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Pulse method of measuring thermal diffusivity and optical absorption depth for partially transparent materials

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Data analysis in the laser-pulse method of measuring thermal diffusivity is based on the solution of the heat diffusion equation, assuming several boundary and initial conditions. The initial condition generally results from considering the material opaque to the laser's radiation. This requires building special absorbing layers on the sample's surface when the material is transparent. A more general solution of the heat diffusion equation was obtained in this paper, in order to simplify sample preparation, and in some cases (high temperatures, sample contamination, etc.) even make possible the measurement of partially transparent materials. Based on an exponential-like absorption of the laser's radiation, the solution includes an absorption-depth parameter p, which describes the material's degree of transparency. A computer-programmable data-reduction procedure, including a least-squares estimation algorithm of nonlinear parameters, was also developed. It permits the simultaneous determination of both the thermal diffusivity and the absorption depth from a single laser-pulse experiment. The accuracy of the method was verified by measuring a sintered glass sample, both with the usual and the new procedures. The results indicate an accuracy level compatible with the current state of the art.

INTRODUCTION

The pulse method of measuring thermal diffusivity (originally named "flash" method and also referred to as "laser-pulse" method) has gained widespread acceptance since it was first introduced in 1961.[1] The basic concept is rather simple; the sample, of typical dimensions in the range of a fraction of 1 cm, shaped in the form of a slab of uniform thickness and initially at a constant temperature, receives a short but powerful pulse of radiant energy at one surface (front face) and the temperature at the opposite surface (rear face) is recorded as a function of time. The value of the sample's thermal diffusivity D is then derived by comparing the recorded temperatures with corresponding theoretical values calculated from the appropriate solution of the heat diffusion equation[2]:

$$\frac{\partial T}{\partial t} = D\,\nabla^2 T, \tag{1}$$

where T is the temperature at any point of the sample at time t.

In order to keep the theoretical treatment between tractable limits, the experiment is arranged so that the geometry of the sample, and the initial and boundary conditions are as simple as possible. Efforts are usually made in order to be able to assume that:

(a) Heat losses from the sample may be neglected;

(b) the sample is homogeneous, and if not isotropic, at least cut with its width along a principal axis of the thermal conductivity tensor;

(c) the incident radiation pulse is uniformly distributed on the surface of the sample;

(d) thermal properties are temperature independent;

(e) the duration of the incident radiation pulse is negligible compared to the time required for the heat to diffuse through the sample;

(f) the radiation is absorbed by a layer of material which is very thin compared to the thickness of the sample.

Assumptions (a)–(c) reduce the mathematical problem to the case of one-dimensional heat flow, i.e., the temperature is a function of time and one space coordinate $T(x,t)$. Assumption (d) keeps Eq. (1) linear, while assumption (e) implies that the incident radiation pulse may be considered instantaneous at $t = 0$. Its heating action can therefore be described by an initial temperature distribution $T(x,0)$. Assumption (f) leads to equating this initial temperature distribution to

$$T(x,0) = Q/(\rho C_P)\delta(x), \tag{2}$$

where $\delta(x)$ is Dirac's delta function, Q is the amount of radiant energy per unit area that is absorbed by the sample, ρ is its density, and C_P is its specific heat capacity at constant pressure. Equation (2) corresponds to the conventions that the sample's temperature is zero before the radiation pulse arrives, and that the front face, where the radiation is absorbed, is placed at $x = 0$, while the rear face, where temperature is sensed, lies at $x = L$ (i.e., L = width of the sample).

Since departures from these ideal assumptions are in practice unavoidable, a large amount of theoretical and experimental work was performed following the introduction of the method in order to evaluate uncertainties and systematic errors, propose corrections, and improve the experimen-

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tal data analysis by finding more general solutions of Eq. (1). A comprehensive list of references to these investigations is given in a review article by Righini and Cezairliyan,[3] to which some more recent references[4] may be added.

The present paper deals with an improvement in the experimental data analysis which permits eliminating assumption (f), by means of considering the partial transparency of the sample, leading to a simultaneous determination of thermal diffusivity and optical absorption depth of the specimen under study. The method here presented helps solve a major experimental problem which appears when the samples under investigation are partially transparent (like, for instance, ceramic or glassy materials, plastics, semiconductors, etc.) avoiding the cumbersome usual solution[5] which consists of building on the front surface of the sample an opaque coating which absorbs the pulse of radiant energy. This coating must be thin enough and well bounded to the surface, in order that it does not add significantly to the diffusion time, and should uniformly absorb the incoming radiation. It should also hold up at high temperatures, both those resulting from the very high power associated with the incoming heating pulse, or during high-temperature thermal diffusivity measurements. Additional difficulties may result at high temperatures since the coatings may become transparent, diffuse into the sample or react chemically with it, thereby affecting its thermal properties. The present method helps solve this "hardware" problem, employing a "software" solution, in order to measure the sample in its unperturbed original state.

THEORY OF THE METHOD

Insofar as the irradiation power of the incoming pulse, typically less than 10 000 W/cm^2, is not as high as to give rise to nonlinear effects[6] in the sample, and as long as the latter satisfies the assumption (b) stated above, an exponential absorption law may be assumed for partially transparent materials. It follows from stating that the number of photons $dN(x)$ absorbed per unit area at a depth x, by a differential layer of material of width dx, is proportional to the number of photons per unit area $N(x)$, arriving at x:

$$dN(x) = -N(x)/p\,dx, \tag{3}$$

where the constant of proportionality $1/p$ is such that p is a typical length called the absorption depth. Straightforward integration of Eq. (3) leads to $N(x) = N(0)\exp(-x/p)$. Assuming that the duration of the incoming radiation pulse is negligible compared to the time required for the heat to diffuse through the sample, the exponential absorption of the radiation can be considered instantaneous, leading to an initial temperature distribution given by

$$T(x,0) = Q/(\rho p C_P)\exp(-x/p)/[1-\exp(-L/p)]. \tag{4}$$

As expected, in the limit in which p tends to zero (opaque materials) Eq. (4) becomes identical to Eq. (2), since $T(x,0)$ tends to zero for all values of $x \neq 0$, but its integral over the space coordinate is constant, and equal to $Q/(\rho C_P)$.

If the initial temperature distribution within a thermally insulated solid, of uniform thickness L, is $T(x,0)$, satisfying Eq. (1) with $\partial T/\partial x = 0$ at $x = 0$ and $x = L$ (adiabatic boundary conditions), the temperature distribution at any later time is given in Ref. 2, p. 101, as

$$T(x,t) = 1/L \int_0^L T(x,0)dx + 2/L \sum_{n=1}^{\infty} \exp(-n^2\pi^2 Dt/L^2)\cos(n\pi x/L)\int_0^L T(x,0)\cos(n\pi x/L)dx. \tag{5}$$

Substituting Eq. (4) into Eq. (5) it follows that for partially transparent materials with an absorption depth p:

$$T(x,t) = Q/(\rho C_P L)\left(1 + 2\sum_{n=1}^{\infty}\frac{[1-(-1)^n\exp(-L/p)]\exp(-n^2\pi^2Dt/L^2)}{[1-\exp(-L/p)][1+(n\pi p/L)^2]}\cos(n\pi x/L)\right), \tag{6}$$

and the temperature at the rear face in the laser-pulse experiment can be expressed by

$$T'(L,t') = 1 + 2\sum_{n=1}^{\infty}\frac{[(-1)^n - \exp(-1/p')]\exp(-n^2t')}{[1-\exp(-1/p')][1+(n\pi p')^2]}, \tag{7}$$

where the primed variables are dimensionless quantities, i.e., T' represents temperature in units of the maximum temperature rise at the rear face, $Q/(\rho C_P L)$; t' represents time in units of $L^2/(\pi^2 D)$, and p' is the absorption depth in units of the sample's width L. In the present case $T'(L,t)$ will be called the normalized-temperature rise, since it takes values from zero through one, stressing the fact that the temperature sensors need not be calibrated, but have only to be linear over the range of the temperature change induced by the laser's pulse.

Figure 1 represents a family of curves of $T'(L,t')$ vs t' for different values of p'. Contrasting with the usual case for opaque materials ($p' = 0$), for which the temperature at the rear face increases continuously from zero, as heat begins to arrive from the front face, where all the radiation was absorbed, partially transparent materials ($p' \neq 0$) present a sudden rear-face temperature rise due to direct photon heating.

The effects produced by internal reflections of the incoming radiation at the sample-environment interfaces have so far been neglected. This additional heating due to the absorption of radiation that otherwise would have left the sample without further consequences, can be treated following the same steps outlined above, starting from the initial temperature distribution given by

$$T(x,0) = \frac{Q\{\exp(-x/p) + R\exp[-(2L-x)/p]\}}{(\rho C_P)[1-(1-R)\exp(-L/p) - R\exp(-2L/p)]}, \qquad (8)$$

where $R < 1$ is the appropriate reflection coefficient of the sample-environment interface.

Equation (8) results from adding to Eq. (4) a similar exponential term, decaying from $x = L$ toward $x = 0$, with the same absorption depth parameter p, and with an initial amplitude, R times smaller than the direct radiation arriving at $x = L$. The whole expression is normalized in order to render its integral over the space coordinate, equal to $Q/(\rho C_P)$. Multiple internal reflections can likewise be treated with no more conceptual difficulties, leading to an easy to add geometric series, and therefore somewhat more complicated mathematical expressions. This matter is not pursued here any further, since it may have some relative importance only for highly reflecting, highly transparent materials [see discussion following Eq. (15) to the end of the section].

Substituting Eq. (8) into Eq. (5) the following generalization of Eq. (7) results, that takes into account a first internal reflection at the rear face of the sample:

$$T'(L,t') = 1 + 2\sum_{n=1}^{\infty} \frac{\{(-1)^n[1 - R\exp(-2/p')] - (1-R)\exp(-1/p')\}\exp(-n^2t')}{[1-(1-R)\exp(-1/p') - R\exp(-2/p')][1+(n\pi p')^2]}. \qquad (9)$$

As expected Eq. (9) becomes identical with Eq. (7) if the reflection parameter R is set to zero.

DATA REDUCTION

Equation (7), or the more general Eq. (9), gives rise to the possibility of deriving from a single experiment both D and p. In order to illustrate this point, and assuming $R = 0$ for the sake of simplicity, the conceptually simplest form of doing this is by determining first the value of p' from the initial normalized-temperature rise $T'(L,0)$ at the rear face of the sample, as follows from Eq. (4) evaluated at $x = L$, i.e., solving for p' the equation

$$T'(L,0) = 1/p' \exp(-p')/[1-\exp(-p')]. \qquad (10)$$

Once p' is known, the value of the thermal diffusivity D can be derived from the rest of the measured temperature history. It is well known[1] that in the particular case in which $p' = 0$, the diffusivity can be calculated from the relation:

$$D = 1.37L^2/(\pi^2 t_{1/2}), \qquad (11)$$

where $t_{1/2}$ is the time required for the rear face to reach half of the maximum temperature rise, i.e., $T'(L,t_{1/2}) = 1/2$. In the case in which $p' \neq 0$, this simple procedure has to be generalized by determining, for instance, the time t_g for which $T'(L,t_g)$ reaches the value $[1 + T'(L,0)]/2$, and calculating the diffusivity from the relation

$$D = 1.37\exp(-0.94p')L^2/(\pi^2 t_g). \qquad (12)$$

Equation (12) is an approximate ($\pm$ 15%) generalization of Eq. (11), that was obtained fitting numerically a set of values of $t'_g = t_g/(L^2/\pi^2 D)$ for different values of p'. This set of values was in turn obained from Eq. (7) by choosing different values of $0 < p' < 1$, and calculating corresponding values of t'_g, that satisfied the condition $T'(L,t'_g) = [1 + T'(L,0)]/2$.

The use of Eqs. (10) and (12) illustrates the simultaneous determination of p' and D, employing only two points (i.e., at $t = 0$ and $t = t_g$) from the rear-face temperature history recorded in a single laser-pulse experiment. Nevertheless, this procedure may not be practical, since the measurement of the rear-face normalized temperature change at $t = 0$ can be quite inaccurate when the direct influence of the incoming radiation on the temperature sensor (thermocouple or radiometer) is important, as was the case in the present work. This annoying effect evidenced by a sharp peak starting a $t = 0$, can be observed in Fig. 2, curve A, showing a typical experimental rear-face temperature curve, for a partially transparent sample.

A more accurate numeric data reduction procedure, that includes the information contained in most of the experimentally recorded rear-face temperature history, was developed. Based on a multidimensional Newton–Raphson iterative algorithm, and programmed on a digital computer, the procedure varies simultaneously both p' and D, regarded as adjustable parameters, fitting Eq. (9) to all the experimental data, except the early portion of the recorded curve, that has been distorted by direct action of the incoming radiation on the temperature sensor. Although algorithms for least-squares estimations of nonlinear parameters are generally known,[7] a brief outline of the procedure used here fol-

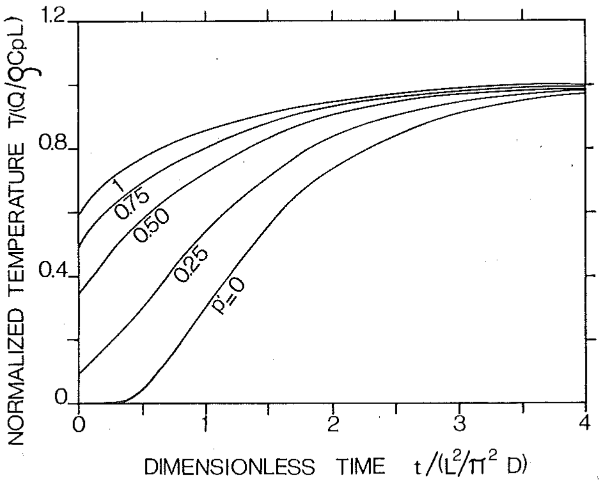

FIG. 1. Rear-face normalized-temperature history of a partially transparent sample, following the exponential absorption of an instantaneous radiation pulse, calculated employing Eq. (7) for different values of the absorption depth p'.

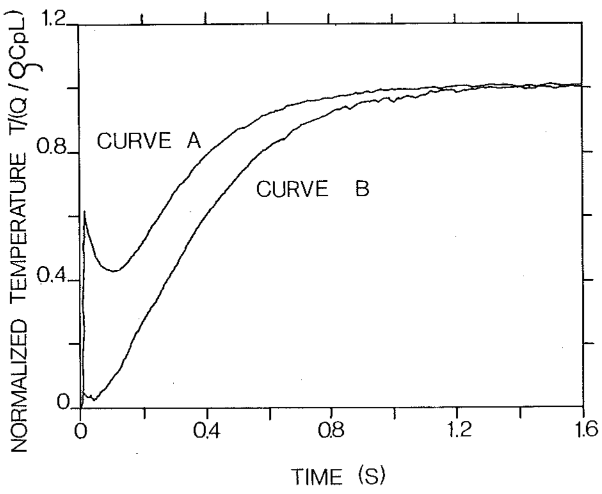

FIG. 2. Experimental rear-face normalized-temperature history. Curve A: a partially transparent sample; curve B: same sample with a thin gold coating on the front face, that absorbs all the incoming radiation.

lows. For every experiment (one laser pulse) the corresponding experimental points (~900) are stored as a digitalized array of normalized-temperature-change values, for equally spaced instants of time. Out of these, a set of approximately 100 points are actually used for the analysis, in order to cut on computing time. These points result from averaging several neighboring points of the original data collection, a process that has to be done with care in order not to distort the recorded curve, but at the same time eliminate unwanted noise. Starting from an initial guess of p' and D, calculated from Eqs. (10) and (12), the computer program calculates the values of $T'(L,t)$ from Eq. (9), and their first derivatives with respect to p' and to D. New values of these parameters are then computed by solving the two equations that result from equating to zero the derivatives with respect to p' and D, of the sum of weighted squares of the differences between the measured and calculated rear-face temperatures, i.e.,

$$\frac{\partial\chi^2}{\partial p'}=0, \quad \frac{\partial\chi^2}{\partial D}=0, \tag{13}$$

$$\chi^2 = 1/(N-2)\sum_{i=1}^{N} 1/(\Delta T'_i)^2[T'(L,t_i)-T'_m(L,t_i)]^2, \tag{14}$$

where N is the number of data points fitted, $\Delta T'_i$ is an estimate of the uncertainty of data point i corresponding to time t_i, and the subscript "m" stands for "measured."

The necessary linearization of the two Eqs. (13) is performed by expanding them in a Taylor series about the initial values of the parameters, keeping only linear terms, as well as dropping terms containing second-order derivatives. In this sense the procedure becomes a multidimensional Newton–Raphson iterative algorithm. Values of $\chi^2 < 1$ indicate acceptably good data fits.

Due to the fact that Eqs. (10) and (12) provide a good initial guess for p' and for D, convergence is rather fast (typically four or five iterations), and little doubt is left about having obtained the physically unique searched-for solution, having avoided a false local minimum that could be caused by inaccuracies in the measured data.

EXPERIMENTAL VERIFICATION

An experimental verification of the presently proposed method was performed employing a laser-pulse thermal diffusivity apparatus developed at INTI.[8] It consists basically of a pulsed glass/Nd laser constructed at INTI, that provides 0.5-ms radiation pulses, with an energy of more than 2 J per pulse. The pulse travels horizontally, and impinges upon the front face of the sample, where a sufficient amount of the radiation is absorbed. The sample is supported in a quasiadiabatic manner by means of spring-loaded thermocouple wires of 0.1 mm diameter (Fig. 3), within a vacuum-tight fused-quartz tube. The quartz tube is in turn within a tubular furnace, that permits heating up the sample to different initial temperatures. For most industrial work that does not require high accuracy, or in case of low thermal diffusivities, the same thermocouple wires that support the sample are used in order to sense the rear-face temperature. In that case, thermal contact between the thermocouple and the sample is improved by means of a tiny amount of conductive paste. In case of more accurate work, or high thermal diffusivity materials, or very thin specimens, a special thermocouple of 0.025 mm diameter is pasted to the rear face of the sample. The thermocouple signal is amplified by means of a low-noise amplifier, digitalized and stored with a microprocessor-controlled data-acquisition system. The data reduc-

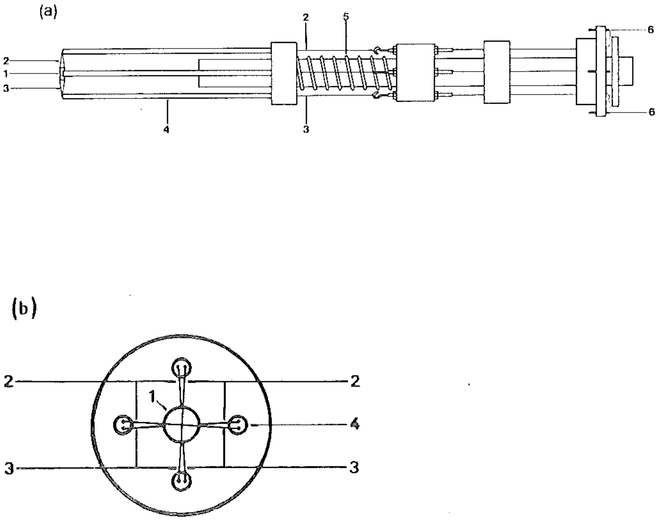

FIG. 3. (a) Schematic view of the sample holder for thermal diffusivity measurements, showing the spring loaded 0.1-mm-diam thermocouple wires that simultaneously support the sample and measure its rear surface temperature. (b) Front view of the sample holder. (1) Sample, (2) chromel, (3) alumel, (4) twin-bore alumina insulators, (5) spring, (6) pins connecting thermocouple legs to the amplifier.

tion procedure described above is then performed, following the transfer of the data to a large computer, to which the system is interfaced.

The overall accuracy of thermal diffusivity measurements carried out with this system, was verified by measuring samples of Standard Reference Materials provided by the National Bureau of Standards (NBS-SRM). These samples of tungsten, stainless steel, and electrolytic iron, covering thermal diffusivity values from 0.037 to 0.7 cm^2/s, were measured with an accuracy better than 5%, and a repeatability better than 1%, for temperatures from 20 to 500 °C.

Since transparent reference samples of known properties were not available, in order to test the procedure here developed for partially transparent materials, a 1-mm-width by 10-mm-diam specimen of sintered glass was measured under two different conditions. First it was irradiated in its original state, recording a rear-face temperature history like the one shown by curve A, Fig. 2. This curve indicates a significant amount of partial transparency, evidenced by the sharp peak recorded right after the laser's pulse ($0 < t < 0.17$ s), caused by direct heating of the thermocouple junction by part of the incoming radiation. This unwanted heating is rapidly dissipated due to the small mass of the thermocouple, and after a short time the recorded signal reflects essentially the sample's rear-face temperature. Employing the data reduction procedure described above, applied to the data points corresponding to times larger than 0.17 s, and assuming the reflection parameter $R = 0$ the resulting values of thermal diffusivity and of absorption depth were

$$D = 0.0043 \pm 0.0001 \text{ cm}^2/\text{s},$$

$$p = 0.0298 \pm 0.0007 \text{ cm}, \tag{15}$$

where the stated uncertainties represent the standard deviation corresponding to a single measurement, calculated from a set of 23 laser-pulse measurements. These uncertainties estimate therefore only the repeatability of the measurements.

Within the stated repeatability limits ($\sim 2.5\%$) the results of analyzing the same data assuming values of R as large as 20%, produced no significant effect on the thermal diffusivity value, since the calculated changes were smaller than 0.2%. On the other hand, using values of R equal to 3%, 10%, and 20% led to corresponding values of the absorption depth p, equal to 0.0297, 0.0295, and 0.0293 cm, also well within the observed repeatability. This can be understood by realizing that an R % reflectance implies a relative temperature increase of R % (with respect to the case $R = 0$), only at time $t = 0$; a proportion that decreases rapidly as heat from the hotter regions of the sample begins to arrive to the rear face.

The sample's optical transmittance at the laser's wavelength, 1.06 μm, was also measured by means of an independent experiment performed with a spectral radiometer, and a corresponding optical absorption depth of 0.0337 ± 0.0007 cm was calculated from the sample's width, under the assumption of exponential absoption. The latter uncertainty was estimated applying error propagation of the sample's width uncertainty, and of the observed repeatability of the spectral-radiometer measurements. Uncertainty estimates from other error sources were not available. The difference between the absorption depths determined by means of the two independent procedures just described, may be taken as an estimate of the accuracy of the method under discussion. It amounts to about $\pm$ 12% of the absorption depth.

After sputtering a thin gold coating on the sample's front face, it was again irradiated and a temperature history, like that shown by curve B, Fig. 2, was recorded. As expected, curve B did not present any longer the sharp peak appearing on curve A. A new measurement of the coated sample with the spectral radiometer indicated a negligible residual optical transmittance. Applying the data reduction procedure described above (with $p = 0$), to the recorded temperature history, the following value of thermal diffusivity resulted:

$$D = 0.004\,29 \pm 0.000\,03 \text{ cm}^2/\text{s}, \tag{16}$$

where the stated uncertainty is the standard deviation corresponding to a single measurement, calculated from a set of 13 laser-pulse measurements. The fact that in this case only one parameter has to be estimated by the least-squares algorithm, accounts for the smaller standard deviation, when compared with the result given in Eqs. (15). The agreement of the values of thermal diffusivity obtained following both procedures is remarkably good, both within the limits of repeatability given by the stated uncertainties, and within the limits of accuracy of the current state of the art of laser-pulse thermal diffusivity measurements. The data fits were also very good, leading to values of χ^2 of about 0.5, which corresponds to differences of less than 2%, between measured and theoretically predicted rear-face normalized-temperature values.

It is interesting to point out that the effect of partial transparency on the apparent thermal diffusivity of a sample, measured with the laser pulse technique, can be quite important. Had this effect been ignored due to the apparent opaque appearance of the sample under visual inspection, and had curve A on Fig. 2 been fitted considering $p = 0$ (excluding the distorted early portion, $t < 0.17$ s) a value of 0.0064 cm^2/s would have resulted for the thermal diffusivity of the sample, i.e., about 50% higher than the expected value.

It may be expected that the attainable accuracy employing the proposed procedure, will decrease as transparency increases (larger values of p'). The rear-face temperature history becomes flatter (see Fig. 1), implying a worsening signal to noise ratio and therefore less significant fits. From this point of view the use of thicker samples is advisable, with a corresponding increase in incoming laser energy in order to maintain a large temperature excursion.

In order to gather some quantitative insight into this matter, and in support to the above conclusion, computer-simulated runs were performed as follows. Simulated experimental data for chosen values of D and p' were calculated employing Eq. (7), and experimental uncertainties generated with a pseudorandom algorithm following a Gaussian distribution were superimposed. The standard deviation of the simulated uncertainties were chosen to amount to about 2%. Applying the data reduction procedure described above, to these simulated data, for values of $p' = 0$, 0.5, and

1, led to values of thermal diffusivity that were off from the expected value by about 1.5%, 2.5%, and 10%, respectively. Corresponding values of p' were off from expected values by about 0.001 in the case of $p' = 0$, and about 2% and 7% in the cases of $p' = 0.5$ and 1, respectively. Furthermore, the simulated 2% noise level is comparable to the observed noise during the present experiments, and these results are consistent with the observed repeatability, corresponding to $p' \simeq 0.3$.

CONCLUSIONS

The results presented in the preceding section indicate a very good agreement between the values of thermal diffusivity that result either from following the usual procedure of building an opaque layer on the front surface of a transparent sample, or employing the new data reduction procedure described in this paper. This new method, which essentially takes into account transparency effects by generalizing the mathematical description of the laser-pulse thermal diffusivity experiment, provides as well a value of the sample's optical absorption depth at the laser's wavelength. It also helps to solve a major experimental problem, simplifying preparation of the sample, since it avoids having to coat one of the surfaces with an appropriate opaque material; a task that may be cumbersome, if not impossible for high-temperature measurements, and which may significantly perturb the sample's properties through chemical reaction or contamination.

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