched to the filter radiometer. A diode laser source input to the integrating sphere, is selected based on the temperature and rough emittance value of the sample. The reflectance is obtained via comparison to a calibrated standard. The second step is a relative radiance measurement of the sample to a BB at the same wavelength. The integrating sphere is removed for the second step. The temperature is then calculated from the results of these two steps. This procedure has the benefit of obtaining the temperature of the sample from the region on the sample identical to that for which the infrared spectral emittance is measured. It also makes use of the steep short wavelength edge of the Planck function for sensitive (and higher accuracy) temperature measurement.

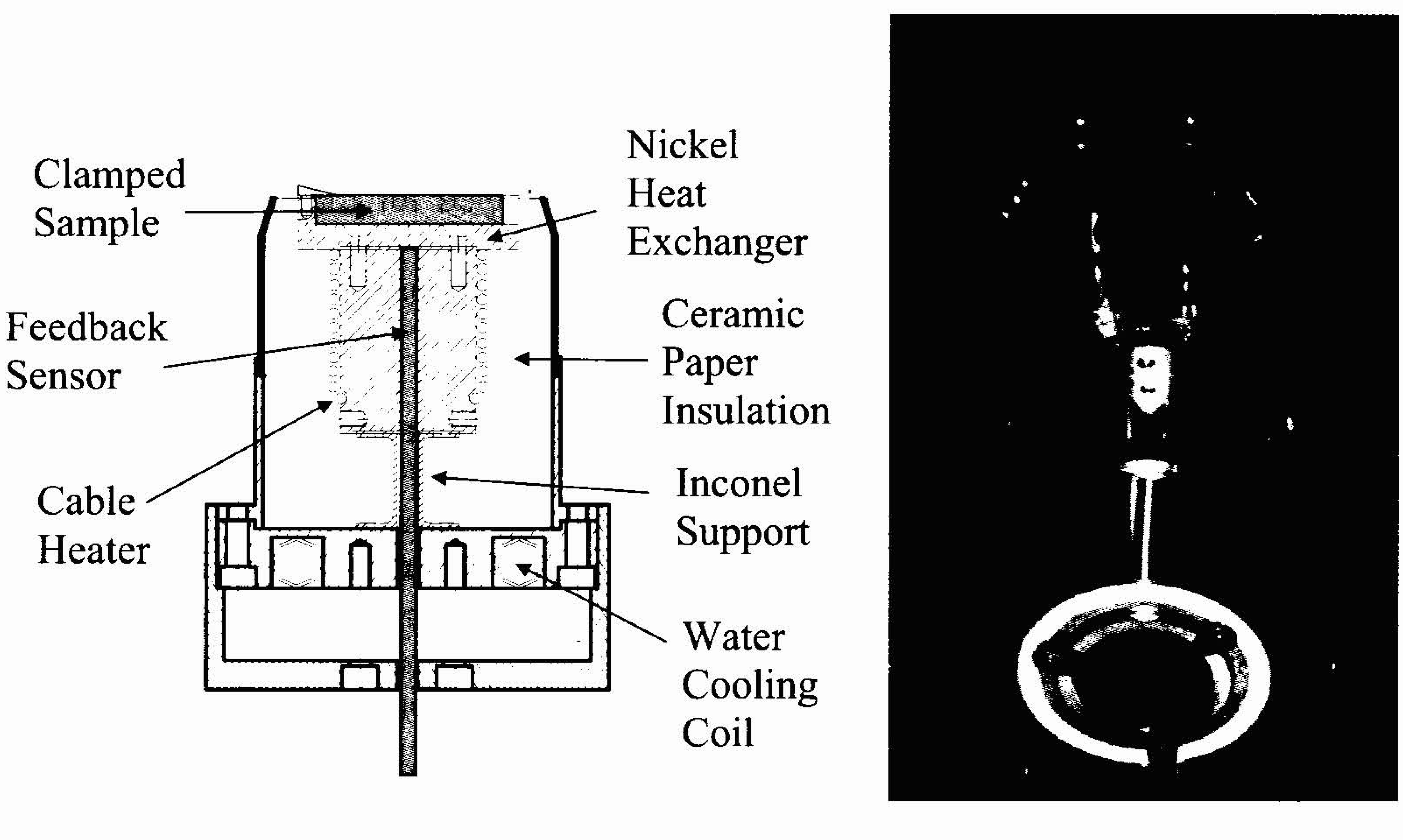

Figure 3. The first of several sample heaters to be built for the emittance characterization facility. It is designed to hold up to 1 inch diameter samples, heat them up to 900 K, and mount onto the integrating sphere reflectometer.

For the purposes of the direct-indirect method comparison, the measurements are performed at 473 K (current maximum of the infrared sphere sample heater/holder). For this temperature, the emitted sample light is insufficient for the non-contact temperature measurement (using the initially configured Si radiometer at 900 nm). Hence the comparison sample is made thick enough to contain a 0.5 mm hole drilled into its side, with a thermocouple mounted inside.

The comparison sample is a hexagonal SiC 1 inch diameter polished disk. The sample heater/holder shown in Figure 3 was used. The sample is inserted and held down with the three spring clamps as shown in the photograph. The thermocouple access is not shown in the sketch and is hidden in the photograph in Figure 3. The heat exchanger is made of nickel with thermoshield and water-cooling to prevent overheating of other components. The maximum sample temperature achievable is about 875 K.

4. COMPARISON RESULTS

Measurements of a SiC sample were performed on the two systems described in Sections 2 and 3. For the indirect emittance system, the FTIR was used with a SiC ceramic source and coated KBr beamsplitter. The infrared integrating sphere uses an MCT detector. For the direct emittance system, the FTIR was also used with a KBr beamsplitter and a DTGS pyroelectric detector. The measurement time for both systems was approximately 1 hour. The measured emittance of the sample obtained from the two systems is shown in Figure 4.

The measurements described here are some of the first ones performed with the direct emittance system. Hence they are preliminary in nature and we have not included error bars or uncertainties. For example, at 200 °C, the sample reflected background radiation may result in significant measurement error for the direct method. We plan to construct a controlled background (black sphere) system to enable more accurate measurements near ambient in the future.[1] Nevertheless the agreement between the methods is very encouraging. The structure seen at short and long wavelengths is noise due to atmospheric absorption (the direct measurement was performed in air, the indirect under purge). The only visible difference (0.02) in the results is in the region of low emittance/high reflectance from 11 μm to 13 μm.

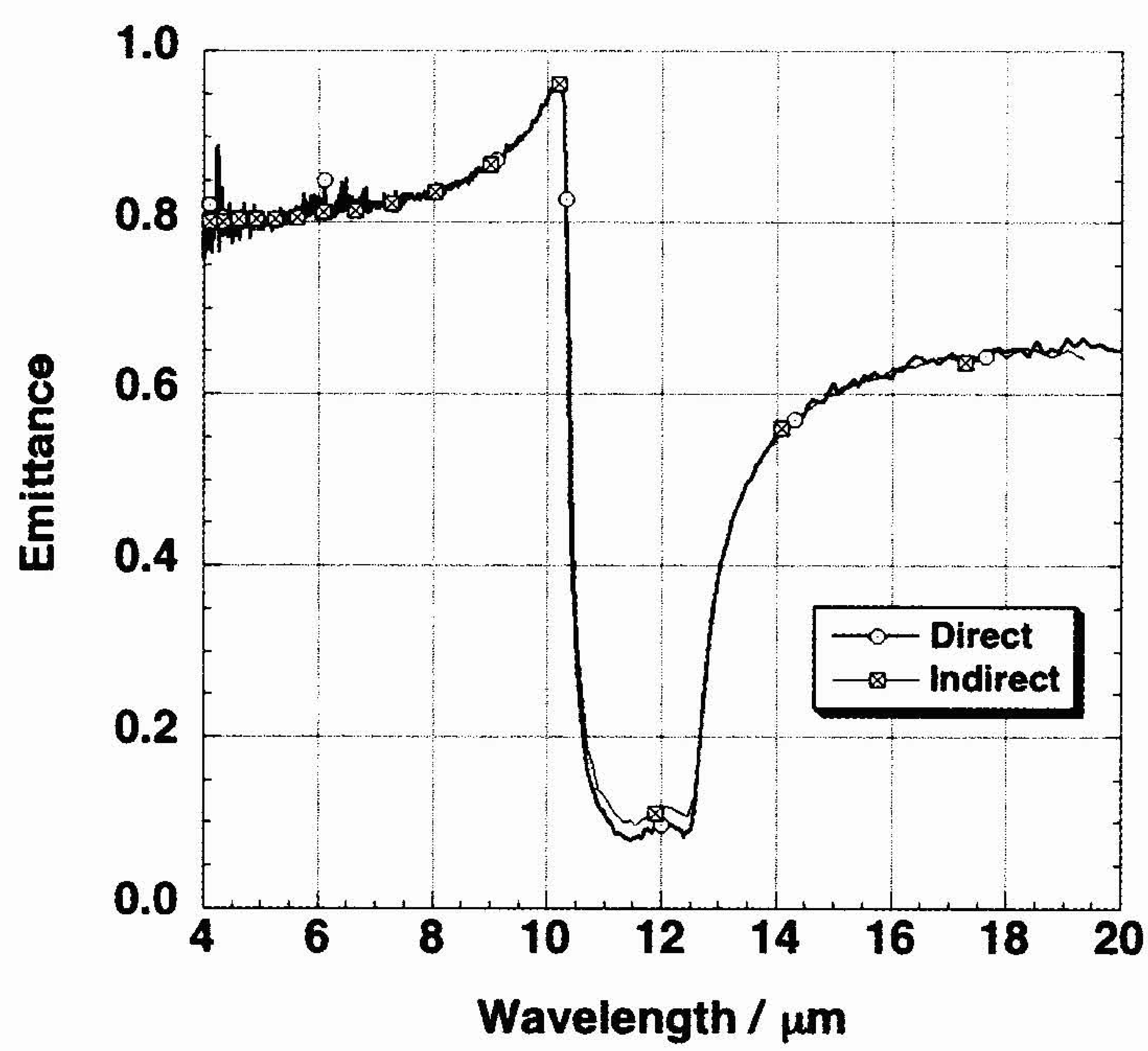

Figure 4. Comparison of the direct and indirect emittance measurement results obtained for a specular (polished) sample of hexagonal SiC at 473 K.

5. CONCLUSIONS AND FUTURE WORK

We have developed independent measurement systems for the characterization of infrared spectral emittance of materials that employ independent methods (direct and indirect). Using these two systems we have performed a comparison of the infrared emittance of a SiC sample from 4 μm to 18 μm. The emittance results obtained from the direct and indirect methods were in good agreement in consideration of the low temperature of 473 K at which the comparison was performed. Other materials are being studied and sources of measurement error are being investigated. Based on this ongoing work, an uncertainty budget will be developed.

The results of the low temperature comparison studies will provide confidence in the results obtained at elevated temperature with the direct emittance system. We anticipate an even greater benefit from the future deployment of the controlled background system, which will have considerable temperature overlap with both the current direct and indirect emittance measurement systems

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[10] Certain commercial equipment, instruments, or materials are identified in this paper to specify the experimental procedure adequately. Such identification is not intended to imply recommendation or endorsement by the National Institute of Standards and Technology, nor is it intended to imply that the materials or equipment identified are necessarily the best available for the purpose.

Address of the Authors:

National Institute of Standards and Technology,
100 Bureau Dr., Stop 8442, Gaithersburg, MD, USA,
tel. 301 975 2344,
fax 301 840 8551,
e-mail: hanssen@nist.gov,
website: www.nist.gov