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# On the photopyroelectric investigation of thermal effusivity of solids. Amplitude vs. phase in the FPPE-TWRC configuration

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The thermal effusivity of two solids (steel and epoxy) was measured by using a combined FPPE-TWRC technique. The suitability of using the amplitude or phase of the PPE signal, as source of information, for backing materials with different values of thermal effusivity was analyzed. The values of thermal effusivity obtained for the investigated materials when using the amplitude and phase are in good agreement. Concerning the accuracy of the data processing, the amplitude seems to be more suitable for good thermal conductors (steel), while the phase offers a better accuracy in the case of low thermal conductors (epoxy).

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## 1. Introduction

During last decades the photopyroelectric (PPE) calorimetry developed many ways in order to obtain values of thermal parameters of condensed matter samples [1, 2]. Two detection configurations ("back" – BPPE, and "front" – FPPE) and two sources of information (amplitude and phase of the complex PPE signal) were mainly combined in order to obtain accurate values of static and dynamic thermal parameters [3]. Technically speaking, some methods are based on the measurement of single values; other alternatives make use of scanning procedures. The second type of investigations is proved to be more precise [4, 6]. When liquid materials are involved in investigations, there are two parameters susceptible to be scanned: the chopping frequency of radiation and the liquid's thickness. In the second case one develops the so called "thermal-wave resonator cavity" (TWRC) method [7-9]. This technique showed recently an increased interest especially due to the high accuracy (relative error around ±1%) of the results obtained for the room temperature values of thermal diffusivity and effusivity of liquids [10-13].

Independent on the detection configuration, the PPE technique is a contact one and, consequently, the thermal contact between the different layers of the detection cell is responsible for the accuracy of the results. When investigating solids, the most important thermal contact is between the pyroelectric sensor and the solid sample. Recently, we proposed a technique in which the FPPE detection configuration is combined with the TWRC method in order to measure the thermal effusivity of a solid material, inserted as backing in the detection cell [14]. The method uses the scan of the FPPE phase as a function of a liquid's thickness, acting as coupling sensor/backing fluid. The value of backing's thermal effusivity resulted from the phase of the FPPE signal and the method proved to be suitable especially when investigating solids with values of thermal effusivity close to the effusivity of the liquid layer [14-15].

In this communication we analyze the suitability of using the amplitude or the phase of the PPE signal as source of information, when backing materials have different values of thermal effusivity.

## 2. Theory and mathematical simulations

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. In the approximation of the one-directional heat propagation and thermally thin limit for the sensor ($exp(\pm\sigma_p L_p)=1\pm\sigma_p L_p$), the normalized complex PPE signal is given by [13, 14]:

$$V = \frac{\left[S(b_{bs}+1)-S^{-1}(b_{bs}-1)\right](\sigma_p L_p)}{(\sigma_p L_p)\left[S(b_{bs}+1)-S^{-1}(b_{bs}-1)\right]+b_{sp}\left[S(b_{bs}+1)+S^{-1}(b_{bs}-1)\right]} \quad (1)$$

where

$$S=exp(\sigma_s L_s),\ \sigma_j=(1+i)a_j;\ \mu=(2\alpha/\omega)^{1/2},\ b_{ij}=e_i/e_j \quad (2)$$

In Eqs. (1)-(2), $\omega$ is the angular chopping frequency of radiation, $\sigma$ and $a$ are the complex thermal diffusion coefficient and the reciprocal of the thermal diffusion length ($a = 1/\mu$), respectively. Symbols "*p*", "*s*" and "*b*" refer to pyroelectric sensor, liquid sample (acting as a coupling fluid in our case) and backing material, respectively. The normalization of Eq. (1) was performed with the signal obtained with the sensor standing alone in air.

In order to compare results obtained with different liquid samples and backing materials, it is useful to perform a second normalization with the signal obtained with very thick liquid sample ($exp(-\sigma_s L_s) = 0$). The result is given by:

$$V_n = \frac{\sigma_p L_p + b_{sp}}{\left(\sigma_p L_p\right) + b_{sp}\left[\dfrac{1 + R_{bs}\exp(-2\sigma_s L_s)}{1 - R_{bs}\exp(-2\sigma_s L_s)}\right]} \qquad (3)$$

where $R_{ij}=(b_{ij}-1)/(b_{ij}+1)$ represents the reflection coefficient of the thermal wave at the "ij" interface.

Mathematical simulation of the normalized amplitude and phase of the PPE signal, respectively, for different $e_b/e_s$ ratios, is presented in Figs. 1 and 2.

As a conclusion to this theoretical section, the normalized PPE signal (Eq.(3)) depends on the thermal effusivity of the backing material; a thickness scan of the amplitude and/or phase of the PPE signal (at constant chopping frequency) will lead to its direct measurement.

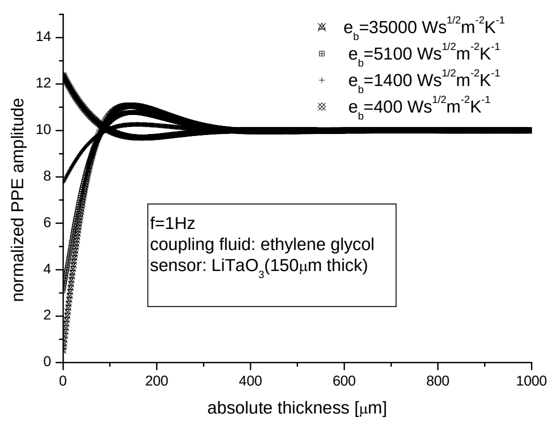


*Fig. 1. Mathematical simulations of the behavior of the amplitude of the FPPE signal, as a function of coupling fluid's thickness for backing solids with different values of thermal effusivity.*

## 3. Experimental results

The experimental set-up and the detection cell in the FPPE-TWRC configuration were largely described before [3, 12-14]. Only some details will be presented here. The pyroelectric sensor, was a 150 μm thick $LiTaO_3$ single crystal ($e_p$= 3.66 × $10^3$ $Ws^{1/2}m^{-2}K^{-1}$; $\alpha_p$= 1.36 × $10^{-6}$ $m^2s^{-1}$), provided with Cr-Au electrodes on both faces. The coupling fluid was water ($e_s$= 16.0 × $10^2$ $Ws^{1/2}m^{-2}K^{-1}$; $\alpha_s$= 14.6 × $10^{-8}$ $m^2s^{-1}$). 1Hz chopping frequency of the incident radiation assures the thermally thin regime for the pyroelectric sensor. The liquid's thickness scan was performed with a step of 30 nm, with data acquisition at each 0.9 μm. Two solid materials were selected as backings: a good (steel) and a bad (epoxy) thermal conductor, respectively.

The results obtained for the amplitude and phase of the FPPE signal are displayed in Figs. 3 and 4. A good agreement with the mathematical simulations was obtained.

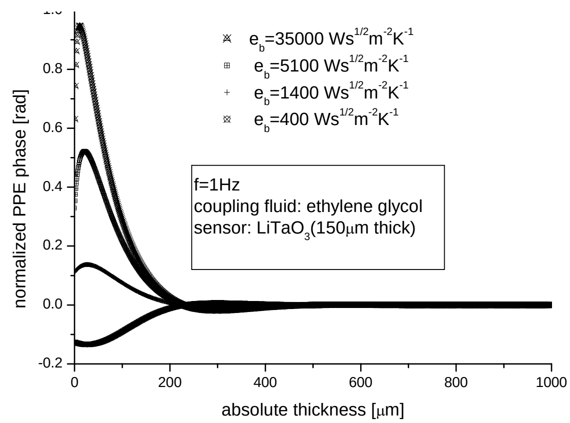


*Fig. 2. Mathematical simulations of the behavior of the phase of the FPPE signal, as a function of coupling fluid's thickness for backing solids with different values of thermal effusivity.*

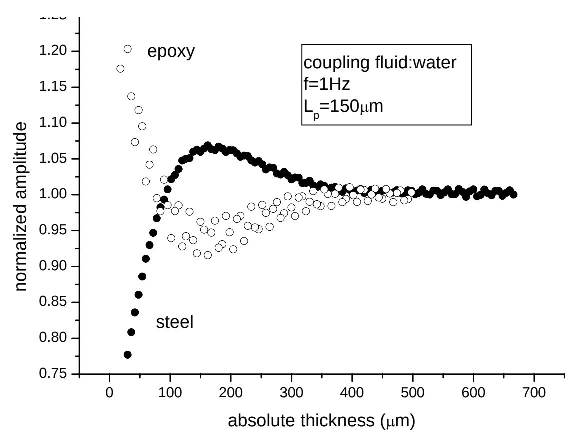


*Fig. 3. Thickness scan of the normalized amplitude of the FPPE signal for detection cells containing water as a coupling fluid and steel and epoxy respectively, as backing materials.*

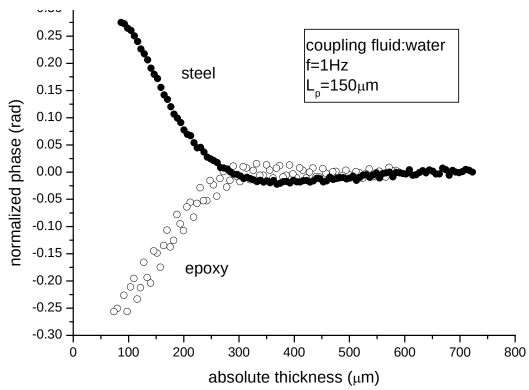


*Fig. 4. Thickness scan of the normalized phase of the FPPE signal for detection cells containing water as a coupling fluid and steel and epoxy respectively, as backing materials.*

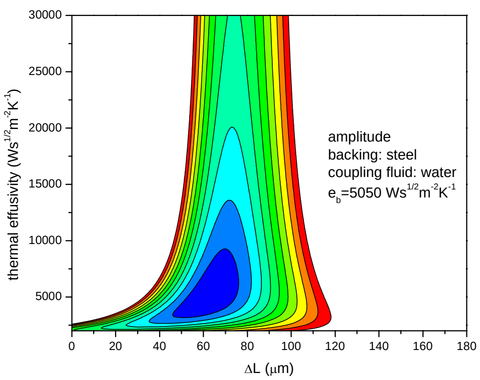


*Fig. 5. Contour map of the precision of the fit performed with Eq. (3) on the experimental amplitude data obtained with steel as backing. X-axis represents the correction term in the measurement of the absolute liquid's thickness.*

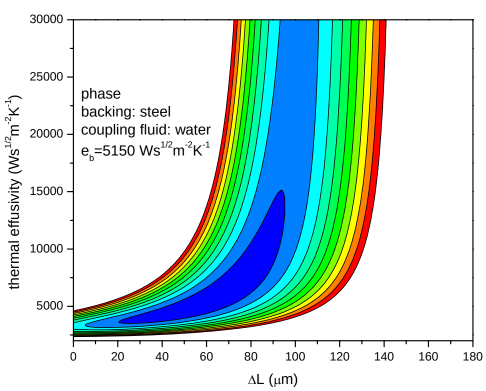


*Fig. 6. Same as Fig. 5, but for the phase of the signal.*

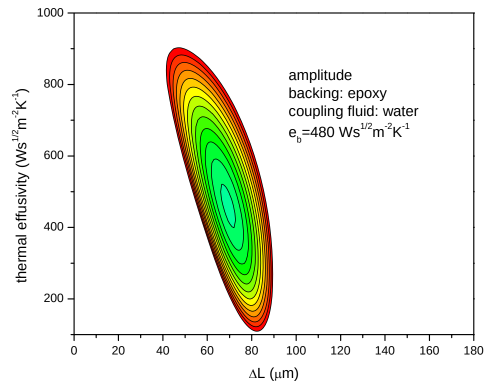


*Fig. 7. Contour map of the precision of the fit performed with Eq. (3) on the experimental amplitude data obtained with epoxy as backing. X-axis represents the correction term in the measurement of the absolute liquid's thickness.*

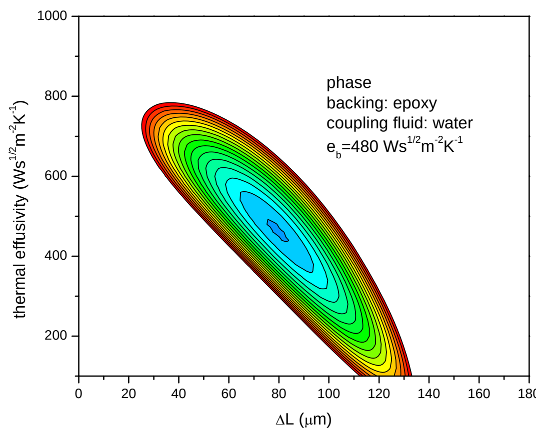


*Fig. 8. Same as Fig. 7, but for the phase of the signal.*

Figs 5-8 contain the results obtained for the thermal effusivity of the two backings, by using both the amplitude and phase as source of information, together with the contour maps for the accuracy of each measurement.

We have to mention that, in order to have a correct comparison for the accuracy of the measurements, the contour maps were displayed in *identical* conditions for the scale range.

## 4. Concluding remarks

The results (values of thermal effusivity) obtained on the investigated materials, by using the amplitude or the phase of the signal are in good agreement: in the case of epoxy, they are the same (e=480 $Ws^{1/2}m^{-2}K^{-1}$) and in the case of steel the relative error is smaller than 2% (e=5100±50 $Ws^{1/2}m^{-2}K^{-1}$).

Eq. (3) represents the complex FPPE signal and an evaluation of the real (amplitude) and imaginary (phase) parts is difficult analytically. In order to check for the suitability of each source of information, one has to compare the 3D “effusivity-thickness-rms” diagrams, or the contour maps (Figs. 5-8). The contour maps indicate that in the case of bad thermal conductors the phase of the signal is more suitable for finding the value of thermal effusivity of the backing. In the case of metals (good thermal conductors) the amplitude of the signal offers a better localization of the backing position and a better accuracy of the fit.

In conclusion, when the thermal effusivity of solid is investigated, by combined FPPE-TWRC method, for a better accuracy, a selection of the source of information (amplitude or phase) is necessary, depending on the value of the thermal effusivity of the backing material.

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