Evaluation of a Method for Measuring Spectral Emissivity at Moderate Temperatures

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High-accuracy measurement of spectral emissivity at moderate temperatures (< 1000 K) is especially challenging due to the low self-exitance of the target and significant reflected irradiance contributions. A ratio-radiometric method has been developed that utilizes a heated equalizing block accommodating thin rectangular samples and a reference blackbody. Analysis has been performed to provide corrections for nonisothermal conditions, apparent emissivity of the cavity, and reflected irradiance from the surroundings. The method is evaluated by measurements in the spectral range 1.4–12 μm at 800 K on the NBS thermal emittance reference materials oxidized Inconel, Kanthal, and a platinum/10% rhodium alloy.

Nomenclature

c_1 = first radiation constant, 1.191×10^8 W·μm^4/m^2·sr
c_2 = second radiation constant, 14,388 μm·K
c_3 = third radiation constant, 2898 μm·K
k = thermal conductivity, W/m·K
L_λ = spectral radiance, W/m^2·μm·sr
R = detector signal ratio, Eq. (3)
S = detector signal proportional to target exitance
Z = detector signal proportional to radiometric zero

Subscripts

b = blackbody or reference blackbody condition
o = background or offset condition
sur = surroundings of the target
t = target condition

Introduction

SPECTRAL emissivity is an important radiation property for heat-transfer analysis and temperature measurement by radiation methods. In the moderate temperature range (600–1000 K), the spectral range of significance is the infrared region between 1.5 μm and (because of practical detection limitations) 15 μm. Most materials of technical interest, such as ceramics and alloys, exhibit nongray behavior in this spectral range and it is, therefore, necessary to have knowledge of spectral behavior for accurate analysis purposes.

Recently, more attention has been given to the use of radiation methods for determination of surface temperatures.[1,2] The primary instrumentation advantage is that of a remotely observed, noncontact technique. These methods are realized using a radiation thermometer (RT), which is broadly defined as a spectral radiometer calibrated to correctly indicate the temperature of a blackbody. In order to determine the temperature of a real material, it is necessary to have knowledge of the target emissivity over the spectral bandpass of the RT in order to infer the surface temperature from the indicated temperature.[3,4] Application of RT's to industrial processes are increasing primarily as a consequence of computer-based process automation to achieve improved productivity and/or quality control and for energy conservation. Commercially available RT's for the moderate temperature range have spectral bandpasses centered about these common values: 0.65, 0.9, 1.5, 2.2, 3.9, and 8–14 μm. In order to provide adequate spectral emissivity data for RT applications, measurements in the near-infrared region will also be useful.

The spectral emissivity is defined as the ratio of the spectral self-exitent radiance of the material to that from a blackbody, identically viewed, and at the same temperature. The most customary measurement process is referred to as a radiometric method wherein radiance is sensed by a suitable radiation detector; calorimetric methods, wherein the conversion of electrical power, for instance, to radiant power is measured are more difficult to apply for spectral radiation properties.[5,6] The most demanding conditions to be experimentally satisfied in the ratio-radiometric method are the isothermal sample/blackbody condition, knowledge of the apparent spectral emissivity of the reference blackbody cavity, and assurance that the target is freely radiating. The latter condition is especially troublesome in the moderate temperature range for low-emissivity materials, since spectral irradiation originating from the surroundings of the target can be reflected into the radiation detector field of view and is significant compared to the target self-exitent radiance.

The objectives of the present study were to develop a method for measuring the spectral emissivity of opaque materials and to evaluate the method through performance analysis and comparison of results on selected, well-characterized materials for which reference-quality data are available. The method was intended to be used for the study of processed primary metal samples (rolled aluminum and steel alloys) that dictated requirements for sample form and size (sheet stock of variable thickness). Furthermore, it was desirable to simultaneously observe several samples since the differences in spectral emissivity between samples was especially interesting.

The principle of the measurement method is described in the following section and the effects due to nonisothermal conditions between the sample and blackbody and to reflected irradiation from the surroundings are analyzed. The experimental apparatus used to obtain the measurements is then described with special attention given to the manner in which the sample and blackbody temperatures are determined. The following section discusses the procedures used to evaluate the performance of the method including results of a Monte Carlo analysis for estimating the apparent emissivity of the reference cavity and of a heat-transfer analysis for confirming the

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isothermal conditions between the sample and blackbody. Finally, the results for three materials are compared with reliable reference data as evidence of reliable determination of spectral emissivity. The reference materials are the high-, medium-, and low-emittance standards (oxidized Inconel, oxidized Kanthal, and Pt/13% Rh alloy, respectively) developed by the National Bureau of Standards (NBS) in the 1960's.

The Measurement Method

Principle of the Method

The normal spectral emissivity ϵ_λ of a surface is defined as the ratio of the normal, self-exitent (emitted) spectral radiance from the surface to that of a blackbody identically viewed and at the same temperature. A narrow-band or spectral radiation detector, when viewing a target, produces an output proportional to the total spectral exitent radiance $L_{\lambda,t}$. If the target is small compared to its surroundings, which are assumed to be isothermal at a temperature T_{sur}, the radiance from the target reaching the detector is comprised of three components

$$L_{\lambda,t} = \epsilon_\lambda L_{\lambda,b}(T_t) + (1-\epsilon_\lambda) L_{\lambda,b}(T_{sur}) + L_{\lambda,o} \quad (1)$$

where $L_{\lambda,b}$ is the blackbody spectral radiance of the target at a temperature T_t. In this relation, the terms represent, respectively, the self-exitent radiance, the reflected portion of the irradiation from the surroundings, and the background accounting for scattering into the optical system and detector offset.

The ratio of the detector signal outputs when identically viewing the target S_t and the reference blackbody cavity S_b, is of the form

$$\frac{S_t}{S_b} = \frac{\epsilon_\lambda L_{\lambda,b}(T_t) + (1-\epsilon_\lambda) L_{\lambda,b}(T_{sur}) + L_{\lambda,o}}{\epsilon_{\lambda,a} L_{\lambda,b}(T_b) + L_{\lambda,o}} \quad (2)$$

The reference blackbody cavity is assumed to have a temperature T_b slightly different than that of the target. Furthermore, the apparent emissivity $\epsilon_{\lambda,a}$ of the cavity is slightly less than unity, making the reflected irradiation term for the denominator of Eq. (2) negligible. The magnitude of $L_{\lambda,o}$ can be measured directly by identically viewing a cold blackbody. The reading obtained, referred to as the radiometric zero Z is subtracted from S_t and S_b, giving

$$R = \frac{S_t - Z}{S_b - Z}$$

$$R = \frac{\epsilon_\lambda L_{\lambda,b}(T_t) + (1-\epsilon_\lambda) L_{\lambda,b}(T_{sur})}{\epsilon_{\lambda,a} L_{\lambda,b}(T_b)} \quad (3)$$

where R is the zero-corrected measured radiometric ratio. Solving Eq. (3) for ϵ_λ, the measurement equation for the target spectral emissivity can be expressed as

$$\epsilon_\lambda = \frac{R\epsilon_{\lambda,a} \times [L_{\lambda,b}(T_b)/L_{\lambda,b}(T_t)]_b - [L_{\lambda,b}(T_{sur})/L_{\lambda,b}(T_t)]_{sur}}{1 - [L_{\lambda,b}(T_{sur})/L_{\lambda,b}(T_t)]_{sur}} \quad (4)$$

The terms in the brackets represent spectral radiance ratios for the reference blackbody-to-target (b) and for the surroundings-to-target (sur). If the reference blackbody and target temperatures were identical ($T_b = T_t$) and the surroundings temperatures were low with respect to the target temperature ($T_{sur} \ll T_t$), the measurement equation would reduce to the simple form, $\epsilon_\lambda = R\epsilon_{\lambda,a}$. Hence, these spectral radiance ratios can be considered as corrections due to nonisothermal conditions between the target and reference blackbody (b) and to reflected irradiation from the surroundings (sur).

Effect of Nonisothermal Conditions

The effect of nonisothermal conditions between the reference blackbody and the target on the spectral emissivity is significant even for small temperature differences, but dependent upon temperature and wavelength conditions. Using Wien's approximation to Planck's law[7,8] for blackbody spectral radiance,

$$L_{\lambda,b} = c_1 \lambda^{-5} \exp(-c_2/\lambda T) \quad (5)$$

the relative radiance change due to a fractional temperature change is

$$\frac{\Delta L}{L} = \frac{c_2}{\lambda T} \frac{\Delta T}{T} \quad (6)$$

With $c_2 = 14388$ μm·K and for a value of $\lambda T = (\lambda T)_{max} = c_3 = 2898$ μm·K, $c_2/\lambda T$ has the value of 5, indicating a strong effect of temperature difference on the radiance. For example, at 1 μm and 800 K, which are representative conditions for the present method, a 1 K difference between the reference blackbody and target will give rise to a relative radiance change of 2.3%, which propagates to a relative error in the emissivity ($\Delta\epsilon_\lambda/\epsilon_\lambda$) of 2.3%. Hence, for high accuracy in determining the spectral emissivity, it is necessary to evaluate the temperature uniformity between the reference blackbody and the target within a fraction of 1 K. This effect becomes most significant at shorter wavelengths and lower temperatures.

The Effect of Reflected Irradiation

The effect of reflected irradiation originating from the surroundings at T_{sur} on the spectral emissivity depends upon the target temperature T_t, wavelength, and the spectral emissivity of the target material ϵ_λ. From Eq. (1), neglecting the background radiance terms, and the definition of spectral emissivity, the effect can be expressed as

$$\frac{\epsilon'_\lambda - \epsilon_\lambda}{\epsilon_\lambda} = \left(\frac{1}{\epsilon_\lambda} - 1\right) \frac{L_{\lambda,b}(T_{sur})}{L_{\lambda,b}(T_t)} \quad (7)$$

where ϵ'_λ is the observed spectral emissivity biased by the irradiation effect. For the condition $\epsilon_\lambda \approx 1$ and/or $T_{sur} \ll T_t$, the effect is negligible. The effect for a target temperature of 800 K and surroundings at 300 K with various values of target emissivity ϵ_λ is represented in Fig. 1. It is evident that the effect is most significant at long wavelengths for low-emissivity (high-reflectivity) materials. In order to make irradiance corrections to the experimental observations with high precision, it is necessary that the surroundings be highly absorbing and of uniform temperature. However, from consideration of the relative radiance change of Eq. (6), but using the Planck law, the isothermal requirement for the surroundings is of the order of 1 K rather than a fractional amount.

Description of the Apparatus

The measurement method is a pseudointegral blackbody technique wherein the sample and reference blackbody cavity are in close proximity in a nearly isothermal region, but are not part of the same body. Target temperatures are measured, but do not require separate control.

The Equalizing Block

The heated component of the sample holder assembly diagrammed in Fig. 2 is a cylindrical copper equalizing block of 114.3 mm diameter and 25.4 mm thickness. The surface has been nickel plated for stable radiative properties and to increase the radiative heat-transfer resistance. Two capped conical cavities of 4 mm diameter extend to a depth of 20.3 mm

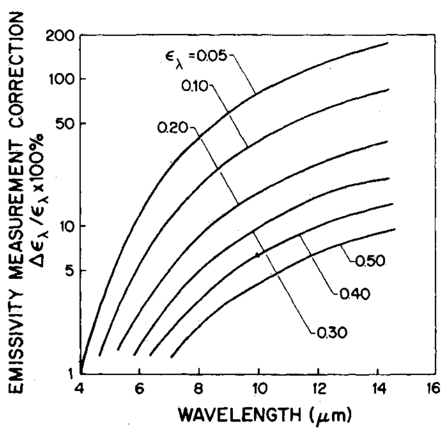

Fig. 1 Emissivity correction due to reflected irradiation for various target emissivities ($T_t = 800$ K, $T_{sur} = 300$ K).

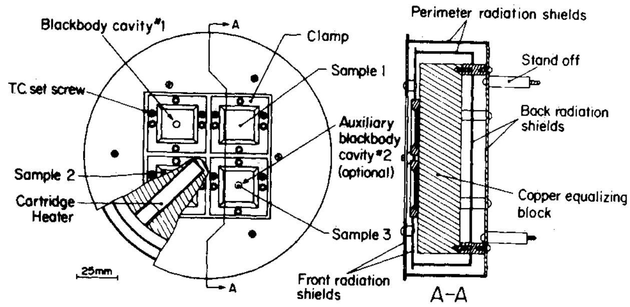

Fig. 2 Front and cross-sectional views of the sample holder assembly.

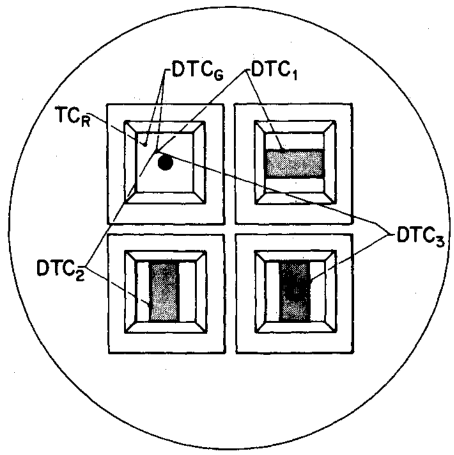

Fig. 3 Arrangement of the reference thermocouple TC_R and the differential thermocouples for the samples DTC_1–DTC_3.

within the block. Cavity 1 is the primary blackbody against which all emissivity measurements are referred. Cavity 2 is an auxiliary cavity, used for comparative radiometric measurements, and can be covered with a sample target if desired. Double radiation shields of polished stainless steel surround the block perimeter and the front and back sides. Square openings are provided on the front-side shielding to permit viewing of the target. The block is supported by stainless steel standoffs, extending through the back-side shielding, the ends of which are grooved to make line contact, thereby increasing thermal contact resistance between the copper block and its mounting frame (not shown).

The block is heated with six 75 W Watlow cartridge-type heaters of 31.8 mm length and nominal diameter of 6.25 mm. To avoid hot spots on the cavity walls and target surfaces, the heaters are positioned symmetrically with respect to the cavities and as far away from the samples as possible. The block is instrumented with Omegaclad iron-constantan (type J) thermocouples. Since the temperature gradients within the copper equalizing block are small, the temperatures of the samples and cavity are determined using the differential thermocouple (DTC) arrangement of Fig. 3. The differential thermocouples DTC_1, DTC_2, and DTC_3 provide a measure of the difference between the three samples and the reference cavity 1 (near the bottom of the conical shape). The differential thermocouple DTC_G provides a measure of the temperature gradient along the depth of the reference cavity 1. The thermocouple TC_R provides an absolute indication of the reference cavity temperature. Details of the sensing junction locations for the sample DTC's are subsequently presented.

The sample holder is enclosed in a water-cooled vacuum chamber (203 mm diameter, 229 mm high) coated inside with diffuse black paint to minimize interreflections. A sodium chloride port window (102 mm diameter, 12.7 mm thick) permits emissometer viewing over the 0.4–15 μm spectral range. Chamber pressure sufficient to minimize convection effects is maintained below 40 μm Hg with a mechanical vacuum pump.

Sample Coupons

Coupons to accommodate the sample targets as shown in Fig. 4 are mounted on the equalizing block. The coupons permit viewing an 18 mm^2 area and are mounted with nickel-plated copper clamps. A hole to accommodate one leg of the differential thermocouple (DTC_1, DTC_2, or DTC_3) is drilled laterally into the coupon for measurement of the sample temperature. To improve thermal contact between the coupon and the sample, the slot is coated with Cotronics 931-graphite adhesive. The sample is then pressed into the slot and the coupon peened near the sample edges to further improve thermal contact.

Optical Arrangement

The optical elements of the emissometer shown in Fig. 5 are mounted on a 25 mm thick aluminum plate (92×138 cm). Radiation from the viewed target is reflected from M-1 onto the spherical mirror M-2, which focuses the image of the target on an aperture (2 mm diameter). Two solenoids are located in a diagonal fashion on the back of planar mirror M-1. Their arrangement allows the system to view the four different targets of the sample holder. The radiation passing through the aperture is modulated with a 1 kHz chopper. Spherical mirror M-5 focuses the aperture image onto the entrance slit of the monochrometer (magnification 2:1).

A Perkin-Elmer model 98 single-beam, single-pass monochrometer with LiF and NaCl prisms spectrally disperses target radiance, with the exit beam being refocused onto the detector element by a 90 deg off-axis ellipsoid M-6. The wavelength setting is determined from a potentiometer reading that is related to a wavelength calibration curve. Further details on the emissometer have been reported previously.[9,10]

Detection and Data Acquisition

Two IR Associates HgCdTe liquid-nitrogen-cooled detectors cover the 1.5–12 μm wavelength range. The 1 kHz modulated signal is input to an Ortec-Brookdeal model 9503 lock-in amplifier. The data acquisition and processing system (DAPS) consists of an HP-3497A datalogger controlled by an HP-85 minicomputer. The HP-85 has a serial interface that is used to send data to a VAX computer for final processing. The datalogger is equipped with two 20 channel multiplexers for voltage measurements and a 16 channel digital actuator for operation of the emissometer solenoids.

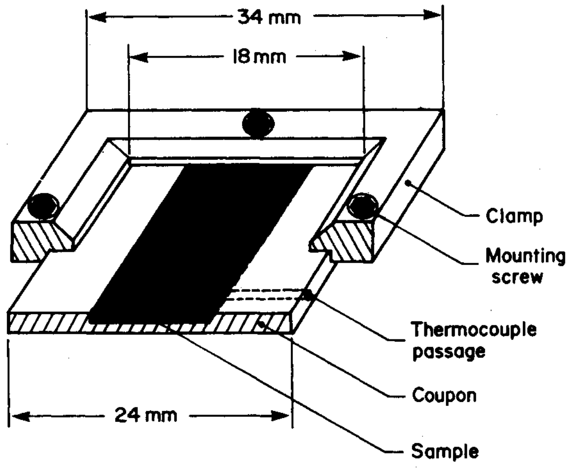

Fig. 4 Cutaway view of the square sample coupon.

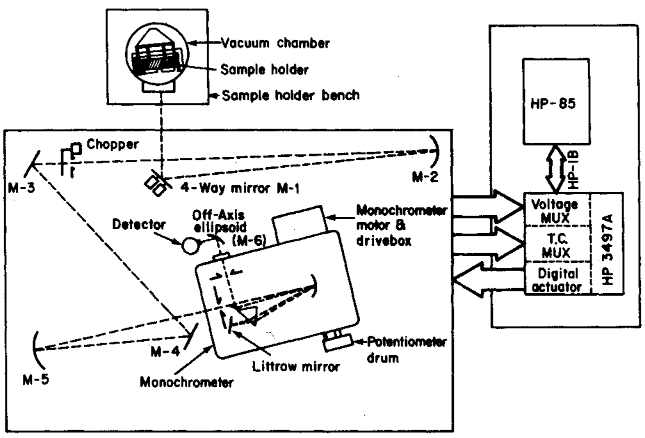

Fig. 5 Schematic of emissometer optical arrangement, detection system, and data acquisition unit.

Performance Evaluation

Reference Blackbody Cavity

A conical geometry was chosen as the reference blackbody configuration primarily because of its favorable directional radiance characteristics. Using a Monte Carlo technique, Polgar and Howell[11] have shown that the apparent emissivity $\epsilon_{\lambda,a}$ of a cone is directionally independent as long as the cone apex is in view. Hence, for a cone half angle of 7.5 deg, the $\epsilon_{\lambda,a}$ is the same for all incidence viewing angles within 7.5 deg of the conical axis. The sample holder utilizes the capped conical cavity of Fig. 6 as the reference blackbody.

A Monte Carlo method was developed for estimating $\epsilon_{\lambda,a}$ of the capped-conical cavity (Fig. 6) having walls with specular-diffuse reflection characteristics. In its present form, the model is restricted to an isothermal condition. The existing cavity, formed in the copper equalizing block, closely approaches the isothermal condition, with maximum relative temperature differences $\Delta T/T$ better than 0.2% as measured between the cone apex and opening.

In the cavity analysis, the wall reflectivity ρ_w is expressed as

$$\rho_w = \rho_s + \rho_d = \rho_d \left[1 + \frac{\rho_s}{\rho_d} \right] \tag{8}$$

where s and d denote the specular and diffuse components, respectively, and the ratio ρ_s/ρ_d is the specular fraction. The cavity was analyzed for wall emissivities $\epsilon_{\lambda,w}$ between 0.80 and 0.96 with $\rho_s/\rho_d = 0$, 0.1, and 0.2. The $\epsilon_{\lambda,a}$ for different wall emissivities $\epsilon_{\lambda,w}$ and ρ_s/ρ_d values were fit to a curve, giving the

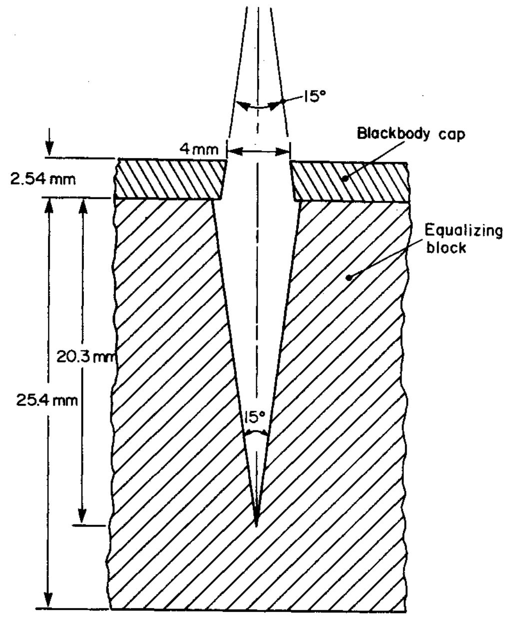

Fig. 6 Cross-sectional view of the capped conical cavity in the equalizing block.

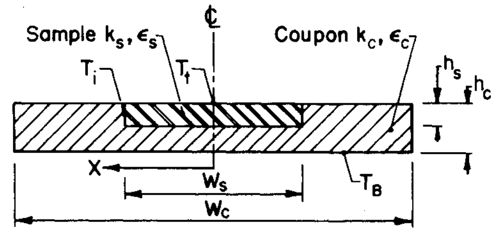

Fig. 7 Two-dimensional heat-transfer model of the sample coupon.

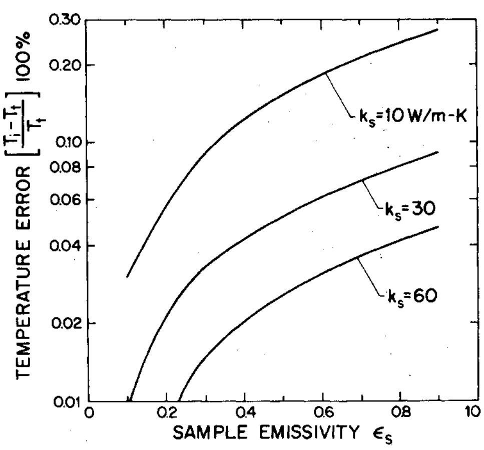

Fig. 8 Indicated temperature errors for various sample thermal conductivity values.

relationship

$$\ln(\epsilon_{\lambda,a}) = \left[0.054 - 0.05 \left(\frac{\rho_s}{\rho_d} \right) + 0.1 \left(\frac{\rho_s}{\rho_d} \right)^2 \right] \ln \epsilon_{\lambda,w} \tag{9}$$

Note that the apparent emissivity increases with the fraction ρ_s/ρ_d, indicating that a high specular fraction is advantageous. However, the value of ρ_s/ρ_d for a surface is determined only by very elaborate testing and is therefore rarely known. In practice, the diffuse condition ($\rho_s/\rho_d = 0$) can be

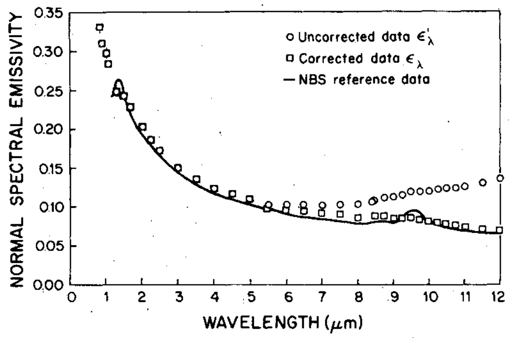

Fig. 9 Normal spectral emissivity of platinum/10% rhodium with and without the reflected irradiation correction.[13]

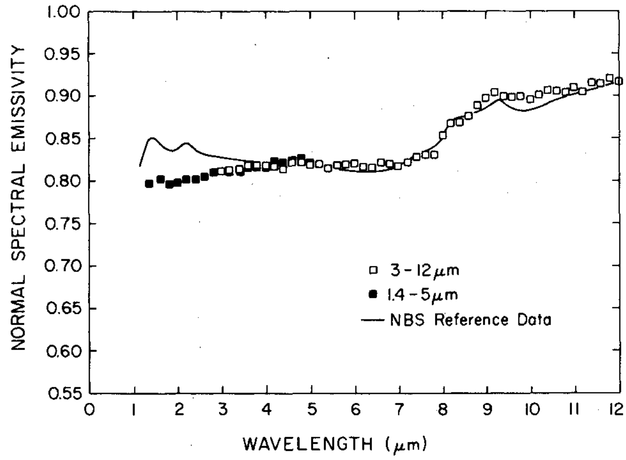

Fig. 10 Normal spectral emissivity of the oxidized Inconel standard compared with NBS reference data (800 K).[13]

closely approximated by roughening the cavity surface and coating it with a flat paint. Also, a near-diffuse surface is more stable as many high-gloss paints degrade after repeated thermal cycling. Therefore, it is better to fabricate and model a black diffuse surface for accurate estimation of the apparent emissivity.

The cavities were formed by drilling into the equalizing block with a conical shaped bit (15 deg apex angle) to a depth of 15 mm, followed by an electric-discharge machining (EDM) process to give the cavity its final depth (Fig. 6). The tip of each apex was punched with a stainless steel pin to provide a true point. The cavity walls were roughened uniformly with a coarse rubbing compound and the tapered caps drilled with the same bit used for the cavities. The cavities and caps were coated with Pyromark 1200, a flat black stovepaint.

To estimate the cavity wall emissivity, a copper coupon was painted in a manner similar to the cavities. Radiance measurements were then performed on the coated coupon at 800 K for the spectral range of interest. From these measurements, the apparent emissivity $\epsilon_{\lambda,a}$ and cavity wall emissivity $\epsilon_{\lambda,w}$ were determined using the results of the Monte Carlo analysis. From the Monte Carlo analysis, assuming diffuse surfaces ($\rho_s/\rho_d=0$), Eq. (9) reduces to

$$\epsilon_{\lambda,a}=\epsilon_{\lambda,w}^{0.054} \qquad (10)$$

given the measured spectral radiances and sample/reference blackbody temperatures, Eqs. (4) and (11) form a system of two equations with two unknowns ($\epsilon_{\lambda,a}$ and $\epsilon_{\lambda,w}$) that can be solved by an iterative technique. The values of $\epsilon_{\lambda,a}$ as a function of wavelength were found to be relatively constant, varying between 0.996 and 0.999 across the 1-15 μm spectral range.

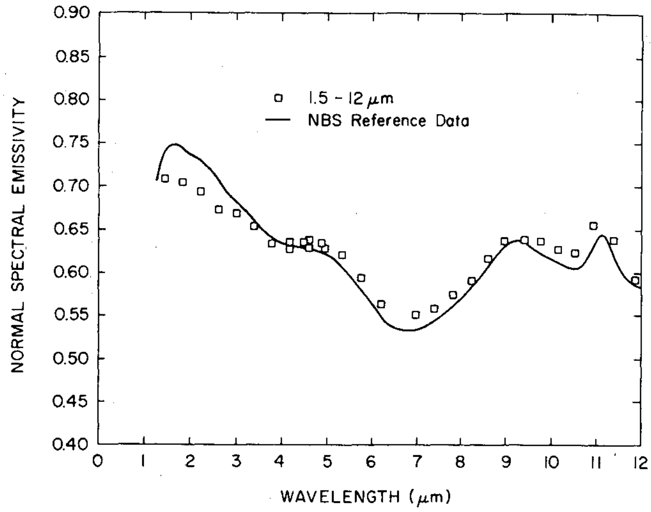

Fig. 11 Normal spectral emissivity of the oxidized Kanthal standard compared with NBS reference data (800 K).[14]

Sample Coupon Analysis

The influence of nonisothermal conditions and temperature errors on the spectral emissivity determination is significant, especially at short wavelengths. A concern with the present coupon arrangement (Fig. 4) is the difference between the actual temperature of the target T_t and the indicated temperature of the thermocouple T_i. A two-dimensional steady-state, finite-difference heat-transfer analysis was performed to estimate the error in determining the target temperature.

The model of the sample coupon is illustrated in Fig. 7. It is assumed that the bottom surface is isothermal at $T_B=800$ K and the edges are adiabatic while the upper sample surface experiences radiative exchange with the chamber walls at 300 K. Sample conductivities k_s of 60, 30, and 10 W/m·K and sample total emissivity ϵ_s between 0.1 and 0.9 were investigated. The remaining parameters were held constant at these representative values: $k_c=360$ W/m·K, $h_c=2.54$ mm, $h_s=1.27$ mm, $W_c=24$ mm, and $W_s=6.6$ mm. The indicated temperature T_i was taken at the sample edge 0.64 mm below the surface and the target temperature T_t at the center of the sample surface.

The conditions that produced the largest temperature gradients were those of low k_s and high ϵ_s. Temperature measurement errors $\Delta T/T=(T_i-T_t)/T_t$ for the various values of these parameters are presented in Fig. 8. The highest $\Delta T/T$ shown, occurring with $k_s=10$ W/m·K and $\epsilon_s=0.9$ is 0.27%. From Eq. (6), at a wavelength of 1.0 μm, the corresponding radiance error $\Delta L/L$ is 4.9%, indicating that the present coupon arrangement is not suitable for materials of low thermal conductivity. The metallic materials of the present study have thermal conductivities in the 60 W/m·K range. The corresponding $\Delta T/T$ for $\epsilon_s=0.9$ is 0.046%, giving a radiance error $\Delta L/L$ of 0.8% at 1.0 μm. This error is for the worst case condition and will decrease for lower ϵ_s and longer wavelengths. It is concluded that the arrangement of a thermocouple located at the side of the sample provides an accurate estimate of the sample target surface.

Reflected Irradiation Effects

The reflected irradiation contribution $(1-\epsilon_\lambda)L_{\lambda,b}(T_{sur})$ to the total radiance becomes significant at low emissivities and long wavelengths. The results from the spectral emissivity measurements of the well-characterized, low-emissivity platinum/10% rhodium surface will be described to demonstrate this effect.

The uncorrected ϵ_λ' values (open circles) are compared with the NBS reference data (solid line) for platinum/13%

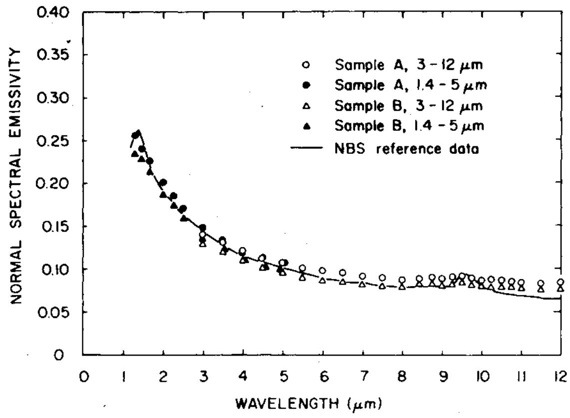

Fig. 12 Normal spectral emissivity of platinum/10% rhodium compared with NBS reference data for platinum/13% rhodium (800 K).[13]

rhodium§ 1-12 μm in Fig. 9. The ϵ_λ' values are systematically greater than the reference values as wavelength increases, whereas the corrected ϵ_λ values (open triangle) correlate well with the NBS data.[13] Note the indifference between ϵ_λ' and ϵ_λ at wavelengths less than 5 μm. The agreement between corrected ϵ_λ values and the reference data supports the accuracy of the correction procedure.

Repeatability Studies

An experiment was performed to determine the repeatability of the measurement apparatus, defined as the standard deviation of several emissivity measurements on a single sample. Good repeatability (low standard deviation σ) implies that the optical alignment, temperature determination, and other factors influencing the measurement can cycle repeatedly without significant change.

The experiment was performed at 800 K and a wavelength setting of 1.0 μm selected because of the high ratio of radiance to temperature change. The value of σ was based on 10 measurement cycles of the data acquisition routine. The test was performed using two metallic samples and the auxiliary blackbody, cavity 2. Results show that the repeatability is always within 0.005 emissivity units and is generally in the vicinity of 0.001.

It should be noted that the observed maximum fluctuation in temperature of any 1 sample during a 10 measurement cycle sequence was 0.55 K. From Eq. (4), the radiance change due to this fluctuation is 1.2% at 1.0 μm. However, repeatability measurements were observed to be an order of magnitude less, indicating that system temperature corrections are reliable.

Detector Uniformity and the Off-Axis Effect

The spatial separation of the targets can give rise to nonsystematic errors in the measurement method. When mirror M-1 is switched to view a different target, the incidence angle between M-1 and spherical mirror M-2 changes, causing a microscopic shift in the central ray of the optical system. If the radiation detector element does not have a spatially uniform responsivity, the image shift will cause a spurious signal change unrelated to target radiance. To assure the system is not influenced by this effect, physically separated sources of equal radiance (cavities 1 and 2) were observed. The experiments showed that with a properly aligned optical system, there are no measurable signal differences when viewing the two blackbodies.

§As discussed subsequently, the difference of rhodium alloying percentages (10 vs 13%) has little effect on the spectral emissivity.

Results on Reference Materials

Spectral Emissivity of Oxidized Inconel

The Inconel stock was obtained from the NBS in the form of a 25.4×254 mm strip of thickness 1.35 mm preprocessed according to the procedures described in Ref. 14. Samples of rectangular shape, 6.35×22.2 mm, were cut from the stock and their spectral emissivities measured at 800 K.

During the Inconel machining process, removal of some surface particles was observed, thus exposing the unoxidized metal. Subsequent emissivity measurements performed on the fabricated samples were significantly lower than the NBS reference data at shorter wavelengths. The damaged oxide layer was suspected as the cause of the differences.

Inconel has a regenerative quality whereby the damaged surface can be restored upon retreatment. Following the procedures described in Ref. 14, the Inconel sample was cleaned in acetone after machining, passivated for 1 min in 10% nitric acid at 316 K, rinsed in distilled water and then acetone. It was placed in a cold furnace, which was brought to 1340 K and held for 24 h; the temperature was then reduced to 1100 K and held for an additional 24 h, after which the sample was allowed to cool in the furnace. After retreatment, the sample was fit to a square copper coupon as shown in Fig. 4.

Figure 10 shows the spectral emissivity of the retreated Inconel measured between 1.4-12 μm at 800 K. The measured values are in good agreement with the NBS reference data, especially over the 4-12 μm range. The retreatment seems to have promoted the formation of a new oxidized surface on the Inconel.

Spectral Emissivity of the Kanthal Working Standards

The Kanthal stock was obtained from the NBS in the form of a 25.4 × 254 mm strip of 1.09 mm thickness preprocessed according to the procedures described in Ref. 14. Samples were fabricated from the stock and their spectral emissivities measured at 800 K.

A rectangular-shaped Kanthal sample was cut to 9.53×22.2 mm and fit to a square nickel-plated copper coupon. During the machining process, a piece of paper was taped over the specimen for protection from scratching. The sample was fit to a coupon as fabricated, with no chemical or heat treatment prior to measurement.

The results of the Kanthal measurements are compared with the NBS reference data in Fig. 11. The numerous inflections of the reference curve are followed closely by the measurement data at wavelengths greater than 2 μm. As with the Inconel, the results agree well at longer wavelengths, being within 0.02 units of the NBS data at wavelengths greater than 3 μm.

The results under 3 μm compare less favorably with the NBS reference data. The fabrication process is again suspect in causing a degradation of the oxide film. While Kanthal has a less fragile oxide film, it lacks the regeneration qualities of Inconel. Therefore, any damage incurred during fabrication cannot be reversed. Also, it is worthy to note that other users of Kanthal reference materials have experienced significant departure from the NBS data (i.e., Ref. 12).

Spectral Emissivity of Platinum/10% Rhodium

The main objective of the platinum/10% rhodium sample measurements (800 K) was to test the apparatus and method on a low-emissivity surface. A second objective was to reproduce surface characteristics of NBS platinum/13% rhodium reference material as closely as possible by treating samples in accordance with procedures outlined in Ref. 13.

Platinum/10% rhodium stock was received from Engelhard Industries in the form of a 22.2 mm square of thickness 0.89 mm. The stock was washed in a hot, all-purpose detergent and tap-water solution, rinsed in deionized water and then in ethyl alcohol, allowed to dry in air, and placed in a covered ceramic crucible suspended only by its corners. The crucible was placed in a nichrome heating element furnace at room temperature and brought to a temperature of 1500 K over a period of 2 h, held at temperature for an additional 2 h, and allowed to cool within the furnace for 2 days. Using a die punch, two circles 6.35 mm diameter were punched from near the specimen corner with a dowel pin. The side opposite the punched surface constitutes the target sample surface. The die and all handling tools were rinsed thoroughly with trichloroethane prior to the punching process. A special copper coupon was fabricated to accommodate this small circular sample configuration.

The results of the platinum/10% rhodium data run are presented in Fig. 12, and compared with NBS reference data for platinum/13% rhodium at 800 K.[13] The agreement between measured and NBS reference data beyond 1.4 μm, being to within 0.02 units, indicates an accurate assessment of the irradiation component and a proper sample fabrication process. Also, the modified coupon arrangement for small circular sample targets has proved reliable.

The difference in the rhodium content of the present sample (10%) and the NBS reference sample (13%) is thought to have little effect on the ϵ_λ values. Emissivity measurements performed on pure platinum[15] have the same spectral contour and are in close agreement with the reference platinum/13% rhodium values.[13] Hence, the 3% rhodium content discrepancy between the present and reference material is of little consequence.

There is an inflection in the NBS data between 9 and 10 μm that does not occur with the present measurements. The cause of the NBS inflection is a silicon deposit on the reference samples originating from the silicon carbide heating elements used in their annealing furnace. The present samples, having been annealed in a nichrome element furnace, are therefore free of this contaminant.

Summary

The emissometer and sample holder performance at moderate temperatures has been investigated and the measurement objective met. The components of the sample holder, vacuum chamber, and optical system have been evaluated through analytical and experimental studies. The major effects influencing measurement accuracy—temperature differences between samples and reference cavities, blackbody quality, and reflected irradiation—have been quantitatively assessed and procedures developed for their correction. The effects due to veiling glare, off-axis operation, and calibration shifts can be avoided through proper procedures or equipment selection.

The accuracy of the method with its various corrections and the ability to generate reliable spectral emissivity data were assessed by the measurement results performed on the NBS reference materials. For oxidized Inconel retreated according to the NBS procedures, agreement between measurements and the reference data for the spectral region beyond 4 μm is excellent. At shorter wavelengths, the measurements are systematically lower and do not exhibit the spectral behavior displayed by the reference data. For the oxidized Kanthal standards, agreement was also very good beyond 3 μm. The differences for the Inconel and Kanthal surfaces at shorter wavelengths are likely due to variations in the surface characteristics of the sample rather than due to errors in the measurement method. For the platinum/rhodium alloy, agreement between the measurements and reference data was within 0.02 emissivity units; this excellent comparison is to be expected since replication of the surface conditions is easy to achieve. Other features of the method were successfully evaluated. The repeatability of the overlapping data in the 3–5 μm range, acquired with two independent prism/detector combinations, supports the reliability of both arrangements. Equal radiance measurements obtained with the two blackbodies indicates no off-axis effects due to physical target separation. For these reasons, the errors for the measurement method are believed to be within 0.02 emissivity units when corrections relating to nonisothermal conditions, apparent emissivity of the reference cavity and reflected irradiance are considered. It has been analytically determined that the isothermal correction for the apparatus is not valid for poor thermal conductors and should therefore be used only for measurement of primary metals.

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