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Emissivity measurements by using sine wave thermal modulation. Radiometric and calorimetric approaches.

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Abstract. This paper presents a new technique for measuring total emissivities at room temperature involving a multifrequency sine wave thermal modulation. The infrared output signal as well as the sample's front side temperature are measured. Experimental data and a one dimensional model are compared, yielding identified values of diffusivity a , Biot number Bi and E , the latter being defined as the ratio of the sample to reference emissivity. Using Bi , emissivity can be also reached, so emissivity of a thermally thick sample can be reached through two different approaches. Results have been obtained for a 5mm thick sample of PVC, and it is shown that they are well concordant with integrated directional measurements.

1 Introduction

When modeling heat transfer the total radiative properties of surfaces have to be known. These properties are highly dependent on the surface conditions. They can not be found easily in the literature, and must be measured in working conditions. Emission measurement techniques may be separated into two groups : radiometric measurements and calorimetric measurements (Sacadura, 1990). Steady state radiometric techniques are not convenient for low temperature emissivity measurements, because of the sample reflected flux. Thermal periodic techniques have been used mainly for measuring thermal diffusivity (Cowan, 1961), (Ranchy et al., 1992), however a periodic radiometric method has been developed for emissivity measurements at room temperature (Matteï et al. 1994). This method is limited to thin sample with known surface temperature. The knowledge of surface temperature is needed for transient or calorimetric emission measurements too. This paper presents a periodic technique for measuring total emissivities of thick materials at low temperature, without emissive surface temperature measurement. This technique allows to reach total emissivity through two different approaches : radiometry and calorimetry.

2 Thermal behavior of a modulated strip shaped sample

Consider a strip shaped sample (Fig. 1) the front side temperature of which, T_{FS} , is modulated :

$$T_{FS}(t) = \bar{T} + T_0 \cos(2\pi f_0 t + \varphi) \quad (1)$$

We define the thermal transfer function of the strip $\tilde{H}(f_0)$ by the relation :

$$\tilde{H}(f_0) = \frac{\tilde{T}_{BS}(f_0)}{\tilde{T}_{FS}(f_0)} \quad (2)$$

where $\tilde{T}_{BS}(f_0)$ and $\tilde{T}_{FS}(f_0)$ are the complex temperature of respectively the back side and the front side of the sample.

\tilde{H} is a complex number, the modulus of which characterizes the temperature attenuation, and the phase, the temperature phase shift between the two sides of the strip. When $\tilde{H}(f)$ is equal to one then we say that the sample is thermally thin.

For a modulated temperature one dimensional conduction model, the heat balance equation can be written :

$$\frac{\partial^2 \tilde{T}}{\partial x^2} - \frac{j2\pi f}{a} \tilde{T} = 0 \quad (3)$$

with boundary conditions :

$$\tilde{T}|_{x=e} = |\tilde{T}_{FS}|e^{jq}, \quad \left. \frac{d\tilde{T}}{dx} \right|_{x=0} = \frac{h}{k} \tilde{T}|_{x=0} \quad (4)$$

leading to :

$$\tilde{H} = \frac{\frac{q \cdot e}{Bi}}{\text{sh}(q \cdot e) + \frac{q \cdot e}{Bi} \text{ch}(q \cdot e)} \quad (5)$$

e is the sample thickness. Bi is the Biot number given by $Bi = h \cdot e / k$. h is the heat transfer coefficient on the sample back side which is the sum of a convective coefficient and a radiative coefficient. q is given by $q = \sqrt{j2\pi f/a}$, where a is the sample diffusivity.

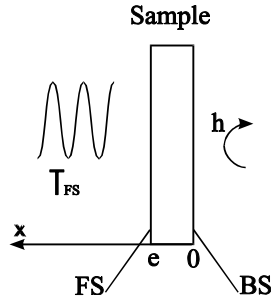


Figure 1. The conduction model.

3 Application to emissivity measurements

3.1 Principle of the method

Consider a total infrared detector facing the back side of the sample the temperature of which is in the form given by Eq. 1. The measured radiative flux is the sum of an emitted flux and a reflected one. At temperature modulation frequency f_0 , the reflected flux is not modulated. If $\tilde{K}(f)$ is the transfer function of the IR detection system (including geometrical parameters), the IR output signal f_0 component is given by :

$$\tilde{U}(f_0) = \tilde{K}(f_0) \cdot \epsilon_s \cdot \frac{\sigma \tilde{T}_{BS}^4(f_0)}{\pi} \quad (6)$$

ϵ_s is the sample back side emissivity. Its geometrical properties depend on the IR detection system optical path.

We note $\tilde{R}(f_0)$ the ratio :

$$\tilde{R}(f_0) = \frac{\tilde{U}(f_0)}{\tilde{T}_{FS}^4(f_0)} \quad (7)$$

If temperature amplitudes are small, $\tilde{T}_{BS}^4(f_0)$ can be linearised, and if front side and back side mean temperature are equal, we can write :

$$\tilde{R}(f_0) = \tilde{K}(f_0) \cdot \frac{\epsilon_s \sigma}{\pi} \cdot \tilde{H}_s(f_0) \quad (8)$$

where $\tilde{H}_s(f_0)$ is the thermal transfer function defined above.

Two measurements of $\tilde{R}(f)$ are carried out for several frequencies : one on the sample and the other one on a thin reference. For each modulation frequency f_i we obtain :

$$\frac{\tilde{R}_s(f_i)}{\tilde{R}_r(f_i)} = \frac{\epsilon_s}{\epsilon_r} \cdot \tilde{H}_s(f_i) \quad (9)$$

or

$$\tilde{R}_{s/r} = E \cdot \tilde{H}_s(f_i) \quad (10)$$

where $E = \epsilon_s / \epsilon_r$.

E , a , and Bi can be identified by fitting the measured values with the model (Siroux et al., 1994).

It is then possible to reach the sample emissivity through two different approaches. The radiometric approach consists in calculating ϵ_s , via E . The calorimetric one consists in calculating via Bi the hemispherical emissivity noted ϵ_s^h of the same surface.

In fact, when the sample is in a vacuum chamber, and surrounded by a black surface dimensions of which are greater than those of the sample, the heat transfer coefficient can be written :

$$h = 4\epsilon_s^h \sigma \bar{T}^3 \quad (11)$$

where \bar{T} is the mean temperature of the sample.

So we can obtain ϵ_s^h using the below formula :

$$\epsilon_s^h = \frac{Bi \cdot k}{4\sigma \bar{T}^3 e} \quad (12)$$

The radiometric approach requires an emissivity reference ϵ_r . On the other hand, the calorimetric approach requires the knowing of the ratio k/e .

3.2 Experimental set-up

The apparatus (Fig. 2) is composed of a vacuum chamber (1), a pumping system (2), sample holders (3), an infrared detector (4) and a data acquisition system (5). A turbomolecular pump system permits to reach pressure near 7×10^{-5} mbar.

Each sample (6) is stock on a modulated sample holder. The latter is composed of a thermoelectric device (3b) squeezed between two metallic plates (3a and 3c). A thermocouple (14) is embedded in the metallic plate which holds the sample. Since this plate is thermally thin, readings are assumed to be representative of the front side sample temperature. A cooling water system sinks the heat created by the thermoelectric devices Joule's effect.

The infrared detector is a thermopile with a KRS5 window. The detector is motorized (12) and can be successively placed in front of the different samples.

A PC data based acquisition system controls the excitation voltage of the thermoelectric device, measures sample temperature $T(t)$ as well as the infrared detection system output $U(t)$. The data are first 100 Hz sampled, amplified and analogically low pass filtered ($f_c = 5$ Hz), then they are numerically filtered using a Hanning low pass filter ($f_c = 0.2$ Hz) and 0.4 Hz sampled again.

The excitation voltage is in the form :

$$V(t) = V_{\text{mean}} + \sum_{i=1}^5 V_i \cdot \cos(2\pi 2^{i-1} f_0 t) \quad (13)$$

where V_{mean} is the mean excitation voltage, f_0 is equal to 0.78 mHz. V_i is chosen in order that the ratio signal/noise is the same over all the spectrum. The number and the amplitude of each modulation component is chosen regarding the signal/noise ratio, the total peak to

peak voltage and the available power. The amplitude of the front side sample temperature is around 12°C peak to peak.

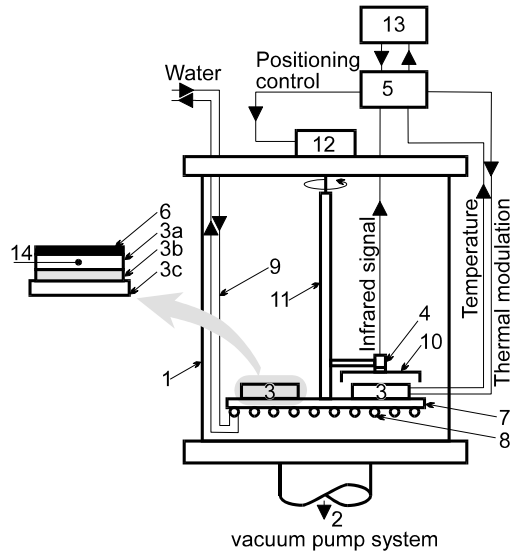


Figure 2. Experimental set-up : 1-vacuum chamber, 2-vacuum pump system, 3-sample holder (3a, 3c-metallic plate, 3b-thermoelectric device), 4-infrared detector, 5-data acquisition system, 6-sample, 7-mounting sample holder, 8-cooling serpentine, 9-cooling tube, 10-detector holder, 11-sectional iron, 12-motor, 13-computer, 14-thermocouple.

The two measured signals $U(t)$ and $T(t)$ are composed of five significant components, a low frequency component (slow variations of environmental temperatures) and a wide band component essentially due to the background noise of the detector. First, $U(t)$ and $T^4(t)$, computed from $T(t)$, are high filtered in order to remove the trend. Then they are processed using a FFT (Fast Fourier Transform) leading to $\tilde{U}(f)$ and $\tilde{T}^4(f)$. The length of record L (around 85 minutes) is chosen so that each component frequency is a multiple of the spectral resolution $\Delta f = 1/L$. Figure 3 shows examples of temperature and infrared signal amplitude spectra.

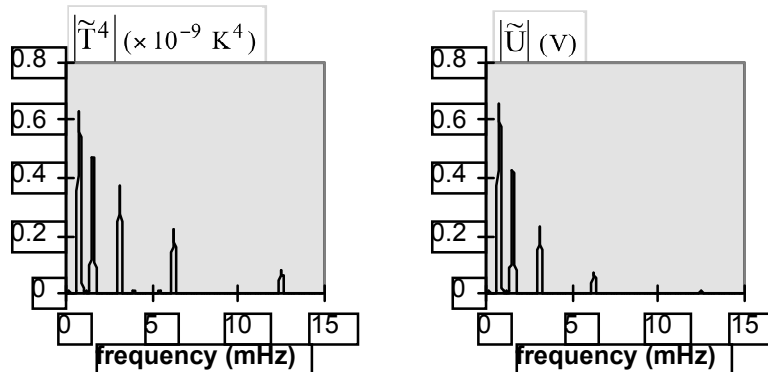


Figure 3. Amplitude spectra for the temperature and for the infrared signal

For the above configuration (Fig. 2), for a sample the emissivity of which is equal to one, and for a mean temperature equal to 300 K, the minimum theoretical detectable signal amplitude of an infrared detector depends on the detector detectivity and the length of record. It can be shown that (Matteï et al., 1996) for the used thermopile and for $L=85$ minutes, this value is around 10^{-6} K. However, other noises than detector noise influence to this value, the main one being the D/A board noise which is around 0.05 % of the full scale.

The statistical error of each component of $\tilde{U}(f)$ and $\tilde{T}^4(f)$ is on the order of 0.1 % for the modulus and 0.05 degrees for the phase. These accuracies have been obtained by making measurements of repeatedly and calculating the standard deviation of the resulting distribution of modulus and phase.

If the sample thickness is not very smaller than the lateral dimensions, then the use of a one dimensional model is questionable. In fact, lateral radiant losses influence on back side temperature and identified parameters are then false.

In order to evaluate this influence, a three dimensional numerical model has been developed using finite differences. According to the heat balance equation, the complex temperature \tilde{T}_i of each elementary volume is linked with the complex temperature of the six contiguous elementary volumes :

$$\tilde{T}_i = \frac{\frac{6}{jC_i} \frac{\tilde{T}_j}{R_j}}{\frac{6}{jC_i} \frac{1}{R_j} + \frac{1}{R_j} \tilde{T}_j} \quad (14)$$

Where R_j is a thermal resistance and C_i a thermal capacity. Since the problem is symmetric, temperatures are computed for only one quarter of the sample (4608 elements have been used), using an iterative method. The infrared signal of a detector facing the surface sample is then calculated and the Biot number and the diffusivity are identified using a one dimensional analytic model. These values are compared with those obtained with a model without lateral losses, the founded differences are 10 % for the Biot number and 0.1% for the diffusivity. So we can note that the identified value of the Biot number is very dependent on the lateral radiant losses. In order to reduce these losses, reflective surfaces have been closely placed in front of the four sides of the sample.

3.3 Results and Discussion

The parameters are identified using the minimization between measured data and calculated ones. In order to take into account the modulus and the phase of data the chosen estimator is the sum of two estimators :

$$C = C_\rho + C_\varphi \quad (15)$$

$$\text{with } C = \frac{\sum_{i=1}^k \frac{1}{(\hat{\varphi}_i - \varphi_i^*)^2}}{k}, \quad C = \frac{\sum_{i=1}^k \frac{1}{(\hat{\varphi}_i - \varphi_i^*)^2}}{k}$$

where $\hat{\varphi}_i$ and φ_i^* are respectively the modulus and the phase of the data, k is the number of data, $*$ is relating to the measured data, $\hat{}$ is relating to the calculated data and φ_i^* is defined

$$\text{by : } \varphi_i^* = \frac{1}{k} \sum_{i=1}^k \varphi_i^*$$

Measurements have been carried out on a PVC sample (44mm x 44mm x 5mm) coated with a paint. The diffusivity and the emissivity of the coating are known. The same paint coated on the sample holder is used as the reference.

Figure 4 (a and b) shows modulus and phase of five experimental data $\tilde{R}_{s/r}(f)$ and calculated values of the product $E \cdot \tilde{H}_s(f)$. The E, a, Bi values shown on this figure are respectively the mean of four values identified by using four trial of measurements of $\tilde{R}_{s/r}$. The statistical errors are estimated via the fluctuations of the identified values. We can first note that we obtain the expected results for E, the sample being coated with the same paint than the reference.

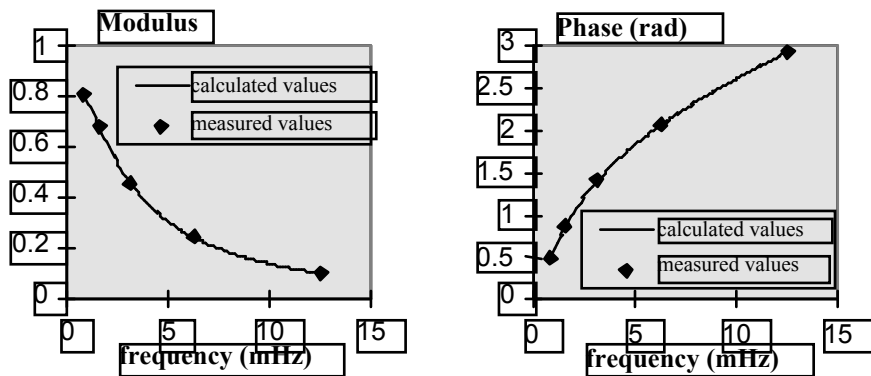


Figure 4. Modulus and phase of measured values $\tilde{R}_{s/r}(f)$ and calculated values $E \cdot \tilde{H}_s(f)$. Identified parameters are : $a = 0.113 \text{ mm}^2\text{s}^{-1}$, $Bi = 0.160$, $E = 1.006$.

In order to verify the calorimetric emissivity ϵ_s^h we have carried out directional emissivity measurements. Assuming that the normal emissivity is 0.97, calculating calorimetric emissivity using directional emissivity integration yields to a close result $\epsilon_s^h = 0.88$.

The above results show that it is possible to access to emissivity of thick materials at low temperature via radiometric and calorimetric approaches. This method must be applied now to different sorts of coatings and materials.

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