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Lock-in thermography versus PPE calorimetry for accurate measurements of thermophysical properties of solid samples: A comparative study

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ABSTRACT

The aim of this paper is to compare the measurement accuracy of photopyroelectric calorimetry in back detection configuration (BPPE) and infrared lock-in thermography (LT) for thermal diffusivity measurement of solid samples. For this purpose, the following materials with well-known thermal properties have been selected: glassy carbon (type G), $LiTaO_3$ crystal and binary II–VI semiconductors (based on CdSe and CdTe). The advantages and drawbacks of the two techniques have been analyzed both theoretically and experimentally.

1. Introduction

Thermal characterization of the materials is very important due to the dissipation of the heat in projected systems e.g. miniaturized semiconducting devices. Thermal parameters are unique for each material, being strongly dependent on the composition, structural characteristics and fabrication process. From an application point of view, thermal diffusivity describes how quickly a material reacts to a change in temperature [1–5]. Thermal diffusivity of solid samples is typically measured with non-contact techniques such as flash method [6] or photothermal radiometry (PTR) [2]. In this work, the thermal diffusivity of some solid samples having well-known thermal properties was determined by means of contact (PPE calorimetry) and non-contact (IR lock-in thermography) techniques. The aim of this paper is to compare the results obtained by two techniques as well as to find out the most suitable theoretical and experimental conditions for improving the accuracy of the results.

The PPE technique has been extensively applied for the study of thermal properties of condensed matter [4,7,8]. The major advantages of this technique are its simplicity, high sensitivity, non-destructive character and adaptation on experimental restrictions for theoretical requirements. In classical BPPE method, for the investigation of solid samples, a coupling fluid must be introduced between the sample and sensor in order to ensure a good thermal contact. This always leads to an error in thermal diffusivity measurement. This fact is well known and it was previously discussed by Salazar et al. [9–11]. They have shown that the results obtained with BPPE technique are always underestimated due to the presence of the coupling fluid between the sample and the sensor. The influence of the coupling fluid in pyroelectric measurements of solids becomes significant especially for high conductive samples and at high modulation frequency of incident radiation. Up to date many different solution have been proposed, often based on non-contact techniques such as photothermal radiometry (PTR) [2,8,12].

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It has been shown that lock-in thermography allows the determination of thermal diffusivity and thermal conductivity of solid samples [13]. The procedure is fast and requires little preparation, i.e. the blackening of the investigated surface (for semi-transparent samples) and a calibration procedure for measurement of thermal conductivity. Thermal diffusivity is being estimated from the phase image obtained after lock-in detection. Infrared lock-in thermography has been also successfully applied to the determination of thermal diffusivity of thin slabs and filaments [14]. However, in the case of thin samples, experimental conditions must be chosen carefully to fulfill theoretical requirements, e. g. the sample should be kept in vacuum to suppress convective heat losses (especially for samples with low thermal diffusivity). Several conditions must be fulfilled in the case of LT technique: (i) the thickness of the blackening layer must be negligible with respect to the sample thickness, (ii) to prevent nonlinear heating effects, small laser powers must be applied, (iii) the sample under investigation should be thermally thick.

The classic BPPE calorimetry with frequency scanning procedure is used for thermal diffusivity measurements of solid samples. The influence of different coupling fluids on the accuracy of the results has been analyzed. The experimental set-up was improved in order to minimize the measurement errors. The parallelism between the sample/camera objective and the influence of the excitation frequency on the results have been also analyzed. The results obtained by two techniques were compared.

2. Material and methods

Several different solid materials were investigated in this work: (i) a glass-like carbon plate (GC) type G [15] having a square shape (thickness 1 mm), (ii) a $LiTaO_3$ crystal with Cr + Au electrodes (thickness 0.4 mm), (iii) a binary II–VI crystal based on CdSe (thickness 1.15 mm) (iv) and a binary II–VI crystal based on CdTe (thickness 1.04 mm). The binary II–VI crystals under investigation were grown from the melt by the high-pressure (150 atm of argon) modified vertical Bridgman method using high purity (99.995%) powders in a graphite crucible. The crystal rods (1 cm in diameter) were cut into about 1.5 mm thick plates. To provide a good thermal contact with the pyroelectric sensor, the surface of the sample was polished in order to be as flat as possible. The samples were first grounded by using a grinding powder (Al_2O_3 with 10 μm diameter) and next polished with the diamond paste (1 μm diameter). During the polishing procedure the thickness of the specimens was controlled at several points to assure the parallelism of the two sides of the samples. The thickness of all specimens was measured with a micrometer with an accuracy of 10 μm.

For the PPE investigations, a modified experimental setup in the back configuration was used (Fig. 1). It consisted of a 300 mW power blue diode laser (λ = 405 nm), a 0.4 mm thick $LiTaO_3$ detector, provided with CrAu electrodes and a SR850 dual-phase lock-in amplifier. The reference signal provided from the internal oscillator of the lock-in was used for the modulation of the incident radiation. In standard BPPE configuration the sample (placed onto the sensor) is directly excited with a modulated radiation. A thin layer of ethylene glycol served as coupling fluid between the sample and the sensor. To improve the thermal contact between the sample and the sensor, a textolite disc was mechanically pressed to the sample. This also prevented the sensor from direct illumination. The modulation frequency of the excitation source was changed in the frequency range 1–15 Hz.

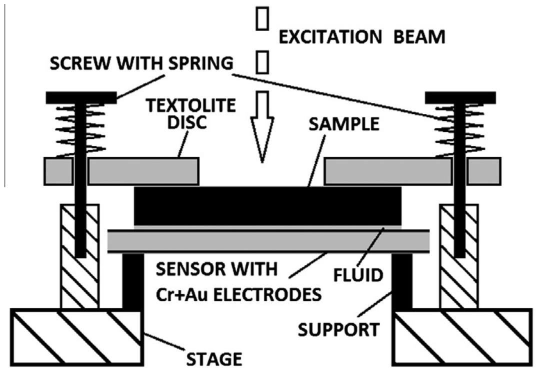

Fig. 1. Modified experimental setup for the BPPE method.

The experimental IR setup included a heat source, a waveform generator, an infrared camera and a computer for data acquisition (Fig. 2). The intensity-modulated optical stimulation was delivered by an Nd:YAG laser (Laser Quantum OPUS, with λ = 532 nm and P = 0.5 W). The IR camera (FLIR 7200 series, with a 256 × 320 pixel array of InSb detectors sensitive in the 1.5–5.1 μm wavelength range, working at a sampling frequency of 100 Hz) recorded the changes in the surface temperature of the specimens. The noise equivalent temperature difference (NETD) of this camera is lower than 20 mK. The signals delivered by the infrared camera and the reference frequency f_0 were sent to the lock-in detection module incorporated into the camera, which outputs the continuous component image (f = 0) as well as the amplitude and

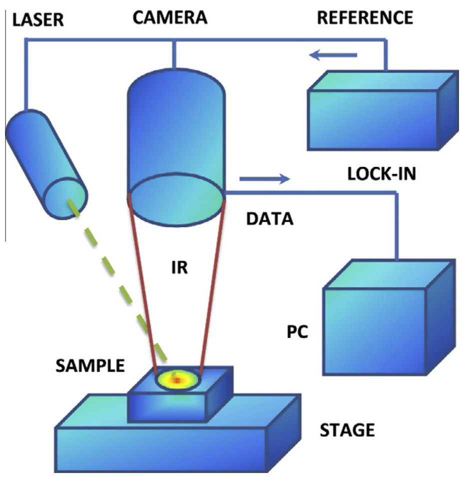

Fig. 2. Experimental setup for the lock-in thermography technique.