

The strong influence of heat losses on the accurate measurement of thermal diffusivity using lock-in thermography

Agustín Salazar,^{a)} Arantza Mendioroz, and Raquel Fuente

Departamento de Física Aplicada I, Escuela Técnica Superior de Ingeniería, Universidad del País Vasco, Alameda Urquijo s/n, 48013 Bilbao, Spain

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In modulated photothermal experiments the lateral thermal diffusivity can be obtained from the slope of the linear relation between the phase of the surface temperature and the distance to the heating spot. However, this slope is greatly affected by heat losses so that the measured thermal diffusivity is overestimated, especially for thin samples of poor thermal conducting materials. In this paper we definitely identify the physical mechanism responsible for the overestimation of the diffusivity as heat conduction to the surrounding gas. Accurate measurements of the thermal diffusivity using the “slope method” have been obtained by keeping the sample in vacuum. © 2009 American Institute of Physics. [doi:10.1063/1.3236782]

It is well known that the lateral thermal diffusivity of solid samples can be obtained from the slope of the linear relation between the phase of the surface temperature and the radial distance to the heating spot, the so-called slope method. However, the accuracy of this method is limited by several issues. In the case of photothermal infrared radiometry (or lock-in thermography) it has been demonstrated that the effect of diffraction leads to an overestimation of the thermal diffusivity of poor thermal conductors, the effect being enhanced for increasing frequencies.¹ On the other hand, the use of low frequencies also produces an overestimation of the thermal diffusivity of poor conductors due to heat losses. This effect is especially dramatic for thin plates and filaments for which overestimations of more than one order of magnitude have been reported.²⁻⁵

In this paper, we analyze the effect of the different mechanisms of heat losses (conduction and convection to the gas, and radiation) on the accuracy of the retrieved thermal diffusivity when using the slope method. We demonstrate theoretically and experimentally that the main disturbing effect is the heat conduction to the gas, a transport method that is usually left aside because of the very low thermal conductivity of the air. Experimental measurements using lock-in thermography, with the sample kept in vacuum, show that an accurate thermal diffusivity value can be obtained even for poor thermal conductors as polymers and biological samples.

Let us consider an opaque and semi-infinite slab of thickness L , illuminated by a focused laser beam of power P_o with a Gaussian profile of radius a (at $1/e^2$) and modulated at a frequency f ($\omega=2\pi f$). The sample is surrounded by air. The geometry of the problem is shown in Fig. 1. In what follows, subscripts s , gf , and gr stand for sample, gas at the front surface, and gas at the rear surface, respectively. The temperature is obtained by solving the heat diffusion equation in each medium with the following boundary conditions:

$$T_{gf}(z=0) = T_s(z=0),$$

$$T_{gr}(z=-L) = T_s(z=-L),$$

$$-K_s \left. \frac{\partial T_s}{\partial z} \right|_{z=0} = -K_g \left. \frac{\partial T_{gf}}{\partial z} \right|_{z=0} + hT_s|_{z=0} - \frac{P_o}{2\pi} \int_0^\infty \delta J_o(\delta r) e^{-(\delta a)^2/8} d\delta,$$

$$-K_s \left. \frac{\partial T_s}{\partial z} \right|_{z=-L} = -K_g \left. \frac{\partial T_{gr}}{\partial z} \right|_{z=-L} - hT_s|_{z=-L},$$

which take into account heat losses due to conduction and convection to the surrounded air, and radiation. The radial dependence of the illuminated surface temperature writes

$$T_s(r) = \frac{P_o}{4\pi K_s} \int_0^\infty \delta J_o(\delta r) \times \frac{e^{-(\delta a)^2/8}}{\beta_s} \frac{(1+G+H)e^{\beta_s L} + (1-G-H)e^{-\beta_s L}}{(1+G+H)^2 e^{\beta_s L} - (1-G-H)^2 e^{-\beta_s L}} d\delta, \quad (1)$$

where δ is the Hankel variable, J_o is the Bessel function of the zeroth order, $\beta^2 = \delta^2 + i\omega/D$, D is the thermal diffusivity, and K is the thermal conductivity. Parameter $G = K_g \beta_g / K_s \beta_s$ governs the contribution of the heat conduction to the surrounding air to the sample temperature, while parameter $H = h/K_s \beta_s$ controls the heat losses by convection and radia-

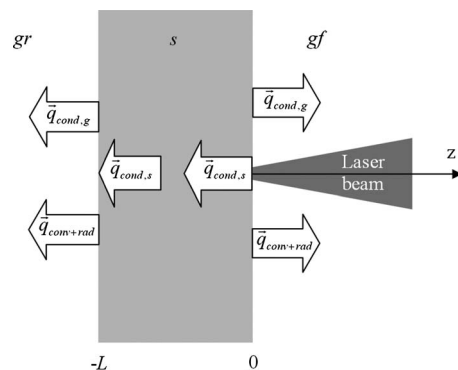


FIG. 1. Geometry of the problem, indicating the heat fluxes (\vec{q}) at each surface.

^{a)}Electronic mail: agustin.salazar@ehu.es.

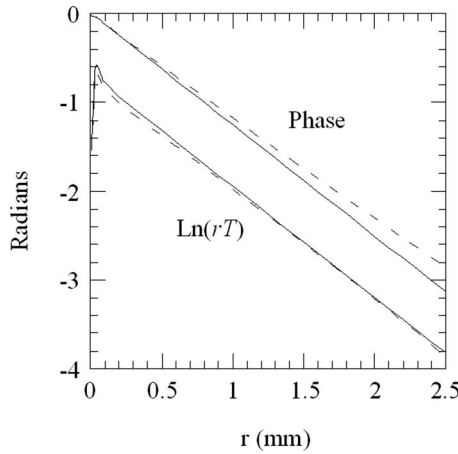


FIG. 2. Simulation of the radial dependence of the natural logarithm of the temperature amplitude multiplied by r and of the phase for a thermally thick sample. Without heat losses (continuous line), with conduction to the gas (dashed line). Data: $D_s=0.1 \text{ mm}^2/\text{s}$, $K_s=0.3 \text{ W m}^{-1} \text{ K}^{-1}$, $D_g=22 \text{ mm}^2/\text{s}$, $K_g=0.026 \text{ W m}^{-1} \text{ K}^{-1}$, $a=50 \text{ }\mu\text{m}$, and $f=0.05 \text{ Hz}$.

tion, being $h=h_{\text{conv}}+h_{\text{rad}}$ the combined heat transfer coefficient.

For measurements performed at room temperature ($T_{\text{amb}} \approx 300 \text{ K}$) and using moderate laser beam powers producing a temperature rise of a few degrees, $h_{\text{rad}} \approx 4\varepsilon\sigma T_{\text{amb}}^3$, where ε is the surface emissivity and σ is the Stefan-Boltzmann constant.⁶ The highest value of the radiative transfer coefficient, which is obtained for an emissivity $\varepsilon=1$, is $h_{\text{rad}} \approx 6 \text{ W m}^{-2} \text{ K}^{-1}$.

For a vertical slab surrounded by air at room temperature and at room pressure, the convective transfer coefficient $h_{\text{conv}} \approx 1.5(\Delta T/b)^{0.25}$, where ΔT is the temperature difference between the sample surface and the surrounding air and b is the slab height.⁷ For instance, by taking rather extreme conditions of a high $\Delta T=10 \text{ K}$ and a short sample $b=1 \text{ cm}$, $h_{\text{conv}} \approx 8.5 \text{ W m}^{-2} \text{ K}^{-1}$, which can be considered as the highest h_{conv} in photothermal infrared radiometry at room temperature. Accordingly, the highest value of combined heat transfer coefficient $h=h_{\text{conv}}+h_{\text{rad}} \approx 15 \text{ W m}^{-2} \text{ K}^{-1}$.

Two extreme cases are considered. In the case of *thermally thick samples* ($e^{-\beta_s L} \approx 0$) Eq. (1) reduces to

$$T_s(r) \approx \frac{P_o}{4\pi K_s} \int_0^\infty \delta J_o(\delta r) \frac{e^{-(\delta a)^2/8}}{\beta_s} \frac{1}{(1+G+H)} d\delta, \quad (2)$$

which allows us to discriminate the influence of the conduction to the surrounding air (G) and the influence of convection and radiation (H) on the surface temperature. Numerical simulations indicate that the influence of convection and radiation with $h=15 \text{ W m}^{-2} \text{ K}^{-1}$ is negligible for samples with $D \geq 0.1 \text{ mm}^2/\text{s}$ and $f \geq 0.05 \text{ Hz}$.

The influence of the heat conduction to the gas cannot be neglected instead. In Fig. 2 we show the lateral scan of the phase and of the natural logarithm of the amplitude of the surface temperature of a thick slab of a poor conducting sample surrounded by air using a low modulation frequency ($f=0.05 \text{ Hz}$). The continuous lines are the results for an isolated sample ($G=H=0$). As can be seen, both are parallel and straight lines from whose slope (m) the thermal diffusivity can be retrieved: $m_{\text{phase}}=m_{\text{Ln}(rT)}=-(\pi f/D)^{0.5}$. The dashed lines show the influence of the surrounding air ($G \neq 0$). As

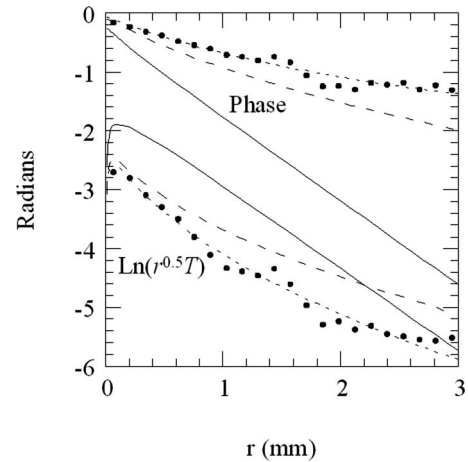


FIG. 3. Simulation of the radial dependence of the natural logarithm of the temperature amplitude multiplied by $r^{0.5}$ and of the phase for a thermally thin sample. Without heat losses (continuous line), with conduction to the gas (dashed line), and with the combined effect of conductive, convective, and radiative losses (dotted line). Data: $D_s=0.19 \text{ mm}^2/\text{s}$, $K_s=0.4 \text{ W m}^{-1} \text{ K}^{-1}$, $D_g=22 \text{ mm}^2/\text{s}$, $K_g=0.026 \text{ W m}^{-1} \text{ K}^{-1}$, $a=50 \text{ }\mu\text{m}$, $f=0.12 \text{ Hz}$, $h=15 \text{ W m}^{-2} \text{ K}^{-1}$, and $L=25 \text{ }\mu\text{m}$. In dots, experimental data obtained at room pressure for a $25 \text{ }\mu\text{m}$ thick PEEK film ($D=0.19 \text{ mm}^2/\text{s}$, $K=0.4 \text{ W m}^{-1} \text{ K}^{-1}$).

can be observed, both slopes increase and therefore the retrieved thermal diffusivity is overestimated. It is worth noting that the change in slope is independent of the frequency but diminishes as the thermal conductivity increases, in such a way that it is negligible for $K_s > 5 \text{ W m}^{-1} \text{ K}^{-1}$, i.e., the overestimation in D is smaller than 1%.

In the case of *thermally thin samples* ($e^{\pm\beta_s L} \approx 1 \pm \beta_s L$) Eq. (1) reduces to

$$T_s(r) \approx \frac{P_o}{4\pi K_s} \int_0^\infty \delta J_o(\delta r) \frac{e^{-(\delta a)^2/8}}{\beta_s} \frac{1}{2G+2H+\beta_s L} d\delta. \quad (3)$$

As before, we analyze separately the influence of G (heat conduction to the gas) and H (heat losses by convection and radiation). Heat losses by convection and radiation produce an increase in the phase slope compensated by a decrease in the slope of the natural logarithm of the amplitude multiplied by $r^{0.5}$ so that the product of both slopes verifies^{3,5} $m_{\text{phase}} \times m_{\text{Ln}(r^{0.5}T)} = -\pi f/D$.

On the contrary, the heat conduction to the gas has a dramatic influence in the case of thin slabs of poor thermal conductors, preventing the use of the slope method to retrieve their thermal diffusivity. In Fig. 3 we show the lateral scan of the phase and of the natural logarithm of the amplitude of the surface temperature multiplied by $r^{0.5}$ with thermal parameters corresponding to a thin slab ($L=25 \text{ }\mu\text{m}$) of polyeter-eter-ketone (PEEK) at $f=0.12 \text{ Hz}$. The continuous lines are the calculations for an isolated sample ($G=H=0$). As can be seen, both are parallel and straight lines with the same slope as for a thick sample. The dashed lines show the strong influence of the surrounding air ($G \neq 0$). It is clear that there is a lost of linearity, which is greater in amplitude and that both profiles are raised if compared to the isolated sample. The dotted lines represent the combined effect of conduction to the air, and convective and radiative losses ($G \neq H \neq 0$), as in a real experiment with the sample in air. Clearly an “average” slope of both phase and amplitude produces a huge overestimation of the thermal diffusivity. This

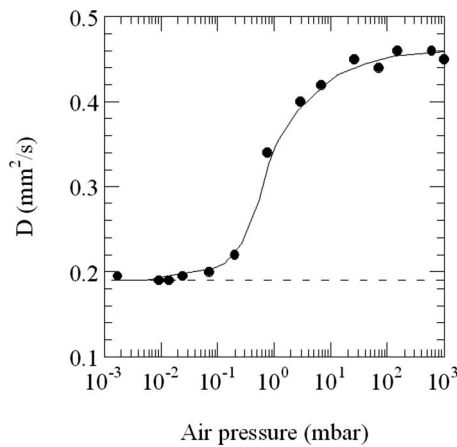


FIG. 4. Thermal diffusivity dependence on air pressure for a 25 μm thick PEEK film ($f=0.12$ Hz). The continuous line represents the simulation from Eq. (3) by taking into account the pressure dependence of K_{air} . The dashed line represents the simulation neglecting the conduction to the gas.

overestimation increases as D , L , and f decrease.

This theoretical approach indicates that an accurate measurement of the thermal diffusivity using the slopes method requires the sample to be kept in vacuum to eliminate the heat conduction to the gas. In order to verify this theoretical prediction we have used a lock-in thermography setup. The sample is heated by an acousto-optically modulated laser beam ($\lambda=532$ nm) focused by a spherical lens of 5 cm focal length. The infrared emission from the sample surface is captured by an infrared camera (3.6–5.0 μm) provided with a lens of 50 mm focal length. This lens has a minimum working distance of 23.5 cm, which gives a spatial resolution of 137 μm . The lock-in software provided with the camera gives the amplitude and phase of the oscillating temperature. In order to increase at the same time the absorption of the exciting light and the infrared emissivity, the samples were covered by a 200 nm thick graphite layer.

The samples were placed in a vacuum chamber with sapphire windows, which are transparent to the infrared radiation. The pressure in the chamber could be controlled in the range between room pressure (10^3 mbar) and 10^{-3} mbar. We have recorded the amplitude and phase profiles of a 25 μm thick PEEK film as a function of the air pressure, at $f=0.12$ Hz. The results at room pressure are shown in dots in Fig. 3, where a good agreement with the calculated profiles containing all the heat loss effects is observed. In Fig. 4 we show in dots the measured values of the thermal diffusivity of the same film as a function of the air pressure inside the vacuum chamber. In order to overcome the influence of convection and radiation, the thermal diffusivity is obtained from the product of the slopes $m_{\text{phase}} \times m_{L_n(r^{0.5T})} = -\pi f/D$. As can be seen, at room pressure the retrieved thermal diffusivity is $D=0.45$ mm^2/s while in vacuum the nominal value of this polymer is obtained, $D=0.19$ mm^2/s .⁸ The dashed line represents the simulated values of the thermal diffusivity neglecting the heat conduction to the gas, which certainly does not reproduce the experimental data. The continuous line depicts the calculated values of the thermal diffusivity using Eq. (3) by including the pressure dependence of the thermal conductivity of the air.⁹ The three heat loss mechanisms have been considered in

TABLE I. Thermal diffusivity (mm^2/s). Uncertainty: $\pm 5\%$.

Material/shape	In air	In vacuum
PEEK/bulk	0.23	0.20
PEEK/film (125 μm)	0.26	0.19
PEEK/film (75 μm)	0.30	0.19
PEEK/film (25 μm)	0.45	0.19
Human hair	0.30	0.14

the calculation. As can be observed, the agreement between the calculated and experimental results is excellent. Note that retrieving the right value of the thermal diffusivity requires lowering the pressure in the chamber below 10^{-1} mbar (four orders of magnitude below room pressure). Actually, for pressures down to 1 mbar the diffusivity barely changes. This can be explained in the framework of the kinetic theory of gases, according to which the thermal conductivity of the air remains essentially pressure independent down to 1 mbar, while below this value the thermal conductivity becomes directly proportional to the pressure.⁹ This result clearly demonstrates that conduction to the gas is the only mechanism responsible for the overestimation of the thermal diffusivity when using the slope method.

Table I summarizes the results of the thermal diffusivity of several samples with different shapes obtained from the slope method. As theoretically predicted, the thermal diffusivity of these poor conductors is overestimated in measurements at room pressure. However, accurate values are obtained when the sample is kept in vacuum. It is worth noting that the conclusions of this work are also valid for filaments, where one-dimensional heat propagation takes place. Moreover, these results can be applied not only with infrared sensors (as is the case of lock-in thermography or photothermal radiometry), but in all modulated techniques sensing the surface temperature (or a quantity proportional to it), e.g., thermoreflectance (optical sensor), photothermal resistance (electrical sensor), etc.

In conclusion, we have analyzed the effects of heat losses on the surface temperature distribution when the sample is heated by a focused and modulated laser beam. The results are conclusive in demonstrating that the conduction to the gas is the main disturbing mechanism. To avoid its effect the sample should be placed in vacuum so the very simple and convenient slope method can still be used.

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