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An extension to the methodology for characterization of thermal properties of thin solid samples by photoacoustic techniques

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ABSTRACT

The paper presents a study of possibilities to extend the present methodology for thermal characterization of thin solid samples by photoacoustic techniques. The present methodology consists of linear fitting of the experimental data to approximate the expressions derived from the composite piston model of photoacoustic response and it is mainly used for calculation of thermal diffusivity of thin samples. The study has shown that the methodology may be extended to calculation of thermal diffusivity, thermal conductivity and thermal expansion coefficients of thin samples by linear fitting in multiple frequency ranges and by analysis of the intersection frequency, which is the frequency where the magnitudes of two components of photoacoustic response in the composite piston model are equal. The analysis of numeric errors of the methodology has revealed the dominant sources and magnitude of the errors, leading to the conclusion that photoacoustic techniques should be carefully used as a tool for extensive thermal characterization of thin samples in the cases when other techniques are not applicable or have larger errors.

1. Introduction

As non-contact and non-destructive material characterization methods, photoacoustic techniques have been used for thermal characterization of thin samples for four decades [1–12]. While photoacoustic techniques were mainly used for calculation of thermal diffusivity of samples [5–12], their application to calculation of thermal conductivity [10–12] and thermal expansion coefficient [13] of samples increases.

The photoacoustic (PA) effect, which is the basis for PA techniques, is generation of sound waves in a sample and its surroundings due to the exposure to modulated optical radiation [2–4]. The usual method for thermal characterization of materials is based on studies of dependencies of the amplitude (amplitude-frequency characteristics, abbreviated as AFC) and the phase delay (phase-frequency characteristics, abbreviated as PFC) of the PA response on the modulation frequency of excitation. The calculation of thermal and thermoelastic properties of the samples based on the experimental AFC and PFC represents “the inverse photoacoustic problem”. The methodologies for solution of the inverse photoacoustic problem depend on the theoretical models of the PA effect, which are in turn determined by the setup of a PA experiment.

The PA effect was discovered and reported by A.G. Bell at the end of the 19th century [14], but the proper explanation was given in the 1970s in some papers of A. Rosenzweig [15–18]. The papers introduced the basic setup of the gas-microphone PA experiment and proposed a theoretical model of the PA effect called “the thermal piston model” [17]. The model considers the indirect mechanism of the PA effect [2], where the optically generated heat in the sample is transferred to the surroundings causing an increase in the temperature and expansion of the nearby gas. In the explanation of the indirect PA mechanism, a thin layer of the expanded gas adjacent to the surface of the sample acts as a “thermal piston” that generates sound waves propagating through the gas in the PA cell. In the reflection configuration of the PA experiment, when the light source and the microphone are placed at the same side of the sample, the thermal piston model successfully explains the PA effect [2,19].

However, during the 1980s, it was demonstrated that the transmission configuration of the PA experiment where the light source and the microphone are placed at the opposite sides of the sample offers many advantages [5]. In the transmission

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configuration, the described indirect PA mechanism was not sufficient to explain the obtained results for all modulation frequencies, which led to the conclusion that direct PA mechanisms have significant contribution to the PA response [5]. Direct PA mechanisms arise because of the thermoelastic waves generated within the sample due to the heat produced by the modulated optical beam [20,21]. In direct PA mechanisms, the sample acts as a “mechanical piston” that generates sound waves propagating through the gas in the PA cell.

In the contemporary literature it is usual to consider the PA response as a consequence of the action of a “composite” (thermal and mechanical) piston, i.e. as a summary effect of indirect and direct mechanisms of PA sound emission [12,22]. The solution of the inverse PA problem based on the composite piston model is a numerically complicated and sensitive task due to the complex and non-linear mathematical expressions describing the influence of the properties of the sample on the PA response [16–20]. A straightforward application of the composite piston model to material characterization would require the nonlinear fitting of complex expressions with a variation of several thermal properties of the sample. The nonlinear fitting is a notoriously intricate and unstable procedure, where the increase in the number of fitting parameters decreases the reliability of the obtained results.

For these reasons, the common methodology for thermal characterization by PA techniques is the application of approximate models, which enables replacing of non-linear fitting by linear fitting in appropriate modulation frequency ranges. The usual approximations of the composite piston model consider the thermodiffusion (abbreviated as TD) component of the indirect PA mechanism as the dominant component in the low modulation frequency range, and the thermoelastic (abbreviated as TE) component of the direct PA mechanism as the dominant component in the high modulation frequency range [5,10,12]. Significant weaknesses of the presented common methodology are a large number of experimental setup parameters that affect the PA response and its applicability only to the frequency ranges where one of the components of the PA response is dominant.

The number of experimental setup parameters that affect the results of the methodology may be reduced by proper handling of multiple PA responses. The study of phase difference (or the ratio of magnitudes) between PA responses of the same sample obtained in reflective and transmission configuration [23] represents a successful improvement of the methodology. Another technique which reduces the number of experimental setup parameters that affect the results of the methodology is normalization of the PA responses of samples with different thicknesses [24,25]. Both proposed techniques enable accurate and reliable calculation of thermal properties in the frequency ranges where one of the components of the PA response is dominant.

However, in the case of materials with large thermal expansion coefficients, wide PA experimental modulation frequency ranges contain subranges where one of the components is dominant, as well as the range where neither of the components is dominant [6,12]. The interest in the extension of the methodology for solution of the inverse photoacoustic problem in such cases has risen with the recently increased application of PA and other photothermal techniques to thermal characterization of thin polymer layers [12] and thin samples of porous materials [26].

This paper presents the studies of possibilities to extend the application of the common methodology for thermal characterization of thin samples to the modulation frequencies where neither of the components is dominant. The initial step is derivation of approximate expressions for the TD and TE components of the PA response that are convenient for the analysis of characteristic features of AFC and PFC of the PA response. The analysis considers both modulation frequency ranges where one of the components dominates, but also the modulation frequency where the magnitudes of the components are equal. The results of the study are relationships between the PA response and thermal parameters of the sample, which enables the solution of the inverse photoacoustic problem. It should be noticed that the subject of the study presented here is the properties of the composite piston model, which describes sound wave pressure as the PA response. Therefore, the study does not deal with the problem of separation of the PA response from the experimentally detected signal, which represents a serious research task for itself, requiring further investigations.

2. Theory

The basic setup for PA studies of thin samples in transmission configuration is shown in Fig. 1. The PA cell is the cavity of an electret microphone. The thin sample, with the thickness l_s and the circular cross-section with the radius R, is placed above the diaphragm of the microphone and fixed to the PA cell by a rubber ring with the inner diameter R_c. On the opposite side of the sample, there is a laser with the optical system which provides uniform irradiation on the sample surface. The sample is either opaque or adequately prepared so that it may be adopted that the light absorption occurs in a very thin layer close to the surface of the sample. The cell is usually filled with air, which is considered to be a much worse heat conductor than the sample, and it may be assumed that the heat flux into the surrounding air is negligible.

Since the PA response consists of the amplitude of the signal recorded by the microphone p and its phase delay with respect to the excitation light beam ϕ, it is convenient to describe it in complex representation $\underline{p} = p \cdot \exp(-i\phi)$, where i is the imaginary unit. As explained in the introduction, the PA response in the transmission configuration is described by the composite piston model as a summary effect of direct and indirect PA mechanisms.

The dominant component of the direct PA mechanism in the case of thin samples is acoustic emission due to the TE bending of the sample (“the drum effect”) [21]. The other direct PA mechanism

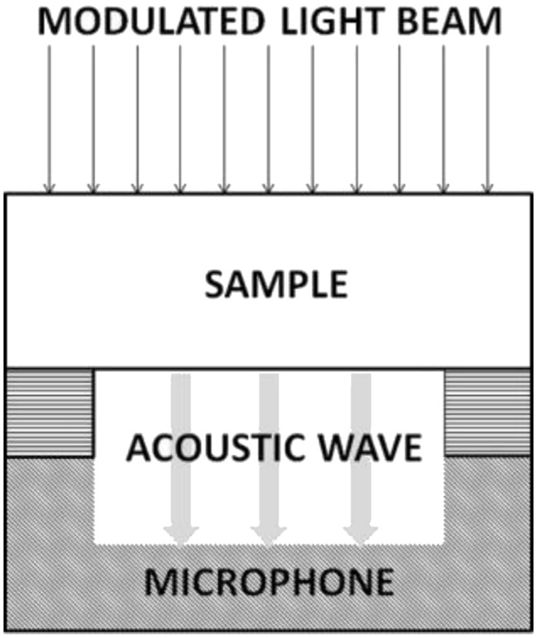

Fig. 1. Basic setup for the PA experiment in the transmission configuration.