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Armco Iron Normal Spectral Emissivity Measurements[1]

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Directional spectral emissivity data in different environments are needed in a great number of scientific and technological applications. In this work, the normal spectral emissivity of Armco iron is studied as a function of temperature under a controlled atmosphere. Emissivity values are calculated by the direct radiometric method. The evolution with thermal cycling, the dependence on temperature, and the effect of surface roughness are considered. Additionally, the electrical resistivity is calculated by using the Hagen–Rubens emissivity relation. This work makes progress in the use of Armco iron as an emissivity reference.

KEY WORDS: Armco iron; electrical resistivity; emissivity; infrared radiation; roughness.

1. INTRODUCTION

The emissivity can be considered as a surface thermophysical property of materials [1, 2]. A great number of scientific and industrial applications (heat transfer, heating efficiency, optical constants, pyrometry, etc.) need availability of experimental values of this physical property as a

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function of wavelength and temperature. However, we know that emissivity measurements are highly sensitive to experimental conditions. Some of them are related to sample characteristics such as composition, microstructure, or surface roughness. Others, such as thermal control, sample thermal gradient, or background radiation control, depend on the experimental setup design. In recent years, many efforts have been carried out in order to minimize these difficulties [3–8]. In our opinion, deficiencies in the control of experimental conditions can give rise to discrepancies in the spectral emissivity.

The lack of a standard is another important problem for the comparison of experimental emissivity values obtained by using different experimental devices. In this way, Armco type iron (99.8% pure iron) was recently proposed as a potential emissivity reference material [9] because it is a commercial standard low cost metal with well-defined and known material properties. Evidently, in order to perform a comparison, a precise definition of the sample surface state (roughness, oxidation state, etc.) is necessary. Thus, it is essential to have good atmosphere control in order to prevent noticeable modifications in the sample surface oxidation state during the measurement time. It is also important to control the sample temperature gradient to establish a correct measurement temperature. Finally, it is necessary to give an account of the importance of the blackbody radiation source position, to guarantee that it has the same optical path as the sample.

We report carefully measured spectral emissivities of Armco samples in order to make progress in the search of an emissivity standard. These measurements can be compared with similar experimental data in the literature. With the intention of checking the obtained emissivity values, we have calculated the Armco iron electrical resistivity by using the Hagen–Rubens emissivity relation. The calculated values have been compared with those values in the literature.

2. EXPERIMENTAL

The emissivity measurements have been obtained by using a new emissometer designed in our laboratory [10]. This apparatus can be used to obtain experimental values of the directional spectral emissivity as a function of temperature and environmental conditions (controlled atmosphere) for opaque samples. The measurements have been performed by using the direct radiometric method. The sample temperature has been controlled by means of a PID electronic system that ensures a surface radial temperature gradient of less than 5 K for the studied sample area. The setup can be evacuated, including the detection apparatus (Bruker's

IFS66v/S Fourier transform infrared spectrometer). KBr windows are used to introduce controlled atmospheres into the sample holder without disturbing the vacuum level in other parts of the experimental device. The uncertainty [10] in the emissivity measurements is less than 4%, except for low emissivity samples at low temperatures and long wavelengths where the uncertainty can increase up to 7% due to the low signal-to-noise ratio.

The samples are discs of 60 mm in diameter and 2 mm in thickness. Two Armco iron samples were measured: one of them (sample 1) was ground by an industrial grinding machine, the other (sample 2) was ground by the same process but, in addition, was manually ground using several grinding papers, the last one being a 1000 grit abrasive paper. In both cases, the sample roughness was measured in four different directions. The mean roughness parameters are shown in Table I. The roughness parameters shown in the table have the usual meaning: R_a is the roughness average, R_z is the average maximum height, and R_t is the maximum height of the profile.

3. RESULTS AND DISCUSSION

3.1. Evolution with Thermal Cycling

With the goal of studying the possible influence of thermal history on the emissivity, several thermal cycles have been performed on both samples: starting at room temperature and heating to 800°C, and then cooling again to room temperature. Normal spectral emissivity (ε) measurements have been carried out during heating between 200 and 700°C in 100°C steps. Before each measurement, the temperature was held constant for 20–30 min to guarantee sufficient thermal stability. In all cases, the emissivity spectra are monotonically decreasing with increasing wavelength, as typically observed in metals [1, 2].

Figure 1 shows a comparison of the emissivity spectra for sample 1 at three different temperatures between the first and fourth thermal cycles. A clear evolution of the spectral emissivity can be observed between both

Table I. Average Surface Roughness of Samples: Roughness Average (R_a), Average Maximum Height (R_z), and Maximum Height of the Profile (R_t)

Sample	R_a (μm)	R_z (μm)	R_t (μm)
1	1.09	7.28	9.94
2	0.080	0.82	1.36

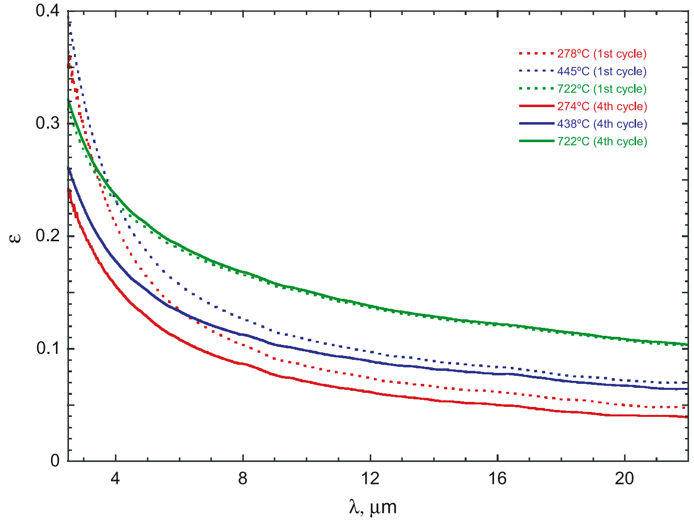

Fig. 1. Armco iron spectral emissivity for sample 1 at several temperatures. Comparison between first cycle (dashed curves) and fourth cycle (solid curves).

cycles. As reported by Bauer et al. [9], we find that after several cycles the emissivity spectra reach a stable state, and the samples show reproducible radiative properties. It is interesting to note that the changes in the emissivity with thermal cycling do not affect the whole spectra in the same way, and that it does not have the same effect at different temperatures. For a better visualization of these differences, Fig. 2 shows the evolution of the emissivity values with cycling number for sample 1 at three different wavelengths (4, 12, and 20μm) and temperatures. At low and medium temperatures, a decrease of the emissivity is observed, whereas at high temperatures, the emissivity does not show any noticeable change. For short wavelengths, the changes observed at low and medium temperatures are more remarkable.

A possible explanation of the observed effects is a slight change of the sample roughness. Nevertheless, roughness measurements performed after the thermal cycles show, in our case, that there are no noticeable changes. It must be remarked that Bauer et al. [9] observed an increase of roughness, although a decrease of roughness should have been observed to explain these effects. For this reason, they also rejected that the emissivity variation was caused by roughness changes. In addition, surface oxidation processes cannot be a valid explanation because they would have produced an increase of the emissivity rather than a decrease. The emissivity evolution could be attributed to the machining process. Thus, the

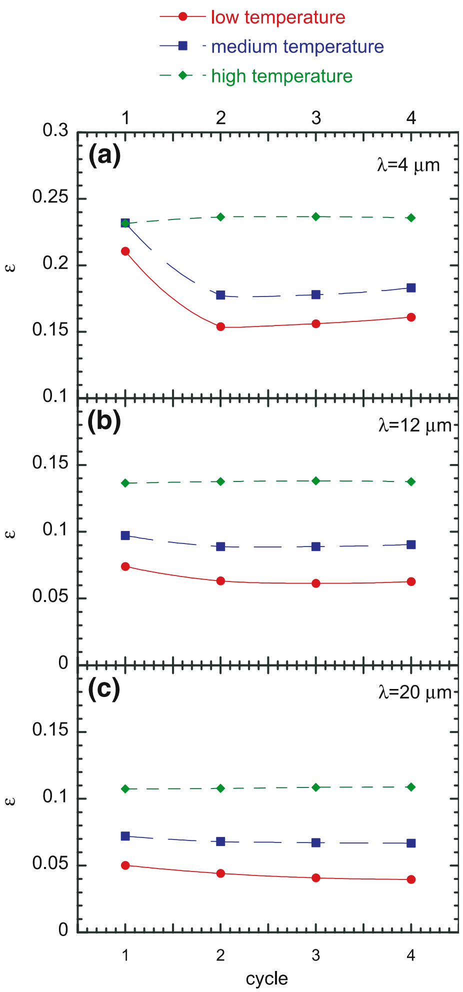

Fig. 2. Effect of thermal cycling on spectral emissivity of sample 1 for three different wavelengths: (a) 4μm, (b) 12μm, and (c) 20μm.

grinding could have created some defects in the sample that are removed during the first thermal cycle, and in this sense, the thermal cycle can be considered as a kind of annealing. Annealing usually involves thermally activated processes, which can progress at a sufficiently high constant temperature. This effect can be seen in Fig. 3, where three of the spectra (measured using sample 1, during the first heating cycle, at 445°C at different times) show a clear evolution towards a final state represented by the

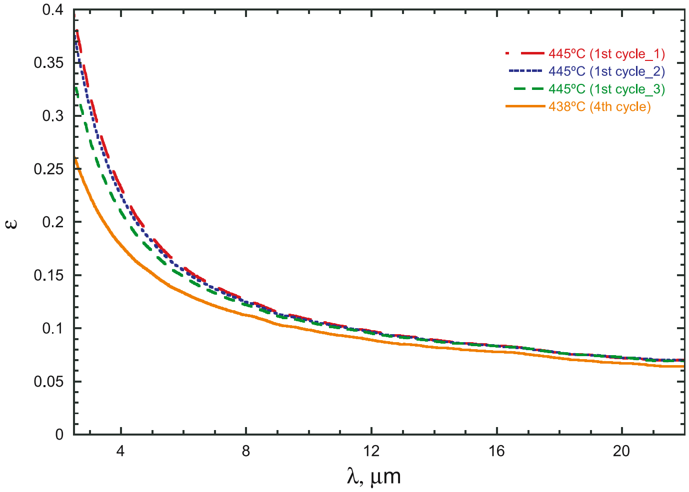

Fig. 3. Three spectral emissivity spectra for sample 1 measured at different times during first cycle at T = 445°C. A spectrum measured during fourth cycle (solid line) is also shown for comparison.

fourth spectrum in the figure (obtained at the same temperature during the fourth cycle).

3.2. Dependence on Temperature

Normal emissivity spectra have been measured at several temperatures for both samples between 200 and 800°C. Figure 4 shows the emissivity results obtained between 2.5 and 22μm at different temperatures. These are the spectra corresponding to the fourth thermal cycle, that is, to the situation when the emissivity has reached its stable state. The two samples show the expected tendency for most metals according to classical electromagnetic theory: the emissivity increases with increasing temperature [1, 4, 7]. For the sake of better visualization of this dependence, Fig. 5 shows the variation of the emissivity with temperature for wavelengths of 4, 10, 15, and 20μm. Both samples show the same qualitative behavior, but in sample 1 the emissivity changes are larger. Furthermore, for long wavelengths, the increase of emissivity is almost linear, with no slope dependence on wavelength. However, for short wavelengths, there is a growing tendency to a nonlinear behavior, especially in sample 2.

It is interesting to note that the linear dependence of emissivity with temperature allows a straightforward accurate extrapolation to temperatures out of the measurement range.

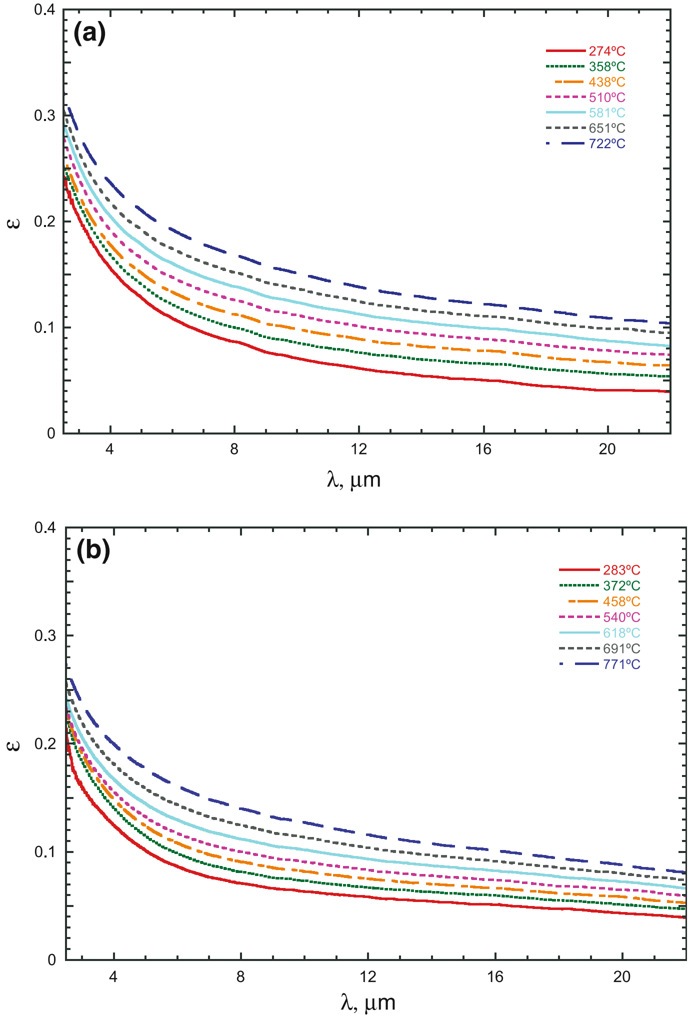

Fig. 4. Armco iron spectral emissivity for different temperatures: (a) Sample 1 and (b) Sample 2.

3.3. Effect of Surface Roughness

Both samples are identical except for their differences in roughness (Table I). Figure 6 shows the comparison for two temperatures between the emissivity of both samples. As is usual in metals [11–13], the smoothest sample (sample 2) has a lower emissivity. The difference between both

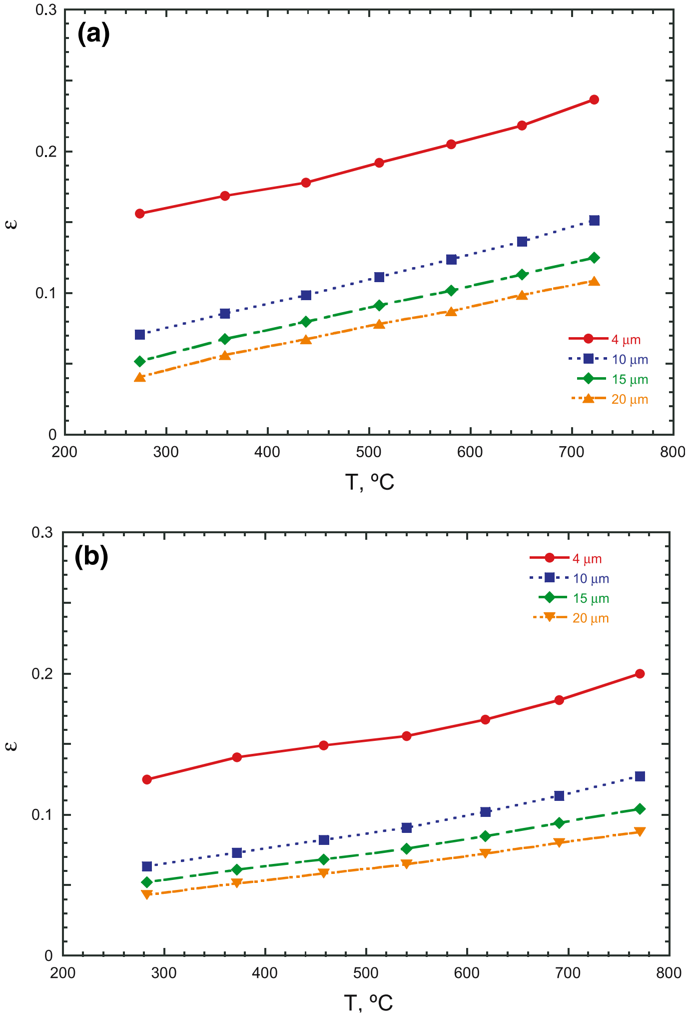

Fig. 5. Effects of temperature on spectral emissivity for several wavelengths: (a) Sample 1 and (b) Sample 2.

samples seems to be dependent of wavelength and temperature. For a better visualization of this effect, the relative difference between both emissivities is shown in Fig. 7 as a function of temperature for several wavelengths. As supported by a recent study by Ghmari et al. [14], the roughness effect must depend on wavelength. For long enough wavelengths compared to the period of the roughness, there must be no differences

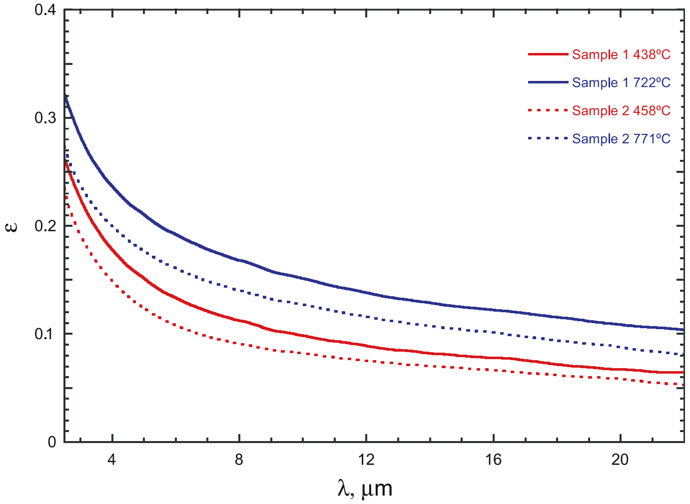

Fig. 6. Effect of surface roughness on spectral emissivity for two temperatures. Solid lines stand for rough sample (sample 1) and dashed line for smooth sample (sample 2).

between the smooth and rough surfaces. In our case, this effect only seems to occur at low temperatures: there are no noticeable differences between the two samples for long wavelengths, while for short wavelengths, there is up to a 15% difference. At higher temperatures, there is a clear difference between both samples in the whole spectra. And, surprisingly, at high temperatures, the difference is larger for long wavelengths than for short ones.

3.4. Electrical Resistivity Determination by Using the Hagen–Rubens Relation

In classical electromagnetic theory, the Hagen–Rubens emissivity relation [1, 2],

$$\varepsilon = 36.5\left(\frac{r_e}{\lambda}\right)^{\frac{1}{2}} - 464\frac{r_e}{\lambda} \quad (r_e \text{ in } \Omega\cdot\text{cm}, \lambda \text{ in } \mu\text{m}) \tag{1}$$

can be used to determine the normal spectral emissivity (ε) as a function of the electrical resistivity (r_e) and wavelength (λ).

In order to apply this relation, we study the smoother sample (sample 2), since the classical electromagnetic theory is only valid for perfectly flat surfaces. On the other hand, only values with $\lambda > \sim 5\mu$m must be considered. Thus, the emissivity spectra shown in Fig. 4b have been fitted using

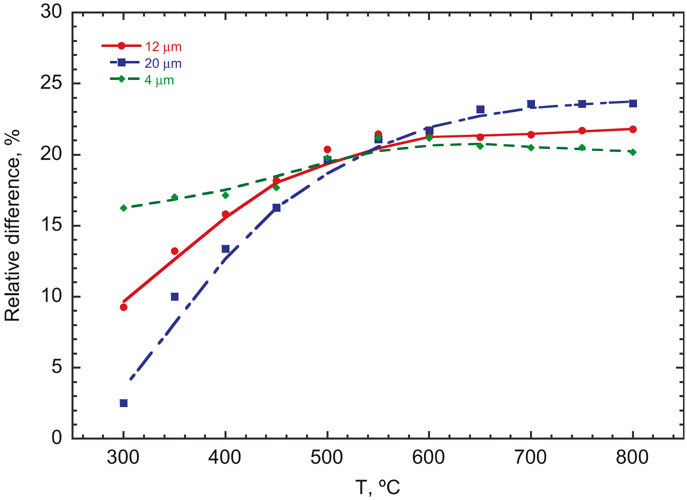

Fig. 7. Effect of temperature on the relative difference between the emissivity spectra of both samples (rough and smooth) at three different wavelengths.

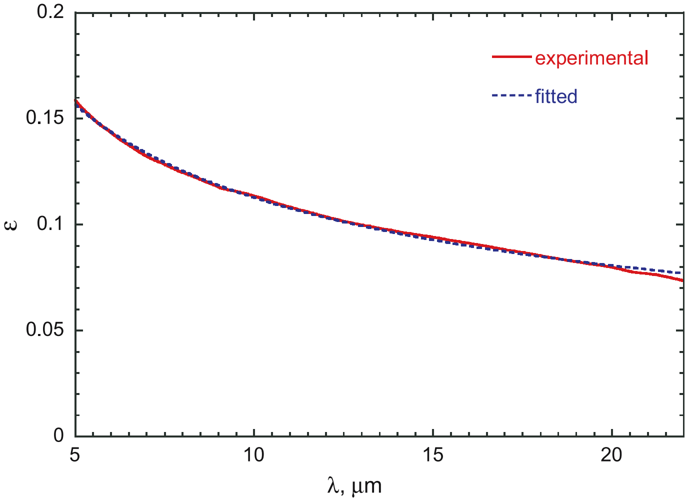

Fig. 8. Spectral normal emissivity for sample 2 at 691°C (solid line) and its corresponding fitted curve (dashed line) using the Hagen–Rubens emissivity relation.

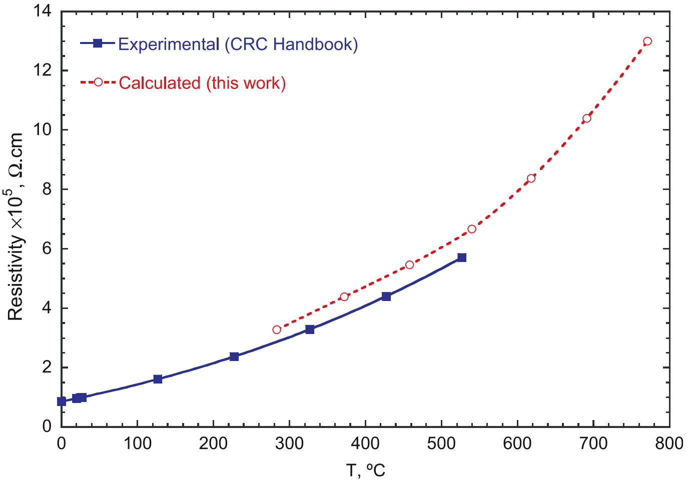

Fig. 9. Electrical resistivity dependence on temperature. Comparison between the electrical resistivity calculated using the Hagen-Rubens relation (dashed curve) and the directly measured values (solid curve) [15].

Eq. (1), with the resistivity being the fitting parameter. The fit between the experimental data and the Hagen–Rubens relation is very good (an example at 691°C is shown in Fig. 8). Furthermore, this allows us to determine the electrical resistivity of the sample. In Fig. 9, the resistivity values obtained are displayed as a function of temperature. The obtained values are in very good agreement with the resistivity data of pure iron in the literature [15]. Differences may be due to the roughness of the sample. Also, the data in the literature correspond to pure iron, and the calculated ones correspond to Armco iron (99.8% iron).

4. CONCLUSIONS

A dependence of the spectral normal emissivity on thermal cycling has been observed. The experimental results suggest that changes in emissivity are related to a thermally activated process in the sample. In order to obtain reproducible emissivities, the sample must be measured after several thermal cycles or after a suitable annealing process.

As the temperature increases, the spectral normal emissivity also increases. The temperature dependence for longer wavelengths is nearly linear. There is an increasing dependence of the spectral normal emissivity on surface roughness, which gets stronger at high temperatures. At low temperatures, emissivity differences due to surface roughness only exist for short wavelengths.

A satisfactory fit between the emissivity spectra and the Hagen–Rubens relation has been observed. It enables a determination of the electrical resistivity of the samples as a function of temperature. The calculated resistivity is in very good agreement with the direct measurement of this physical property.

The paper provides new measurements for the potential use of Armco iron as a reference material for emissivity measurements. Evidently, these data can be used as reference values by other researchers, but in order to improve and establish Armco iron as a standard, more experimental data by other laboratories would be desirable.

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REFERENCES

1. R. Siegel and J. Howell, *Thermal Radiation Heat Transfer*, 4th Ed. (Taylor and Francis, Washington, 2002).
2. Y. S. Touloukian and D. P. DeWitt, *Thermal Radiative Properties: Metallic Elements and Alloys, Thermophysical Properties of Matter* (IFI/Plenum, New York, Washington, 1970), Vol. 7.
3. R. M. Sova, M. J. Linevsky, M. E. Thomas, and F. F. Mark, *Infrared Phys. Technol.* **39**:251 (1998).
4. H. Oertel and W. Bauer, *High Temp. High Press.* **30**:531 (1998).
5. O. Rozenbaum, D. De Sousa Meneses, Y. Auger, S. Chermanne, and P. Echegut, *Rev. Sci. Instrum.* **70**:4020 (1999).
6. M. Kobayashi, M. Otsuki, H. Sakate, F. Sakuma, and A. Ono, *Int. J. Thermophys.* **20**:289 (1999).
7. T. Furukawa and T. Iuchi, *Rev. Sci. Instrum.* **71**:2843 (2000).
8. B. Zhang, J. Redgrove, and J. Clark, *Int. J. Thermophys.* **25**:423 (2004).
9. W. Bauer, M. Rink, and W. Gräfen, presented at *9th Int. Symp. Temperature and Thermal Measurements in Industry and Science* (TEMPMEKO), Cavtat, Dubrovnik 2004 (2004).
10. L. del Campo, R. B. Perez-Saez, M. Tello, X. Esquisabel, and I. Fernandez, *Rev. Sci. Instrum.* (submitted).

11. W. Sabuga and R. Todtenhaupt, *High Temp. High Press.* **33**:261 (2001).
12. M. Misale, C. Pisoni, and G. Tanda, *Heat Technol.* **6**:97 (1988).
13. C. D. Wen and I. Mudawar, *Int. J. Heat Mass Transfer* **47**:3591 (2004).
14. F. Ghmari, T. Ghbara, M. Laroche, R. Carminati, and J. J. Greffet, *J. Appl. Phys.* **96**:2656 (2004).
15. D. Lide, ed., *Handbook of Chemistry and Physics,* 75th Ed. (CRC Press, Boca Raton, Florida, 1994), pp. 12–40, 12–41.