Infrared Spectral Emissivity Characterization Facility at NIST

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ABSTRACT

A new facility for the measurement of spectral emittance (emissivity) of materials that employs a set of blackbody sources is being built at NIST. This facility has also been used to investigate the capabilities of Fourier transform (FT) spectrometers to characterize the spectral emissivity of blackbody sources. The facility covers the spectral range of 1 µm to 20 µm and temperatures from 600 K to 1400 K. The principle of operation involves the spectral comparison of an unknown source with a group of variable temperature and fixed point reference sources by means of the FT spectrometer and filter radiometers. Sample surface temperature is measured by non-contact method using a sphere reflectometer. The current reflectometer setup allows measurements of opaque samples, but it is planned to include semitransparent materials at a later stage.

Keywords: spectral emittance, spectral emissivity, infrared, blackbody, sphere reflectometer,

1. INTRODUCTION

To meet the growing needs for data and standards of emittance of materials, a facility for the characterization of infrared spectral emittance has been under development at NIST for the past several years.[1] The facility is designed to provide emittance measurement capability in support of the requirements of a wide number of industries and applications including metals, glass, semiconductor, and plastics processing, aerospace and defense, energy, remote sensing, building and fire research, medicine, etc.

The facility is part of the Fourier Transform Infrared Spectrophotometry Laboratory devoted to the characterization of the optical properties of solid materials. The Laboratory covers the infrared spectral range of 1 µm to 100 µm, with particular emphasis on the 2 µm to 20 µm region. It is built around several commercial Fourier transform infrared (FTIR) instruments. Custom specialized accessories have been developed to enable transmittance and reflectance measurements of a wide variety of sample types and under the variable control of measurement geometry, beam polarization and sample temperature. [2,3,4,5] Methodologies and new techniques have been developed for high accuracy measurements.[6,7] In addition to the directly measured quantities, these have also been implemented for other properties such as refractive index and Mueller matrix elements. With the advent of the new emittance facility, we have added direct emittance to the suite of measured properties.

The emittance facility is envisioned to be comprehensive for the characterization of solid samples, specular and diffuse, opaque and transparent, at temperatures from 250 K to 1400 K, wavelengths from 1 µm to > 20 µm, and angles from 0° to > 75°. The development of the emittance facility is proceeding in 3 stages: Stage 1 - opaque samples, 600 K < T < 1400 K; Stage 2 – opaque samples, 290 K < T < 600 K; Stage 3 – transparent samples. Stage 1 has recently been completed.

2. MEASUREMENT METHOD AND INSTRUMENTATION

For the direct measurement of spectral emittance of a material, one must compare the spectral radiance of a sample to that of a blackbody at the same temperature. This requires several system and measurement components: (1) blackbody

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sources at the appropriate temperature, and a means of determining the temperature; (2) sample hardware for heating and manipulation, along with (3) a means of accurate temperature measurement; (4) a spectrometer such as an FTIR; and (5) an interface optics system for viewing the blackbody and sample and switching between them. The emittance characterization facility consists of several major subsystems designed to accomplish these requirements. These are shown schematically in Figure 1. A reference source system consists of several blackbodies covering different temperature regions that can be selected via a translation stage. A sample system consists of several interchangeable heater mounts and manipulation stages. The interface optics is not shown except for the rotatable mirrors, #1 for source selection and #2 for detector selection. The emitted radiation is measured with an FTIR Spectrometer or filter radiometer, as selected by means of mirror #2 and a translation stage. As the primary means of sample temperature measurement, an integrating sphere reflectometer with a calibrated reference sample is used. The additional components shown selectable by mirror #1 are planned for the low temperature Stage 2: a liquid N_2 blackbody and sample inside a blackbody reflectometer.

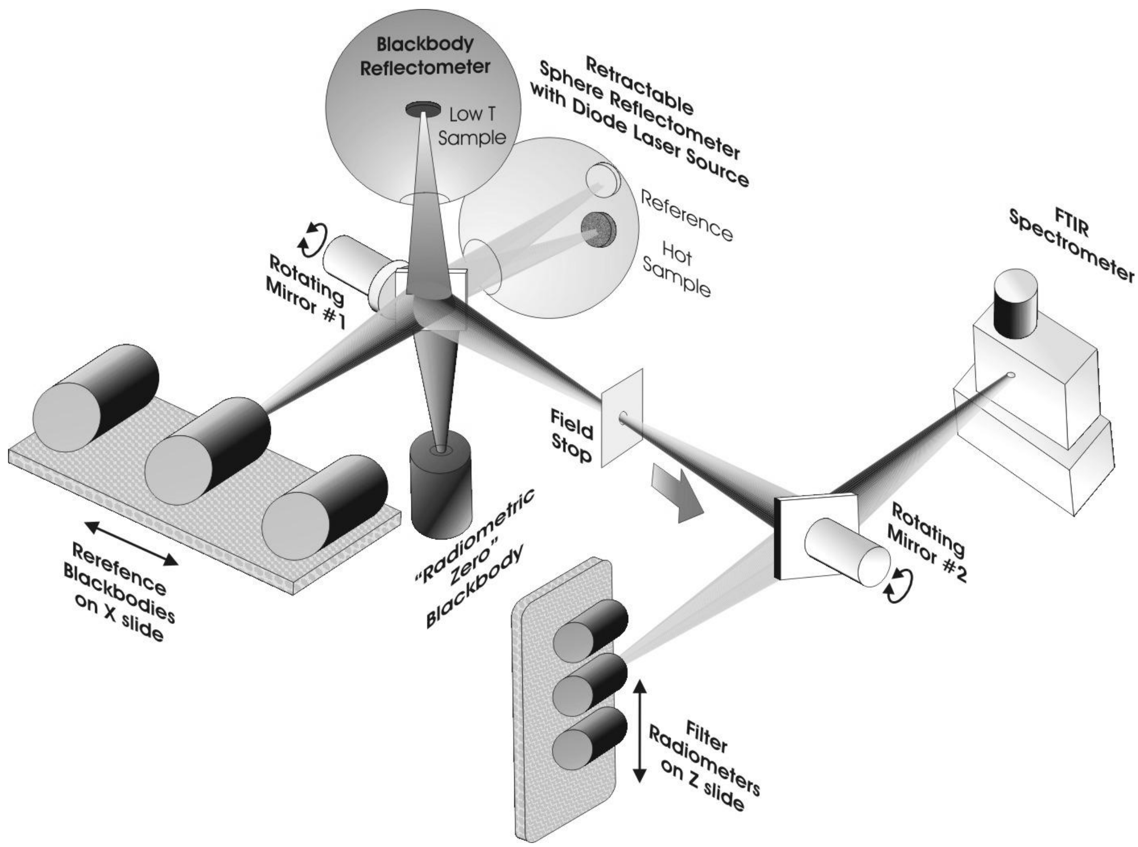


Figure 1. Schematic of Infrared Spectral Emittance Characterization Facility. Radiation from blackbody reference sources (to the left) is compared with that from a sample (located opposite the blackbodies) via rotation of a selection mirror #1. It proceeds through a set of interface optics (not shown) and is measured with an FTIR Spectrometer or filter radiometer, selected by means of mirror #2. For sample temperature measurement, a sphere reflectometer with a calibrated reference sample is used. The additional components shown are planned for low temperature measurements: mirror #1 will select a liquid N_2 blackbody and a sample mounted inside a blackbody reflectometer.

An overview of key components of the facility (including all Phases) and their relationship is shown in Figure 2. The temperature ranges from right (250 K) to left (1400 K). At the top are listed the fixed-point blackbody sources that are used to define the temperature scale according to ITS-90. The emittance facility has two fixed-point furnaces with interchangeable crucibles that contain In, Sn, Zn, and Al, Ag, and Cu, respectively. In addition the facility has four variable temperature blackbodies: a low temperature blackbody controlled by a fluid bath recirculator, and three blackbodies containing water, cesium and sodium heat pipes. Between these are shown four filter radiometers: InSb at 3700 nm, InGaAs at 1550 nm, Si at 900 nm, and Si at 650 nm. These radiometers have a dual purpose: first to transfer the temperature scale from the fixed-points to the variable temperature blackbodies, and second to perform the measurements together with the integrating sphere reflectometer for sample temperature determination. For monitoring the stability of the blackbodies, platinum resistance thermometers are used below 900 K, and a silicon optical pyrometer above 850 K. In addition, an InGaAs pyrometer may be built for Phase 2. For sample temperature measurement contact thermometers are used for lower temperatures. For temperatures above 500 K and especially for poorly conducting samples, we employ a non-contact method described below that includes the use of an integrating sphere reflectometer. For Phase 3, a black sphere reflectometer will be constructed.

2.1. Reference Source System

The blackbody reference source system consisting of six blackbodies and control electronics is shown mounted on a motorized translation stage in Figure 3. The variable temperature blackbodies have matching temperature-controlled apertures, water cooling and gas purge lines, and temperature sensors within both the heating elements and heat pipes. They are specifically constructed to provide excellent stability and uniformity, and an optimized cavity design with a high emittance paint coating. At the same time the design provides sufficient area, solid angle and consequent flux to serve as reference for sample spectral emittance characterization with apertures up to 12 mm and FOV of f/7, and to fit with a limited footprint onto the base optical table and within its own purged light-tight box.

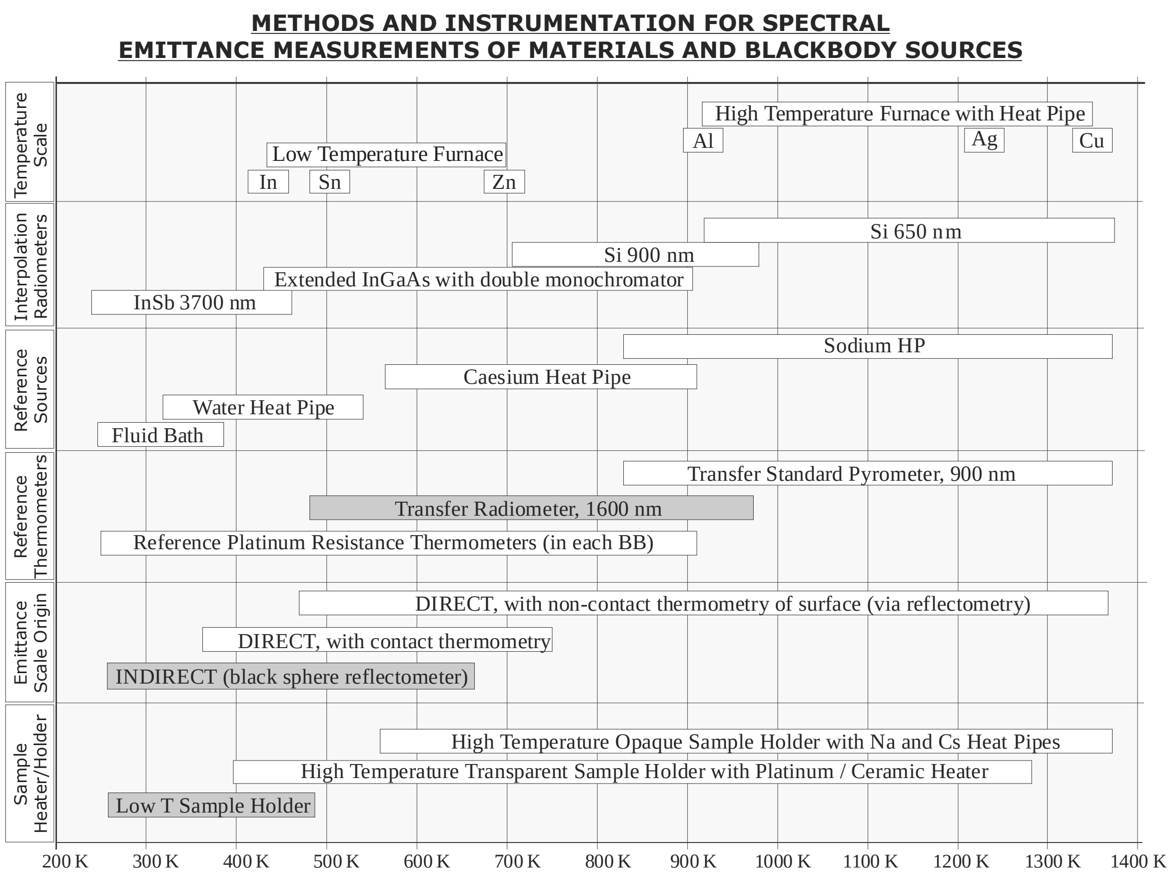


Figure 2. Overview of key elements of the emittance characterization facility showing the temperature range coverage and corresponding relationship to each other. Elements for Phases 2 and 3 are shown in gray.

The fixed-point blackbodies need to be stable (provide a freeze "plateau") for a sufficient period of time to allow comparative measurements of them with each other or variable temperature blackbodies by either the filter radiometers or the FT spectrometer. A freeze plateau of 3 hours for our Al fixed-point is shown in Figure 4. The ultimate plateau limit has not yet been determined, as each measurement was purposely terminated due to considerations other than the fixed-point behavior. Similar performance has been observed for the Ag fixed-point. The others have yet to be tested.

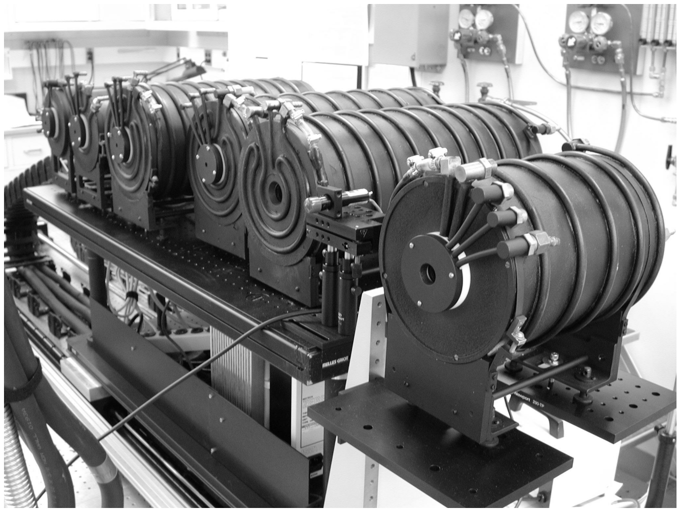


Figure 3. Early photo of blackbody reference source system. From front to back: low T fixed-point BB, high T fixed-point BB, sodium heat pipe blackbody, cesium heat pipe blackbody, water heat pipe blackbody, and T-controlled liquid blackbody. The blackbodies and control electronics are mounted on a translation stage.

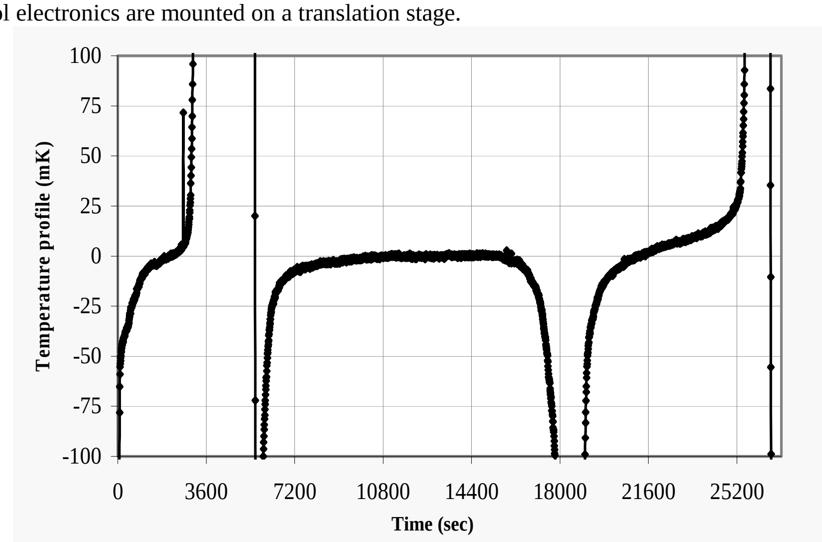


Figure 4. Melt / freeze curve of the Al fixed point blackbody source in emittance characterization facility. The change in radiance temperature of the fixed-point cavity as measured by a Si transfer pyrometer is plotted over a period of 7.5 hours as the fixed point is driven through a melt-freeze-melt sequence. In this case the fixed-point provided 3 hours of stable output for use in temperature scale transfer or sample characterization.

2.2. Sample System

For the sample component of the emittance facility, several sample mounts with different heater designs will be used. A schematic and photo of the first one constructed is shown in Figure 5. The heater element is wrapped around a solid nickel heat exchanger, surrounded by insulation. A central access hole contains a Pt resistance thermometer (PRT) for heater/sample temperature monitoring. The base is water-cooled. The unit is designed for use in air from 300 K through 900 K, for samples up to 1 inch in diameter. The entire unit is sized to be compatible with mounting on the integrating sphere used for accurate sample temperature determination. This heater is used for the sample emittance measurement discussed in the Section 3.

Additional heaters have been built including one designed with appropriate central clearance for holding and heating transparent samples. This heater is designed for temperatures approaching 1300 K. Further we are building a pair of heaters for opaque samples that employ small heat pipes (Cs and Na) for maximum efficiency and greatest temperature uniformity.

For good conducting materials and for moderate temperatures, the use of contact thermometers built into the sample heater or touching the sample by various means can produce an acceptable temperature measurement. But for temperatures greater than 500 K and especially for poorly conducting samples, non-contact methods can produce more accurate results. We have followed the approach taken by IMGC in Italy.[8] We use a visible/near-infrared integrating sphere to make a high accuracy reflectance measurement of the sample at the temperature of interest and under optimized wavelength and geometry conditions. From this, via Kirchoff's Law, we obtain the sample emittance at a single or narrow band of wavelengths. Then the integrating sphere is removed and a relative spectral radiance measurement is made via comparison to a blackbody source at the same wavelength(s) and temperature. From these two measurement results, the sample temperature is calculated. The primary advantage of this method is that the measurements are performed directly on the sample region of interest – the center of the sample surface.

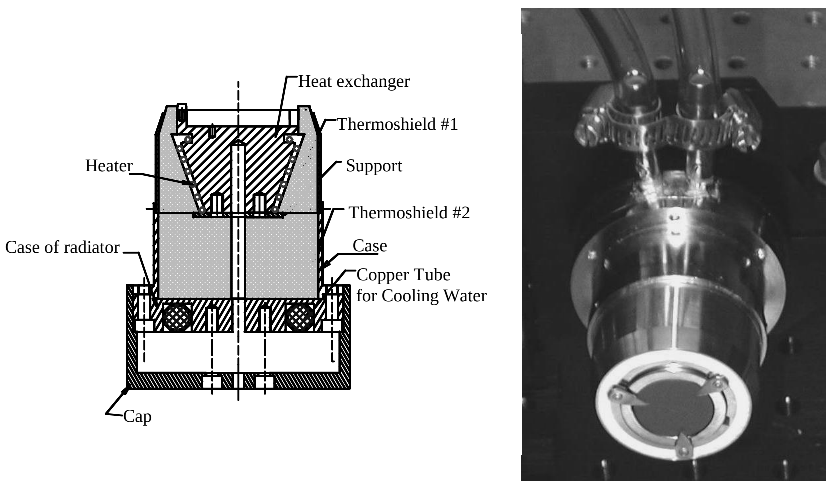


Figure 5. The first of several sample heaters to be built for the emittance characterization facility. It is designed to hold up to 1 inch diameter samples, heat them up to 900 K, and mount onto the integrating sphere reflectometer.

The integrating sphere reflectometer is shown in Figure 6(a), (b) and (c): a schematic of the sphere and measurement geometry, sample/source insert, and assembled sphere, respectively. The sphere is a hemispherical-directional design, in which one compares the directional flux (at 0° or 11°) reflected off a hemispherically illuminated sample to that of a calibrated reference. The sphere has a diameter of 10 inches and is coated with a high reflectance sintered poly-tetraflouroethylene shell (except for the sample/source insert shown in (b) which has a BaF_2 coating). Diode laser and halogen lamp sources are coupled via fiber optics to a nearly Lambertian diffuser at the sphere wall located between the sample and reference ports. Two triangular-shaped baffles, sized to minimize perturbation of the sphere geometry, prevent direct illumination of the sample or reference. An external mirror in the interface optics system is used to alternate viewing the sample and reference. The sphere design reflects the results of considerable modeling and analysis described in previous work.[9,10] Characterization of the sphere performance including the measurement of sample temperature are underway.

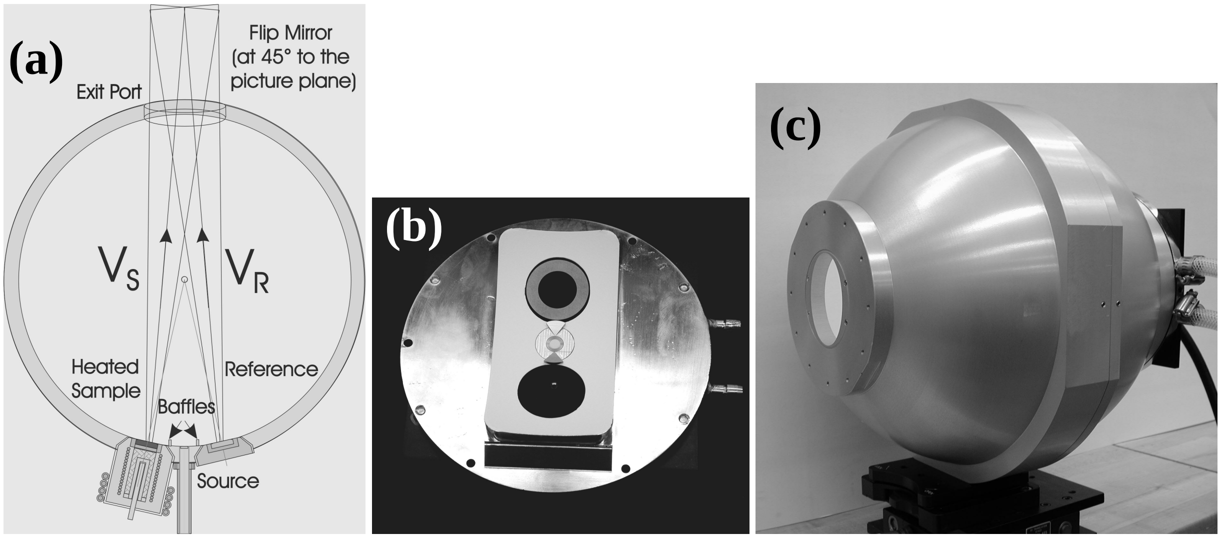
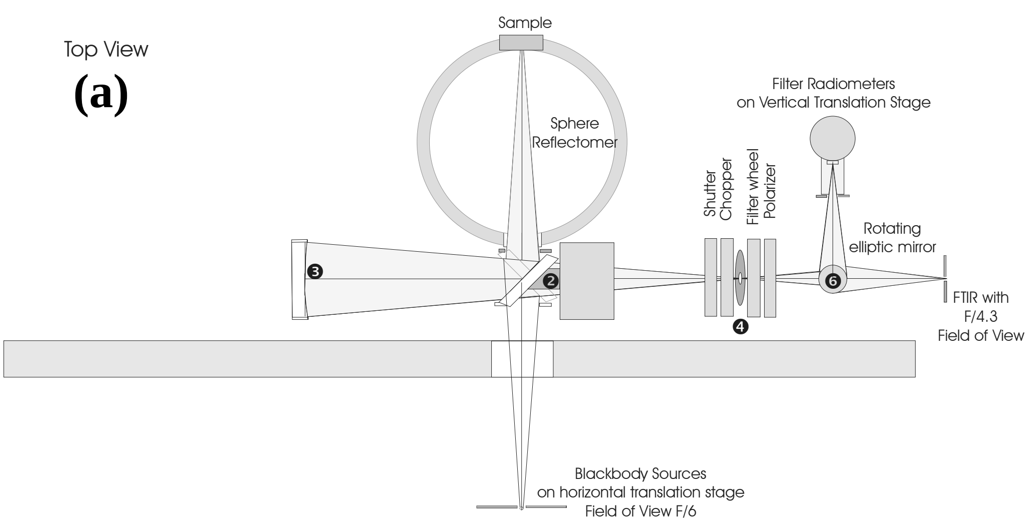


Figure 6. Integrating sphere reflectometer used for sample temperature measurement. In (a) the sphere provides a hemispherical-directional reflectance measurement via a ratio of measured signals V_s and V_r from the heated sample and a calibrated reference. These are baffled from a nearly Lambertian source mounted between them. These elements are all part of a removable and interchangeable water-cooled sample/source insert (b) that matches the sphere wall when assembled as seen in (c).

2.3. Interface Optics System

The source radiation is selected and transferred to the detection system via a set of interface optics shown in Figure 7. The all-reflective optics include four low-scatter mirrors (two flat, one elliptic and one toroidal) in an arrangement to minimize aberrations. The first mirror is mounted on a rotation stage that allows selection between sample (and reference), blackbody sources, liquid N_2 blackbody and potential future blackbody reflectometer. The primary field stop is a water-cooled aperture located with a filter wheel, polarizer, shutter and chopper. After redirection by a second flat mirror, the elliptic mirror on another rotation stage is used to underfill the FT emission port or one of a set of filter radiometers mounted on a vertical translation stage. Not shown are several lasers and visible sources that are used, together with kinematic mounting of each optical component, for both forward and backward alignment.



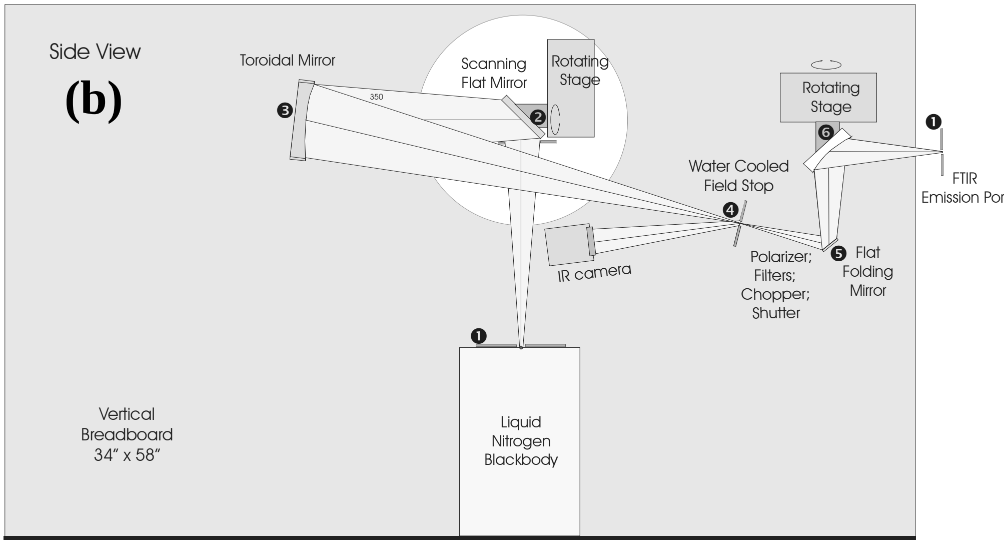


Figure 7. Top (a) and side (b) views of the emittance facility interface optics mounted on a common breadboard. (1) Temperature controlled apertures at the object and image planes; (2) rotatable selection mirror for source selection; (3) primary toroidal mirror; (4) primary filed stop with water-cooled aperture, filter wheel, polarizer, chopper and shutter and alignment camera; (5) flat folding mirror; and (6) rotatable elliptic mirror for detector selection.[11]

Size-of-source (SSE) measurements were performed on the interface optics to evaluate the potential errors due to scattering effects.[12] The SSE represents the relative contribution to the measured signal due to radiation from outside the nominal measured area of the source. This can lead to significant measurement error when comparing sources with differing backgrounds. In particular, for the integrating sphere reflectance measurement, the relative proximity of the source to the sample could be a problem. The SSE measurement can be used to evaluate and potentially correct the errors and provide uncertainty contributions. The results for our interface optics system are summarized in Figure 8. The relatively low value of 0.025 % means that for both relative radiance and reflectance measurements, other sources of error will dominate the uncertainty budget. We have built into the optics system additional equipment for in-situ periodic monitoring of the SSE.

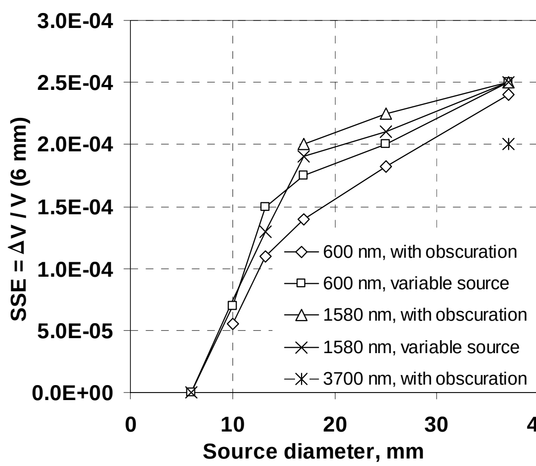


Figure 8. SSE characterization of the interface optics (up to the field stop). Results for both variable source and central obscuration methods at several wavelengths are consistent and show acceptable performance (Figure 4 from Reference 12).

2.4. Detection System

The primary spectral detector of the emittance characterization facility is a Fourier transform spectrometer. Currently a modified Bomem DA3 is used.[11] It is equipped with a complete set of detectors and beam-splitters for coverage of the visible through far-infrared spectral ranges. However, the primary emphasis is on the 1 µm to 20 µm range. In addition, a set of filter radiometers listed in Figure 2 are also used. These radiometers contain temperature-stabilized narrow-band filters with high ($>10^8$) out-of-band rejection, spatially uniform and temperature-stabilized (where appropriate) detector elements, and low-noise highly linear amplifiers. These radiometers have two purposes. First, they are used to transfer the temperature scale from the fixed-point to the variable temperature blackbodies and to monitor and characterize a number of blackbody parameters including short and long term stability, spatial and angular uniformity, linearity etc. The second purpose is to perform the sample temperature measurement together with the sphere reflectometer as described in Section 2.2

2.5. Control System

A large number of electronic instruments including motion controllers, power supplies, voltmeters, amplifiers, water bath recirculators and spectrometers, are used to operate the numerous system components and monitor a wide array of signals. Most of these electronics are in turn monitored or controlled by custom computer software written using Labview.[11] The measurement programs form a suite that includes the ultimate measurement and calculation of sample spectral emittance. The emittance program controls the FT spectrometer and radiometer measurements of blackbody and sample radiances as well as motion of the blackbody and mirror stages. Several examples of experiments performed using these programs are given in Section 3.

3. EVALUATION OF FACILITY PERFORMANCE

After construction, assembly and testing of the emittance facility component systems, we have begun to make system level performance tests including preliminary sample emittance measurements. After determination of the sample and blackbody temperatures, the basic measurement of relative spectral radiance is made in the following fashion. We employ the common practice of performing 3 measurements: along with any particular unknown sample or blackbody, we measure two “known” blackbodies at different temperatures (typically one near to that of the sample and one near ambient). The second blackbody measurement is used to subtract out the FT self-emitted radiance component. The spectral radiance of the unknown is then given by

$$L_C(\nu) = \mathrm{Re}\left[\frac{[V_C(\nu) - V_B(\nu)]}{[V_A(\nu) - V_B(\nu)]}\right][L_A(\nu) - L_B(\nu)] + L_B(\nu) \ , \tag{1}$$

where, $L(\nu)$ is spectral radiance, $V(\nu)$ is the FT measured complex spectrum, A is the higher T blackbody, B is the lower T blackbody, and C is the unknown. The spectral radiance of the blackbody is taken from Planck’s law and the emissivity as calculated or otherwise determined. The unknown spectral emittance is simply obtained from L by ratioing to the Planck function.

The measurements are performed in repeated cycles of an *A*-*B*-*C*-*B*-*A* sequence, to reduce effects of drift. An initial study and optimization of drift was performed. The total time for each experiment was 1 hour, with the number of cycles varying from 2 to 20 resulting in individual measurements of from 1 to 10 minutes. We measured the Cs heat pipe blackbody at approximately 500 °C, comparing it to itself (as both *C* and *A*). The H_2O heat pipe blackbody was used at 100 °C (as *B*). It was found that the 20 cycle experiment provided the result with the best stability. The mean value of the 20 cycles is shown in Figure 9. Hence drift of the system is essentially eliminated if one measures for very brief periods but in cycles repeated correspondingly many times. This is feasible because of the motorized blackbody stage and automation of the entire measurement process.

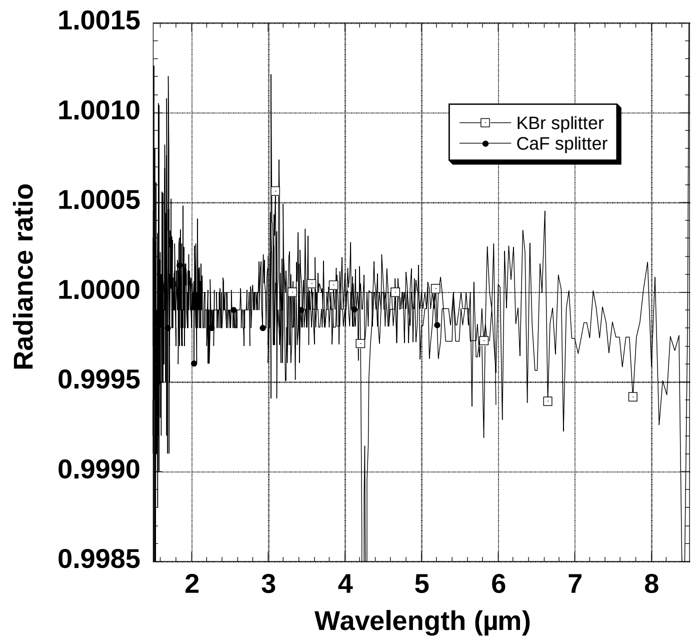


Figure 9. Stability measurement of the emittance characterization facility viewing a 500 °C blackbody source compared to itself, with a 100 °C blackbody background reference. The results of two separate measurement sets are overlayed.

We have performed a number of the spectral radiance experiments comparing both our fixed-point and variable temperature blackbodies. For each blackbody we have previously obtained calculated values of emissivity from modeling studies. One goal is to validate or replace these emissivity values with measurement results. We intend to do this by seeking self-consistency of experimental results for operation of the variable temperature blackbodies at a number of temperatures in comparison with each other and the fixed-point blackbodies as well as temperature readings of the blackbody temperature sensors, and separate emittance measurements of the blackbody cavity materials.

An example of one of the experimental results is shown in Figures 10 and 11 for our Na heat pipe variable temperature blackbody. The spectral radiance obtained from Equation 1, and using the Al fixed-point (at 660.32°C) as the high T reference and the H_2O heat pipe at 100 °C as the low T reference. The measured curve is overlayed with the Planck curve for a temperature of 657.49 °C as given by the blackbody's internal PRT. The spectral effective emissivity obtained from the ratio of the two curves in Figure 10 is shown in Figure 11. The measurements were not performed with blackbody purging resulting in some spurious water vapor structure in the spectra. Elsewhere in the spectra, the very high effective emissivity represents a good result.

The most accurate results are to be expected for comparison of sample to reference with closely matched flux levels, such as the case of Figures 10 and 11. Further experiments with larger temperature differences between the sources will be performed to evaluate the linearity of the system and map out the limitations of the instrumentation and methodology. We need to maximize the workable temperature difference range over which we can obtain accurate blackbody results. Nevertheless, by having a set of blackbodies that almost completely cover the temperature range from 250 K through 1400 K, we can closely match radiance levels for any arbitrary sample.

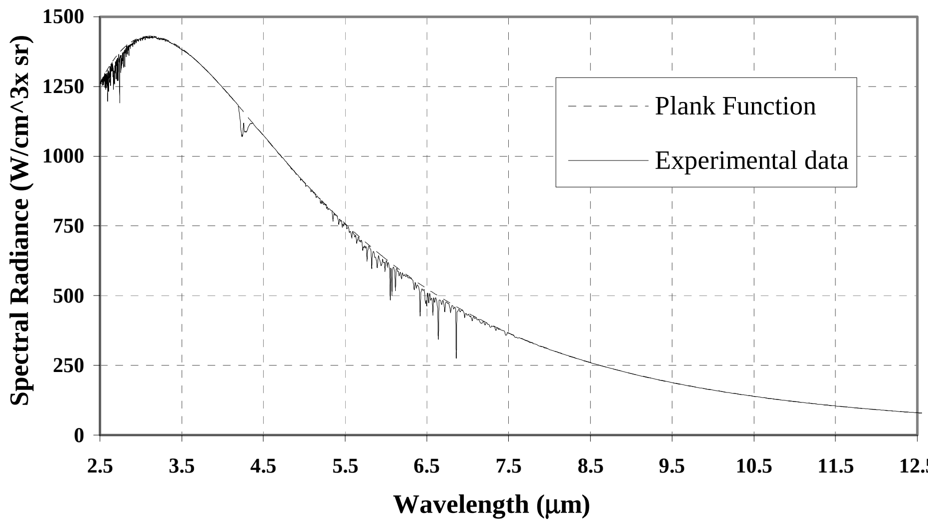


Figure 10. Measured spectral radiance and calculated Planck curve for the Na blackbody with a PRT reading 657.49 °C using an MCT detector, measured relative to the Al fixed-point and H_2O heat pipe blackbodies, no purge.

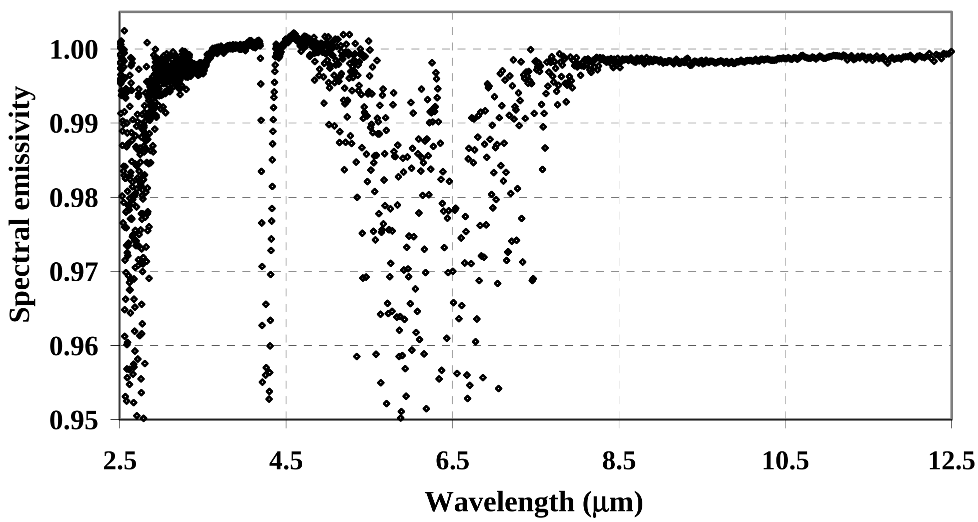


Figure 11. Na heat pipe blackbody effective spectral emissivity calculated from spectral radiance and a PRT reading of 657.49 °C, ratio of curves in Figure 10.

Finally we show the results of two sample measurements using the same procedure as before, except that the selection mirror #1 (Figure 1) is also brought into play. A commercial large area flat plate source (with a built in thermometer) with nominal emissivity of 0.95 was measured at the 500 °C setting. The measured apparent emissivity as shown in Figure 12 is seen to vary substantially from the nominal value depending on wavelength. An oxidized Ni sample measurement result is shown in Figure 13. The sample temperature was measured using the built in PRT of the sample mount. The emittance facility measurement curves labeled “direct” are qualitatively compared to room temperature data from an infrared integrating sphere measurement performed on the system described in Reference 3. The peak at 4.3 µm is a true sample feature and not due to atmospheric CO_2. Fringes due to the oxide layer become prominent at longer wavelengths. In both Figures the sharp structure in the 5 µm to 7 µm region and below 3 µm is the result of remnant water vapor in the system. Whether additional efforts (beyond current purging) will be required to reduce these residual water vapor effects is yet to be determined.

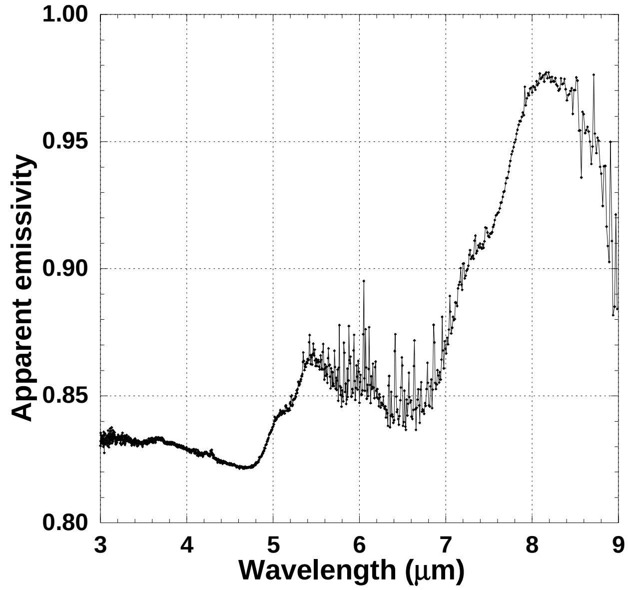


Figure 12. Apparent emissivity of a commercial flat plate source with a nominal emissivity of 0.95, for a temperature setting of 500 °C.

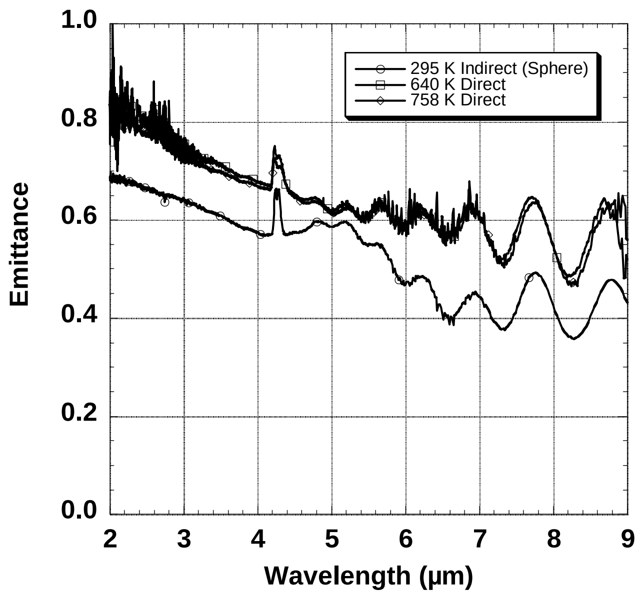


Figure 13. Emittance of oxidized Ni at several temperatures as measured via the direct method at the emittance facility compared to an indirect method measurement at ambient temperature using an infrared integrating sphere reflectometer.

4. SUMMARY

We have recently completed construction of the first phase of a new facility for the direct characterization of the infrared spectral emittance of samples at temperatures initially ranging from 600 K to 1400 K for wavelengths from 2 μm to 20 µm. Individual elements of the facility have exhibited excellent performance and initial sample measurements indicate the potential for the facility to meet its goal of providing high quality emittance measurements and data. The design and construction effort have taken several years to complete. The testing and evaluation will take a considerable time as well. We can expect the ultimate capabilities and measurement accuracies for emittance to be enhanced by the complementary measurement capabilities of the existing and improved reflectance and transmittance measurement instrumentation through a variety of mechanisms including inter-comparison of methods.

5. REFERENCES

[1] L.M. Hanssen, S.G. Kaplan, and S.N. Mekhontsev, "Fourier transform system for characterization of infrared spectral emittance of materials," Proceedings of the 8th International Symposium on Temperature and Thermal Measurements in Industry and Science (TEMPMEKO 2001), Berlin, June 19-21, 2001, pp 212-221.

[2] S.G. Kaplan and L.M. Hanssen, "Infrared regular reflectance and transmittance instrumentation and standards at NIST," Analytica Chimica Acta **380**, 303-310 (1998).

[3] L.M. Hanssen and S.G. Kaplan, "Infrared diffuse reflectance instrumentation and standards at NIST," Analytica Chimica Acta **380**, 289-302 (1998).

[4] D.J. Dummer, S.G. Kaplan, L.M. Hanssen, A.S. Pine, and Y. Zong, "High-quality Brewster's angle polarizer for broadband infrared application," Applied Optics **37**, no. 7, 1194-1204 (1998).

[5] S.G. Kaplan and L.M. Hanssen, "FTIR-based ellipsometer using high-quality Brewster-angle polarizers," Proc. SPIE **3754**, 285-293 (1999).

[6] L.M. Hanssen, "Integrating sphere method for absolute transmittance, reflectance and absorptance of specular samples," Appl. Opt. **40**, 3196-3204 (2001).

[7] L.M Hanssen and K.A Snail, "Integrating spheres for mid- and near infrared reflection spectroscopy", in Handbook of Vibrational Spectroscopy, J.M. Chalmers and P.R. Griffiths (Eds), John Wiley & Sons, Ltd, Volume 2, pp. 1175 - 1192 (2002).

[8] M. Battuelo and T. Ricolfi, "A technique for deriving emissivity data for infrared pyrometry," High Temp.-High Press. **21,** 303-309 (1989).

[9] A.V. Prokhorov, S.N. Mekhontsev, and L.M. Hanssen , "Evaluation of performance of integrating spheres for indirect emittance measurement," Proceedings of the 8th International Symposium on Temperature and Thermal Measurements in Industry and Science (TEMPMEKO 2001), Berlin, June 19-21, 2001, pp. 277-282.

[10] Prokhorov, A., Mekhontsev S., and Hanssen L., "Monte Carlo modeling of an integrating sphere reflectometer," Appl. Opt. **42**(19), 3832-3842 (2003).

[11] 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.

[12] S.N. Mekhontsev and L.M. Hanssen, "Low scatter optical system for emittance and temperature measurements," in *Temperature: Its Measurement and Control in Science and Industry, Volume 7*, ed. D.C. Ripple, AIP pp. 693-698 (2001).