Device for characterization of thermal effusivity of liquids using photothermal beam deflection

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We propose and study a novel optoelectronic device for thermal characterization of materials. It is based on monitoring the photothermal deflection of a laser beam within a slab of a thermo-optic material in thermal contact with the sample under study. An optical angle sensor is used to measure the laser deflection providing a simple and experimental arrangement. We demonstrate its principle and a simple procedure to measure thermal effusivity of liquids. The proposed device could be implemented into a compact sensor head for remote measurements using electrical and fiber optic links. © *2007 American Institute of Physics.* [DOI: 10.1063/1.2793501]

I. INTRODUCTION

Several measurement techniques using photothermal phenomena allow for the study of different thermal and optical characteristics of several materials, either in the gas, liquid, or solid state.[1] Commonly, photothermal techniques are based on the induction of heat in a material with high-energy optical radiation and monitoring the induced heat flux process by an appropriate method. Several techniques for photothermal analysis of materials are based on the photoacoustic effect.[2–4] Others are based on the mirage effect or photothermal beam deflection[5–7] and photothermal radiometry.[8,9] Photothermal measurements can be carried out in situations where other techniques are not useful. For example, they can be used to perform spectroscopy in opaque and rough materials.[1]

To date, most measurements based on photothermal techniques are limited to the laboratory. Only a few proposals for compact and rugged instruments suitable for field measurements have been published (see, for example, Ref. 10). We are interested in developing portable instrumentation systems suitable for remote sensing of thermal parameters of materials, in particular, of liquids. The basic thermal characteristics of materials can be summarized by their thermal diffusivity and thermal effusivity.[11] These parameters can be determined in the laboratory using different methodologies, by either the photoacoustic effect, photothermal beam deflection method, or other more conventional optical techniques.[12–14] The opportunity to apply photothermal radiometry for thermal characterization of liquids has been also demonstrated recently.[15]

Photothermal beam deflection methods are very sensitive to monitor heat flux in photothermal related phenomena. The heat flux across an optically transparent material (gas, liquid, or solid) is indirectly determined by measuring the angle of deflection of a laser beam traveling through the material. These methods are usually limited to the laboratory because their implementation involves relatively long distances. The reason is that the angular deflection of the laser beam is measured by triangulation using a position sensitive detector, so that in order to achieve high sensitivity the position detector must be placed far from the deflection zone (typically in the order of one meter).

Compact devices based on photothermal beam deflection could be realized using optical angle sensors. Instead of being sensitive to the position of the spot of a laser, they are sensitive to the angle of the laser beam, thus, long triangulation distances within the arrangement are not required. Moreover, compact and versatile arrangements could be obtained if optical fibers are used for delivering light, either for heat generation through optical radiation or for photothermal beam deflection measurements. Hence, we suppose that the use of an optical angle sensor and fiber optic technology could make the photothermal beam deflection method a suitable candidate for the development of compact and versatile sensors for thermal characterization of materials.

In this work, we propose and study a novel device based on the photothermal beam deflection method. The main idea is to measure optically generated heat flux within a slab of a thermo optic material that is in thermal contact with the material to be characterized in order to perform remote determination of the effusivity of liquids. The paper is organized as follows: In Sec. II we review the basics of the theory of photothermal beam deflection; in Sec. III we briefly discuss the measurement of the optical beam deflection by a recently developed optical angle sensor; Sec. IV describes the experi-

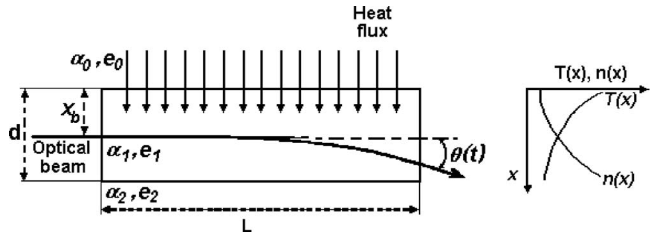

FIG. 1. Geometry of the photothermal deflection in a slab of a transparent thermo-optical material.

mental setup used to test the feasibility of the proposed device and presents the results to date. Finally, Sec. V is devoted to conclusions.

II. HEAT FLUX MONITORING BY PHOTOTHERMAL BEAM DEFLECTION

In the presence of heat flux by conduction there is a spatial gradient of the temperature field T. The refractive index of all materials is a function of temperature. Thus, a spatial gradient of the refractive index n is determined by the heat conduction processes in the material. Some materials are optically transparent and have a relatively large value for the thermo-optical coefficient $\partial n/\partial T$. If a thin optical beam traveling through a region of variable refractive index, the beam deflects. The deflection angle can be related to the gradient of the refractive index and therefore to the heat flux. If the change of refractive index is small over the cross section of the optical beam, the angle of deflection[16] can be approximated by

$$\theta(t) = \frac{1}{n}\frac{\partial n}{\partial T}\int_{\text{path}} ds \nabla_{\perp} T(\mathbf{r},t), \tag{1}$$

where $\nabla_{\perp} T(\mathbf{r},t)$ is the gradient of the temperature field perpendicular to the path of the beam.

In this work, we investigate the application of the photothermal deflection of a laser beam traversing the slab of a thermo-optical material as a sensor for the thermal characterization of liquids contacting with this slab.

A. Photothermal beam deflection in a one dimensional heat flow

Let us assume a one dimensional heat flow in which the temperature field varies only along the x direction and is constant in the x-z plane (see Fig. 1). This case corresponds to the temperature field within a half of the slab of material. If an optical beam is sent parallel to the surface of the material, it deflects towards the coldest region that is in the direction of the heat flux.

In photothermal techniques the heat is supplied by optical irradiation. For transparent materials the heat generation implies the addition of an optically absorbing thin coating. In several of the well established photothermal techniques used for material testing, the supplied heat is modulated in time.[1] In practice, one may use an intensity modulated laser or a mechanical chopper. If the heat source varies harmonically in time, it generates monochromatic thermal waves in the material, which greatly simplifies the analysis of the heat flow. Also, some techniques use a Dirac-pulse type of a heat source. These techniques are usually realized in practice by using pulsed lasers. For sensor development purposes, it is convenient to restrict the heat source to step pulses. In this case one needs only to switch on and off a continuous wave optical source.

Thus, let us consider the heat flow within the slab of a material caused by a step pulse applied to one of its surfaces. Let us consider a material with thermal diffusivity α_1 and thermal conductivity k_1 filling up the half space $x>0$. If a constant heat flux Q is applied to the surface (at $x=0$) and time $t=0$, the derivative of the temperature field with respect to x ($T_x \equiv \partial T/\partial x$) is given by (see Appendix)

$$T_x(x,t \geqslant 0) = -\frac{Q}{k_1}\left[1 - \operatorname{erf}\left(\frac{x}{2}\sqrt{\frac{1}{\alpha_1 t}}\right)\right]. \tag{2}$$

The angle of deflection induced on a thin optical beam propagating within the half space $x>0$ is obtained by using Eqs. (2) and (1).

In a practical device we must have a slab of thermo-optic material of length L and a finite thickness d. The thermal properties of the material in contact with the slab will modify the heat flux field with respect to that described by Eq. (2). The thermal parameters involved in the heat flux in the presence of interfaces of different materials are the thermal diffusivity α and thermal effusivity e. These parameters are related to the thermal conductivity k, density ρ, and heat capacity c of the material by

$$\alpha = \frac{k}{\rho c} \quad \text{and} \quad e = \sqrt{\rho c k}. \tag{3}$$

B. One dimensional heat flow in a slab of finite width

Let us consider a slab of a transparent thermo-optic material of length L and width d as shown in Fig. 1. Let us also suppose that the slab has thermal diffusivity and effusivity α_1 and e_1, respectively, and that it is in thermal contact at $x=0$ with a material of thermal diffusivity and effusivity α_0 and e_0. At the surface $x=0$, a constant heat source is turned on at $t=0$ and turned off at a later time $t=t_{\text{off}}$, whereas at the surface $x=d$ the thermal contact takes place with the material of thermal diffusivity and effusivity α_2 and e_2, as shown in Fig. 1. We will also assume that an optical beam propagates parallel to the slab axis at a distance x_b from the heated surface ($x=0$).

Let us suppose that the heat flux generated by the source is uniformly distributed over the surface of the slab at $x=0$. If $L \gg d$, we can approximate the heat flow as a one dimensional flow within the slab, except near the side walls of the slab. In this work only the normal component of the photodeflection signal is considered. In this case Eq. (1) can be simplified. For a small optical beam deflection it can be represented in the following form:

$$\theta(t) \approx \frac{L}{n}\frac{\partial n}{\partial T}\frac{\partial T}{\partial x}. \tag{4}$$

The temperature field and its derivative with respect to x due to a step pulse applied at the surface of the slab will be modified from the semi-infinite medium because of the reflections between the two interfaces of the slab. As can be seen in the appendix, the T_x field due to a heat step at $t=0$ is

$$T_x^S(x,t) = -\frac{Q}{k_1}\sum_{n=0}^{\infty}(R_2R_1)^n\left[1-\mathrm{erf}\left[\frac{(2nd+x)}{2}\sqrt{\frac{1}{\alpha_1 t}}\right] - R_2\left(1-\mathrm{erf}\left\{\frac{[2(n+1)d-x]}{2}\sqrt{\frac{1}{\alpha_1 t}}\right\}\right)\right], \quad (5)$$

where R_1 and R_2 are the reflection coefficients of thermal waves at $x=0$ and $x=d$, respectively, and the superscript S is used to indicate that a step-on pulse is considered. The reflection coefficients can be written explicitly as

$$R_1 = \frac{1-b_{10}}{1+b_{10}} \quad \text{and} \quad R_2 = \frac{1-b_{12}}{1+b_{12}}, \quad (6)$$

where $b_{10}=e_0/e_1$ and $b_{12}=e_2/e_1$.

Now, if the heat source is turned off at $t=t_{\mathrm{off}}$, we may think that a thermal step-on pulse of a negative amplitude is added at $t=t_{\mathrm{off}}$. Then the solution for a step pulse of finite duration is

$$T_x(x,t) = T_x^S(x,t) - U(t-t_{\mathrm{off}})T_x^S(x,t-t_{\mathrm{off}}), \quad (7)$$

where $U(t)=1$ for $t>0$ and $U(t)=0$ for $t\leq 0$.

The photothermal deflection of the laser beam across the slab is then obtained using Eqs. (4) and (7).

If we keep all parameters fixed except of the external medium, that is medium 2, we can consider $T_x(x,t)$ as a function of e_2 only, i.e., a function of the thermal effusivity of the external medium. Thus, if the slab, heat source, laser beam, and detection electronics are built within a single arrangement, this scheme can be used to measure and sense the effusivity of a material by simply bringing it into thermal contact with the slab.

III. MEASUREMENT OF PHOTOTHERMAL BEAM DEFLECTION

We are interested in developing an inexpensive and compact instrument based on the scheme described above. A key element towards this goal is the beam deflection sensing method. Photothermal deflection techniques used in the laboratory commonly use position-sensitive detectors and sense the beam deflection by triangulation. However, for high sensitivity, a long distance between the detector and the deflection zone is required. Recently, an angle-sensitive device that offers a comparable sensitivity to the maximum possible sensitivity of the triangulation scheme has been proposed.[17] The main advantage of these type of device is that it can be placed as close as possible to the deflection zone and maintain the highest sensitivity to angle deflections.

The optical deflection sensor consists of a film resonator formed by a thin air gap between two optical prisms. The laser beam enters the angle-sensitive device and is partially reflected and transmitted at the film resonator as depicted in Fig. 2. The power carried by the reflected and transmitted beams is a function of the angle of incidence of the input beam. Therefore, registering the power of either the reflected or transmitted beam can be used to sense microdeflections of the laser beam. The reflected and transmitted beams are complementary, that is, the sum of their optical power for any angle of incidence is constant and equal to the optical power of the incoming beam. The reflected and transmitted beams can both be registered by independent photodetectors and the output signal could be defined as the difference divided by the sum of the two signals, in analogy to position sensitive detectors. However, for simplicity in this work we used only the reflected beam for beam deflection sensing. We measured the optical power of the reflected beam with a single photodetector. The output signal (in volts) was registered by a computer using a commercial voltmeter. The sensitivity and range of measurement depends on the film resonator width and on the angle of divergence of the laser beam (assumed to be a gaussian beam). Details can be found in Ref. 17.

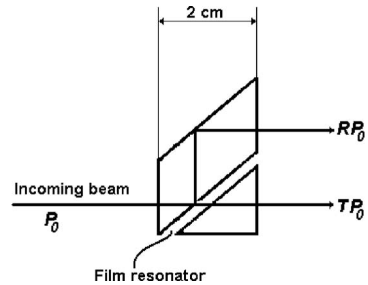

FIG. 2. Schematic layout of the film resonator angle-sensitive detector. R and T are the reflected and transmitted beams, respectively.

We assembled an angle sensor with two prisms made of BK7 glass forming an optical resonator whose length was about 2.8 cm. A special mechanical mount was fabricated in order to hold and adjust the resonator width. The angle sensor was adjusted for maximum sensitivity for the particular laser beam used in our experimental setup. In Fig. 3 we show the output signal as a function of the deflection angle of the probe laser beam. The range of angle variation in the graph essentially exceeds the values of the deflection angles in our photothermal experiments. Therefore, these results can be used for selection of the resonator working point and calibration purposes in experiments described in the next section.

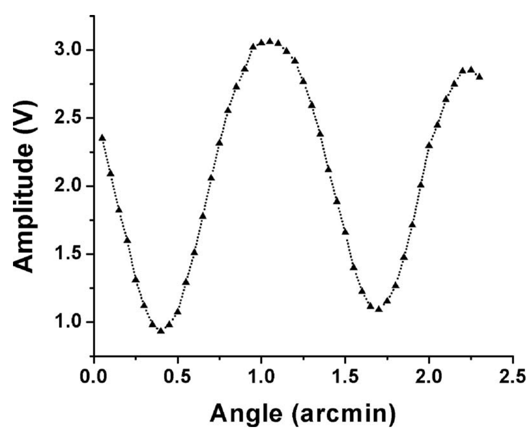

FIG. 3. Normalized response of the angle sensor to a laser diode input beam.

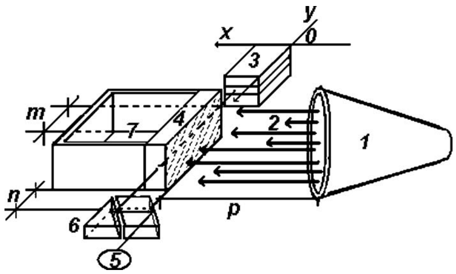

FIG. 4. Experimental setup; (1) 75 W lamp, (2) incident radiation, (3) laser diode, (4) slab of acrylic, (5) photodetector, (6) angle-sensitive detector, and (7) liquid container.

An interesting possibility in our device would be to deliver the probe optical-beam with an optical-fiber. To this end, we tested the angle-sensor response to an optical beam delivered through a collimator coupled to a standard multimode optical fiber (50 μm core diameter) and obtained similar curves. However, a noticeable reduction in angle sensitivity and additional noise appeared owing to the multimode character of the output beam. Future work will be devoted to achieve better sensitivity using single-mode fiber devices.

IV. EXPERIMENTAL SETUP AND RESULTS

The experimental results obtained in this work were carried out in the laboratory using the setup schematically shown in Fig. 4. It consists of a 75 W incandescent lamp (1), a 1 mW laser diode emitting a beam of circular cross section (3), a thick slab of acrylic with dimensions of $66\times30\times14$ mm^3 (4), photodetector (5), the assembled angle sensor (6), and a liquid container (7). The laser beam is sent through the center of the slab (4) as shown in the figure and is detected by the angle sensor. A black coating was added to the external surface of the acrylic slab to absorb the radiation from the incandescent lamp and generate a heat flux across the slab.

The heat flux generated could be controlled to some degree by changing the distance p between the lamp and the surface of the slab. The choice of the distance was based on two reasons. The first one is to provide the uniform illumination of the region of measurement. The second reason is to restrict the intensity of the illumination to prevent convection processes in liquids. Based on these reasons, the distance p in our experiments was not less than 10 cm.

The distance from the end face of the slab to the angle sensor could be reduced as much as desired without loosing sensitivity. As already mentioned, for simplicity, we only used one detector and registered the reflected beam from the resonator to sense angle deflections of the probe laser beam. The output signal from the photodetector was registered in the computer using a six digit voltmeter.

Two types of experiments were carried out. In the first case, the lamp was turned on and the photothermal deflection signal was registered for 800 s. We will refer to this experiment as a "heat step experiment." In the second type of experiments, the lamp was turned on for 240 s and then turned off, registering the photothermal signal during 800 s. We refer to this type of experiment as a "heat pulse experiment."

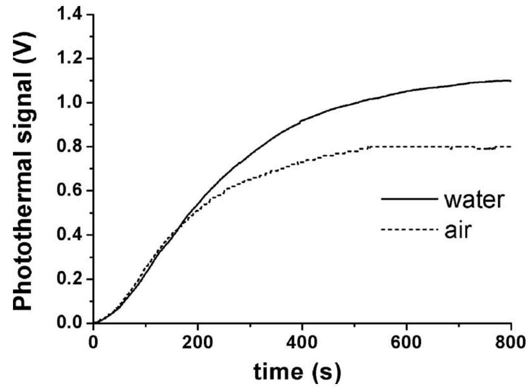

FIG. 5. Heat-step experimental results for the acrylic slab.

Some of the curves for both types of experiments are shown in the following figures. In these figures we plot the photothermal signal versus time. By photothermal signal we mean the change of the output-voltage at the photodetector. This is proportional to the change in angle of the probe beam as long as this is a small fraction of 1 arc min (see Fig. 3).

For comparison, we also plot the curves for T_x at the center of the slab versus time predicted by Eq. (7). For the theoretical curves we assumed the following parameters: $\alpha_{\text{acrylic}}=0.11\times10^{-6}$ m^2 s^{-1}, $e_{\text{acrylic}}=150$ W s$^{1/2}$ m^{-2} K^{-1}, $e_{\text{air}}=5.5$ W s$^{1/2}$ m^{-2} K^{-1}, $e_{\text{water}}=1500$ W s$^{1/2}$ m^{-2}K^{-1}, and $e_{\text{oil}}=500$ W s$^{1/2}$ m^{-2} K^{-1}.

In Fig. 5 we show the results of a heat step experiment for air and water in the container and for $p=14$ cm. In Fig. 6 we plot the curves predicted by Eq. (7). It can be noticed that the theoretical curve of the x-component of the gradient of the temperature field reproduces qualitatively the behavior of the experimental curves for both cases, i.e., for air and water. Notice that the photothermal signal indicates the propagation of the heat flux from the irradiated surface of the slab towards the colder surface. The difference between the experimental curves for both cases (air and water) allows for the calculation of the effusivity. We can estimate from this experiment that one requires a minimum of time to differentiate between the effects of the effusivities of air and water. In this particular setup this value is about 300 s.

Figure 7 shows the results obtained for the same case as in the previous figures but based on the heat pulse experi-

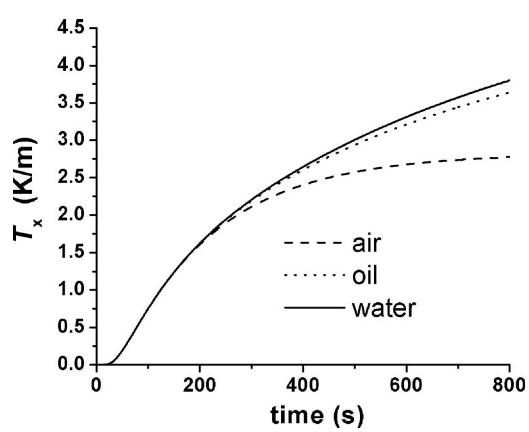

FIG. 6. Theoretical results for the heat step process in the acrylic slab.

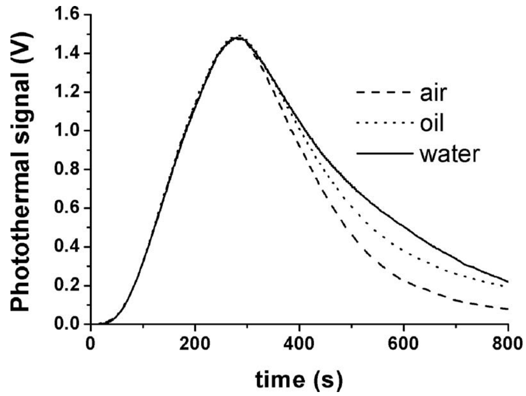

FIG. 7. Heat pulse experimental results for the acrylic slab.

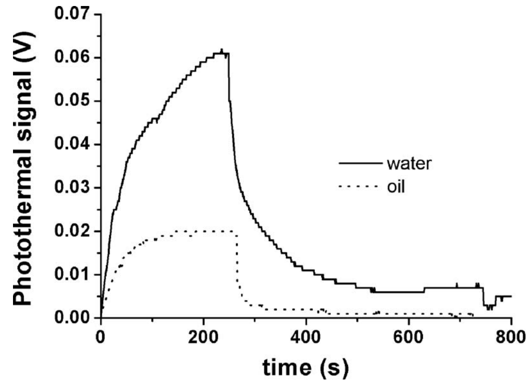

FIG. 9. Heat pulse experimental results.

ment. In Fig. 8, the theoretical model shows an immediate decrease in the field temperature as the irradiation is turned off. A good correspondence is observed between the experimental and theoretical curves. In this case the effect of the different effusivities is also clearly noticeable.

Next, we describe the case of a glass slab $(52\times 42 \times 8\ \mathrm{mm}^3)$ as the thermo-optic material. Figure 9 shows the results for a heat pulse experiment using oil and water in the container. The theoretical model differs from the experimental curves in the relative response between oil and water (see Fig. 10). The calculations were made for the glass with the thermal diffusivity and effusivity $\alpha_{\mathrm{glass}}=0.87\times 10^{-6}\ \mathrm{m}^2\ \mathrm{s}^{-1}$ and $e_{\mathrm{glass}}=1502\ \mathrm{W\ s}^{1/2}\ \mathrm{m}^{-2}\ \mathrm{K}^{-1}$. Also, the response of the photothermal deflection is much smaller than that obtained for the acrylic slab. The reason is that the thermo-optical coefficient of the glass is smaller than that of the acrylic. Unfortunately, the experimental curves for air were not reproducible and therefore they are not shown; we attribute this to a strong contribution from heat convection.

V. SUMMARY AND CONCLUSIONS

We proposed a novel heat flux sensor based on the photothermal beam deflection of a laser beam within a slab of a thermo-optical material and an optical angle sensor. The introduction of the optical angle sensor in the device allows for a compact optical arrangement. This fact, together with the use of fiber optics to deliver the pump light, may allow to implement all the elements of the device into a compact form. Remote measurements may be possible and would require a fiber optic link for light delivery and electrical links for the photodetectors. We implemented an experimental prototype in the laboratory for preliminary testing and demonstrated the feasibility of such a device. In the laboratory, we used the laser beam directly from a laser diode. Our first attempt to replace the direct laser beam by the output beam from a fiber optic collimator showed a strong loss of sensitivity owing to the multimode propagation through the fiber. Future work will include the use of single-mode fiber devices in order to achieve adequate sensitivity.

We investigated the possibility of determining the thermal effusivity of a liquid in contact with one side of the thermo-optic slab of the device. A simple procedure based on applying a heat step pulse to the slab by optical irradiation on the other side of the slab and monitoring the heat flux transient has been proposed. Preliminary results showed that the photothermal beam deflection signal as a function of time can be used to distinguish liquids with different thermal effusivity.

A simple theoretical model of the photothermal beam deflection signal as a function of time was given and a good qualitative correspondence with the experimental curves was found. Using the theoretical model it may be possible to optimize the device parameters to measure thermal effusivities of liquids or gases. However, it will be necessary to consider the possible contribution of the heat flux by convection in the case of gases. With this arrangement, the device

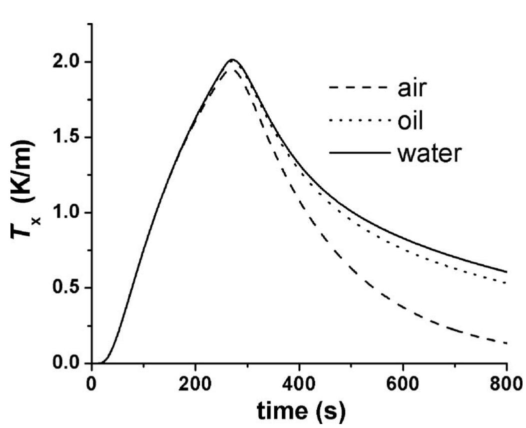

FIG. 8. Theoretical results for the pulse process in the acrylic slab.

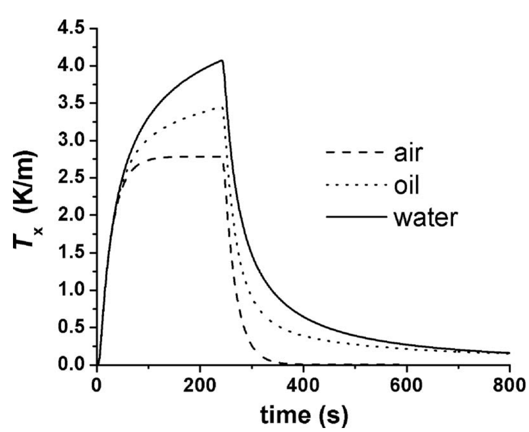

FIG. 10. Theoretical results for the heat step process in the glass slab.

may be advantageous also in determining the heat transfer coefficient of gases. Further work will be devoted to other applications in this field.

ACKNOWLEDGMENTS

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APPENDIX: THEORETICAL MODEL

Let us first consider the temperature field due to a plane heat step pulse applied to the surface of a medium occupying the half-space $x>0$. The heat source at $x=0$ is

$$Q_s(t)=\begin{cases} Q & \text{for } t\geqslant 0\\ 0 & \text{for } t<0.\end{cases}$$

Let us assume the material has a thermal diffusivity α_1 and a thermal conductivity k_1. The temperature field satisfies the diffusion equation,

$$\frac{\partial^2 T}{\partial x^2}-\frac{1}{\alpha_1}\frac{\partial T}{\partial t}=0. \tag{A1}$$

The heat flux q at any point within the slab is given by

$$q=-k_1\frac{\partial T}{\partial x}\equiv -k_1 T_x. \tag{A2}$$

Differentiating Eq. (A1) with respect to x gives

$$\frac{\partial^2 T_x}{\partial x^2}-\frac{1}{\alpha_1}\frac{\partial T_x}{\partial t}=0. \tag{A3}$$

Thus the heat flux also satisfies the diffusion wave equation. We have the following boundary conditions:

$$T_x(x,0)=0, \tag{A4}$$

$$T_x(0,t)=\begin{cases} -\dfrac{Q}{k_1} & \text{for } t\geqslant 0\\ 0 & \text{for } t<0.\end{cases} \tag{A5}$$

The solution for a semi-infinite medium of this boundary value problem is in Ref. 18,

$$T_x(x,t\geqslant 0)=-\frac{Q}{k_1}\left[1-\operatorname{erf}\left(\frac{x}{2}\sqrt{\frac{1}{\alpha_1 t}}\right)\right], \tag{A6}$$

where $\operatorname{erf}(x)$ is the error function,

$$\operatorname{erf}(s)=\frac{2}{\sqrt{\pi}}\int_0^s \exp(u^2)du.$$

Now, let us consider the case of a slab of a thermo-optic material of width d between two semi-infinite media as shown in the figure. We will number the three media from right to left as medium 0, 1, and 2, with thermal diffusivities and effusivities α_0, α_1, and α_2 and e_0, e_1, and e_2, respectively, as shown in the Fig. 11.

We place the origin of our coordinate system at the surface of the slab, that is, at $x=0$. The temperature field and the heat flux along the x-axis satisfy the diffusion equation. Now, however, we must satisfy additional boundary conditions at $x=d$.

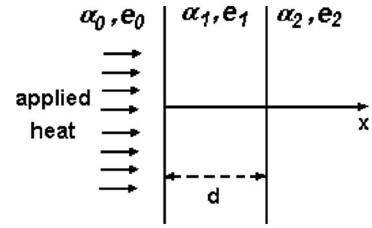

FIG. 11. Geometry considered in the theoretical model.

One way of doing this is to use the concept of thermal waves. We may regard the step pulse as the superposition of monochromatic thermal waves. Each thermal wave is reflected back and forth from the two interfaces: $x=0$ and $x=d$. The reflection coefficients at the first and second interfaces are

$$R_1=\frac{1-b_{10}}{1+b_{10}} \quad \text{and } R_2=\frac{1-b_{12}}{1+b_{12}}, \tag{A7}$$

where $b_{10}=e_0/e_1$ and $b_{12}=e_2/e_1$, respectively.

Since this reflection coefficient does not depend on the frequency, the step response function reflects without additional distortion from the second interface. We can write down the field T_x at some point within the slab as the superposition of all the step pulses originating at mirror sources located to the left and to the right of the slab. For example, the first reflection of the step pulse at $x=d$ generates a step pulse traveling to the left with an amplitude $-R_2$ times the amplitude of the initial step pulse (the minus sign is due to the fact that the pulse is traveling in the opposite direction and the spatial derivative is of the opposite sign). This reflected pulse appears as generated at a mirror source located at $x_1^-=2d$. This second pulse is then reflected at $x=0$ and generates the third pulse traveling to the right with an amplitude R_2R_1 times the initial amplitude. The equivalent mirror source for this third pulse is at $x_1^+=-2d$. Again this pulse is reflected at $x=d$, and so on. Following this procedure we see that the mirror sources to the right of the slab are at $x_n^-=2d,4d,6d,\cdots$ with the amplitude factors $-R_2$, $-R_2(R_2R_1)$, $-R_2(R_2R_1)^2,\cdots$. The mirror sources to the left of the slab are at $x_n^-=-2d,-4d,-6d,\cdots$ with the amplitude factors R_2R_1, $(R_2R_1)^2$, $(R_2R_1)^3,\cdots$.

Adding the initial step pulse to all the reflected step pulses traveling to the right gives

$$-\frac{Q}{k_1}\sum_{n=0}^{\infty}(R_2R_1)^n\left\{1-\operatorname{erf}\left[\frac{(2nd+x)}{2}\sqrt{\frac{1}{\alpha_1 t}}\right]\right\}. \tag{A8}$$

Adding up all the step pulses traveling to the left gives

$$\frac{Q}{k_1}R_2\sum_{n=0}^{\infty}(R_2R_1)^n\left\{1-\operatorname{erf}\left[\frac{[2(n+1)d-x]}{2}\sqrt{\frac{1}{\alpha_1 t}}\right]\right\}. \tag{A9}$$

Adding the latter two contributions gives Eq. (5).

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