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Temperature-Resolved Infrared Spectral Emissivity of SiC and Pt–10Rh for Temperatures up to 900°C

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This article reports the first comprehensive results obtained from a fully functional, recently established infrared spectral-emissivity measurement facility at the National Institute of Standards and Technology (NIST). First, sample surface temperatures are obtained with a radiometer using actual emittance values from a newly designed sphere reflectometer and a comparison between the radiometer temperatures and contact thermometry results is presented. Spectral emissivity measurements are made by comparison of the sample spectral radiance to that of a reference blackbody at a similar (but not identical) temperature. Initial materials selected for measurement are potential candidates for use as spectral emissivity standards or are of particular technical interest. Temperature-resolved measurements of the spectral directional emissivity of SiC and Pt–10Rh are performed in the spectral range of 2–20 μm, over a temperature range from 300 to 900°C at normal incidence. Further, a careful study of the uncertainty components of this measurement is presented.

KEY WORDS: infrared; platinum–10% rhodium; silicon-carbide; spectral emissivity; spectral emittance; 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 [1,2]. This facility is designed to provide reflectance and emittance measurement

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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 described within this manuscript is part of the Fourier Transform Infrared Spectrophotometry Laboratory devoted to the characterization of the optical properties of solid materials, which has been built around several commercial Fourier transform infrared (FTIR) spectrometers. Although the covered spectral infrared range is much larger, the main emphasis is on the 2–20 μm region. 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 [3–5].

A sphere reflectometer paired with an existing Bomem FTIR [6] marks the newest addition to the emittance facility, which adds direct emittance to the suite of measured properties. The main purpose of the sphere reflectometer is to provide emittance information of the test material at each temperature of interest and at the specific wavelengths of operation of the radiometers. The sphere reflectometer is used to enable more accurate optical temperature sensing with a radiometer instead of relying on the temperature measured with a thermocouple installed inside the sample.

This setup is envisioned to be comprehensive for the characterization of solid samples, specular and diffuse as well as opaque and transparent, at temperatures from 520 to 1670°C, at wavelengths from approximately 1–20 μm, and at angles from 0° to >75°. After extensive testing and characterization of the facility, the first comprehensive temperature-dependent spectral emittance results have been obtained for SiC and Pt–10Rh, which are both potential candidates for emittance standard reference materials, from room temperature (RT) to 900°C at normal incidence.

2. MEASUREMENT METHOD AND INSTRUMENTATION

For direct measurement of the spectral emittance of a material as reported within this work, the spectral radiance of a sample has to be compared to that of a blackbody (BB) at the same or similar temperature. The components required for such a measurement can be grouped as follows:

- BB sources (as references) at the appropriate temperature as well as a means of determining the temperature
- Sample hardware for heating and manipulation

- Accurate sample-temperature measurement
- A spectrometer based on a grating/prism monochromator, interferometer, or a circular variable filter (CVF)
- Interface optics for viewing and switching between blackbodies and samples.

The emittance characterization facility provides all the required components. A schematic overview is given in Fig. 1. The components most relevant for the spectral emittance measurements will each be briefly described within the following sections. A more detailed description of the entire facility and its capabilities is given in Ref. 2.

The most critical point when performing direct emittance measurements is to know the temperature of the sample and the reference BB as accurately as possible, since the spectral radiances are compared to each other. While temperature monitoring and control for the reference BBs is well established and considerable expertise in that field has been built up at NIST over the years, accurate measurement of the sample temperature

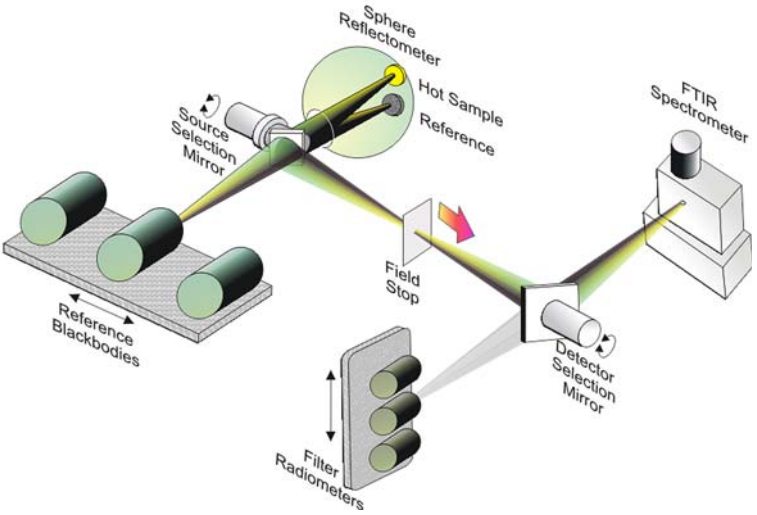

Fig. 1. Schematic of the infrared spectral emittance characterization facility. Radiation from blackbody (BB) reference sources (to the left) is compared with that from a sample (located opposite the blackbodies) via rotation of a flat source selection mirror. The radiation proceeds through a set of interface optics (not shown) and is measured either with a filter radiometer or with a FTIR spectrometer, selected by means of the elliptical detector selection mirror. For the temperature-dependent emittance measurement of opaque samples, a sphere reflectometer with a calibrated reference sample is shown.

is equally critical but more difficult. To minimize the error in the sample-temperature measurement, a sphere reflectometer is used in our setup.

For materials with high thermal conductivity and for moderate temperatures, the use of contact thermometers such as thermocouples built into the sample, i.e., by machining a small cavity into the side of the disc-shaped sample, can produce acceptable results. For temperatures higher than 200°C and especially for poorly conducting samples, non-contact methods can produce more accurate temperature results.

Therefore, we use an integrating sphere reflectometer to make high-accuracy reflectance factor measurements of the sample at the temperature of interest by comparing the reflected light of a hemispherical light source off the sample to that reflected off of a calibrated reference sample at room temperature (RT). Although only the sample reflectance factor is measured directly with the sphere, the sample emittance is obtained via Kirchhoff's law at the single wavelength of the measurement.

After the sphere is removed from the optical path, the known sample emittance is utilized to determine the sample temperature with a radiometer (operating at the same wavelength), which has to be calibrated against a BB reference source. The advantage of this method is that the reflectance and radiance temperature measurements are performed directly on the same sample region, thus reducing the uncertainty related to possible spatial non-uniformities of the sample.

2.1. Blackbody Reference Sources

The variable temperature blackbodies used as spectral radiance reference sources are a Cs heat-pipe BB for the 300–650°C range, a Na heat-pipe BB covering the 550–1100°C range, and a H_2O BB which is used as a stable room-temperature source. All blackbodies have temperature-controlled apertures, water-cooling, and purge-gas lines; temperature sensors are installed in both the heating elements and in the heat pipes. They are mounted on a motorized translation stage to automatically switch from one source to another.

Temperatures of the blackbodies have also been measured by means of an InGaAs radiometer calibrated against fixed-point sources. The spectral emissivity of the reference blackbodies has been evaluated by direct comparison of their spectral radiance with the set of fixed-point blackbodies. The spectral emissivities of the fixed-point crucibles have been calculated from the measured emissivity of the graphite crucible material and the geometry of the cavities using Monte Carlo analysis [7, 8]. The emittance of the BB cavities was found to be greater than 0.9995 over the wavelength range of interest.

2.2. Sample Heaters

Two different sample heaters have been used for these measurements: a Cs heat-pipe heater and a Na heat-pipe heater almost matching the temperature ranges of the reference BBs. Each of the small heat pipes is temperature controlled with a center-mount Pt resistance thermometer (PRT) and has a water-cooled base. They are built to hold opaque, disc-shaped samples up to 19 mm (3/4 in.) diameter, and the entire unit has been designed to fit the sphere reflectometer for temperature-dependent sample emittance measurements.

2.3. Sphere Reflectometer for Temperature Measurements

The integrating sphere reflectometer has a hemispherical-directional design, in which the directional flux (at 8°) reflected off a hemispherically illuminated sample is compared to that of a calibrated reference sample. The sphere has a diameter of 25.4 cm (10 in.) and is coated with a high reflectance sintered polytetrafluoroethylene (PTFE) shell. A chopped halogen lamp source is coupled to a nearly Lambertian diffuser, located in the sphere wall between the sample and the reference ports, via fiber optics. To prevent direct illumination of the sample or the reference, two specially designed triangular-shaped baffles have been installed in the sphere and an external mirror in the interface optics is used to alternate viewing the sample and the reference.

Different samples (both high/low and specular/diffuse reflectance) can be used as reflectance standards to match the sample as closely as possible. The spectral reflectance of these reference materials has been calibrated at NIST [9] in the 0.6–2.5 μm range.

As this technique is designed for heated samples, chopping of the light source is needed to distinguish between the light reflected off the sample or reference (ac component) and the light emitted into the sphere by the sample itself (dc component). The overall reflected light measured with a radiometer is fed to a lock-in amplifier for signal recovery. Further details of the sphere reflectometer can be found in Refs. 10 and 11.

Reflectance measurements for specular samples (SiC and Pt–10Rh) in the sphere were performed at an angle of view of 8°. This angle is sufficient to guarantee that, as viewed by the detector, the sample is fully illuminated with light from the sphere. The same angle was later used for measuring the thermodynamic sample temperature with a radiometer using the emittance results from the sphere measurement.

2.4. Radiometers for Sample-Temperature Measurements

Two different filter radiometers based on Si and InGaAs photodiodes with band-pass filters at 905 and 1550 nm, respectively, have been used for detecting the signal for the sphere-reflectance measurements as well as for the non-contact sample-temperature measurement using the respective sphere emittance result. The InGaAs radiometer contains a temperature-stabilized narrow-band filter and a temperature-stabilized detector. The signals of both radiometers are amplified with highly linear, low-noise amplifiers and fed to digital voltmeters (DVM). As the goal is to measure the sample temperature, it does not matter what wavelength this is done at, except to the extent it affects the temperature measurement itself: (a) the shorter the wavelength, the smaller the temperature uncertainty for a given reflectance uncertainty and (b) both reflectance and radiance need to be measured at the same wavelength, i.e., both nonzero. Since material properties vary spectrally, having several wavelengths at hand allows greater flexibility in accommodating the sample characteristics. For SiC and Pt–10Rh, the two radiometers at 905 and 1550 nm were able to meet these requirements but for greater versatility a small monochromator (especially useful for item (b)) is also available, which was not used for the recent measurements.

2.5. FTIR for Spectral Detection

The main spectral detector of the emittance characterization facility is a modified, dry-air purged Bomem DA3 Fourier transform (FT) spectrometer. 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 for the emittance measurements is on the 2–20 μm range and the FTIR was used with a KBr beamsplitter and a DTGS (deuterated triglycine sulfate) pyroelectric detector throughout.

3. EXPERIMENTAL DETAILS AND RESULTS

After determination of the sample and BB temperatures, the relative spectral radiance is calculated from the results of three measurements: along with the sample, two reference blackbodies at different temperatures are measured. The temperature of one BB is chosen to match the sample temperature as closely as possible, whereas the second one is close to room temperature. This second BB measurement is used to subtract out the FT self-emitted radiance component. The spectral radiance of the sample is given by the real part $\Re$ of the complex FT spectra by

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

where $L(\nu)$ is the spectral radiance, $V(\nu)$ is the FT measured complex spectrum, A represents the higher T BB, B represents the ambient T BB, and C denotes the sample. The spectral radiance of the blackbodies is given by Planck's radiation law and the emissivity as obtained from calculation or comparison with a fixed-point cavity. The unknown spectral emittance of the sample is obtained from L by its ratio to the Planck function.

The measurements are performed in repeated cycles of an A–B–C–B–A sequence to reduce the effects of drift. Depending on the level of emitted radiation from the sample at different temperatures, the number of measurement cycles was adjusted to optimize the signal-to-noise ratio. Most of the measurements were done with 12 cycles using 100, 50, and 100 individual FT scans for A, B, and C, respectively. The average FT measurement time for 12 cycles was approximately 2 h. Spectral emittance measurements with the Bomem FTIR have been performed at normal incidence. The implementation of a polarizer to facilitate measurements at off-normal angles is planned as a follow-on step.

All room-temperature emittance measurements are obtained from an indirect reflectance measurement facility at our Spectrophotometry Laboratory [12,13] and have been added for better comparison of the temperature-dependent results.

3.1. Silicon Carbide (SiC)

The silicon-carbide sample used is a 2.3mm thick disc with a 19 mm diameter of pressureless sintered silicon carbide of approximately 95% theoretical density. A piece of β-SiC made in the 1980s was evaluated by Slavin and Quinn [14]. A similar material has been used for the present investigations. The measured surface is polished and has a cavity hole with a 0.7 mm diameter and a depth of 8 mm drilled into the side of the disc for contact temperature measurements.

Although we considered SiC to be reasonably stable when heated in air since it did not show any change after heating to 200°C in earlier measurements, we discovered a change in the optical properties of the sample after heating in air to 600°C. SiC develops a stable "passivation" oxide layer, which does affect the room-temperature emissivity before and after heating to 600°C but seems to stabilize afterwards, since the room-temperature emittance remained unchanged after repeated heating to 850°C. The effect of this change in emittance is profound, as can be seen in Fig. 2.

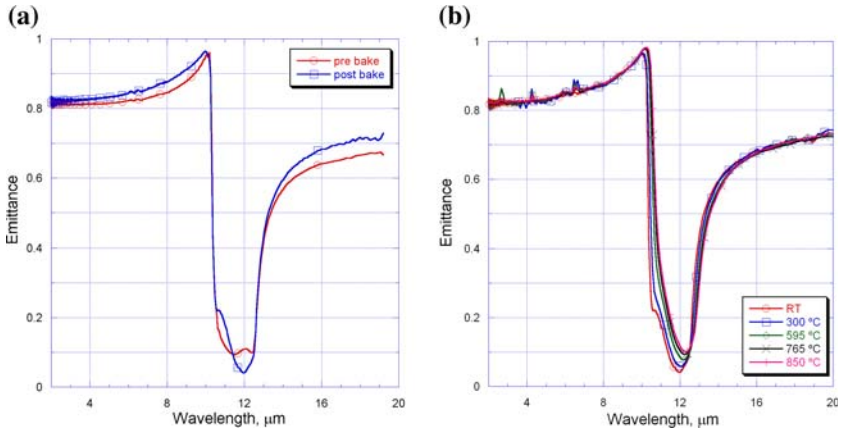

Fig. 2. (a) β-SiC before [13] and after baking at 600°C measured at room temperature. Emittance changes are due to the build-up of a 'passivating' oxide layer which alters the optical properties of SiC at almost all wavelengths and (b) temperature-resolved spectral emittance for β-SiC with the stable oxide layer.

As described earlier, the temperature-dependent emittance for SiC was measured by means of a hemispherical-directional sphere reflectometer from room temperature up to a maximum temperature of 850°C. The reference standard was a specular gold mirror with a nominal reflectance of 0.972 at 905 nm and 0.978 at 1550 nm (reflectance numbers are average values of s- and p-polarized components).

The maximum temperature was determined by the maximum operating temperature of the Na heat-pipe heater used. Since SiC is a moderate thermal conductor and because of radiative and convective heat losses from the sample surface, the heater temperature is always greater than the sample temperature. The heater reaches its maximum temperature of operation when SiC is at 850°C. The temperature dependence of the sample emittance measured with the sphere reflectometer is shown in Table I.

As can be seen, the emittance change of SiC at 905 nm is only 0.8% from room temperature to 850°C. However, larger changes in emittance are observed for other wavelengths, as can be seen from the spectral emittance results in Fig. 3.

3.2. Platinum–Rhodium (Pt–10Rh)

The platinum–rhodium sample used for these measurements is a 2.3-mm thick disc with a 19 mm diameter of Pt–10%Rh ZGS purchased from Noble Metals NA, West Chester, Pennsylvania. The Pt–Rh alloy is

Table I. Emittance Results for β-SiC and Pt–10Rh at 905 and 1550 nm Obtained with a Hemispherical-Directional Sphere Reflectometer

β-SiC			Pt–10Rh		
Temperature (°C)	905 nm	1550 nm	Temperature (°C)	905 nm	1550 nm
25	0.814	0.818	25	—	0.202
300	0.812	0.816	300	0.326	0.223
600	0.809	—	600	0.318	—
765	0.808	—	900	0.308	—
850	0.808	—			

No InGaAs measurements were possible for temperatures higher than 300°C due to saturating dc component of the signal.

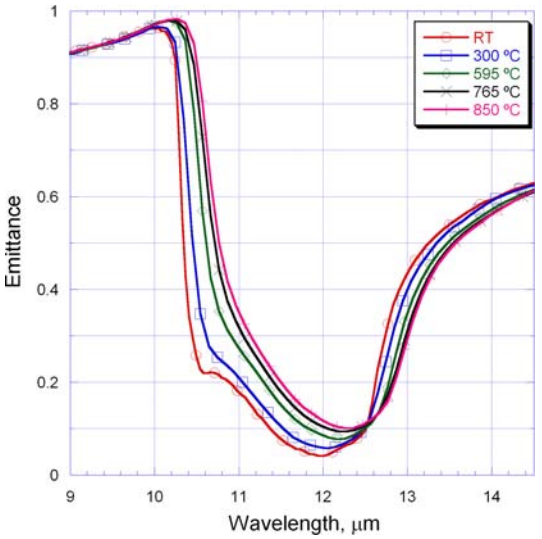

Fig. 3. Close-up of the absorption-edge region from 10 to 14 μm of β-SiC. Emittance isotherms in this region are slightly shifting towards longer wavelengths with increasing temperatures, and crossover is observed at about 12.6 μm.

zirconia-grain-stabilized (ZGS) to prevent the formation of large grains during heating treatment of the material. The surface used for measurements was polished, and the same cavity hole as with SiC was drilled in the sample.

In order to avoid any unwanted alterations of the sample during the measurements, the Pt–10Rh sample was first annealed in a tube furnace to stabilize the material. Starting with a cold furnace, the temperature

was raised to 1100°C (about 100°C higher than the highest anticipated measurement temperature) over a period of 4 h and held at this temperature for another 4 h. Cooling the sample back to room temperature took also 4 h. After visual inspection under a microscope, the sample was cycled a second time at the same temperatures and stable times to check for heat stability of the sample. Before, during, and after the heat treatment, the room-temperature reflectance was checked by means of an integrating sphere reflectometer. After stabilization of the RT reflectance as well as the grain size and distribution in the microscope, the sample was considered to be stable.

Figure 4(a) shows the RT emittance of Pt–10Rh before and after stabilizing, as well as a microscope picture of the surface texture after baking (Fig. 4(b)). As with SiC, the gold mirror was used as a reference standard for the sphere reflectance factor measurements. Due to better heat conductivity and thermal contact with the heater, Pt–10Rh measurements were feasible for temperatures up to 900°C. The results from the sphere reflectometer measurements are listed in Table I. Please note that no emittance result can be presented for RT at 905 nm due to insufficient signal levels. Direct spectral emittance measurements for platinum–rhodium have been performed at the temperatures stated above, and the results are graphically presented in Fig. 5.

Albeit almost invisible in the plot, the emittance isotherms for 600 and 900°C crossover near 2.6 μm. Unfortunately, due to the poor signal-

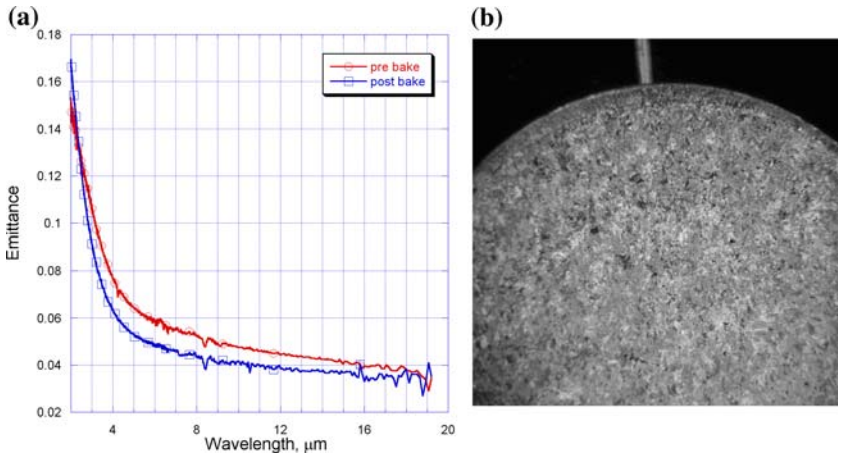

Fig. 4. (a) Room-temperature emittance of Pt–10Rh before and after baking at 1100°C for 4 h and (b) microscope image of the sample surface after baking; the thermocouple can be seen as the vertical marker on top of the sample.

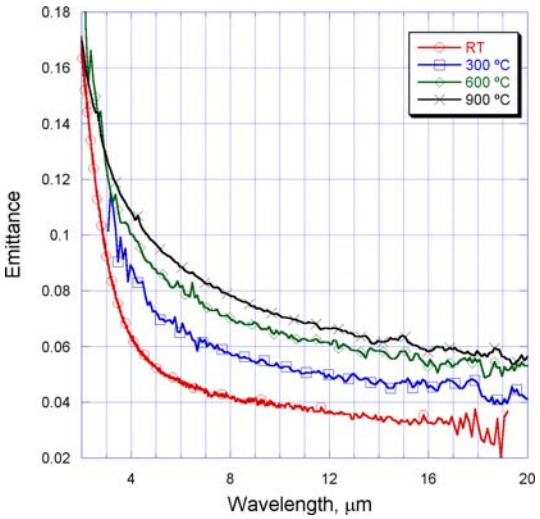

Fig. 5. Spectral emittance of Pt–10Rh at RT, 300°C, 600°C, and 900°C in the wavelength range from 2 to 20 μm.

to-noise ratio for low temperatures and short wavelengths, no clear statement regarding such a crossover for 300°C is possible.

4. DISCUSSION

4.1. Silicon Carbide

Although the possibility of an oxide layer on SiC is not new, we did not anticipate the change in emittance of SiC after the first heating cycle up to 600°C, since the sample was heated prior to 200°C for some earlier reflectance measurements and did not show any changes at this temperature. Apparently this increase in temperature was insufficient or the heating time was too short to reach a stable oxide layer. The change in optical properties is easily noticeable from the room-temperature comparison measurements, since the emittance outside the absorption-edge region (roughly 10–14 μm) has increased. The Christiansen wavelength has also shifted to shorter wavelengths, and the structure at the bottom of the absorption 'dip' has been altered.

After formation of the 'passivating' oxide layer, the sample was very stable and did not show further changes or build-up for any other heating cycles. By looking at the temperature-dependent spectral emittance result for SiC, little or no change can be seen in the 2–10 μm wavelength band

for SiC at temperatures up to 850°C. This is also confirmed by the emittance data obtained with the sphere reflectometer at 905 and 1550 nm.

According to our results, the temperature has the greatest influence on the emittance of SiC at the Christiansen point and in the absorption region. The former is shifted towards longer wavelengths (from about 9.98 μm at room temperature to 10.26 μm at 850°C) and to higher absolute emittance values. Temperature-dependent changes of the latter are even larger; the emittance at the absorption edge shifts by more than 0.4 μm at the $\varepsilon = 0.5$ level towards longer wavelengths and the maximum absorption decreases with temperature. Such shifting of the reflection band for SiC has also been reported by Roy et al. [15]. As a result, crossover of the emittance isotherms is observed at around 12.6 μm. This effect, known as the x-point, has also been found for other materials such as tungsten [16], but is still not theoretically understood. For wavelengths between 16 and 20 μm, the emittance isotherms for SiC again become identical and indistinguishable.

A literature survey has revealed little data concerning the temperature-dependent emittance of SiC. A collection of available data in the late 1960s at various temperatures (600–1100°C) is available from Touloukian [17], but the reported emittance numbers in the near IR up to the absorption band vary from 0.85 to 1, preventing any detailed comparison.

Newer data from Neuer and Jaroma-Weiland [18] report spectral emissivities of SiC in the 1.3–9 μm range at $T = 900$–1440°C. They observe temperature-related differences in emittance of 0.84–0.9 at about 6 μm and an overall decrease from 0.98 to 0.9 within the 1.3 to 6 μm region.

Our own published data from earlier measurements [13] are inapplicable for comparison, as the sample has since been 'passivated' with a stable oxide layer due to additional heating.

4.2. Platinum–Rhodium

Similar to the situation with silicon carbide, no data are reported for the temperature-dependent emittance of platinum–10% rhodium. The National Bureau of Standards (NBS) utilized Pt–13Rh as a normal spectra emittance standard (SRM 1402 to 09) at 800 to 1400 K and from 1 to 15 μm [19]. Other normal spectral emittance data also from NBS for the same material (but computed from electrical resistivity) can be found in Ref. 20.

For comparison reasons it would be better if the sample composition were the same, which is not the case. Furthermore, our Pt–10Rh sample is zirconia-grain-stabilized to resist grain growth and contamination at high

temperatures, while our existing SRM 1403 sample shows a tendency to large grains after heating and the surface becomes anisotropic.

At 3.79 and 5.06 μm, pure platinum data at 500°C from an intercomparison between the National Physical Laboratory (NPL), United Kingdom and Istituto di Metrologia "Gustavo Colonnetti" (IMGC), Italy have been reported [21]. Although they used pure platinum samples and our measurements do not explicitly give numbers for 500°C, the intercomparison results fit very well to our emittance measurements. NPL results are closer to our 600°C isotherm, whereas the IMGC numbers are close to our 300°C result. However, a mean value from the intercomparison fits well with the 300 and 600°C values from this work.

The recent results for the temperature-dependent spectral emittance show a strong effect on emittance in the 2–20 μm range from room temperature up to 900°C. The emittance increases with temperature and at 4 μm we obtain 0.089 at 300°C, 0.100 at 600°C, and 0.109 at 900°C. The SRM numbers at 4 μm for the closest matching temperatures are 0.102 at 527°C and 0.124 at 827°C. As with SiC, the isotherms for 600 and 900°C intersect at about 2.8 μm. Unfortunately due to low signal, no useful data at 300°C are available at this wavelength.

Both literature sources also report such a crossover effect. The data given in Touloukian intersect twice at about 1.9 and 2.2 μm and the SRM values report an intersection of the 1100 and 1400 K isotherms at about 3.2 μm.

5. UNCERTAINTY ANALYSIS

The overall uncertainty analysis splits into two large groups: the sample-temperature measurement with the radiometer including the sphere reflectometer and the spectral radiance comparison with the FTIR.

5.1. Sample-Temperature Measurement

The first uncertainty component is the repeatability of a temperature comparison using the radiometer. Several measurements of the same sample at the same temperature have been used to calculate the uncertainty from the mean value and its standard deviation.

Similar to the temperature comparison, we tested the repeatability of the reflectance measurement within the sphere with multiple measurements and used the standard deviation in the mean as an uncertainty. This component also accounts for all light source related issues like drift of the source, instability of the source, etc.

The sphere-reflectometer measurement itself is split into different contributing components that are different for the sample and the reference standard. For the heated sample, alignment and the temperature dependence of the reflectance are considered. Alignment (as well as for the reference standard) has been extensively tested and checked for the sphere reflectometer, and signal-related uncertainties have been calculated. The temperature dependence of the reflectance is sample related, and a maximum temperature variation of ±5°C is used as an estimate of the signal variation. The change in emittance due to this ±5°C temperature variation is used as the uncertainty.

For the reference standard, uncertainties are taken from the calibration report, which splits uncertainties into repeatability, wavelength, and linearity contributions. However, the gold mirror used as a reference standard within this work, exhibits a difference of about 0.02% for 905 nm in reflectance for *s*- and *p*-polarization. Since unpolarized light was used for the present study, the mean reflectance of *s* and *p* was used.

Although the sphere-reflectometer measurement is a direct comparison of the reflectance factor of the sample to that of a standard, optical influences within the sphere (such as mounted baffles), imperfection in the wall reflectance, a non-Lambertian source, etc. will only cancel out if the sample and reference are optically identical. In prior investigations we compared a perfectly diffuse sample to a perfectly specular one to obtain a value for the combination of these effects. The maximum effect was found to be less than 1% in reflectance of the sample. Since all samples measured within this study and the reference are specular, the uncertainty arising from the sphere reflectometer can be expected to be smaller than 20% of the maximum value found.

Another uncertainty comes from the calibration of the radiometer with the fixed-point BBs. Such studies have already been performed, and the established uncertainties for the calibration itself, the interpolation between temperatures, and the alignment at the reference source are taken into account.

Finally, the size-of-source effect (SSE) contribution from the interface optics has already been tested for earlier measurements by using a variable size source.

5.2. Spectral Radiance Comparison

Repeatability of the spectral radiance comparison is the largest contribution to the overall uncertainty of Pt–10Rh but is a moderate contribution for SiC. We checked the repeatability of single measurements with the FT and obtained the values of the standard deviation. The effect is large

for Pt–10Rh since the spectral radiance of the sample is very low compared to that of the BB. A similar effect can be seen in the non-linearity of the detector, which is much larger for Pt–10Rh than for SiC.

For both calibration of the RT reference and the reference BB, the temperature uncertainty and the uncertainty in the cavity emittance are accounted for. The temperature uncertainty has been determined in earlier measurements, and the cavity emissivity has been calculated to be >0.9995 and half of the remainder (difference from one) is assigned as the uncertainty.

As the last component, we estimate the uncertainty due to non-linearity of the DTGS pyroelectric detector. To estimate this contribution, we used data from two experiments we carried out on Pt–10Rh at 600°C. Though the sample was at the same temperature, the temperature of the reference BB was varied from matching the sample temperature (600°C) to a significantly lower temperature of 370°C. Comparison of the difference in spectral emittance results was used to estimate the non-linearity uncertainty component.

The specific values for each component, the combined standard uncertainty for the spectral emittance measurement, and the expanded uncertainties for Pt–10Rh and SiC at 600°C are listed in Table II.

6. SUMMARY

This work presents the first comprehensive temperature-dependent emittance results from the newly established spectral emittance facility at NIST. To improve the sample surface temperature measurement, a hemispherical-directional sphere reflectometer was added to the setup and recently characterized. This sphere reflectometer enables sample emittance measurements up to a maximum temperature of 1000°C.

The experiment was used to measure temperature-resolved spectral emissivities of β-SiC and Pt–10Rh in the 2–20 μm range. Both materials are of particular interest as potential candidates for emittance standard materials. The emissivity of Pt–10Rh was found to be more temperature dependent than SiC. The latter only shows a significant temperature dependence of emittance in the absorption region from 10 to 14 μm, and both materials exhibit a crossover of emittance isotherms at individual wavelengths. An uncertainty budget was calculated for the sphere reflectometer and the spectral radiance comparison with the FTIR spectrometer yielding a total expanded uncertainty of $\pm$2.92% for Pt–10Rh and $\pm$0.93% for SiC.

Table II. Uncertainty Budget of the Spectral Directional Sample Emittance Measurement including the Sphere Reflectometer

Uncertainty budget of sample spectral emissivity	Type	Pt–10Rh at C 600° (%)	SiC at 600°C (%)
Sample-temperature calibration at 905 nm			
Repeatability of temperature comparison	A	0.05	0.00
Sample reflectance			
Repeatability of reflectance comparison	A	0.03	0.03
Sample			
Alignment	B	0.19	0.19
Temperature	B	0.05	0.00
Reflectance reference			
Calibration	B	0.09	0.09
Alignment	B	0.19	0.19
Sphere reflectometer	B	0.20	0.20
Radiometer calibration			
Calibration at FP	B	0.01	0.01
Interpolation	B	0.01	0.01
Alignment	B	0.00	0.00
SSE of interface optics	B	0.04	0.04
Spectral radiance comparison with FTIR			
Repeatability of spectral radiance comparison	A	1.40	0.30
Calibration of room-temperature reference BB			
Temperature	B	0.01	0.01
Spectral emissivity	B	0.03	0.03
Calibration of reference BB			
Temperature	B	0.01	0.01
Spectral emissivity	B	0.03	0.03
Non-linearity of the FT detector	B	0.23	0.03
Combined standard uncertainty of spectral emissivity		1.46	0.47
Expanded uncertainty ($k=2$)		2.92	0.94

All reported uncertainties are signal-related (relative) and based on a sample temperature of 600°C and the Si-radiometer.

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