Comparison of mid-infrared absorptance scales at NMIJ and NIST

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Abstract. National Institute of Standards and Technology (NIST) realizes scales for infrared absolute absorptance using the integrating-sphere method. National Metrology Institute of Japan (NMIJ) realizes an independent scale for infrared spectral emissivity equivalent to absorptance traceable to the ITS-90. Comparisons of these scales in the mid-infrared part of the spectrum have been performed successfully for the first time and the results are reported here. The results of the comparison show agreement is lying within uncertainty levels for the majority of values measured for opaque materials, and the level of equivalence of the NMIJ and NIST scales.

Introduction

Infrared optical properties such as absorptance are essential in the fields of both radiometry and radiation thermometry. Under the Mutual Recognition Agreement, the metrological equivalence of national measurement standards based on comparisons between the national metrological institutes has increased over time. Recently, the CCT (Consultative Committee for Thermometry) – Working Group 9 is organizing international comparisons of thermophysical properties including emissivity (emittance) of solids. Under such circumstances in this study, a comparison of spectral absorptance scales near room temperature in the mid-infrared portion of the spectrum from 5 μm to 12 μm at NIST and NMIJ based on fundamentally different methods has been performed for the first time. The normal spectral emissivity scale of NMIJ is realized by a direct comparison method between a sample and the reference blackbody cavities traceable to the ITS-90 [1, 2]. On the other hand, NIST realizes the same scale by an integrating sphere based absolute measurements of directional-hemispherical reflectance and transmittance[3].

Measurement techniques and apparatus

Both facilities at NIST and NMIJ employ Fourier transform infrared spectrometers (FTS), however, major differences exist between the techniques used to realize these scales. NMIJ applies the conventional direct technique that consists in measuring the ratio of the spectral radiance of the sample to that of a blackbody radiator at the same temperature and at the same wavelength to derive emissivity values. On the other hand, NIST uses a direct absolute technique with an improved integrating sphere method for measuring spectral reflectance and transmittance of materials. Absolute absorptance is indirectly obtained when the sum of the absolute reflectance and transmittance is subtracted from unity based on Kirchoff's law, which is written in the form for an opaque surfaces which is of primary concern in this study

$$\alpha(\lambda;\theta,\phi) = 1 - \rho(\lambda;\theta,\phi;2\pi) = \varepsilon(\lambda;\theta,\phi), \qquad (1)$$

where α is spectral-directional absorptance, ρ is the spectral-directional-hemispherical reflectance, and ε is spectral-directional emissivity, at wavelength λ, incident angle of radiation θ, ϕ, respectively.

Detailed descriptions of the apparatus of both NMIJ and NIST are found in references[1-3]. Here only brief introductions are presented here.

NMIJ: The spectrometer is a custom designed Michelson interferometer. For calibration of the response of the FTS, two reference blackbody cavities are installed. One is a liquid nitrogen cooled cavity and the other is a variable temperature one operated at 100 ^{o}C. The solid sample attached to the holder is heated from the backside by direct contact with the circulating thermostatic fluid. The temperature of the heating fluid is measured with a calibrated PRT sensor placed close to the backside surface of the sample. For minimal uncertainty the samples were measured at 100 ^{o}C. To reduce the effects of absorption by air and convective heat exchange, all of the components are assembled and operated in vacuum. The combined standard relative uncertainty for samples with good thermal conductivity is typically around 1 % for high emissivity values and less than 5 % for low emissivity values.

NIST: The spectrometer is a bench-top FTS with external beam output that is interfaced to a custom diffuse gold-coated integrating sphere with an MCT detector. Measurements of absolute reflectance and transmittance are performed at semi-normal incidence (8^{o}) using custom developed methods described in References [3] and [4]. The samples were measured at an ambient temperature of 24 ^{o}C, determined by a Pt thermometer. The expanded uncertainty (k=2) for reflectance and transmittance measurements is 3 % of the values for samples of unknown scattering nature, and 0.3 % for specular samples.

We prepared several kinds of solids and coated surfaces as the transfer samples of the comparison. The samples were 45 mm diameter discs of approximately 1 mm thickness. The samples were measured first at NMIJ and then sent to NIST for measurement.

Results and discussions

Figures 1 to 3 summarize the results measured by NMIJ and NIST for the solids and the coated samples in which transmittance can be disregarded. Figure 1 shows the results for a silicon dioxide sample having a roughened surface. The values measured by the two methods agree to within the combined uncertainty over the entire wavelength range. The discrepancy is at most 0.02 for an absorptance value of 0.6 around 9 μm, which is comparable to the NMIJ uncertainty level of emissivity. Figure 2 illustrates the results obtained for the silicon nitride sample, which is a standard reference material provided by the JFCC (Japan Fine Ceramics Center). It is also seen that the difference between NMIJ and NIST gradually increases as the absorptance values decreases. The discrepancy is about 0.03 when the absorptance value is at its minimum of 0.2 around 10.5 μm. This trend is consistent with an increase in the uncertainty of the radiance measurement for low emissivity materials near room temperatures. Figure 3 shows the results for a black paint, a Heat-resistant black manufactured by Asahi-paint, coated on a copper substrate. It is found that the paint maintains an absorptance value higher than 0.9 and the values observed by NMIJ and NIST are in good agreement over the entire wavelength range. The maximum discrepancy is less than 0.01 and within the combined uncertainty.

Summary

In this study a comparison of spectral absorptance (emissivity) scales in the middle-infrared wavelength region near room temperatures at NIST and NMIJ has been performed for the first time. NIST and NMIJ have established independent scales based on basically different approaches. NIST employs absolute reflectance and transmittance measurement using an improved IR integrating sphere. NMIJ applies a radiance comparison method with reference blackbody radiators traceable to the ITS-90. The results observed by the different approaches almost agree for opaque solid materials within their combined uncertainty levels. The results observed for other materials and details of uncertainty analysis will be also given in the presentation.

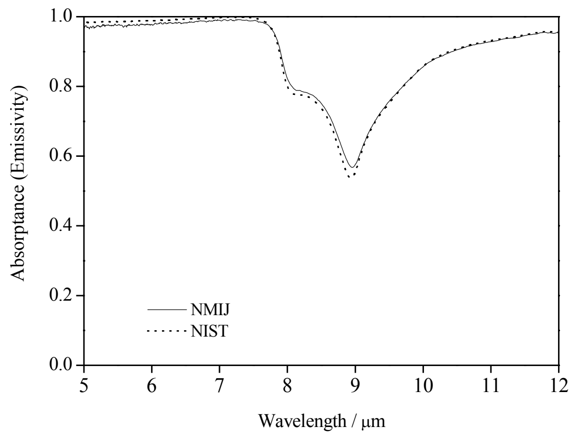

Figure 1. (Semi-) normal spectral absorptance (emissivites) of Silicon dioxide observed by NMIJ and NIST

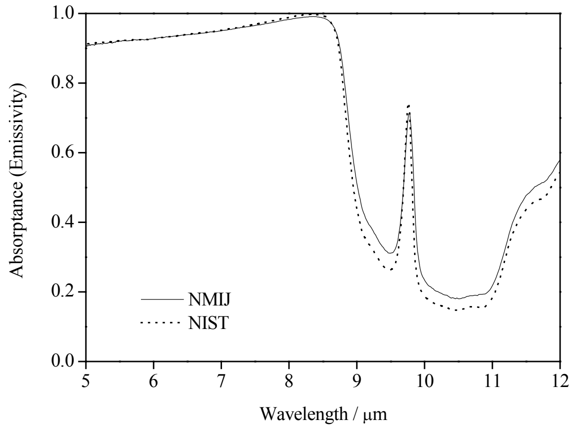

Figure 2. (Semi-) normal spectral absorptance (emissivites) of silicon nitride observed by NMIJ and NIST

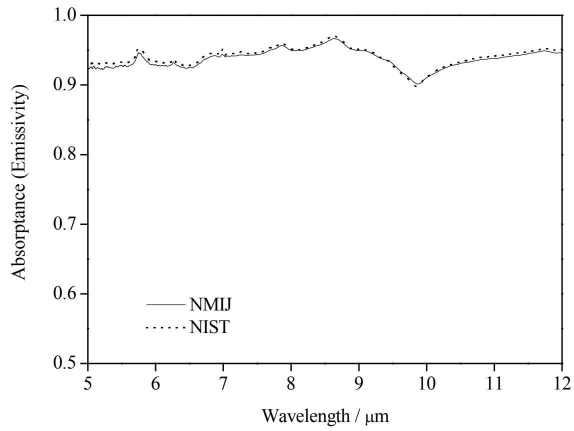

Figure 3. (Semi-) normal spectral absorptance (emissivites) of the black paint (Heat-resistant black, Asahi-paint) on copper substrate observed by NMIJ and NIST

References

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