



Improved thermal effusivity measurements of solids using the photopyroelectric technique in the front configuration



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ABSTRACT

The photopyroelectric (PPE) technique in the front configuration consists of illuminating one surface of a pyroelectric slab while the other surface is in contact with the test sample. This method has been widely used to measure the thermal effusivity of liquids. However, in the case of solid samples the PPE signal is affected by the thin coupling fluid layer used to guarantee the thermal contact. Recently, a thickness scan of the coupling fluid layer has been proposed to extract the thermal effusivity of the solid. However, this method is delicate since it needs absolute values of the fluid thickness. In this work, we propose a classical frequency scan of the PPE signal in the front configuration, which only depends on two parameters: the effusivity of the solid and the thickness of the coupling fluid layer. It is demonstrated that both quantities are not correlated and that the sensitivity to the thermal effusivity of the solid is very high. PPE measurements performed on a set of calibrated solids covering a wide range of effusivity values (from polymers to metals) confirm the ability of the method to obtain the thermal effusivity of solids accurately. Moreover, this method is specially suited to measure the temperature dependence of the thermal effusivity across phase transitions.

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1. Introduction

The photopyroelectric (PPE) technique was introduced by Mandelis and Zver in 1985 [1]. Since then, it has been extensively used to measure the thermal properties of solid and liquids due to its outstanding signal to noise ratio. In a PPE device, the test sample is in thermal contact with a pyroelectric slab, which senses temperature variations. In the back configuration the free surface of the sample is illuminated by a modulated light beam, while in the front configuration the free surface of the pyroelectric sensor is illuminated. In both configurations, the PPE current produced by the pyroelectric sensor depends on the thermal diffusivity (D) and effusivity (e) of the sample under study.

The PPE technique in the back configuration, with thermally thick sample and sensor, allows measuring D and e simultaneously from simple linear relations [2,3]. This method has been widely used to measure these thermal properties in liquids across phase

transitions (see Ref. [4] and references therein). However, in the case of solid samples, a very thin grease layer must be used to assure the thermal contact between the sample and the detector. It has been demonstrated that even a few microns thick fluid layer produces an underestimation of D and e , which is higher for good thermal conductors [5]. To overcome this underestimation it has been proposed to use a transparent pyroelectric sensor and a transparent coupling fluid, together with a self-normalization procedure [6]. In this modified setup, the amplitude and phase of the self-normalized PPE signal behaves linearly as a function of the square root of the modulation frequency, but unlike in the standard back configuration with an opaque sensor, the slope does not depend on the fluid layer. This means that the thermal diffusivity can be obtained accurately, but the price to be paid is losing information on the thermal effusivity of the solid.

The PPE technique in the front configuration was introduced by Dadarlat and coworkers [7]. In the case of thermally thick sample and sensor, it provides an accurate method to measure the thermal effusivity of liquids [8]. The method consists of heating the free surface of the pyroelectric sensor and recording the PPE signal twice, first with the liquid wetting the back surface of the detector

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and then with the bare detector. The normalized PPE signal is obtained as the ratio of both signals, which is independent of the instrumental factor (the frequency dependence of the detection electronics), of the electrical properties of the sensor and of the laser intensity. It is demonstrated that the frequency scan of this normalized signal is highly sensitive on the thermal effusivity of the backing liquid [9,10]. This method cannot be applied straightforward to solid sample, since the coupling fluid layer modifies the normalized PPE signal, so the thermal effusivity cannot be obtained accurately.

An alternative procedure to overcome this issue is to change the frequency scan by a thickness scan of the coupling fluid layer at a fixed frequency [11]. It is shown that this PPE signal is sensitive to the thermal effusivity ratio between coupling fluid and backing solid [12–14]. This method has been used to characterize the thermal effusivity of two families of semiconductors when varying the doping level [15,16]. However, this method is delicate to manage since it requires knowledge of the absolute thickness of the coupling fluid layer. Moreover, it is not useful for measuring the temperature dependence of the thermal effusivity of solids, which is one of the most relevant applications of PPE calorimetry.

In this work, we propose a frequency scan of the PPE signal in the front configuration of the following three-layer system: pyroelectric sensor, coupling fluid and test solid. In order to eliminate the instrumental factor this PPE signal is normalized to that obtained with the bare pyroelectric detector. Provided the thickness and thermal properties of the pyroelectric sensor, as well as the thermal properties of the fluid are known, it is demonstrated that this normalized PPE signal only depends on two unknown parameters: the solid effusivity and the thickness of the coupling fluid layer. Moreover, it is shown that both parameters are uncorrelated and that the normalized PPE signal is highly sensitive to the thermal effusivity of the solid sample. Accordingly, by fitting the experimental frequency scan of the normalized PPE signal to the theoretical model, which includes the effect of the fluid layer, the thermal effusivity of the solid is obtained. PPE measurements performed on a set of calibrated solids covering a wide range of effusivity values (from polymers to metals) confirm the ability of the method to obtain the thermal effusivity of solids accurately. Moreover, it is shown that this method is specially suited to measure the temperature dependence of the thermal effusivity of solids across phase transitions from a single heating or cooling run.

2. Theory

Let us consider a three-layer stack made of an opaque pyroelectric slab of thickness L_p , a fluid layer of thickness L_f and a thick solid sample. The free surface of the pyroelectric detector is

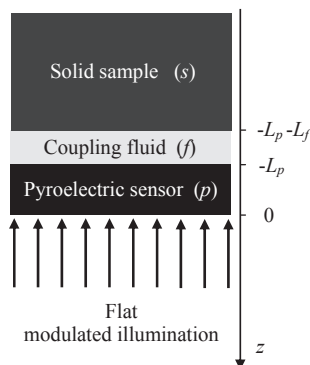


Fig. 1. Geometry of the photopyroelectric cell in the front configuration.

illuminated by a laser beam whose intensity I_0 is modulated at a frequency f ($\omega = 2\pi f$). The geometry of the problem is shown in Fig. 1. The PPE signal (S) is proportional to the spatially averaged temperature of the pyroelectric slab $\langle T_p \rangle$ [17],

$$S = ab\langle T_p \rangle = ab \frac{1}{L_p} \int_{-L_p}^0 T_p(z) dz, \quad (1)$$

where a is a frequency-independent factor that depends on the physical properties of the detector (pyroelectric coefficient, dielectric constant and permittivity) and b is a frequency-dependent factor that accounts for the influence of the detection electronics.

The temperature of the pyroelectric slab is obtained by solving the heat diffusion equation for the three-layer system shown in Fig. 1. Heat losses at the front surface of the pyroelectric sensor are neglected. The temperature at each layer is given by

$$T_p(z) = Ae^{q_p z} + Be^{-q_p z} \quad (2a)$$

$$T_f(z) = Ce^{q_f(z+L_p)} + Ee^{-q_f(z+L_p)} \quad (2b)$$

$$T_s(z) = Fe^{q_s(z+L_p+L_f)}, \quad (2c)$$

where $q = \sqrt{i\omega/D}$ is the thermal wave vector. Subscripts p , f and s stand for pyroelectric detector, coupling fluid and sample respectively. Constants A , B , C , E and F are obtained from the boundary conditions at the interfaces:

(a) Temperature continuity:

$$T_p|_{z=-L_p} = T_f|_{z=-L_p} \quad T_f|_{z=-(L_p+L_f)} = T_s|_{z=-(L_p+L_f)} \quad (3a)$$

(b) Heat flux continuity:

$$K_p \frac{dT_p}{dz} \Big|_{z=-L_p} = K_f \frac{dT_f}{dz} \Big|_{z=-L_p} \quad K_f \frac{dT_f}{dz} \Big|_{z=-(L_p+L_f)} = K_s \frac{dT_s}{dz} \Big|_{z=-(L_p+L_f)} \quad (3b)$$

(c) Illumination at the front surface:

$$K_p \frac{dT_p}{dz} \Big|_{z=0} = \frac{I_0}{2}. \quad (3c)$$

By substituting Eq. (2) into Eq. (3), the temperature of the pyroelectric slab is obtained. Then, from Eq. (1), the PPE signal (S) is obtained. For normalization purposes, i.e. to eliminate the frequency dependence of the detection electronics, the PPE signal obtained for the three-layer system is divided by the PPE signal obtained for the bare pyroelectric slab. Note that the temperature of the bare pyroelectric slab is obtained from Eq. (2a) with adiabatic boundary condition at the rear surface together with illumination at the front surface:

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