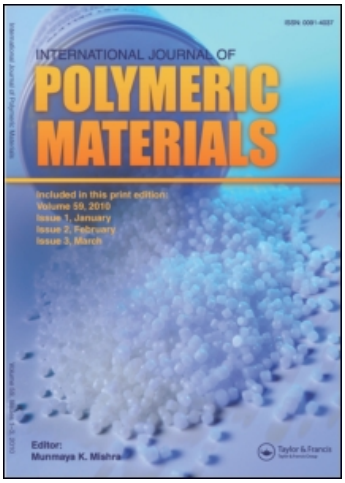

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Self-normalized photoacoustic thermal diffusivity measurements of dental resins

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SELF-NORMALIZED PHOTOACOUSTIC THERMAL DIFFUSIVITY MEASUREMENTS OF DENTAL RESINS

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A new self-normalized photoacoustic technique, using the Open Photoacoustic Cell configuration, to carry out measurements of thermal diffusivity of materials is presented. This new methodology involves a linear fitting procedure of the photoacoustic amplitude signal. To carry out the analysis one requires the ratio of signal amplitudes from two samples of the same material and of different thicknesses. This rationing procedure leads to the elimination of the usual requirement for instrumental transfer-function normalization. The thermal diffusivity for three dental resins is measured with this simple methodology and very good agreement is found with values for similar materials reported by means of more complicated techniques.

Keywords: photoacoustic thermal diffusivity, methodology, dental resins

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I. INTRODUCTION

Photothermal techniques have found extensive applications in various areas of science and engineering. Nowadays they are used in a wide range of scientific disciplines to carry out studies of diverse properties of condensed matter [1–3]. Among the main applications of these techniques, widely used is the thermal characterization of substances, both in liquid and solid phases [4–10]. In particular, the Open Photoacoustic Cell (OPC) configuration is becoming increasingly popular [9–12].

In this article a new measurement methodology based on the OPC configuration and using the photoacoustic signal amplitude as a function of the modulation frequency is reported. A frequency scan is carried out in the photothermally thick regime [13]. The procedure involves implicit signal normalization by taking the ratio of the photoacoustic amplitude frequency responses of two samples of different thickness. With this self-normalization procedure the instrumental transfer function is eliminated. The new methodology is applied to the measurement of the thermal diffusivity of three dental resins.

II. THEORY

In consideration of the optically opaque (surface-absorption) limit [13] it can be shown that the complete photoacoustic signal in the OPC configuration is given by the expression [11]

$$\delta P = \frac{Y}{\sigma_g \sigma_s} \frac{1}{\sinh(L\sigma_s)} \left[1 + \frac{2A}{\sigma_s} \left\{ \cosh(L\sigma_s) - \frac{L\sigma_s}{2} \sinh(L\sigma_s) - 1 \right\} \right] \qquad (1)$$

In this equation Y is a coefficient involving geometrical parameters, as well as optical and thermal properties. The parameter A involves the linear expansion coefficient α_T, its magnitude determine the thermoelastic behavior of the sample. L is the sample thickness, and $\sigma_j = (1+i)\sqrt{(\pi f/\alpha_j)}$, $j = g, s$, is the complex thermal diffusion coefficient, where f is the optical-source modulation frequency and α_j is the thermal diffusivity of the j-th substance. The letter g refers to the gas inside the photoacoustic chamber; s refers to the optically absorbing sample.

Equation (1) is too complicated to be used for analytical purposes. More useful are some approximations based on the value of the dimensionless parameter $x = (f/f_c)^{1/2}$, where $f_c = \sqrt{(\alpha_s/\pi L^2)}$ is the critical frequency. This is the frequency at which the thermal diffusion length $\mu_s = |\sigma_s|/\sqrt{2}$ is equal to the thickness of the sample. Depending on the value of the parameter x, the photothermal behavior of the sample lies between the thermally thin ($x << 1$)

and the thermally thick regime ($x >> 1$). Furthermore, depending on the thermal linear expansion coefficient and thickness of the sample (included in the parameter A in Eq. (1)), a thermoelastic contribution may appear at high modulation frequencies in the thermally thick regime [12]. If this contribution can be separated out at higher frequencies, as is the case with our experiments, it may be theoretically ignored. Then the OPC amplitude, Eq. (1), is simplified substantially in the thermally thick regime [9–11]

$$\delta P_{TG}(f) = c(f)\frac{e^{-a\sqrt{f}}}{f} \tag{2}$$

Here, the coefficient $c(f)$ contains the instrumental transfer function and is independent of the samples thickness. The parameter a is defined as $a = L(\pi/\alpha_s)^{1/2}$. By taking the ratio of Eq. (2) with itself for two different sample thicknesses L_1, L_2, it is clear that the term $c(f)$ is eliminated, yielding the simpler expression

$$R = \frac{\delta P^2_{TG}}{\delta P^1_{TG}} = e^{-B\sqrt{f}} \tag{3}$$

The parameter B is defined as $B = (L_2 - L_1)(\pi/\alpha_s)^{1/2}$. It is clear that the thermal diffusivity of the sample can be obtained from knowledge of this parameter. The measurement procedure involves rationing the photoacoustic amplitudes obtained in transmission experiments from two samples of different thicknesses and plotting *vs.* the square root of the modulation frequency, followed by the corresponding linear best-fit on a semi-log scale to obtain the slope of the straight line.

III. EXPERIMENTAL

The experimental set up is shown in Figure 1. It consisted of an infrared laser diode source with a fiber-optic pigtail (Omnicrome Corporation, model OPC-A00l-FC/100, 830 nm), operating at 200 mW. The photoacoustic transmission configuration was realized by using the sample to cap (and hermetically seal) the chamber of a commercial electret microphone with a built-in pre-amplifier. The photoacoustic signal due to the front-surface-generated thermal wave transmitted through the sample was obtained as a function of the modulation frequency. It was then fed to a lock-in amplifier, LIA (Stanford Research model SR830) for further amplification and demodulation. The laser intensity was modulated by using the internal oscillator of the LIA to drive the laser power supply *via* a TTL communication port.

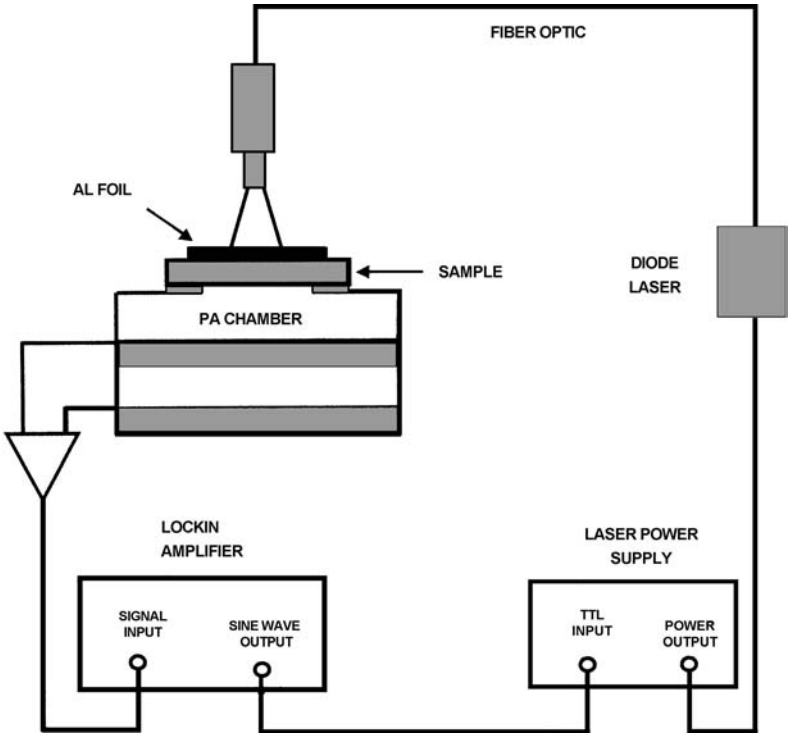

FIGURE 1 Schematic representation of the OPC configuration experimental set-up used for self-normalized thermal diffusivity measurements.

IV. RESULTS AND DISCUSSION

The methodology was applied to the measurement of the thermal diffusivity of three dental resins (Tab. 1). The resin samples were prepared in a disk form (1 cm in diameter) with thicknesses between 80 to 200 μm (Tab. 1). To satisfy the surface absorption condition, a piece of aluminum foil (17 μm-thick) was attached with thermal paste to the various specimens. The thermal thickness of the foil was thus negligible compared to the sample thicknesses. Figure 2 shows typical signal amplitudes for the studied dental resins. Figure 3 shows the graph of the ratio of the two amplitudes (thick sample: numerator; thin sample: denominator) on a semi-log scale as function of the square root of modulation frequency for one of the resins. The decreasing ratio in the low-frequency portion of this graph is due to the fact that both samples are thermally thick and the thermal-wave attenuation rate of the photoacoustic signal amplitude from the thick solid is faster than that of the thin solid. This makes the amplitude ratio decrease. This behavior is followed by the

TABLE 1 Thermal diffusivities for three dental resins, measured by using the presented methodology and the corresponding values for three similar dental resins reported in the literature

Dental resin (*Commercial name*)	*Thickness* cm ($\times 10^{-2}$)	$\Delta L = L_2 - L_1$ cm ($\times 10^{-2}$)	α cm^2/s ($\times 10^{-2}$)
3M	$L_1 = 1.39 \pm 0.04$ $L_2 = 1.87 \pm 0.05$	0.48 ± 0.09	0.45 ± 0.09
Degufill H	$L_1 = 0.86 \pm 0.03$ $L_2 = 1.70 \pm 0.05$	0.84 ± 0.08	0.34 ± 0.06
Degufill (Auto)	$L_1 = 0.90 \pm 0.05$ $L_2 = 1.70 \pm 0.03$	0.80 ± 0.08	0.09 ± 0.07
Bonfill	–	–	0.12 ± 0.02 [15]
Adaptic			0.68 ± 0.03 [15]
Prisma-Fil Dentsply	–	–	0.50 ± 0.02 [16]

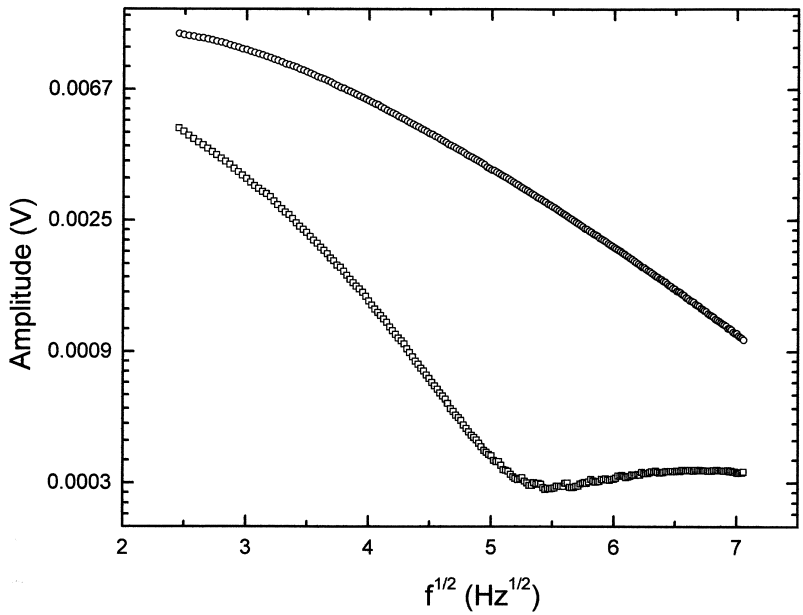

FIGURE 2 Typical photoacoustic signal amplitude for the resins used in this work. Circles correspond to a Degufill (auto) sample resin (0.0090 cm thickness); squares correspond to another Degufill sample (0.0170 cm thickness).

thermoelastic domination of the signal at high modulation frequencies [14]. Here the oscillatory thermal expansion of the thin sample is proportionally smaller than that of the thick sample ($\Delta L_j \propto L_j$). As a result, the acoustic

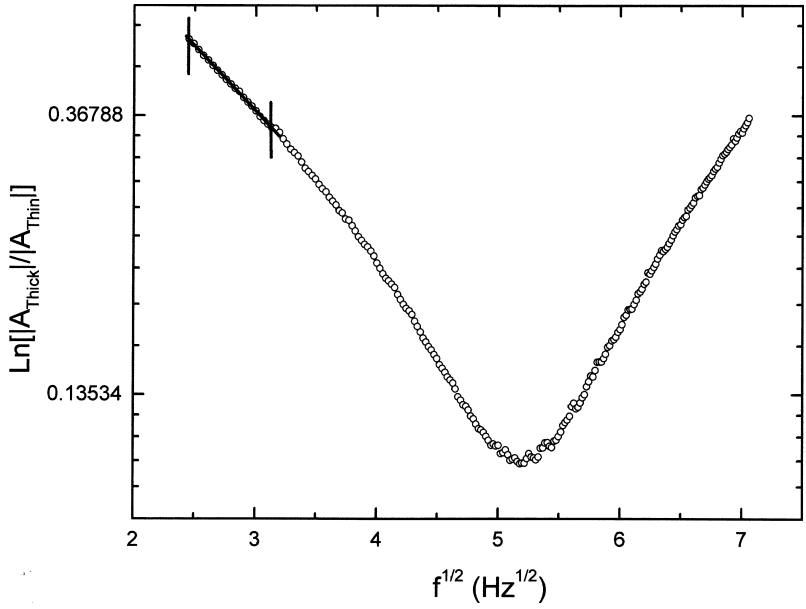

FIGURE 3 Photoacoustic signal amplitudes ratio for the Degufill (auto) resins samples of Figure 2. The linear behavior in the thermally thick regime is delimited by two vertical lines. The continuous straight line is the best fit to Eq. (3) within this region.

vibration amplitude in the photoacoustic chamber is stronger for the thick sample and the ratio reverses its direction. The turning point of the curve in Figure 3 can be used, in principle, to determine the coefficient of thermal expansion of the material constituting the two samples.

The linear behavior in the thermally thick region is as predicted from Eq. (3). The superposed straight-line represents the theoretical best fit to the same equation. The results of the analysis are summarized in the Table 1. A comparison of the thermal diffusivity values measured in this work with corresponding values reported in the literature [15, 16] for similar dental resins reveals good agreement (see Tab. 1). The simplicity of the presented technique is evident as is compared with the methodologies used to obtain the values reported in Refs. [15] and [16]. One of this methodologies (Ref. [15]) involves measurements of the temperature evolution on a rectangular prism molded specimen, with a thermocouple embedded in it as immersed in a thermal bath. The other methodology (Ref. [16]) is a photoacoustic technique using the phase delay signal and involves a complicate normalization procedure to eliminate the other mechanism taken place together with the thermal diffusion mechanism. It is worth to say that all of the

described methodologies has 2 digits of precision, however the precision of the presented methodology can be improved by suppressing the thermoelastic contribution. For to do this a compromise with the sample's thickness is required.

V. CONCLUSIONS

We have presented a new methodology for measuring the thermal diffusivity of solid materials by using the OPC configuration. This new methodology involves a self-normalization procedure by virtue of which the instrumental transfer function is eliminated. This characteristic represents the major accuracy advantage and simplicity of the analysis over other photoacoustic techniques based on the OPC configuration. In principle, the technique is applicable to all opaque solid materials, provided one is working in the purely thermally thick frequency regime with no thermoelastic overlaps. Two samples of the same material of different thicknesses are needed in order to implement the technique. The extended use of dental resins for restoration and the rapid development of new dental materials make it important to implement simple new methodologies to characterize critical thermal loads in dental practice. Our new methodology appears to be suitable for simple, rapid characterization of dental materials. Toward this goal a detailed statistical study is necessary in order to define the precision and accuracy of this new methodology.

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