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Application of the thermal flash technique for low thermal diffusivity micro/nanofibers

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The thermal flash method was developed to characterize the thermal diffusivity of micro/nanofibers without concern for thermal contact resistance, which is commonly a barrier to accurate thermal measurement of these materials. Within a scanning electron microscope, a micromanipulator supplies instantaneous heating to the micro/nanofiber, and the resulting transient thermal response is detected at a microfabricated silicon sensor. These data are used to determine thermal diffusivity. Glass fibers of diameter 15 μm had a measured diffusivity of 1.21×10^{-7} m^2/s; polyimide fibers of diameters 570 and 271 nm exhibited diffusivities of 5.97×10^{-8} and 6.28×10^{-8} m^2/s, respectively, which compare favorably with bulk values. © *2009 American Institute of Physics.* [DOI: 10.1063/1.3086310]

Micro- and nanofibers are one-dimensional structures with diameters ranging from several nanometers (i.e., nanofibers) to ~100 μm (i.e., microfibers). These materials offer promise for use in nanolasers,[1] photonics,[2] photovoltaics,[3] thermoelectrics,[4] sensors and actuators,[5] functional semiconductor electronic components,[6] and polymer electronics.[7] Accurate thermal characterization of micro/nanofibers is critical for their integration into these and other applications. The development of a rapid, simple, accurate, and reliable method to measure their thermal properties would facilitate their use and expand fundamental understanding of nanoscale thermal transport.

For measurement of the thermal properties of micro/nanostructures, various steady-state methods have been proposed.[8,9] These techniques use the steady-state flux of heat through a nanowire from a heater to a sensor to determine thermal conductivity. However, thermal contact resistances in these systems are difficult to accurately estimate.[10] Some transient thermal measurements have been employed in which an estimation of thermal contact resistance is unnecessary.[11–14] These methods use the change in electrical resistance of a thermal sensor to measure the transient thermal response to a sudden application of heat. This approach, however, requires the micro/nanowires to be electrically conductive or to be coated with a thin, electrically conductive material. The positioning and attachment of micro/nanostructures to required sensors further complicate this approach.

This work reports on the development of a transient thermal measurement technique for one-dimensional micro/nanostructures which is administered within a scanning electron microscope (SEM). For this thermal flash method, thermal contact resistance is inconsequential and various complexities typically associated with this type of measurement are minimized. While this method examines the thermal diffusivity rather than conductivity, the conductivity may be obtained though the knowledge on the material density and specific heat. In the particular case of micro/nanofibers, if it can be assumed that the density and specific heat are similar to their corresponding bulk values, then the thermal diffusivity can be used to calculate thermal conductivity. The technique builds on previous work to measure the mechanical properties of micro/nanowires[15] and expands upon the laser flash method in which a sudden application of radiant heat is applied to a sample after which a transient change in temperature is detected.[16] A wire-wrapped micromanipulator supplies instantaneous heating to one end of the micro/nanostructure; the temperature response is then detected along the micro/nanofibers using a microfabricated sensor.[17] The transient electrical resistance of the sensor allows calculation of thermal diffusivity. The technique is validated for low diffusivity materials by measuring the properties of both glass and polyimide micro/nanofibers.

Figure 1 shows the experimental configuration and Fig. 2 depicts a SEM image of the setup for the case of a polyimide microfiber. A doped silicon cantilever acts as the thermal sensor[17] and is employed in a Wheatstone bridge circuit along with three precision potentiometers; the bridge is balanced prior to the measurement. Inside the SEM (FEI XT Nova nanoLab 200), a micro/nanofiber is positioned between the thermal sensor and an aluminum prop, which serves to keep the sample suspended in vacuum, with the help of a micromanipulator (Kleindiek MM3A). If necessary, electron beam ion deposition of platinum is performed using a microdelivery gas-injection system to form a bond between the micro/nanostructure and the sensor. The micromanipulator, which is wrapped with a 0.127 mm diameter polyimide-

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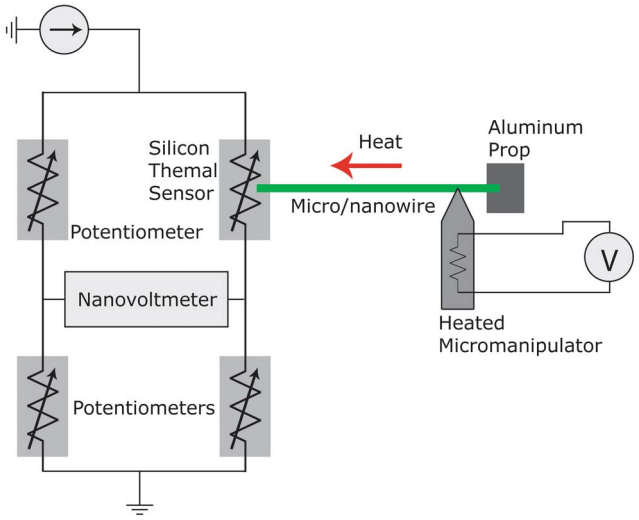

FIG. 1. (Color online) A schematic of the thermal flash method.

coated nichrome wire through which a current is sent, is then moved close to the micro/nanofiber. The electron beam of the SEM is turned off and the micromanipulator is heated to a temperature not exceeding the glass transition temperature of the polyimide fiber and then brought into sudden contact with the suspended structure. The relatively large size and high thermal conductivity of the micromanipulator justify the approximation that it can be modeled as a high temperature thermal reservoir, therefore maintaining a constant temperature at the point of contact. Heat flows along the micro/nanofiber to the sensor, causing the Wheatstone bridge to become unbalanced. The transient voltage at the center of the bridge is monitored with a nanovoltmeter (Keithley 2182A). The amount of heat flowing along the micro/nanofiber in the other direction toward the aluminum prop does not influence the final determination of thermal diffusivity since knowledge on the absolute magnitude of the heat flux is not required for the analysis as described below.

An analytical solution representative of the experiment was derived from the heat equation and used to determine the thermal diffusivity of the micro/nanofibers from the data. The normalized temporal variation in temperature $\bar{T}(t)$ at the contact point between the micro/nanofiber and the sensor can be expressed as[18]

$$\bar{T}(t) = 1 + 2\sum_{n=1}^{\infty} (-1)^n e^{\alpha_f n^2 \pi^2 t / l_f^2}. \tag{1}$$

Here, the subscript f refers to the micro/nanofiber; l_f is its length, and α_f is its thermal diffusivity. The analytical model assumes that at time t equals zero, the heated micromanipulator touches the micro/nanofiber, resulting in an instantaneous temperature rise at the point of contact. The micro/nanofiber is modeled as being in perfect contact with the silicon thermal sensor, which itself is affixed to a large silicon body that is assumed to be at constant temperature. Equation (1) was simplified from its original form by assuming that

$$\frac{k_s}{k_f}\sqrt{\frac{\alpha_f}{\alpha_s}} \gg 1 \quad \text{and} \quad \sqrt{\frac{\alpha_f}{\alpha_s}} \ll \frac{l_f}{l_s}, \tag{2}$$

where the subscript s now refers to the sensor and k is thermal conductivity. These assumptions are applicable for low thermal conductivity/diffusivity materials and for micro/nanofibers of sufficiently long length. Since the transient variation in temperature is required to extract thermal diffusivity and not the magnitude of temperature itself, an offset due to thermal contact resistance will not influence the final result. Experimentally, the voltage measured at the center of the Wheatstone bridge also follows the change in temperature experienced by the sensor. Consequently, the normalized voltage difference measured as a function of time may also be modeled by Eq. (1), and the analytical solution can be used in conjunction with the normalized experimental voltage data to extract the thermal diffusivity of the micro/nanofiber. A curve fitting procedure minimizes the sum of the squared errors between the analytical solution and the experimental data.

The experimental technique was verified using phosphate-based glass available commercially (Mo-Sci Corp.) and polyimide micro/nanofibers. The polyimide nanofibers were prepared by first electrospinning the polyamic acid precursor solution (DuPont). The electrospinning apparatus was operated with 10 kV electrical potential applied between the spinning nozzle and grounded collector that are separated by 15 cm air gap. The collected fibers were found to be mostly devoid of solvents. The completion of solvent removal and imidization reaction was carried out by stepwise heating of the fibers in vacuum at 50 °C for 24 h, 100 °C for 2 h, 175 °C for 1 h, and 350 °C for 1 h. An example of the experimental data used to extract thermal diffusivity is shown in Fig. 3 for the case of the glass microfibers. Here, the exponential rise in voltage versus time is shown to be well modeled by the analytical solution discussed previously. The thermal diffusivity data are summarized in Fig. 4. For the 15 μm diameter glass fibers with lengths varying from 403 to 490 μm, thermal diffusivity was determined to be $(1.21 \pm 0.16) \times 10^{-7}$ m^2/s. The error was taken to be the standard deviation of the eleven measurements made. Note that even potential inaccuracies associated with the length measurement of the microfiber of $\pm 10\%$ still resulted in a thermal diffusivity within the error bars given. As a comparison, a bulk form of the glass sample was measured using the numerical mirage method,[19] giving a

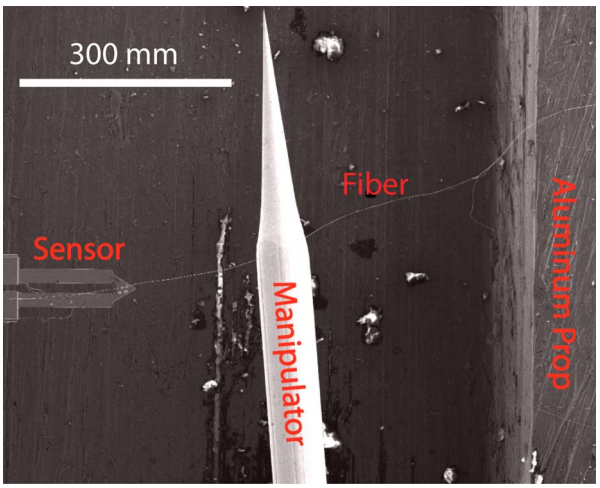

FIG. 2. (Color online) An SEM image of the thermal flash measurement for a polyimide micro/nanofiber.

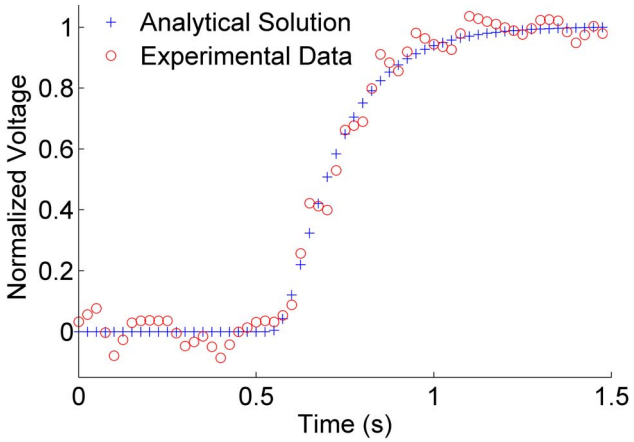

FIG. 3. (Color online) Voltage vs time for a thermal measurement on a 15 μm diameter glass microrod, showing the experimental data after a curve fit with the analytical solution.

result of $(1.2 \pm 0.3) \times 10^{-7}$ m^2/s, providing confidence in the technique. Two different sets of polyimide fibers of 570 and 271 nm diameter were also measured with lengths varying from 163 to 272 μm. The resulting thermal diffusivities were determined to be $(5.97 \pm 0.71) \times 10^{-8}$ and $(6.28 \pm 0.63) \times 10^{-8}$ m^2/s for the 570 and 271 nm fibers, respectively. It should be noted that the 271 nm fibers are not expected to exhibit significant effects of enhanced thermal boundary scattering or other nanoscale mechanisms that might be present in sub-100 nm fibers, and therefore their thermal diffusivity should be comparable to that of the 570 nm diameter fibers. A bulk form of the polyimide was measured using the dynamic plane source technique,[20]

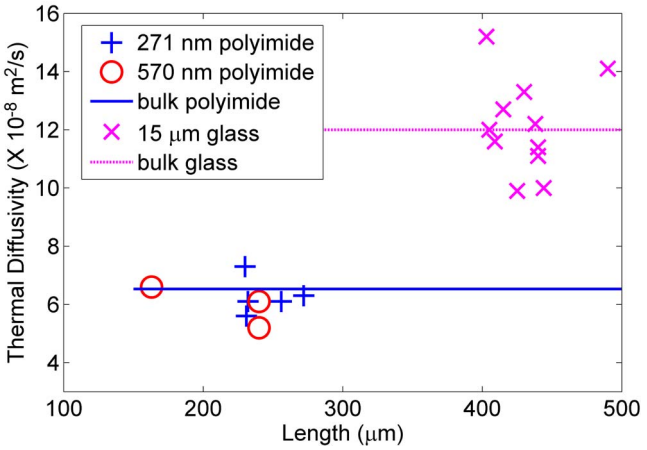

FIG. 4. (Color online) Thermal diffusivity obtained in the thermal flash measurements vs length of wire used for various materials.

resulting in a thermal diffusivity of $(6.53 \pm 0.22) \times 10^{-8}$ m^2/s, which compares well with the measurements obtained using the thermal flash method.

The successful application of the thermal flash method described herein validates this simple, reliable, and accurate technique for characterization of low thermal diffusivity, one-dimensional micro/nanostructures; the measurements conducted on phosphate glass and polyimide micro/nanofibers concur with bulk characterization of similar materials. This technique is attractive because thermal contact resistance is inconsequential and, compared to various other methods, many complexities are minimized. Although measurements thus far have been restricted to low thermal diffusivity materials, the analysis may be appropriately rederived for high thermal diffusivity micro/nanofibers.

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