A Simple High-Sensitivity Radiometer in the Infrared for Measurements of the Directional Total Emissivity of Opaque Materials at Near-Ambient Temperatures

A. Pantinakis^{1, 2} and N. Kortsalioudakis¹

Received November 20, 2000

A novel device for direct determination of the total directional emissivity of solid surfaces at near-ambient temperatures is described. The method of operation is based on comparing the radiation emitted by the surface with that emitted by a reference. The primary sensor is a solid-state, thermopile detector, and the signal recovery is achieved by phase-sensitive detection techniques, using a mechanical chopper. The device is simple and does not use any cooling. Experimental results are presented using a test sample of clear float glass, kept at 323 K (50°C), with black paint on a float glass substrate as the reference. These results, likely the first for the directional total emissivity of float glass at such low temperatures obtained without cooling, compare well with values in the literature.

KEY WORDS: directional total emissivity; float glass; radiometry; thermal infrared, thermopile detector.

1. INTRODUCTION

In applications where the accurate determination of heat transferred and/or lost is required, the angular dependence of emissivity and, more specifically, the directional total emissivity (DTE) must be known. This has led to many studies in a wide range of applications related to energy saving materials used in buildings and solar technologies [1-7].

There are basically two methods of obtaining the DTE of materials. In the first method, the DTE is determined indirectly, from measurements

¹Department of Sciences, Physics Section, Technical University of Crete, 73100 Chania, Greece.

² To whom correspondence should be addressed. E-mail: apostol@science.tuc.gr

of directional reflectivity [5, 7–9]. In the second method, the DTE is determined directly by holding a sample at a fixed temperature and measuring, with a broad-band detector, the amount of radiation the sample emits at direction θ [4, 10–12]. However, for the accurate determination of DTE with this method, the stray radiation reflected off the surface of the sample must be eliminated. At high temperatures, this does not constitute a serious problem, whereas at near-ambient temperatures (where the sample emits less thermal radiation), emissivity measurements may become inexact. So far, there exist several studies where for the emissivity measurement at room temperature, the sample is enclosed within a liquid nitrogen-cooled vessel, to avoid stray radiation and allow a measurable signal to be obtained [1, 10, 11, 13].

More recently, studies have appeared, which propose methods of measuring DTE at near-ambient temperatures without cooling [12, 14, 15].

In the present work, a novel apparatus is described for measuring DTE of opaque materials at near-ambient temperatures without using any cooling. As shown below, with our experimental setup the stray reflected radiation does not appreciably affect the values obtained, and this theoretical prediction is supported by the experimental results presented here.

With this setup, test results were obtained for a float glass sample at 323 K (50°C) over a range of angles from 10 to 70° from the normal to the sample surface. The results presented here fall into two groups. First, the capability of the apparatus was examined by measuring relative values of DTE as a function of angle, i.e., the angular shape of DTE. This is achieved by self-referencing (where the sample itself acts as the reference), and thus a separate reference material is not required. In this way, the experimental procedure is simplified and experimental uncertainties (e.g., due to temperature differences) are reduced. Our relative results are then compared to results derived for float glass at the NPL [16].

Having established the capability of our apparatus, it was then used to obtain absolute values of DTE. For this, a reference is required. In the experiment reported here, the reference is black paint on a float glass substrate. Finally, our absolute values of DTE are compared to absolute values in the literature [4, 8, 11, 16–18].

2. EXPERIMENTAL

2.1. Equipment

A schematic diagram of our apparatus is shown in Fig. 1. It consists of three main components, which are described below.



Fig. 1. Schematic of the apparatus: (1) thermally insulated chamber, (2) sample/reference holder, (3) diaphragm I, (4) mechanical chopper, (5) diaphragm II, (6) thermopile detector, (7) digital thermometer I, (8) lock-in amplifier, (9) digital thermometer II, (10) chopper drive unit, and (11) PC connected to lock-in. L1=10 cm, L2=3 cm, L3=6 cm, L4=5 cm, R=10 cm.

2.1.1. Sample/Reference and Their Positioning System

The apparatus consists of the sample and the reference, which emit infrared radiation. They are enclosed in a thermally insulated chamber whose interior is painted black to reduce stray radiation. The sample/ reference can be rotated around a vertical axis and the radiation for the sample/reference emitted at a given direction leaves the chamber through a 10-mm-diameter hole. The vertical axis of rotation of the sample/reference can also be displaced vertically to remove the sample/reference from the field of view of the detector. One half of a piece of a float glass with dimensions of 5 cm \times 8 cm \times 4 mm is used as the sample. The other half is painted black and serves as the reference. To keep the sample/reference at a fixed temperature, above ambient (around 50°C), resistive heating is used. It is implemented by "sandwiching" flat resistors between a ceramic plate and a sheet of copper onto which the sample/reference are attached. The ceramic plate isolates the sample/reference from the environment, and the copper ensures their homogeneous heating.

2.1.2. Detection System

The primary sensor is a thermopile detector (EG&G Heimann Optoelectronics, Type TPS434). The infrared radiation from the sample reaches the detector after being guided by diaphragms and mechanically chopped by a chopper with black blades. The TPS434 thermopile detector, which converts the infrared radiation to a voltage, has a comparatively high sensitivity of 36.6 V/W [19]. It consists of a number of thermocouples connected in series, forming a sensitive area of less than 1 mm². Because the experiment is carried out in air, the atmospheric absorption of water vapor and carbon dioxide must be taken into consideration. Calculations showed that the atmospheric absorption reduces the radiation reaching the detector by about 2% around 15 μ m (dominant band of CO₂) and by about 1% around 6.3 μ m (dominant band of H₂O). For this reason, the overall effect of atmospheric absorption can be ignored for our measurements. Finally, the optical alignment of the system is carried out using a He–Ne laser.

2.1.3. Signal Recovery System

The signal recovery system is based on phase-sensitive detection techniques, as the infrared radiation power reaching the detector is of the order of 10^{-9} W. The ac voltage of the thermopile detector, due to the chopping, is fed to the signal input of a lock-in amplifier (LIA, Stanford Research Systems, Model SR510), the serial output (RS232) of which is monitored by a PC for recording and storing the data.

2.2. Experimental Procedure

The quantities to be measured for the purpose of our experiment, in the manner described in Section 3, are (a) the output voltage of the LIA, when the sample is in front of the detector at each angle of emission, from 10 to 70° every 10°; (b) the LIA output voltage for the reference at 10°; (c) the LIA output voltage for the chamber wall, when the sample and the reference are lifted away from the field of view of the detector; and (d) the temperatures of the sample/reference, the chamber, and the room.

The above-mentioned temperatures are continuously monitored. They do not vary appreciably during the experiment (as shown in Fig. 2) and so the variations are not included in the calculation of DTE. The temperature of the sample/reference is monitored by a type K thermocouple attached to the sample/reference surface. The chamber and room temperature are monitored by using digital thermometers. All temperatures are recorded and stored during the experiment.

Before initiating measurements, one must ensure that the system has reached thermal equilibrium. This takes about 2 h. Then the optical chopper is switched on and operated at 11 Hz, while at the same time the goniometric device is set to 10° (with respect to the normal to the sample surface) as the initial angle of emission.



Fig. 2. Variation of temperature with time for (a) the sample, (b) the inner wall of the chamber, and (c) the air outside the chamber, during an experimental run.

After these preparations, the system is ready to take measurements. The experimental values are taken in the following order. First, the signal from the sample at each angle is taken. Then the sample holder is raised so that the reference is now in view and the signal is taken. The measurement with the reference in position is taken only at 10°. At the end, the sample holder is raised further, the wall of the chamber is now in view, and its signal (emitted near normal) is taken. To reduce random effects, 10 values at each angle are taken, over a time of several minutes. The statistical analysis of these data is performed by a suitable on-line program compiled for this purpose, to obtain the mean value and the uncertainty of DTE at each angle.

3. THEORETICAL MODELING

Theoretical modeling of the experiment described below is based on the work of Jones and Pantinakis [10, 11], where a full description of the model is given. The signal recovery method is based on phase sensitive detection as described in Ref. 20. According to the modeling, if V_x denotes the output voltage of the thermopile detector where the sample S, reference R, or wall W is in view (from now on, S, R, and W are referred to as "sources"), then

$$V_X = r(\Phi_X^{\text{open}} - \Phi_X^{\text{closed}}), \qquad X = S, R, W$$
(1)

where Φ_x^{open} is the radiant power absorbed by the thermopile detector when the chopper blades are open (sources in view), and Φ_x^{closed} is the power when the blades are closed (blades in view), and *r* is the detector response.

 Φ_x^{open} consists of two terms:

$$\Phi_X^{\text{open}} = C_{1X} E_{1X} + C_{2X} E_{2X} \tag{2}$$

The first term in Eq. (2), $C_{1X}E_{1X}$ is the radiant power emitted by the source and absorbed by the thermopile. E_{1X} is the radiant power that would have been absorbed by a nonselective detector in the absence of any diffraction.

The factors C_{iX} (i = 1, 2) involve spectral quantities, all of which are assumed not to vary appreciably during the experiment. The second term, $C_{2X}E_{2X}$, is the stray radiant power that arises from beyond the chopper blades, and so is chopped. Under our experimental conditions, this quantity is very small with respect to the first term.

 Φ_x^{closed} is given by

$$\Phi_X^{\text{closed}} = C_{BX1} E_{BX1} + C_{BX2} E_{BX2}$$
(3)

The first term, $C_{BX1}E_{BX1}$, is the radiant power emitted by the surface of the chopper blade that reaches the detector. The second term, $C_{BX2}E_{BX2}$, is the stray radiation reflected off the surface of the chopper blade that reaches the detector.

We write the source terms in the form

$$E_x = \varepsilon'_x \sigma T^4_x \Omega \tag{4}$$

where ε'_x is the DTE of source X, σ is the Stefan–Boltzmann constant, T_x is the absolute temperature of the surface of source X, and Ω is a geometrical factor.

The final expression for the DTE ε'_{s} is [10, 11]

$$\varepsilon_{\rm S}' = \varepsilon_{\rm R}' \left(\frac{C_{\rm R}}{C_{\rm S}}\right) \left(\frac{T_{\rm R}}{T_{\rm S}}\right)^4 \left(\frac{V_{\rm S} - V_{\rm W}}{V_{\rm R} - V_{\rm W}}\right) M \tag{5}$$

Equation (5) can be simplified for the experiments described here. The multiplier M consists of a set of ratios of radiant power as they appear in Eqs. (2)–(4). Under our experimental conditions, M is nearly 1 and can be ignored. The C_s factor relates to spectral quantities for the sample S and C_R for the reference R. In general, the C_R and C_s factors are rather difficult to calculate. However, in our experiment, S and R are both chosen to be high-emissivity materials (S, glass/ R, black paint on glass), and it turns out that $(C_R/C_s) \approx 1$.

Finally, as far as temperature is concerned, S and R have practically the same temperature, since they are one piece of the same material with half of the surface painted black.

With these approximations, the following simplified equation is obtained for the calculation of DTE for the experiment described here:

$$\varepsilon_{\rm S}' = \varepsilon_{\rm R}' \left(\frac{V_{\rm S} - V_{\rm W}}{V_{\rm R} - V_{\rm W}} \right) \tag{6}$$

where $\varepsilon'_{\rm S}$ is the (unknown) DTE of the sample, $\varepsilon'_{\rm R}$ is the (known) DTE of the reference, and $V_{\rm S}$, $V_{\rm R}$, and $V_{\rm W}$ are the measured thermopile voltages from the sample S, the reference R, and the wall W, respectively.

4. RESULTS AND DISCUSSION

The graphical representation of our results, for a self-referenced sample of float glass, is shown in Fig. 3. The x axis represents the angle of emission θ (with respect to the normal to the sample surface). The y axis represents the normalized DTE of sample (the normalization has been done by dividing all values with the first value at 10°).

The data points with the error bars depict the normalized values, i.e., the ratio $\varepsilon'_{\rm s}(\theta)/\varepsilon'_{\rm s}$ (10°), obtained from Eq. (6) by measuring $V_{\rm s}(\theta)$, $V_{\rm s}$ (10°), and $V_{\rm w}$. The dashed curve shows the normalized data derived from reflectivity data for the float glass, obtained at NPL [16], and they are used here for comparison. Table I shows the corresponding values for the specific angles of emission. Comparisons with NPL data show that our apparatus is capable of measuring the angular dependence of DTE (i.e., the shape of DTE with angle) with good accuracy. It is seen that the "shape" of DTE of the sample can be obtained without knowing the emissivity of the reference, in fact, without using any separate reference materials. The importance of knowing the shape of DTE is that it allows one (a) to test the accuracy of a new apparatus (radiometer) and (b) to calculate the fraction of the radiation emitted within a range of angles compared with the total radiation emitted in the range from 0 to 90°.



Fig. 3. Normalized directional total emissivity of float glass—comparison of this work with NPL data [16].

For absolute measurements of DTE, a reference is required. From data in the literature for mat enamel black paints [21], a value of 0.93 ± 0.01 (at 1 SD) was derived for the DTE (near normal) of the reference used in the experiment (mat enamel black paint on glass substrate). In Fig. 4, the results of this work for the DTE of float glass are compared with three types of data in the literature: (a) measured values for the DTE

| | DTE (normalized) | |
|------------------|----------------------|----------------------------|
| Angle (θ) | This work (measured) | NPL data [16] (calculated) |
| 10 | 1.000 ± 0.033 | 1.000 |
| 20 | 0.996 ± 0.031 | 0.999 |
| 30 | 0.991 ± 0.033 | 0.997 |
| 40 | 0.989 ± 0.029 | 0.990 |
| 50 | 0.970 ± 0.033 | 0.972 |
| 60 | 0.906 ± 0.030 | 0.930 |
| 70 | 0.847 ± 0.031 | 0.836 |

Table I. Comparisons with Literature Data



Fig. 4. Absolute values of directional total emissivity of float glass: (1) measured DTE (this work; (2) measured DTE [10, 11]; (3) measured DTE [4]; (4) calculated DTE from spectral reflectivity data [16]; (5, 6, 7) calculated DTE from values of n and k in Refs. 8, 18, and 17, respectively.

of glass [4, 10, 11]; (b) published values of optical constants n and k [8, 17, 18] [from n and k the directional spectral emissivity was obtained and then it was integrated from 5 to 20 μ m (which is the spectral range of our detector) to obtain DTE]; and (c) measured values for the float glass of spectral reflectivity at 10° at the NPL [16]. Kramers–Kronig analysis was performed on these data to obtain n and k. To obtain DTE the procedure described in b was followed.

As shown in Fig. 3 and Table I, the normalized values of DTE obtained in this work compare well with those derived from the data from NPL. The absolute values of DTE, shown in Fig. 4, generally agree with previously measured values in the literature, although it may be argued that a small systematic difference exists. This may be due to a systematic uncertainty in the absolute value of the DTE of the reference material used. However, other effects may also exist but are outside the scope of this work. For example, the effect of different types of float glass being used by different investigators may be important. This may be suggested by both the uppermost curve in Fig. 4 (No. 4, obtained from indirect measurements, where a reference is not used) and the lowest curve (No. 7, obtained

from the most recent values, in the literature, of the optical constants n and k, for pure SiO₂).

5. CONCLUSIONS

In the present work, a novel experimental setup for measuring the DTE of opaque materials at near-ambient temperatures is presented. Starting from first principles [22] and taking into consideration recent studies in the field of thermal radiometry [10–12], a novel apparatus was designed and constructed to operate without cooling. The material studied here was float glass at 323 K (50°C), and the DTE was measured over a range of angles from 10 to 70°, with respect to the normal from the sample surface. The measurements were achieved by using phase-sensitive signal recovery means and an advanced solid-state thermal detector (thermopile). With this setup, two types of measurements were performed. Relative values of DTE were obtained (angular shape) to test the capability of the apparatus. The results agree with data obtained from the NPL [16] within our experimental uncertainty. As can be seen from the uncertainty analysis in the Appendix, the dominant uncertainty is due to the very weak voltage signals. Next, absolute values of DTE for float glass were obtained. The reference used was mat black-enamel paint on a float glass substrate. The value of DTE for the reference was taken to be 0.93 + 0.01 near normal as derived from data on black paints [21]. The results (absolute values) of the DTE for the float glass were compared with results in the literature [4, 8, 10, 11, 16-18]. In conclusion, both relative and absolute values presented here show that it is possible to obtain reliable measurements of DTE near ambient temperatures (323 K) without using cooling.

APPENDIX : UNCERTAINTY ANALYSIS

From Eq. (6) it can be seen that the uncertainties in measuring the voltages $V_{\rm S}$, $V_{\rm R}$, and $V_{\rm W}$ introduce an uncertainty in the emissivity $\varepsilon'_{\rm S}$. Also, the uncertainty in $\varepsilon'_{\rm R}$ affects $\varepsilon'_{\rm S}$. Because the voltage signals are of the order of $\approx 10^{-8}$ V, 10 measurements are taken for each $V_{\rm S}$, $V_{\rm R}$, $V_{\rm W}$, at each angle of emission. The mean value for each voltage is given by

$$\bar{V}_{\rm S} = \frac{1}{N} \sum_{i}^{N=10} V_{\rm S_i}, \qquad \bar{V}_{\rm R} = \frac{1}{N} \sum_{i}^{N=10} V_{\rm R_i}, \qquad \bar{V}_{\rm W} = \frac{1}{N} \sum_{i}^{N=10} V_{\rm W_i}$$

The standard deviation for each voltage (of a single observation about the sample mean) is given by

$$\begin{split} \delta V_{\rm S} &= \sqrt{\frac{1}{(N-1)} \sum_{i}^{N=10} (V_{\rm S_i} - \bar{V}_{\rm S})^2}, \qquad \delta V_{\rm R} = \sqrt{\frac{1}{(N-1)} \sum_{i}^{N=10} (V_{\rm R_i} - \bar{V}_{\rm R})^2}, \\ \delta V_{\rm W} &= \sqrt{\frac{1}{(N-1)} \sum_{i}^{N=10} (V_{\rm W_i} - \bar{V}_{\rm W})^2} \end{split}$$

Therefore, the mean value of DTE is given by

$$\bar{\varepsilon}_{\rm S}' = \varepsilon_{\rm R}' \left(\frac{\bar{V}_{\rm S} - \bar{V}_{\rm W}}{\bar{V}_{\rm R} - \bar{V}_{\rm W}} \right)$$

The total uncertainty (at 95% level of confidence) is given by the law of superposition of component uncertainties [23]:

$$\delta \bar{\varepsilon}_{\rm S}' = 2 \sqrt{\left(\frac{\partial \varepsilon_{\rm S}'}{\partial V_{\rm S}} \, \delta \bar{V}_{\rm S}\right)^2 + \left(\frac{\partial \varepsilon_{\rm S}'}{\partial V_{\rm R}} \, \delta \bar{V}_{\rm R}\right)^2 + \left(\frac{\partial \varepsilon_{\rm S}'}{\partial V_{\rm W}} \, \delta \bar{V}_{\rm W}\right)^2 + \left(\frac{\partial \varepsilon_{\rm S}'}{\partial \varepsilon_{\rm R}'} \, \delta \varepsilon_{\rm R}'\right)^2}$$

where

$$\delta \overline{V}_{\rm S} = \frac{\delta V_{\rm S}}{\sqrt{N}}, \quad \delta \overline{V}_{\rm R} = \frac{\delta V_{\rm R}}{\sqrt{N}}, \quad \text{and} \quad \delta \overline{V}_{\rm W} = \frac{\delta V_{\rm W}}{\sqrt{N}}$$

are the standard uncertainties of the mean values \overline{V}_{S} , \overline{V}_{R} , and \overline{V}_{W} , and $\delta \varepsilon'_{R}$ is the standard uncertainty (I-SD level) in the emissivity of the reference.

For relative measurements of DTE a reference is not required and $\delta \varepsilon'_{R}$ is ignored.

The results presented in this work are of the form

$$\bar{\varepsilon}_{\rm s}' \pm \delta \bar{\varepsilon}_{\rm s}'$$

ACKNOWLEDGMENTS

The financial assistance of the State Scholarships Foundation of Greece to one of us (N.K.) is gratefully acknowledged. The valuable help and the technical support of Mr. W. Schmidt and Mrs. C. Berkner at EG&G Heimann Optoelectronics in Germany for the thermopile detector are also acknowledged.

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