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Measurement of thermal properties of microfluidic samples using laser point heating thermometry

Sun Rock Choi, Dongsik Kim *

Department of Mechanical Engineering, POSTECH, Pohang 790-784, Republic of Korea

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Abstract

This work introduces a novel method to measure the thermal conductivity, diffusivity, and heat capacity of a microfluidic sample having a small-volume of the order of 1 μl. The method is based on a new concept named laser point heating thermometry (LPHT). The method employs point heating of a thermocouple tip using a focused laser beam. As the suggested scheme utilizes external optical heating of a temperature probe, a relatively large amount of heat can be generated at a localized spot. Furthermore, the temperature of the heated spot can be effectively monitored from thermoelectric potential without undesirable electrical interference in the heating and sensing circuitry. In addition to the thermal-property measurement, this simultaneous point heating and point sensing technique can be employed in a variety of thermal-sensing applications including velocity and motion measurement.

Keywords: Thermal conductivity; Thermal diffusivity; Heat capacity; Microfluidic sample; Laser point heating thermometry

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1. Introduction

Thermal properties such as thermal conductivity, diffusivity and heat capacity are fundamental in engineering systems that involve heat transfer as well as in scientific studies. Particularly, the need for thermal analysis of a microfluidic sample is rapidly growing as liquid samples of tiny volume are frequently used in microfluidics and bioengineering applications [1–3]. In this regard, this work proposes a novel method to measure thermal transport properties of a small-volume fluid sample in the microliter range. Numerous schemes have so far been developed to measure the thermal properties of fluids. Among them, the transient hot-wire method is regarded as one of the most standard techniques to determine the thermal conductivity and diffusivity of a fluid sample [4]. In the transient hot-wire method, a thin metallic wire typically several centimeters in length is immersed in the sample fluid and electrically heated by Joule heating while the resistance variation monitors the thermal response. Accordingly, the method requires a sample volume at least hundreds of milliliters, which is often unallowable for expensive bio-samples and functional materials. It is also obvious that microfluidic applications necessitate thermal analysis of microfluidic samples as they constantly pursue integration of multiple functionalities on a single substrate having a small dimension.

Measurements of thermal conductivity and diffusivity require two main elements: heating and sensing (thermometry). As the thermal penetration depth $\sim \sqrt{\alpha t}$ (α: thermal diffusivity, t: time) is generally small compared to the dimension of the heating element, conventional measurement schemes cannot be applied to a small sample volume because the majority of the supplied heat is consumed for heating up the heating/sensing element, not the sample. It is thus apparent that a method based on point heating and point sensing (PHPS) principle is the most ideal for local property measurement. Recently, a microcalorimetric technique using the Peltier effect, which relies on the PHPS principle, has been suggested [5]. In the method, a thermocouple (TC) tip serves as a PHPS element. However, in the case of the Peltier sensor, both heating and sensing are based on the same physical principle, i.e. thermoelectric effect. The method therefore has intrinsic interference between heating and sensing signals. To avoid the interference, the heater and sensor should be either spatially or temporally separated, which often makes *in situ* monitoring of the thermal response of a heating element at the same location impossible, resulting in difficulties in data analysis.

In this work, a novel method entitled the laser point heating thermometry (LPHT) is proposed to measure the thermal con-

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* Corresponding author. Tel.: +82 54 279 2179; fax: +82 54 279 3199.
E-mail address: dskim87@postech.ac.kr (D. Kim).

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ductivity and diffusivity of a microfluidic fluid sample. The key to the technique is optical point heating of a tiny thermometer, for example a thermocouple tip. Consequently, a small fluid element can be selectively heated with simultaneous monitoring of the thermal response. The experiment employs a focused laser beam to heat a microthermocouple tip and the real-time temperature response is detected by the Seebeck voltage signal from the junction. A multiparameter-fitting scheme is utilized to simultaneously determine the thermal conductivity, and diffusivity.

2. Theory

2.1. Heat conduction

Fig. 1 shows the principle of the LPHT technique schematically. As the TC tip generally has a much greater conductivity than the fluid, uniform temperature distribution in the TC tip can be assumed. In addition, if the heat loss through the TC wires is negligible, the temperature of the TC can be expressed analytically as [6]:

$$T = \frac{q}{4\pi a k}\left\{\frac{1+ac_1}{c_1} - \frac{2a^2c_1^2c_2^2}{\pi} \times \int_0^\infty \frac{e^{-\alpha u^2 t/a^2}}{[u^2(1+ac_1)-ac_1c_2]^2+[u^3-uac_1c_2]^2}\,du\right\} \tag{1}$$

and

$$c_1 = \frac{h}{k}, \qquad c_2 = \frac{4\pi a^3 \rho C}{M_s C_s}$$

where a, C_s, and M_s are the size, heat capacity, and mass of the TC tip, respectively, and C, k and ρ are the heat capacity, thermal conductivity and density of the fluid sample, respectively (q: heat transfer rate, h: contact thermal conductance at the TC tip–fluid interface).

The above equation provides a simple analytical tool to determine the thermophysical properties of the liquid sample. However, analysis of the rigorous temperature field indicates that the heat loss through the TC wire significantly deteriorates the accuracy, up to about 20%. Therefore, our suggested scheme uses the full numerical solution to the heat conduction problem, considering heat conduction in the TC tip and wires. An FEM (finite element method) scheme is employed to obtain the numerical solution, assuming the convective heat transfer in the liquid is negligible. The temperature field taking the heat conduction loss through the TC wire is displayed in Fig. 2. Comparison of the results in Fig. 2(a) with those in Fig. 2(b) manifests that heat conduction through the wires substantially modifies the temperature field and therefore the numerical procedure is necessary to determine the thermal properties.

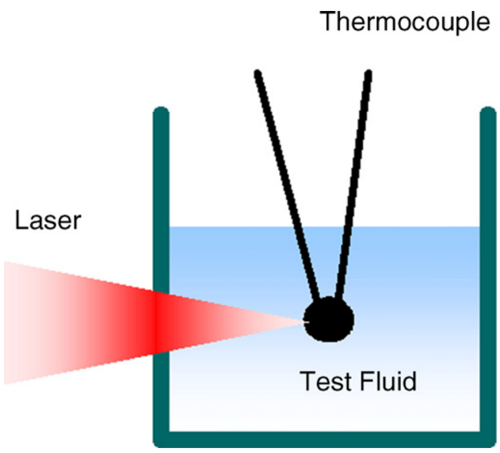

Fig. 1. Principle of the LPHT technique.

2.2. Thermal-property determination

Since the amount of heat flow cannot be quantified precisely in an optical-heating method such as this work, the temperature is normalized by the maximum value at a reference time t_f:

$$\Theta(t) = \frac{T(t)}{T(t_f)} \tag{2}$$

If the size and heat capacity of the TC tip are known, the temporal variation of the normalized temperature becomes a function of thermal conductivity and thermal diffusivity only. Therefore, the normalized transient temperature together with a multiparameter-fitting algorithm yields the properties. Determination of the tip size using a microscope is possible but may cause random error because of the non-uniformity in the shape of a commercial TC tip. The exact value of the heat capacity is also unknown. Therefore, the fitting parameters include the size and heat capacity of the tip. The difference function F between the theoretical and experimental temperature data (Θ_n^{the} and Θ_n^{exp}) is thus minimized numerically:

$$F(k, \alpha, \ldots) = \sum_{n=1}^{N} (\Theta_n^{\text{the}} - \Theta_n^{\text{exp}})^2 \tag{3}$$

In the present study, the quadratic interpolation and the Powell's method were utilized as single-parameter and multiparameter optimization schemes, respectively [7].

Since the time scale of temperature variation is related to the sensitivity of thermal-property measurement, it is important to determine the heating-pulse duration t_f properly. Fig. 3 shows the measurement sensitivity as a function of the pulse duration for three different thermal conductivity values. The sensitivity can be expressed as:

$$S = \frac{1}{E}\frac{\mathrm{d}E}{\mathrm{d}k}, \qquad E = \sum_{n=1}^{N} (\Theta_n)^2 \tag{4}$$

Based on the sensitivity analysis in the range of the thermal conductivity of typical liquids 0.05–0.6 $\mathrm{W\,m^{-1}\,K^{-1}}$, the pulse duration was determined to be 50 ms.

3. Experiment

Fig. 4 displays the schematic diagram of the experimental setup. An $\mathrm{Ar^+}$ ion laser is used as a heat source. The temporal

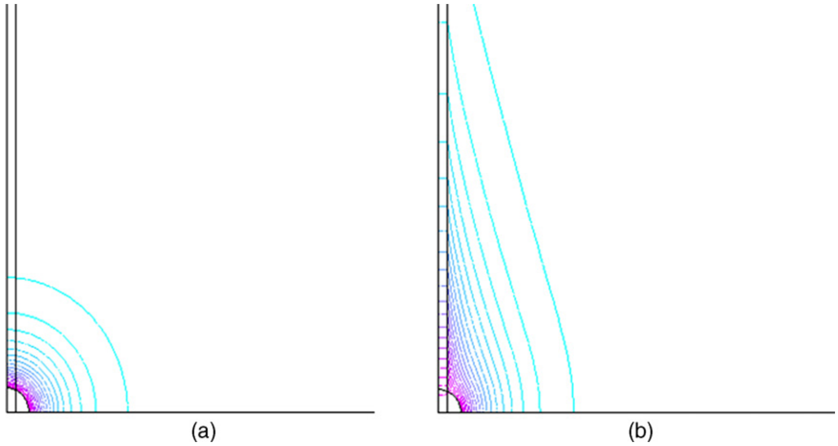

Fig. 2. Temperature contours showing the heat loss through TC wire at $t = 50$ ms (arbitrary units): (a) without heat loss and (b) with heat loss (tip diameter: 55 μm, wire diameter: 25.4 μm).

Table 1
Experimental results

Test liquids	Experiments		Literature [9]	
	α ($\times 10^{-8}$ m^2 s^{-1})	k (W m^{-1} K^{-1})	α ($\times 10^{-8}$ m^2 s^{-1})	k (W m^{-1} K^{-1})
Ethylene glycol	9.06	0.254	9.39	0.252
1-Propanol	6.90	0.166	8	0.172
2-Propanol	6.31	0.148	6.6	0.156
Ethanol	9.33	0.183	8.8	0.182
Methanol	10.6	0.216	10	0.202
Glycerol	8.93	0.273	9.35	0.292
Toluene	11.9	0.137	8.9	0.132

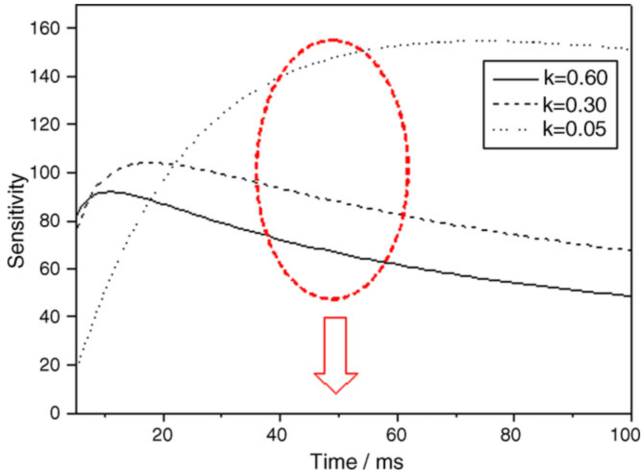

Fig. 3. Sensitivity of the measurement as a function of the heating-pulse duration.

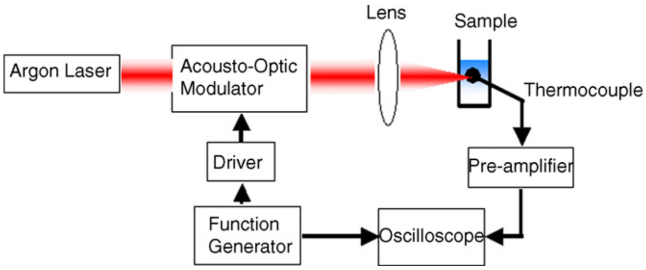

Fig. 4. Schematic diagram of the experimental setup.

shape of the laser beam is controlled by an AOM (Acousto-Optic Modulator) and a function generator. The laser beam is focused on the TC tip by a lens and a PC-controlled micropositioning system. The TC signal is 500 times amplified by an amplifier and fed to an oscilloscope or a lock-in amplifier. The TC is a commercial product from OMEGA (E-type, CHROMEGA®/Constantan) and 25 μm in diameter [8]. The laser beam irradiates the TC tip for 50 ms with the real-time thermal response being recorded. To remove the random electrical noise, the experimental data have been averaged until the random fluctuation in the signal becomes negligible (<1%). The laser intensity is adjusted so that the maximum temperature increase is below 10 K. Seven commonly used liquids have been selected as test samples (see Table 1).

4. Results and discussion

The size and heat capacity of the TC tip were determined by the multiparameter-fitting method to be 46.1 μm and 2.92 J m^{-3}, respectively. The determined size shows good agreement with the results of microscope inspection and the heat capacity is also close to the mean value of the two metals.

Fig. 5 exhibits the temporal variation of the temperature response normalized by the maximum value at 50 ms for ethylene glycol. The line and markers denote the numerical prediction with fitted parameters and the experimental data, respectively. For ethylene glycol, the best-fit values of thermal

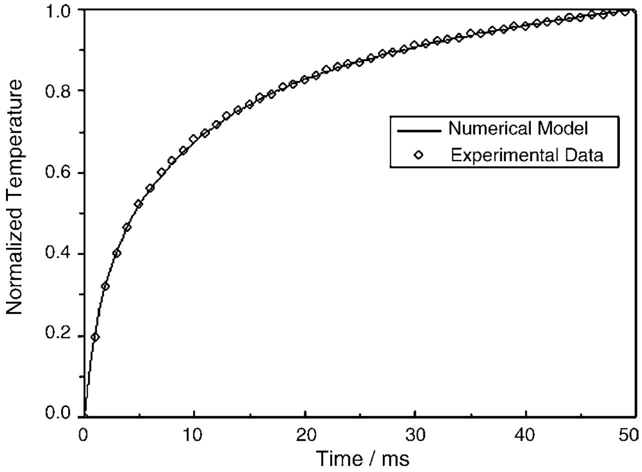

Fig. 5. Temperature increase for ethylene glycol.

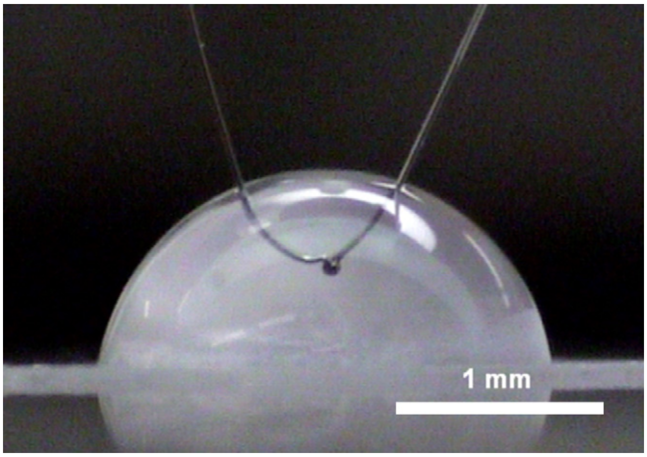

Fig. 7. Photograph of a microdroplet with an inserted TC tip. The laser illumination comes through the transparent glass substrate.

conductivity and thermal diffusivity that minimize the difference between numerical prediction and experimental data are 0.254 W m^{-1} K^{-1} and 9.06×10^{-8} m^2 s^{-1}, respectively, while the corresponding literature values are 0.252 W m^{-1} K^{-1} and 9.39×10^{-8} m^2 s^{-1} [9]. The deviation from the reference values are 0.8% and 3.5% for thermal conductivity and diffusivity, respectively. The results for other liquids are summarized in Table 1 and Fig. 6. Fig. 6 demonstrates that the LPHT method can be used to measure the thermal properties to a reasonable accuracy. It is also shown that the measurement sensitivity is better for thermal conductivity than that for heat capacity.

Uncertainty analysis discloses that the main uncertainty of measurement stems from the non-uniformity of the tip shape. It is noted that the thermocouple used in the measurement is a commercial product whose tip shape is not completely uniform. Therefore, further work is currently on-going to fabricate microthermocouples using the standard MEMS process.

The thermal penetration depth is of the order of $\sqrt{\alpha t_f} =$ 100 μm. Consequently, assuming that 1 mm sample dimension is required, a sample volume on the order of 1 mm^3 = 1 μl is sufficient for measurement. This requirement for the sample volume is obviously far smaller than that of the conventional technique such as the transient hot-wire method. To demonstrate the ability to handle microfluidic samples, water and ethylene glycol samples were prepared in the form of a microdroplet on a glass substrate as shown in Fig. 7. The results obtained from the microdroplet samples are identical to those summarized in Table 1, which confirms that the proposed method can be applicable to a small-volume fluid samples of the order of 1 μl.

In addition to the dc heating mode, preliminary experiments were conducted in the ac heating mode as shown in Fig. 8. However, the measurement in the ac mode results in an increased error, compared to dc mode. It is believed that the increased uncertainty is due to convective heat loss.

It is obvious that the LPHT technique suggested in the work can also be utilized to measure pressure (vacuum), flow, and motion (acceleration) since the thermal-property of a fluid is generally a strong function of those parameters. Furthermore, as the LPHT-based sensors can be miniaturized and integrated into microscale devices using laser diodes, it bears strong potential in microfluidics and its applications.

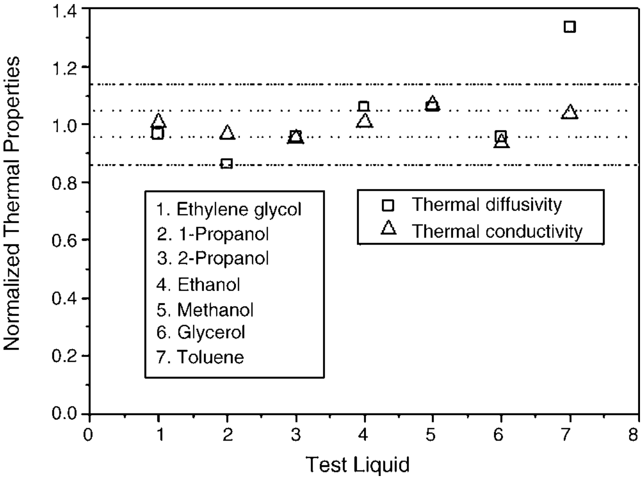

Fig. 6. The thermal properties normalized by the literature values [9]: dotted lines and dash-dotted lines represent 4.5% and 14% deviations, respectively.

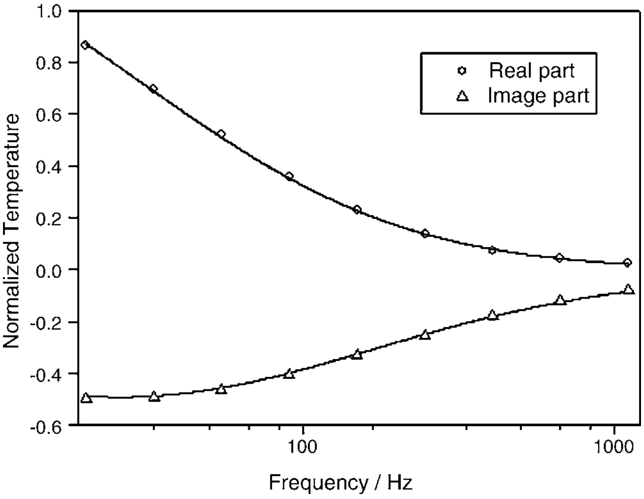

Fig. 8. Frequency dependence of temperature response: the symbols and lines denote the measured data and the theoretically fitted results, respectively.

5. Conclusion

In this work, a novel method to measure the thermal properties of a microfluidic sample has been proposed based on a new concept named laser point heating thermometry. Measurements of the thermal conductivity of seven different liquids demonstrate that the technique is currently capable of determining thermal properties of a microfluidic sample with an uncertainty of ∼10%. The main source of the uncertainty has bean identified to be the non-uniformity of the TC tip, which can be eliminated by incorporating a microfabrication process in the future. It is evident that the suggested laser point heating technique can be applied to a variety of thermal-sensing applications other than the thermal-property measurement.

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