Infrared Physics & Technology 69 (2015) 96–101

Contents lists available at ScienceDirect

Infrared Physics & Technology

journal homepage: www.elsevier.com/locate/infrared

Apparatus for measuring the emittance of materials from far infrared to visible wavelengths in extreme conditions of temperature

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HIGHLIGHTS

- New emissometer design for measuring optical properties at extreme temperatures.
- Procedure to calibrate a blackbody with two spectrometers.
- Emissivity spectra of ruby and $NdGaO_3$ from far infrared to visible wavelengths.
- Platinum optical response versus temperature.

ARTICLE INFO

Article history:
Received 5 December 2014
Available online 2 February 2015

Keywords:
Infrared emissometer
Emittance spectra
Blackbody calibration
Ruby
$NdGaO_3$
Platinum

ABSTRACT

A computer controlled circular turntable equipped with a blackbody reference and an integrated axisymmetric heating system based on a CO_2 laser is at the heart of the reported device. It allows performing emittance measurements in the spectral domain ranging from far infrared up to visible light and in a wide range of temperature. The apparatus includes two spectrometers and was built to achieve optimal experimental conditions of measurement, i.e. environmental stability and single optical path for the acquisition of the thermal fluxes. The specific design of the apparatus is firstly described; applied procedures for the characterization of the blackbody reference, laser heating and the retrieval of the emittance spectra are given after. Finally measurements obtained for ruby, $NdGaO_3$ and platinum are presented to illustrate the capacities of the apparatus.

1. Introduction

The knowledge of accurate thermal and optical properties of materials in extreme conditions of temperature is of interest in both industrial and scientific fields. In the manufacturing sector such as the glass industry, the need to master thermal heat transfer in the plants is mandatory to achieve efficient energy use and optimization of the costs [1,2]. The quality of products necessitating high temperature treatments is also often related to the accuracy of the control of temperature. When pyrometers are used to do this task, the knowledge of the emissivity of the material is generally a prerequisite for an accurate measurement of temperature. In the geosciences field, emittance spectra are also unavoidable data to be able to understand heat transfer and transformation occurring in the earth mantle [3,4]. Several devices [5–14] have been already published around this subject but most of them are limited to the characterization of the emissivity of opaque materials in restricted spectral ranges. In this paper the technical details of an experimental apparatus designed to measure the emittance spectra of opaque and semi transparent materials in the whole spectral range where thermal emission is produced (i.e. from far infrared up to visible wavelengths) is reported. The possibility to measure the infrared response of materials up to their liquid state, in such a wide spectral range, is of particular interest in material science. This allows for instance to characterize phase transition mechanisms of multiferroic compounds via the knowledge of the temperature dependence of their polar lattice dynamics.

The procedures to qualify the sensitive parts of the instrument such as the blackbody reference furnace, the CO_2 laser heating system and the method used to measure and compute the spectral emittance are given in a second part. Finally, the temperature dependences of the normal spectral emittance of a typical set of semitransparent and opaque materials (ruby, $NdGaO_3$, platinum) are reported in order to show the capacities of the apparatus.

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http://dx.doi.org/10.1016/j.infrared.2015.01.011
1350-4495/

2. Apparatus description

A schematic representation of the experimental device is given in Fig. 1.

As seen in the illustration, the blackbody reference and the CO_2 laser heating system are placed on a turntable whose position is fixed by a rotary stage (Aerotech® ALAR-150), the whole being controlled remotely by a computer. The blackbody is a PYROX PY8 furnace including a lanthanum chromite cylindrical cavity with a lateral circular aperture (blackbody target) of 8 mm of diameter. The heating system can be switched with the help of a quick lock system between a standard resistive ceramic plate heater ($T < 1200$ K) and laser heating. Extreme temperatures up to 2500 K can be easily achieved by using a CO_2 laser. The apparatus integrates a 500 W (Coherent K500) laser having a nearly axisymmetric power distribution with Gaussian profile and a diameter of 12 mm at $1/e^2$. This kind of heating is a must for the characterization of semitransparent materials because it works in such a way that it keeps a relatively cold surrounding around the sample (no necessity of water cooling) and avoids the flux pollution induced by more standard heating systems thanks to the single wavelength heating at 10.6 μm. To improve the temperature homogeneity of the sample, the laser beam is separated in two parts of equal power by a beam splitter, and a set of mirrors redirects the energy on both sides of the sample (Fig. 1). As shown on the representation of the apparatus, to avoid damage of the spectrometers, the laser beam does not impact the sample at normal incidence but with an angle that forbids the reflected beam to enter inside the spectrometers. If the sample thickness is less than few millimeters, this heating configuration ensures the axial homogeneity of the temperature. The Gaussian heating profile is at the origin of radial thermal gradients. The choice of a laser beam with a large diameter and the selection of an adequate aperture (1 mm of diameter) for the acquisition of all the fluxes is enough to ensure temperature homogeneity in order to perform accurate measurements. The turntable works as a lighthouse, it allows maintaining always a constant heating of the sample even during the rotation phases. This particular geometry makes also possible the successive placement of the sample and of the blackbody reference at the focus point of the optical system collecting the fluxes. To minimize optical aberrations, the optical path is kept as simple as possible; a single off-axis parabolic mirror is used to transport the emitted energy toward the spectrometers. In order to cover the widest spectral range in a given time, we have designed a configuration with two spectrometers. The first one is a Bruker Vertex 80v that works under vacuum. This instrument is used to acquire emittance spectra in far and mid infrared ranges. The second one, a Bruker Vertex 70 is purged with dry air and works between mid infrared and visible spectral ranges. The optical configurations used to acquire the spectra in the whole spectral range are given in Table 1.

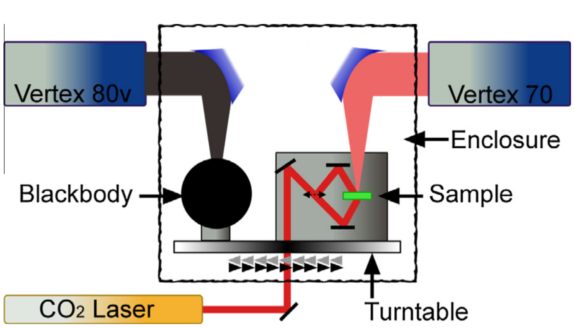

Fig. 1. Schematic representation of the emissivity apparatus including two Bruker spectrometers (Vertex 80v and Vertex 70).

The acquisition is made by pairs; the reference flux is measured with the first spectrometer and the sample flux with the second spectrometer and vice versa in the next round. To improve the environmental stability of the system, the whole optical system is placed into a purged enclosure, which also maintains the amounts of water vapor and CO_2 at a constant low level. The optical paths outside the spectrometers are purged with dry air. The inputs of the spectrometers are equipped with a Si window for the measurements in the far infrared range (below 500 cm^{-1}) and a KBr window for wavenumbers above 500 cm^{-1}. In practice, and due to the fact that the spectrometers are maintained at a constant temperature slightly higher than the room temperature, it is necessary to perform a supplementary measurement to account for a parasite flux originating from the instrumental surrounding.

2.1. Blackbody reference

The accuracy of the emissivity measurements is directly impacted by the quality of the blackbody reference and the accurate determination of its temperature. In the following we present two procedures to determine the thermodynamic temperature T_{BB} of the flux emitted by the blackbody furnace and its emissivity E_{BB}. Determination of the thermodynamic temperature T_{BB} with a good precision is achievable by using the spectrometers and a supplementary thermometer. The supplementary thermometer does not need to measure accurate temperatures; one just needs a linear response in the temperature range used for the calibration. The spectral domain used in the procedure must also be selected with great care; there is no constraint on the emissivity value but one must have the guaranty that there is no temperature dependence in the selected temperature and spectral ranges. With these hypotheses the method of determination of T_{BB} necessitates only measurements of the flux F emitted by the blackbody furnace at three temperatures: the temperature T_{BB} at which the reference needs to be calibrated (flux $F(T_{BB})$), a colder temperature $T_{BB} - \Delta T_{BB}$ (flux $F(T_{BB} - \Delta T_{BB})$) and a hotter one $T_{BB} + \Delta T_{BB}$ (flux $F(T_{BB} + \Delta T_{BB})$). The next step is to compute the following ratios from the three measured fluxes:

$$R^{-}_{\Delta T_{BB}}(\sigma) = \frac{F(T_{BB} - \Delta T_{BB})}{F(\Delta T_{BB})}, \quad (1)$$

$$R^{+}_{\Delta T_{BB}}(\sigma) = \frac{F(T_{BB} + \Delta T_{BB})}{F(T_{BB})}. \quad (2)$$

In these expressions, σ is the wavenumber indicating the spectral dependence. These ratios allow to get rid of both the transfer function of the spectrometer and true emissivity of the furnace. The last step is to fit the ratios with the following model:

$$R_P^{T\pm\Delta T}(\sigma) = \frac{\exp\left(\frac{hc\sigma}{kT}\right) - 1}{\exp\left[\frac{hc\sigma}{k(T\pm\Delta T)}\right] - 1} \quad (3)$$

h stands for the Planck constant, c is the speed of light and k represents the Boltzmann constant. The fit can be performed by minimizing the following objective function:

$$g(T) = \sum_{i=0}^{N}\left[\left(R_P^{T(1+A)}(\sigma_i) - R^{+}_{\Delta T_{BB}}(\sigma_i)\right)^2 + \left(R_P^{T(1-A)}(\sigma_i) - R^{-}_{\Delta T_{BB}}(\sigma_i)\right)^2\right]. \quad (4)$$

The value $A = \frac{\Delta T_{th}}{T_{th}} = \frac{\Delta T_{BB}}{T_{BB}}$ is a known constant ratio resulting from the choice of the temperature interval used for the calibration. T_{th} and ΔT_{th} are the temperature measured by the thermometer and the temperature gap, respectively. This optimization problem has a single adjustable parameter whose solution is the thermodynamic temperature T_{BB} of the blackbody reference. The procedure