Steady heat conduction-based thermal conductivity measurement of single walled carbon nanotubes thin film using a micropipette thermal sensor

R. Shrestha,¹ K. M. Lee,¹ W. S. Chang,² D. S. Kim,³ G. H. Rhee,⁴ and T. Y. Choi^{1,a)}

¹*Department of Mechanical and Energy Engineering, University of North Texas, Denton, Texas 76203, USA*

²*Department of Nanomechanics, Korea Institute of Machinery and Materials, 156 Gajungbuk-no, Yuseong-gu, Daejeon 305-343, Korea*

³*Department of Mechanical Engineering, POSTECH, San 31 Hyoja-Dong, Nam-Gu, Pohang, Gyungbuk 790-784, Korea*

⁴*Department of Mechanical and Information Engineering, University of Seoul, 163 Siripdaero, Dongdaemun-gu, Seoul 130-743, Korea*

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In this paper, we describe the thermal conductivity measurement of single-walled carbon nanotubes thin film using a laser point source-based steady state heat conduction method. A high precision micropipette thermal sensor fabricated with a sensing tip size varying from $2\text{ }\mu\text{m}$ to $5\text{ }\mu\text{m}$ and capable of measuring thermal fluctuation with resolution of $\pm 0.01\text{ K}$ was used to measure the temperature gradient across the suspended carbon nanotubes (CNT) film with a thickness of 100 nm . We used a steady heat conduction model to correlate the temperature gradient to the thermal conductivity of the film. We measured the average thermal conductivity of CNT film as $74.3 \pm 7.9\text{ W m}^{-1}\text{ K}^{-1}$ at room temperature. © 2013 American Institute of Physics. [<http://dx.doi.org/10.1063/1.4792841>]

I. INTRODUCTION

Carbon nanotubes (CNTs) have novel properties with many applications in nanotechnology, which have made it a subject of intense research. They exhibit extraordinary strength, electrical properties, and thermal properties, and therefore have wide applications in electronic and optoelectronic, biomedical, energy, and other fields of material science. For example, CNTs have been demonstrated for potential use in display applications (e.g., CNT field emission displays), being an excellent source of electron beams. Due to one-dimensional nanostructure and superior electrical conductivity, CNTs have been used to develop various electrochemical sensors. With mechanical properties being calculated stronger than steel,¹ they showed a potential for applications in architectural field to strengthen the tensile properties of building materials and there have been researches in progress for building body armor made of CNT fibers. Most importantly, for next-generation integrated circuits, a thin film of CNT will be of great use as a heat sink device to remove localized heat from the hot spots in microchips.² Therefore, determination of thermal transport properties in thin films will help understand the heat conduction mechanisms in the material and would benefit both academic research and numerous industrial applications of CNTs. However, experimental verification of physical properties of materials at micro-scale has always been a challenging task because of sample preparation and probing setup.³

Some experimental results for thermal conductivity of multi-walled CNT films have been reported with values varying from $1.52\text{ W m}^{-1}\text{ K}^{-1}$ to $83\text{ W m}^{-1}\text{ K}^{-1}$.^{2,4-6} Some of the techniques employed in the thermal conductivity measurement of CNTs are the 3-omega method,⁶ Joule heating

analysis,^{2,7} pulsed photothermal reflectance technique,⁵ and comparative technique.⁸ However, the results obtained from these techniques require clarification. For example, various methods using strip/wire heater^{6,9} did not consider the thermal resistance of the interfaces between films and heating strip/wire or heat sinks.³ Also, the heat loss from the bulky sensing tip of the thermocouple wires is relatively larger than the heat flow in thin films which leads to the uncertainty in the measured thermal conductivity of the films.

In the present paper, we provide the solutions to these problems by introducing noncontact heating of the film surface by a laser point source. The CNT film for this experiment was prepared on a polycarbonate substrate containing arrays of $50\text{ }\mu\text{m}$ diameter through holes by the vacuum filtration method. The thickness of the CNT film suspended on the polycarbonate substrate is much smaller than the diameter of the hole on the polycarbonate. Therefore, the heat transfer through the film can be considered as one-dimensional radial heat conduction⁵ and simple Fourier's law can be employed for its thermal conductivity determination. A micropipette thermal sensor¹⁰⁻¹² with a tip size varying between $2\text{ }\mu\text{m}$ and $5\text{ }\mu\text{m}$ used in the experiment had a negligible amount of heat loss through the sensor tip, and the heat loss due to radiation from the sample surface was also demonstrated to be low. The temperature gradient across the two spots along the radial direction was measured with the micropipette thermal sensor at a fixed laser power irradiation to determine the thermal conductivity of the suspended CNT film. Furthermore, this method can be extended to measure thermal conductivities of thick solid samples. As a final step, a thermal modeling and simulation based on 2D steady state heat conduction should be employed to find the thermal conductivity. One drawback is that this method is valid for in-plane thermal conductivity measurement; out-of-plane thermal conductivity cannot be measured using this technique.

^{a)} Author to whom correspondence should be addressed. Electronic mail: choi@egw.unt.edu. Tel.: 1-940-565-2198.

FIG. 1. Fabrication steps of a pipette thermal sensor: (a) an empty pipette with less than $1\ \mu\text{m}$ opening; (b) a schematic outline of the filled pipette; (c) after filling and beveling; (d) after PVD thin film coating of nickel; (e) a prototype micropipette thermal sensor; (f) SEM image of beveled tip of micropipette prior to PVD thin film deposition.

II. EXPERIMENTAL METHOD

A. Fabrication of pipette sensor

The micropipette sensor is prepared from a thick-wall borosilicate glass with outer diameter of $1.5\ \text{mm}$ and inner diameter of $0.86\ \text{mm}$. The glass is pulled to a micropipette with the tip diameter less than $1\ \mu\text{m}$ and taper length of 5 to $7\ \text{mm}$. The micropipette is filled with a lead-free soldering alloy that contains mostly tin (Sn). The tip is then beveled¹⁰ to remove the surplus Sn metal that results from the filling process and to form a smooth and uniform surface at the glass-Sn alloy interface. The scanning electron microscope (SEM) image of the beveled pipette is shown in Figure 1(f). The beveling step also enhances the electrical contact between the inner metal (Sn alloy) and the outer metal (a thin film of nickel (Ni)) that was coated in the next step using physical vapor deposition (PVD) technique^{13–15} to produce a thermocouple junction of 2 to $5\ \mu\text{m}$.

To prevent the unwanted thermoelectric effect due to dissimilar metals, the same materials of wires (i.e., Ni and Sn alloy) were used to form lead wires that connect the sensor with a voltmeter (Keithley 2182, Nano Voltmeter). Moreover, the cold junction formed by the lead wires from the sensor and connecting wires of voltmeter was maintained at constant temperature of $298\ \text{K}$ by using an isothermal block controlled by an in-house temperature controller integrated with an automated data logging system with LabVIEW. By calibration¹⁰ of 2 different sensors, fabricated by different PVD coating conditions, the Seebeck coefficients of 5.67 and $7.44\ \mu\text{V/K}$ were determined. Figure 1 shows the whole process of the fabrication of the pipette sensors.

B. Preparation of CNT film by vacuum filtration

To produce uniform films of single-walled nanotubes, we adopted a vacuum filtration method that involves vacuum filtering a dilute suspension of nanotubes in a solvent over a porous alumina filtration membrane (Whatman, $200\ \text{nm}$ pore size, $47\ \text{mm}$ diameter). We started by mixing arc-discharge single-walled carbon nanotubes (SWNTs) with $1\ \text{wt. \%}$ aque-

ous sodium dodecyl sulfate to make a highly dense SWNT suspension with a typical concentration of $1\ \text{mg/ml}$. This highly concentrated SWNT suspension was then ultrasonically agitated using a probe sonicator for $10\ \text{min}$, followed by centrifugation to separate out undissolved SWNT film. The as-produced suspension was further diluted by a factor of 30 with deionized water. This solution was then vacuum-filtered through a $60\ \mu\text{m}$ thick alumina membrane. As the solvent falls through the pores, the nanotubes are trapped on the surface of the filter, forming an interconnected network. The density of this network can be controlled with high precision by controlling the volume of dilute suspension filtered through the membrane. The SWNT film is released by dissolving filtration membrane in wet chemicals and transferred to the perforated polycarbonate substrate to make a freestanding SWNT film. The SEM image of one edge of the CNT film is shown in Figure 2.

C. Experimental setup for measurement of thermal conductivity of CNT film

The schematic of the experimental setup to measure the thermal conductivity of the CNT film fabricated on the

FIG. 2. SEM image showing the CNT film.

FIG. 3. Experimental setup for thermal conductivity measurement of a CNT film with a micropipette sensor.

polycarbonate substrate is shown in Figure 3. A class B laser at 633 nm was irradiated at the center of the CNT film suspended on a 50 μm -diameter hole. The temperature difference at two radial positions was measured using various pipette sensors of 2–5 μm sensing tip with Seebeck coefficient of 7.44 $\mu\text{V/K}$. We have conducted repeated experiments for the same CNT film under the same experimental conditions using different sensors with Seebeck coefficient of 5.67 $\mu\text{V/K}$ to ensure the measurement accuracy.

An optical setup was prepared to guide the laser from the laser source positioned horizontally at a certain elevation from the surface of the working table. A combination of antireflection coated lenses was used to produce a collimated and expanded ray of beam which was then diverted at 90° by a beam splitter towards the CNT film. A 20 $\times$ super-long working distance objective lens (Mitutoyo MPlan Apo SL) was used to convert this diverted laser into a Gaussian laser of $1/e^2$ diameter of 3 μm at the focal plane. The laser spot diameter was determined by using an optical image of the laser beam and assuming the laser beam is Gaussian. We performed a

digital image processing and deduced the laser spot diameter at the focal plane. A white light from a high intensity illuminator (Edmund optics MI-150) was guided with an optical fiber and delivered through an objective lens to illuminate the sample placed at the focal plane. The CNT film on the polycarbonate substrate was positioned horizontally in between two objective lenses at the focal plane of 20 $\times$ objective lens with the use of an XYZ motorized stage. In a similar approach, a motorized stage was used to precisely control the movement of the pipette sensor to position at a desired location on the surface of the thin film. Special care was taken to land the sensor on the CNT film as slight extra pressure may break the film.

A variable neutral density filter was used to control the incident power of the laser at a desired level. The film was placed at the focal point of the objective lens which was confirmed with a clear image obtained with the charged-coupled device (CCD) camera as shown in Figure 4(a).

The laser was illuminated at the center of the film as shown in Figure 4(b) and then turned off momentarily to position the pipette sensor. Then the sensor was brought to a position (Figure 4(c)) such that its radial position, r , was larger than 6 μm ($r > 6 \mu\text{m}$) in order to avoid the direct laser illumination at the tip of the sensor.

Laser was then irradiated at a fixed power level and at steady state the voltage generated by the sensor was recorded using a voltmeter (Nano Voltmeter, Keithley 2182). It was observed that the measured voltage signal jumped to a significantly higher value when the laser was irradiating with sensor touching the film as compared to the baseline when the sensor was at the same position but not in contact with the film. This implies that the sensor is in fact measuring the temperature rise due to heat conduction across the film. In order to minimize the contact resistance between the sensor and the film, 1 μm of additional vertical movement of the sensor was provided by the motorized stage. At this position the maximum saturated voltage signal was recorded by the voltmeter which confirmed that the sensor and the film were in good contact. For consistency in measurement, the same process was repeated to measure the voltage signals at different locations on the film.

The process was repeated to measure the voltage generated at location 2. With a known Seebeck coefficient of the

FIG. 4. Measurement of temperature with the pipette sensor. (a) CNT film at focal plane, (b) laser shined at the center of the film, (c) pipette sensor at position 1, (d) pipette sensor at position 2, and (e) schematic model showing the heat distribution in a radial direction.

sensor used, the voltages recorded at the two positions were then converted to temperature difference. Figure 4(e) is the schematic of the model to demonstrate the radial heat conduction. It was measured and confirmed with the pipette sensor that the temperature at radius $r > 25 \mu\text{m}$ was approximately close to room temperature. The radial position of the sensor was determined by images taken by the CCD camera.

D. Laser power measurement

The absorbed power (P_{absorbed}) was obtained from the conservation of energy. This implies the amount of incident energy is equal to the sum of the absorbed, reflected, and transmitted energy. A digital power meter (THORLABS PM100D) synchronized with the computer was used to record the laser irradiation power at different positions 1, 2, 3, and 4 as shown in Figure 3. Laser power measured at position 1 is the direct measure of the transmitted power ($P_{\text{transmitted}}$), whereas value at position 2 is the total incident power (P_{incident}). Power measured at position 3 is used to determine the loss in the objective lens, while power at position 4 is the power of reflected light ($P_{\text{reflected}}$) taking into accounts all the losses in objective lens and beam splitter. From a separate measurement it was determined that the beam splitter transmits 45% and reflects 55%. Together with the measured power at position 1 (i.e., transmitted power), the absorbed power was calculated according to the relationship $P_{\text{absorbed}} = P_{\text{incident}} - P_{\text{reflected}} - P_{\text{transmitted}}$.

With the measured absorbed power, radial positions, and the temperature difference at two locations, thermal conductivity of the CNT film was calculated using the Fourier's equation for radial steady-state heat conduction (Eq. (1)). It should be noted that essentially all the absorbed power is transferred through the film in radial direction because the loss due to convection, radiation, and finite tip size (energy loss through the tip) is negligible (see more details of error analysis in Sec. III):

$$\dot{Q} = \frac{2\pi kL(T_1 - T_2)}{\ln(r_2/r_1)}, \quad (1)$$

where $\dot{Q}$ is the power absorbed, k is the thermal conductivity of CNT film, L is the thickness of CNT film, r_1 and r_2 are radii of two locations, and T_1 and T_2 are the temperatures at two radial positions.

III. RESULTS

A. Thermal conductivity measurement of CNT film

As the suspended CNT film was confined within a 50 μm -diameter circle, there was a necessity to use the smallest tip pipette sensor to measure the temperature precisely within a limited region. Voltages at two different locations, 8 μm apart in radial directions, were measured. Using all the powers measured at the four locations, the power absorbed by the CNT film was determined as 65.60 μW (intensity of 929 W/cm^2) which was about 75% of the total irradiated power. The radial position of the two locations was determined by counting the pixels in between the locations and

center of the CNT film. The picture taken with CCD camera was calibrated with respect to SEM image and it was determined that 1 pixel in the image from CCD camera was equivalent to 0.5 μm . Then using Eq. (1), we determined the thermal conductivity to be 75.0 $\text{W m}^{-1} \text{K}^{-1}$. The experiment was repeated at the same power level using the same pipette sensor but this time the temperature difference was measured for two new locations. The thermal conductivity of the film was calculated as 76.4 $\text{W m}^{-1} \text{K}^{-1}$. We conducted repeated experiments with the same sensor with a different laser power. Evidently, the value of the power absorbed increased to 79.2 μW (intensity of 1121 W/cm^2), but the percentage power absorption by the film remains approximately the same. The thermal conductivity of CNT with this experiment was calculated as 73.5 $\text{W m}^{-1} \text{K}^{-1}$.

A similar experiment was completed using a pipette sensor with different sensitivity (5.67 $\mu\text{V}/\text{K}$). From this measurement the thermal conductivity was determined to be 72.4 $\text{W m}^{-1} \text{K}^{-1}$. The thermal conductivity of the CNT film determined by two different sensors while differing the power level to irradiate the film were approximately the same value. Therefore, an average value of 74.3 $\text{W m}^{-1} \text{K}^{-1}$ was accepted as the thermal conductivity of the film. These results were obtained with sensor tips with various sizes (2–5 μm) and the deviation from the mean thermal conductivity value is within the measurement uncertainty. It should be noted that we have measured temperatures at multiple points on the surface of the film and any two temperatures at two different radial positions returned the same thermal conductivity within a certain uncertainty (see the error analysis in Sec. III B). Owing to the simplicity of theory in obtaining thermal conductivities of suspended thin films, fitting of the experimental temperature data points with those of simulation is not needed.

B. Error estimation for thermal conductivity

The uncertainty in the calculation of the thermal conductivity is associated with uncertainty in measurement of power absorbed, radial position, thickness of the film, and temperature difference. The uncertainty in thermal conductivity (k) was calculated using the root-sum-squared method¹⁶ which is expressed as

$$u_k = \left[\left(\frac{\partial k}{\partial Q} u_Q \right)^2 + \left(\frac{\partial k}{\partial R} u_R \right)^2 + \left(\frac{\partial k}{\partial L} u_L \right)^2 + \left(\frac{\partial k}{\partial \Delta T} u_{\Delta T} \right)^2 \right]^{1/2}, \quad (2)$$

where u_Q is the uncertainty in power measurement = $\pm 0.8\%$; u_R is the uncertainty in radial position = $\pm 2.0\%$; u_L is the resolution of SEM = uncertainty in thickness = $\pm 2.0\%$; $u_{\Delta T}$ is the uncertainty in temperature measurement = $\pm 10.2\%$

Uncertainty in power and thickness measurement are the errors due to measuring instruments, whereas errors in radial position and temperature are associated with measurement errors which were determined statistically by calibration of CCD image and pipette sensor. The uncertainty in temperature measurement was determined using the relationship ΔT

$= \Delta V/S$, where ΔT , ΔV , and S are temperature difference, voltage difference, and Seebeck coefficient of sensor, respectively. Error in ΔT was then calculated using the expression

$$u_{\Delta T} = \left[\left(\frac{\partial \Delta T}{\partial \Delta V} u_{\Delta V} \right)^2 + \left(\frac{\partial \Delta T}{\partial S} u_S \right)^2 \right]^{1/2}, \quad (3)$$

where $u_{\Delta V}$ is the resolution of Kithley nano-voltmeter $= \pm 73$ nV; u_S is the uncertainty in Seebeck coefficient $= \pm 0.15$ μ V/K.

In addition, error in temperature measurement is also aroused by the temperature difference across the cross section of measuring sensor tip. It was determined that average temperature difference of 0.12 K would occur across the sensing tip when the total temperature gradient was 1.26 K at two positions of the sample. This results in 9.5% of uncertainty in the temperature measurement due to the effective sensing tip of 2 μ m. Thus, overall uncertainty in temperature measurement was calculated to be 10.2%.

Finally, the uncertainty in k value was determined using Eq. (2) as $u_k = 10.6\%$. It is evident that the uncertainty in temperature measurement has significant contribution in the calculation of the uncertainty in thermal conductivity. Therefore, to reduce the uncertainty and thus increase the accuracy in measurement, we need to produce sensors with small sizes. We determined that by reducing the sensor tip size to 1 μ m, the uncertainty in thermal conductivity can be reduced to 6.6%.

To confirm that the heat transfer in the film was mostly due to the radial heat conduction in the film, the natural convection and radiation heat loss was also evaluated. When the laser was fixed at 79.2 μ W (highest incident power used during the experiment), the sensor detected that the laser would produce a temperature of 300 K at the center of the film. Since the experiment was conducted at a room temperature of 294 K, the mean film temperature to calculate the convection heat transfer was estimated at 297 K. At this mean film temperature Rayleigh number was determined at 2.79×10^{-4} which confirmed that there will be natural convection. Using the Lienhard¹⁷ assumption, we determined the convective heat transfer coefficient at the mean film temperature of 297 K for air to be 22.85 W m⁻² K⁻¹. Then using Newton's law of cooling the heat loss due to the natural convection was determined to be 0.27 μ W which was only 0.34% of the absorbed power 79.2 μ W. Similarly, using Stefan Boltzmann law, the heat loss to the surrounding from the film surface was calculated as 0.07 μ W which was only 0.09% of the total power absorbed in the film. Therefore, the heat transfer by natural convection and radiation was ignored and the temperature difference measured by the sensor was confirmed as the sole effect of the radial heat conduction in the film. Since the temperature measurement is a differential one (i.e., $T_1 - T_2$), the uncertainty due to the thermal resistance between the sensor tip and the thin film will be cancelled. This way, the thermal conductivity of the suspended thin film could be relatively accurately determined.

The heat loss through the sensor tip can be estimated by measuring the temperatures along the pipette including the tip

and the sample in contact with the tip. The heat loss can then be calculated by multiplying the measured temperature difference along the pipette by the theoretical thermal resistance of $\sim 10^5$ K/W along the pipette. This resistance can be obtained by assuming the dominant heat transfer occurs through the pipette core metal (Sn alloy). Considering 1D heat conduction through Sn alloy, we express the thermal resistance through the pipette as follows:

$$R_{pipette} = - \int_{T_{tip}}^{T_{\infty}} \frac{dT}{q_x} = \int_0^{L_{pipette}} \frac{dx}{k A_c}, \quad (4)$$

where k is the thermal conductivity of Sn alloy, $L_{pipette}$ is the length along the pipette, and $A_c = \frac{\pi}{4} D_x^2$ in μ m² (cross-sectional area) with the cone angle of 5°. However, the heat loss through the pipette will not affect the measurement accuracy because the temperature gradient between the two points on the sample does not change assuming that the thermal contact resistance between the tip and the sample remains the same. The same contact resistance and the heat loss will give rise to the same temperature difference between the tip and the sample. This same temperature difference will be subsequently cancelled in calculation of thermal conductivity as shown in Eq. (1). The same thermal contact resistance can be accomplished by applying a slight extra pressure beyond the saturated temperature signal. We expect the heat loss through the pipette sensor is negligible as compared to the absorbed power, which will not significantly affect the accuracy of the measurement. In order to verify the negligible heat loss through the tip, we have performed a separate experiment using a polished silicon wafer which is expected to exhibit lower thermal contact resistance due to the better surface condition of silicon than CNT film. The experimentally obtained thermal conductivity agrees well with the literature value within 5%, which implies the heat loss through the tip can be neglected even with the lower thermal contact resistance.

IV. CONCLUSIONS

We have measured the thermal conductivity of single-walled carbon nanotube thin film with thickness of 100 nm at room temperature using a micropipette thermal sensor and a steady-state heat conduction-based method. The obtained thermal conductivities of the CNT film using the measurement data varied from 72.4 W m⁻¹ K⁻¹ to 76.4 W m⁻¹ K⁻¹. From the error analysis the uncertainty in the obtained values for thermal conductivity was determined to be 10.6%. Therefore, the mean value of 74.3 W m⁻¹ K⁻¹ $\pm$ 7.9 W m⁻¹ K⁻¹ was accepted as thermal conductivity of the CNT film at room temperature. We also calculated that the uncertainty in measurement can be reduced to 6.6% by using micropipette sensors with a tip size of 1 μ m which will be our future goal in order to improve the measurement accuracy. The method developed in this study is applicable to any suspended thin films such as CNT films used in this research and measure the in-plane thermal conductivity. However, this method cannot be used for thick films as we have assumed one-dimensional radial heat conduction. The developed methodology herein

enables high accuracy for temperature measurements in a localized region with a size of a few microns.

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