On the sensitivity of FPPE – TWRC method in thermal effusivity investigations of solids

Dorin Dadarlat, Mihaela Streza, Mircea N Pop and Valer Tosa
National Institute for Research and Development of Isotopic and Molecular Technologies, 65-103 Donath, 400293 Cluj-Napoca, Romania
E-mail: nicolae.dadarlat@itim-cj.ro

Abstract. The front detection configuration (FPPE) together with the thermal-wave-resonator-cavity (TWRC) method was used for direct measurement of the thermal effusivity of solid materials inserted as backings in the FPPE detection cell. It was demonstrated that the normalized phase of the FPPE signal has an oscillating dependence as a function of sample’s thickness. The paper presents experimental results on solid materials, with various values of thermal effusivity (Cu, brass, steel, bakelite, wood). A study of the sensitivity of the technique for different liquid/backing effusivity ratios is performed. The highest sensitivity was obtained when investigating solids with values of thermal effusivity not far from the effusivity of the liquid layer of the detection cell.

1. Introduction
During the last decades, the photopyroelectric (PPE) calorimetry developed many ways in order to obtain the values of the thermal parameters of condensed matter samples [1, 2]. In principle, one may combine detection configurations (back or front), sources of information (PPE amplitude or phase) and scanning parameters (chopping frequency or sample’s thickness), in order to obtain one dynamic thermal parameter (usually, thermal diffusivity or effusivity) [3, 4].

It is well known that, when investigating solid samples, it is not easy to obtain accurate quantitative results, due to the influence of the coupling (sample-sensor) fluid. The PPE calorimetry is a contact technique and the coupling fluid, always necessary when investigating solids, leads to uncontrolled errors.

If we focus our attention on the front photopyroelectric (FPPE) configuration, it was largely used in the past to obtain thermal parameters of liquid and solid samples [1, 2, 5, 6]. Mandelis and co-workers [1, 7, 8] proposed, during the last decade, the so called thermal-wave resonator cavity (TWRC) method, which, in the back (BPPE) configuration, showed to be very accurate for investigating thermal properties (especially thermal diffusivity) of liquids [5, 6, 9, 10].

Recently, we proposed an alternative technique that combines the FPPE configuration with the TWRC method [11]; in this configuration, by monitoring the thickness of a fluid sample, the thermal effusivity of a solid material used as backing in the detection cell can be measured. In such a way the coupling fluid is not anymore a disturbing factor, but its properties can be used in order to obtain the value of the thermal effusivity of a solid.

In this paper we perform an investigation on the sensitivity of the coupled FPPE-TWRC technique in measuring the thermal effusivity of solids, as a function of liquid/backing effusivity ratio.
2. Theory

In the FPPE configuration, the radiation impinges on the front surface of the pyroelectric sensor, and the sample, in good thermal contact with its rear side, acts as a heat sink. The detection cell is composed by 4 layers: air, (directly irradiated) sensor, liquid sample and solid backing.

In the approximation of the one-directional heat propagation, and thermally thin limit for the sensor \( \exp(\pm \sigma L_p) = 1 \pm \sigma L_p \), the normalized complex PPE signal is given by [11]:

\[
V_n = \frac{\sigma_p L_p + b_p}{\sigma_p L_p + b_p} \left( \frac{1 + R_{sb} \exp(-2\sigma_s L_s)}{1 - R_{sb} \exp(-2\sigma_s L_s)} \right)
\]

where

\[
S = \exp(\sigma L_s), \quad P = \exp(\sigma L_p), \quad \sigma_j = (1 + i) \mu_j, \quad \mu = (2\alpha/\omega)^{1/2}, \quad b_{ij} = e_i/e_j
\]

In equations (1) and (2), \( \omega \) is the angular chopping frequency of radiation, \( \sigma \) and \( \alpha \) are the complex thermal diffusion coefficient and the reciprocal of the thermal diffusion length \( (\alpha = 1/\mu) \), respectively. Symbols \( p \), \( s \) and \( b \) refer to pyroelectric sensor, liquid sample and backing material, respectively. \( R_{ij} = (b_{ij} - 1)/(b_{ij} + 1) \) represents the reflection coefficient of the thermal wave at the \( ij \) interface.

Equation (1) was obtained after a double normalization: first with the signal obtained with empty sensor directly irradiated and then, with the signal obtained with very thick liquid sample \( (\exp(-\sigma L_s) = 0) \). Equation (1) indicates that the normalized PPE signal depends on the thermal effusivity of the backing material and a thickness scan of the amplitude and/or phase of the PPE signal (at constant chopping frequency) can lead to its direct measurement.

Mathematical simulations of the phase of the FPPE signal as a function of liquid sample’s thickness are displayed in figure 1.

![Figure 1. Mathematical simulations (equation (1)) of the behaviour of the normalized PPE phase as a function of the absolute thickness of the sample.](image)

3. Experimental results

The experimental set-up was largely presented elsewhere [4, 6, 11]. We will give here only some particular details of the experiment. The modulation frequency was 1 Hz, the thickness variation step was 0.9\( \mu \)m and ethylene glycol was used as liquid layer. Solid samples (copper, glass, bakelite, wood), with values of thermal effusivity covering a large range (350-37000 Ws\(^{1/2}\)/m\(^2\)K) were inserted in the detection cell as backing materials. The values of the thermal effusivity were obtained by optimizing the fit performed with equation (1), with the effusivity of the backing as fitting parameter.
The results obtained for the behaviour of the phase of the FPPE signal as a function of liquid’s thickness, together with the values obtained for the thermal effusivity are displayed in figure 2.

Figure 2. Normalized PPE phase as a function of sample’s thickness for four solid backing materials with different values of thermal effusivity: Cu, glass, bakelite, wood.

Figures 3 and 4 represent contour maps of the precision of the fits for two materials with very different values of thermal effusivity.

Figure 3. Contour map of the precision of the fit performed with equation (1) on the experimental data obtained on Cu backing. X-axis represents the error in the measurement of the absolute liquid’s thickness. Conclusion: good localization of the position of the backing and rather low precision (relative error ± 30%) in the value of thermal effusivity.

Figure 4. Contour map of the precision of the fit performed with equation (1) on the experimental data obtained on bakelite backing. X-axis represents the error in the measurement of the absolute liquid’s thickness. Conclusion: bad localization of the position of the backing and rather good precision (relative error ± 5%) in the value of thermal effusivity.

4. Concluding remarks
The front PPE configuration, coupled with the TWRC method, represents a suitable alternative for the direct measurement of thermal effusivity of solid materials. The value of the thermal effusivity of the solid material used as backing in the detection cell was obtained by performing a sample’s (liquid) thickness scan of the phase of the complex FPPE signal. The accuracy of the measurement (compared with results reported in the literature) is dependent on the signal-to-noise ratio (about 1000 in our experiment), on the thickness variation step (0.9 µm), but also on the ratio of the liquid/solid
effusivities. As revealed in figures 3 and 4, the best accuracy was obtained when the $e_s/e_b \approx 1$ and, at this stage of investigation, it is of about 90%. For backings with large values of thermal effusivity (metals), the method shows accuracies of only about 50%-70%, due to the saturation of the signal for thermally thin regime of the liquid (see equation (1)).

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