

Validation of the Infrared Emittance Characterization of Materials Through Intercomparison of Direct and Indirect Methods

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Abstract A comparison of the spectral directional emittance of samples as a function of wavelength was performed at the Fourier Transform Infrared Spectrophotometry (FTIS) and the Advanced Infrared Radiometry and Imaging (AIRI) facilities at NIST. At the FTIS, the emittance is obtained indirectly through the measurement of near-normal directional-hemispherical reflectance (DHR) using an infrared integrating sphere. At the AIRI, the normal directional emittance is obtained directly through the measurement of the sample spectral radiance referenced to that from blackbody sources, while the sample is located behind a black plate of known temperature and emittance. On the same setup at the AIRI, the normal emittance at near ambient temperatures is also measured indirectly by a “two-temperature” method in which the sample spectral radiance is measured while the background temperature is controlled and varied. The sample emittance measurements on the comparison samples are presented over a wavelength range of 3.4 μm to 13.5 μm at several near-ambient temperatures and for near-normal incidence. The results obtained validate the two independent capabilities and demonstrate the potential of the controlled background methods for measurements of the radiative properties of IR materials.

Keywords Directional reflectance · Emissivity · Emittance · Fourier spectrometer · Hemispherical reflectance · Integrating sphere · Radiance · Radiance temperature

1 Introduction

Measurements of such infrared (IR) optical properties of materials as directional-hemispherical reflectance (DHR) and transmittance are well established at many

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national metrological laboratories, including NIST [1], providing vital data for numerous applications in science and technology. Now these measurements are the most common way of obtaining the spectral directional emissivity (SDE) data, required for non-contact temperature measurements and target discrimination. At the same time, great variability in measurement conditions such as sample temperature, the spectral range of measurements, the geometry of incident, and collected radiation fluxes, as well as in the characteristics of sample optical properties (such as the character of reflected and transmitted radiation, polarization properties, etc.), makes meeting the ever-growing customer demands extremely challenging.

In recent years, the requirements of direct radiometric measurements of spectral radiance and radiance temperature of thermal emission sources in the $3\text{ }\mu\text{m}$ – $14\text{ }\mu\text{m}$ range have resulted in improved experimental techniques, with several laboratories claiming uncertainties in the neighborhood of $0.02\text{ }^{\circ}\text{C}$ to $0.05\text{ }^{\circ}\text{C}$ at near-ambient temperatures, which correspond to 0.02% to 0.05% in spectral radiance [2,3]. If such an approach could be extended to surface emitters (rather than cavity emitters with their inherently higher emissivities), this may potentially enable similar or even smaller uncertainties in the SDE metrology compared with indirect techniques based on DHR measurements.

The following section briefly discusses hardware and experimental methods employed at both the FTIS and AIRI facilities. The comparison measurement results are presented and analyzed in Sect. 3.

2 Measurement Methods

2.1 FTIS/Integrating Sphere Reflectometer System

The FTIS integrating sphere instrument is used to measure absolute reflectance and transmittance of solid material samples, specular and diffuse, opaque and transparent, over a spectral range of $1\text{ }\mu\text{m}$ to $18\text{ }\mu\text{m}$. Custom absolute methods have been developed for use with the sphere and are described in detail elsewhere [1,4]. The measurement system includes a sample heater unit that can control sample temperatures between $15\text{ }^{\circ}\text{C}$ and $200\text{ }^{\circ}\text{C}$. The heater is designed to allow both transmittance and reflectance measurements. Details of the heater can be found in Ref. [5], which describes a comparison of reflectance measurement results from the integrating sphere system with a separate FTIS spectral emissivity system designed primarily for temperatures above $300\text{ }^{\circ}\text{C}$.

The samples measured in this study were at an ambient temperature of $23\text{ }^{\circ}\text{C}$ and not temperature controlled for the reflectance measurements. The emissivity is obtained simply from energy conservation and Kirchhoff's law:

$$\varepsilon(\lambda, \theta_i, T) = \alpha(\lambda, \theta_i, T) = 1 - \rho(\lambda, \theta_i, T) - \tau(\lambda, \theta_i, T), \quad (1)$$

where λ is the wavelength, θ is the direction angle, T is the sample temperature, ε is the directional spectral emissivity, α is the directional spectral absorptance, ρ is the DHR, and τ is the directional-hemispherical transmittance (DHT) (which is equal

to zero for this study). Some instrumentation measures the hemispherical-directional reflectance (HDR) and transmittance (HDT) factors, which are equivalent, by reciprocity, to the DHR (and DHT). For opaque samples, as the ones in the comparison, the transmittance is zero and the correspondingly simplified version of Eq. 1 is used in this work.

2.2 AIRI/Controlled Background Plate System

The AIRI facility [6] was designed to characterize extended area sources, with radiating areas up to 300 mm × 300 mm, for their spectral radiance and emissivity as a function of temperature, wavelength, position over the radiating area, and background temperature. Figure 1 depicts a schematic diagram and photo of the spectral calibration setup for customer BB sources. The diagram shows the sample plate (or customer blackbody) facing the IR spectroradiometer, with an intervening controlled background plate (CBP), similar to the one previously used successfully at the CSIRO [7]. The photo shows the same setup. Large horizontal and vertical stages (in the front of the photo) carry the plate (or blackbody) for mapping, and a tilt stage is used for angle adjustment. The spectroradiometer, partially hidden by the CBP, is mounted on a large translation stage in order to view additional adjacent reference blackbodies outside the picture. The CBP, held in an aluminum frame, is essentially a sandwich of two black plates, the temperatures of which are independently controlled. The two methods described below are based on control of the background temperature.

The first method, which we will call the absolute radiance method (ARM), involves radiometric measurements of the spectral radiances of the sample and the enclosure (background) and also requires knowledge of the sample surface temperature. The simplified measurement equation, taking into account only self-emitted and reflected background radiation, is

$$L_s = \varepsilon_s P(T_s) + (1 - \varepsilon_s) L_E, \quad (2)$$

where T_s is the sample temperature (known from contact measurements), $P(T_s)$ the radiance of a perfect blackbody at the sample temperature T_s (as defined by the Planck equation), and L_s and L_E are experimentally measured radiances of the sample and the enclosure (background), respectively. From Eq. 2, the emissivity is given by

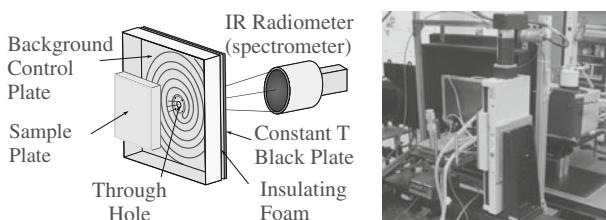


Fig. 1 Schematic and photograph of the arrangement used to realize the controlled background methods of emissivity measurements

$$\varepsilon_s(T_s) = \frac{L_s - L_E}{P(T_s) - L_E}. \quad (3)$$

2.3 Radiometric Radiance Comparison: Sample Temperature

Utilizing the sample emittance results from the sphere reflectometer, the sample surface temperature can be calculated by a radiometric comparison of the spectral radiance of the sample to that of a blackbody source of known emittance and temperature. Starting off with Planck's radiation law for spectral radiance and taking care of the spectral responsivity of the filter radiometer as well as the blackbody cavity spectral emittance, the measurement equation for this radiometric comparison can be written as

$$\frac{V(\lambda, T)}{V_{BB}(\lambda, T_{BB})} = \frac{\varepsilon(\lambda, T) \left(e^{\frac{c_2}{\lambda T_{rad}}} - 1 \right)}{e^{\frac{c_2}{\lambda T_{BB}}} - 1}, \quad (4)$$

where $V(\lambda, T)$ and $V_{BB}(\lambda, T_{BB})$ are the filter radiometer signals for the sample and the blackbody and $\varepsilon(\lambda, T)$ is the sample emittance from the reflectometer measurement.

Another approach is to measure the apparent radiance of the sample and the enclosure at two different background temperatures, maintaining the sample at the same temperature. We will refer to this method as the two (background) temperature method (2TM). For the two measurements, Eq. 2 becomes

$$L_{s1} = \varepsilon_s P(T_s) + (1 - \varepsilon_s) L_{E1} \quad (5)$$

and

$$L_{s2} = \varepsilon_s P(T_s) + (1 - \varepsilon_s) L_{E2}, \quad (6)$$

where the indices 1 and 2 denote the first and second background temperatures, respectively. The emissivity can be solved for and is given by

$$\varepsilon_s = 1 - \frac{L_{s1} - L_{s2}}{L_{E1} - L_{E2}}. \quad (7)$$

Knowledge of the sample's absolute temperature is not required, although it is important to maintain its surface temperature constant or measure its change to introduce necessary corrections.

3 Results and Discussion

In the first experiment, an extended area blackbody SR-80A-HMIS [8], manufactured by CI Systems, and a sample coupon of its surface, shown in Fig. 2, were measured using the ARM method (AIRI facility) and the integrating sphere reflectometer (FTIS),

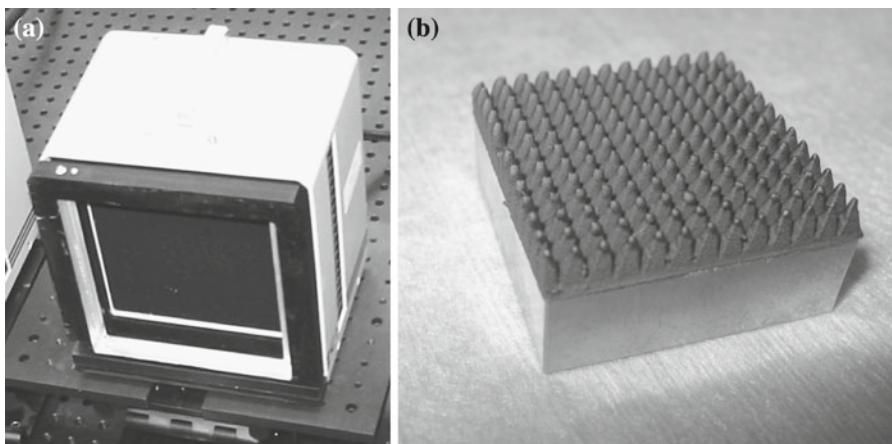


Fig. 2 Blackbody SR-80A and its radiating surface sample

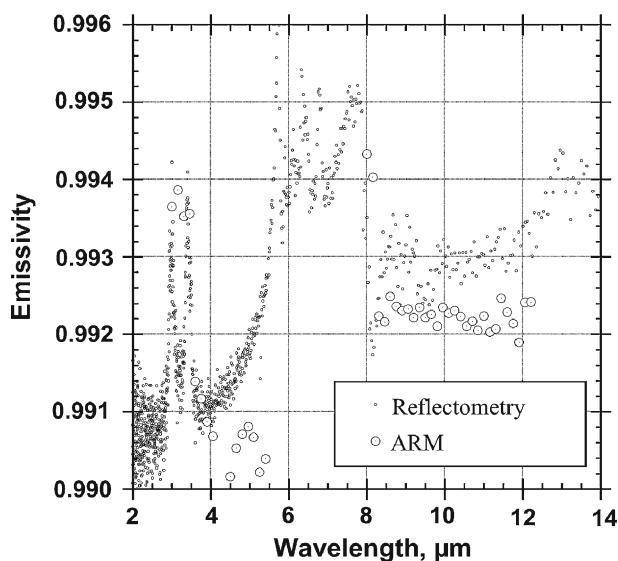


Fig. 3 Comparison of BB spectral emissivity values obtained via reflectometric and radiometric (ARM) measurement methods

respectively. In Fig. 2b, one can see surface features that are designed to enhance the emissivity to achieve the nominal value of 0.99 as listed in the technical specifications.

The integrating sphere reflectance measurements were performed on the coupon at room temperature (23°C). The radiometric measurements were performed on the blackbody at the temperature set point of 70°C , which was used in the calculations as the true surface temperature. Since the AIRI facility does not currently allow purging of the optical path, the associated data shown were taken only in the atmospheric transmission windows of $3.4\text{ }\mu\text{m}$ to $5.4\text{ }\mu\text{m}$ and $8\text{ }\mu\text{m}$ to $12\text{ }\mu\text{m}$. A comparison of the reflectometry and the ARM determined normal spectral emissivities is shown in Fig. 3.

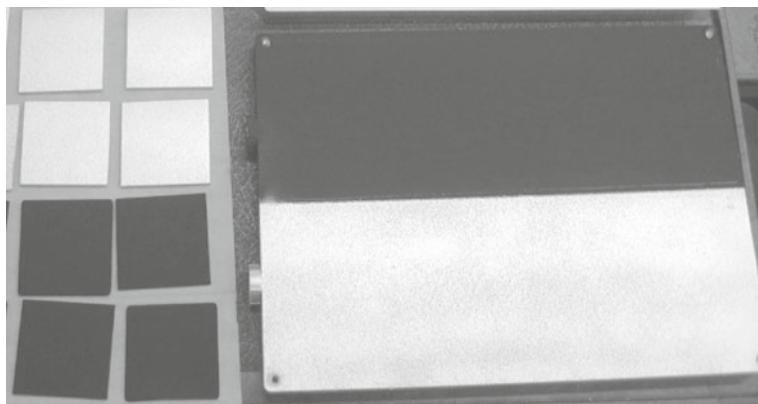


Fig. 4 Sample plate and coating witness samples, used in the second set of measurements

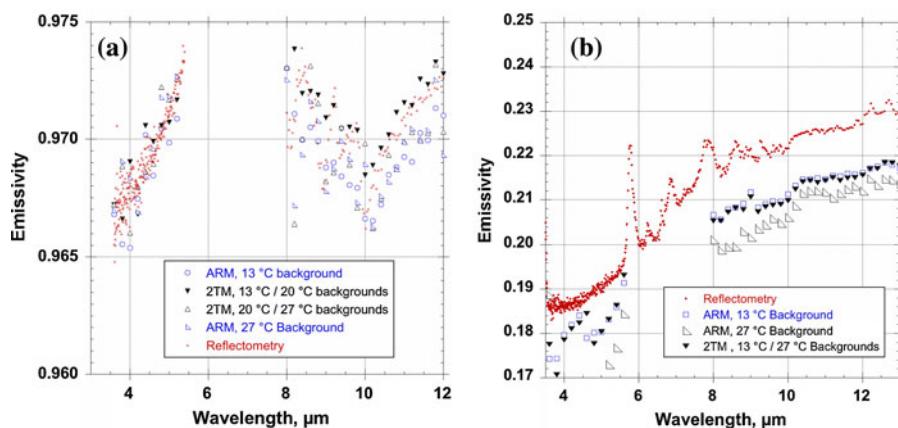


Fig. 5 Measurement results for (a) black paint and (b) grey paint surfaces

Both sets of data exhibit the same spectral variations, and agree to within 0.001 over most of the spectra, which is the expanded ($k = 2$) uncertainty of the reflectometry data.

Another set of measurements was undertaken with a 200 mm \times 280 mm temperature-controlled sample plate, and 50 mm \times 50 mm witness samples painted with diffuse grey and diffuse black paints, as shown in Fig. 4.

Figure 5 below summarizes the results of the measurements, including the reflectometry measurement performed using the sphere at the FTIS facility, and both the ARM and 2TM methods at the AIRI facility. The results demonstrate close agreement of all three methods for the black paint surface. In the case of the grey paint surface, the spread in the results is greater, with better agreement of the ATM and 2TM methods, but offset from the reflectometry results. This offset is less than the expanded uncertainty of the reflectometry results.

We are in the process of performing numerical modeling of different sources of uncertainty for the 2TM and ARM methods, which will help to identify the major ones and improve the performance of the facility. The components of uncertainty of the comparison already established include (a) the expanded ($k = 2$) uncertainties of the reflectometry measurements, which are 0.001 and 0.023 for the black paint and grey paint coupons, respectively; (b) the spreads of the coupon paint emittance values of approximately 0.002 and 0.010 for the black paint and grey paint coupons, respectively; and (c) the non-uniformities of the target plate emissivity of approximately 0.001 and 0.010 for the black paint and grey paint coupons, respectively. Combined, these factors can easily account for the differences seen in the comparison results.

4 Conclusions

We have performed the first experiments aimed at a comparison of measurements using two facilities, the AIRI and the FTIS, which have overlapping capabilities in the measurement of the SDE of IR materials at near-ambient temperatures. The results obtained validate the two independent capabilities and demonstrate the potential of the controlled background methods for measurements of the radiative properties of IR materials.

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8. 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