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# Note: Thermal conductivity measurement of individual poly(ether ketone)/carbon nanotube fibers using a steady-state dc thermal bridge method

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Customized engineered fibers are currently being used extensively in the aerospace and automobile industries due to the ability to "design in" specific engineering characteristics. Understanding the thermal conductivity of these new fibers is critical for thermal management and design optimization. In the current investigation, a steady-state dc thermal bridge method (DCTBM) is developed to measure the thermal conductivity of individual poly(ether ketone) (PEK)/carbon nanotube (CNT) fibers. For non-conductive fibers, a thin platinum layer was deposited on the test articles to serve as the heater and temperature sensor. The effect of the platinum layer on the thermal conductivity is presented and discussed. DCTBM is first validated using gold and platinum wires (25 $\mu$m in diameter) over a temperature ranging from room temperature to 400 K with $\pm 11\%$ uncertainty, and then applied to PEK/CNT fibers with diverse CNT loadings. At a 28 wt. % CNT loading, the thermal conductivity of fibers at 390 K is over 27 $\mathrm{Wm^{-1}K^{-1}}$, which is comparable to some engineering alloys.
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Engineered fibers are currently widely used in airplanes, automobiles, and sports equipment due to their excellent mechanical performance, low weight, high flexibility, and good thermal stability.[1,2] Accurate understanding of the thermal conductivity of high-performance fibers is imperative for their development, integration, and application. Typically, the transverse dimension of polymeric fibers ranges from 1–100 $\mu$m,[2] which makes many of the conventional thermal conductivity measurement techniques, such as the hot guarded plate method and the transient plane source method, not available any more.

Zhang *et al.*[3,4] developed the T-shaped method to measure the thermal conductivity of a single carbon fiber by suspending the sample over a thin platinum hot wire. The reliability and accuracy of this method were, to a large extent, limited by the thermal contact resistance between the sample and the hot wire and the uncertainty related to the geometry. Inspired by the laser flash method, Demko *et al.*[5] proposed a thermal flash method that utilized a micromanipulator to supply heat and a microfabricated sensor to determine the temperature response. This approach, however, was only suitable for fibers with low thermal diffusivity. Resorting to pulsed laser or Joule heating, several transient electrothermal techniques[6–8] have been employed to study the thermal diffusivity of thin fibers and CNT micro bundles. In this type of approach, the thermal conductivity is calculated from the known sample density and specific heat, which are commonly assumed to be the same as that of the bulk material. Moreover, even though the transient electrothermal method recently has been improved to directly test the thermal conductivity, the results were still significantly influenced by the value of the applied current and the heating or cooling mode, especially for temperature-dependent thermal conductivities.[9,10] In addition, based on the harmonic self-heating, thin wire-shaped 3-omega method[11] and its derivative methods such as 2-omega method[12,13] were in principle available; however, this method has not yet been validated on non-conductive fibers.

The steady-state dc thermal bridge method was initially proposed[14] to study metallic films.[15,16] This approach, however, has not been verified for microscale fibers and more importantly non-conductive materials. Based on electrical heating and electrical-thermal sensing, the present work develops the DCTBM to measure the thermal conductivity of both conductive and non-conductive, high and low thermally conductive composite fibers (CF).

Figure 1 illustrates the experimental principle of the DCTBM, in which the sample is suspended over two heat sinks and attached to the electrodes. As the current passes through the sample, it is heated at steady state and the resistance changes correspondingly. This change can be accurately recorded using a four-probe method. For highly conductive materials, the sample serves as both a heater and thermometer; for poor or non-conductive materials, an additional metallic coating can be used to both serve as a heater and thermometer. Utilizing a one-dimensional heat conduction model and combining this with the boundary conditions $T(x = 0) = T_0$ and $T(x = L) = T_0$, in which $L$ is the effective length of

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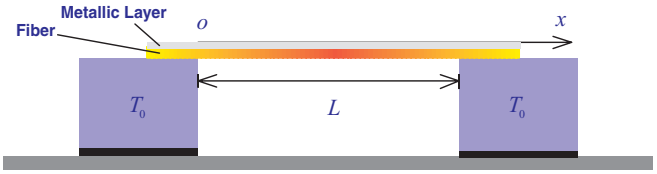


FIG. 1. (Color online) Schematic of the DCTBM.

the sample, the temperature distribution can be determined as

$$T = T_0 + \frac{VI}{2\lambda A}x - \frac{VI}{2\lambda LA}x^2, \quad (1)$$

where λ is the thermal conductivity, $I$ is the current, $V$ is the voltage of the sample, and $A$ is the cross-sectional area. The average temperature rise can be written as

$$\Delta T = \frac{VIL}{12\lambda A}. \quad (2)$$

Therefore, the thermal conductivity can be acquired from the applied heating power, the corresponding temperature rise, and fiber's geometric parameters. The sample resistance is measured at the fixed temperature at different currents. Through extrapolating, the intrinsic resistance (without Joule heating) can be acquired at the corresponding temperature. The resistance-temperature relationship, $\Delta T = f(\Delta R)$, needs to be calibrated before the testing of thermal conductivity for diverse samples. Note that the applied dc should be appropriate to ensure that the temperature rise is not too high, typically below 5 K.

The experimental system is described in Fig. 2. By high performance silver paste (PELCO, Ted Pella), the two ends of fibers were bonded on two separate copper sheets attached to an alumina substrate through a thermal tape (3M). To reduce the heat loss due to air convection, the sample was placed in a vacuum chamber (ST-100, Janis) with base pressure $1 \times 10^{-6}$ Torr. The voltage was recorded by a digital multimeter (Agilent 34410A) and the current was recorded by a resistance box (RS-201W, IET LABS) with the other digital multimeter. The sample temperature was controlled through an

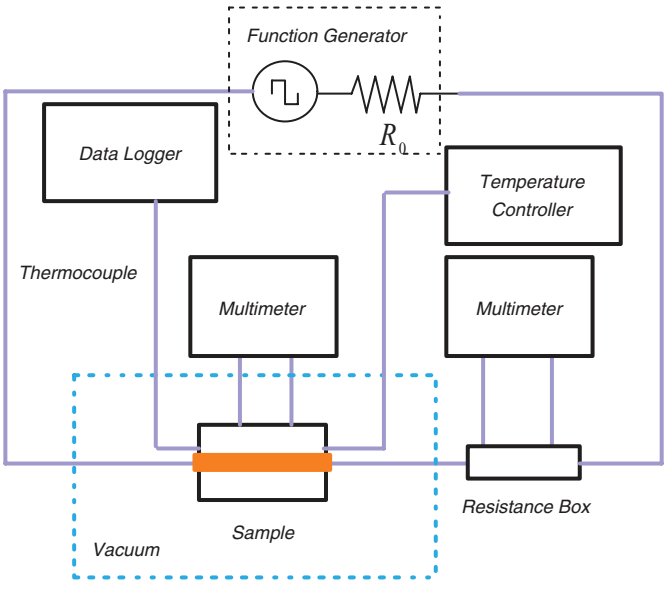


FIG. 2. (Color online) Schematic of experimental system.

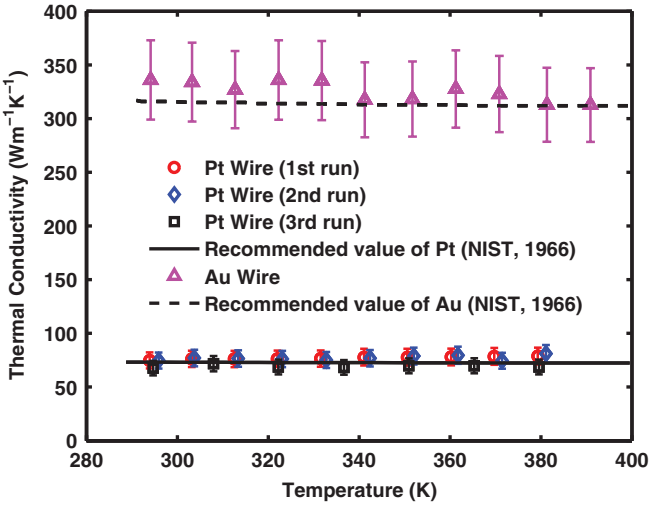


FIG. 3. (Color online) Temperature-dependent thermal conductivity of Pt and Au wires compared to NIST recommended values. The error bar is 11%.

autotuning temperature controller (331 Lakeshore) and then measured using a data logger (Agilent 34970A). To accurately determine the temperature, two T-type 80 $\mu$m thick thermocouples (RTD Company) were attached on the alumina substrate. The applied current was supplied by a function generator (Agilent 33220A).

The experimental technique was initially verified using commercial Pt (99.95%) and Au (99.99%) from Surepure Chemetals, Inc. Figure 3 shows the thermal conductivity of Pt and Au wires over a temperature range of 295–400 K, compared to the NIST recommended values.[17] Overall, the derivations are within ±11%, which is considered to be the experimental uncertainty of the present approach.

The PEK/CNT fibers were processed using a dry-jet wet-spinning approach.[18] The tested CNT weight ratios included 5%, 10%, 20%, and 28%. For CNT loading lower than 20%, fibers were not very conductive. Pt was selected as the coating layer in view of its stable temperature-resistance relationship and lower thermal conductivity (compared with Au). By using an electron-beam evaporator, the thickness was controlled to be around 10 nm and lower deposition rate 0.2 Å $s^{-1}$ was preferred to form a high-quality coating. The resistance after coating was around $10^4$ to $10^5\Omega$. Before testing, each sample was first annealed at 380 K for a few hours, which made the resistance of the metallic coating more stable and consistent.

The thermal conductivity of the PEK/CNT fibers is shown in Fig. 4. Poly(ether ketone) is a type of plastic resin that is nearly thermally and electrically insulating, while individual CNTs exhibit a very high thermal conductivity ($10^3$ W $m^{-1}$ $K^{-1}$). The increased CNT loading enhances thermal conductivity. Note that for 28 wt. % CNT containing fibers, the sample with Pt (CF-28-Pt-1 and CF-28-Pt-2) and without Pt (CF-28) coating have been tested and compared with each other (listed in Table I). Even though the resistance, thickness, and length of these samples differ from each other, a consistent thermal conductivity has been observed, which implies that the coating of metallic layer is a reliable and effective approach.

Due to the deposition of the platinum film on the sample, it is necessary to determine the impact on the thermal

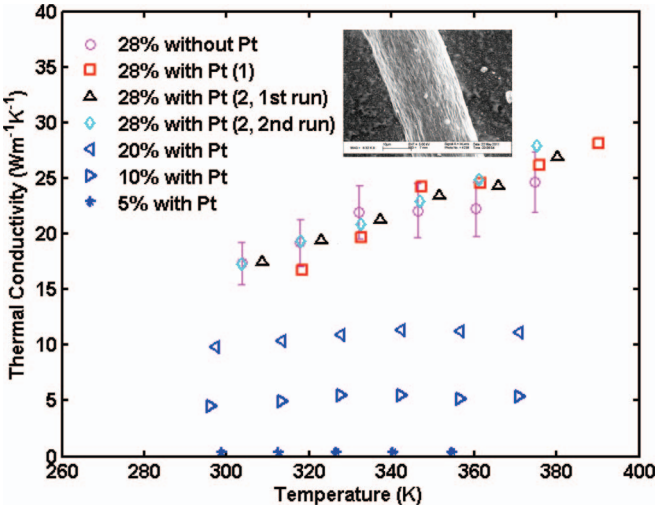


FIG. 4. (Color online) Temperature-dependent of thermal conductivity of PEK/CNT fibers with diverse CNT loadings.

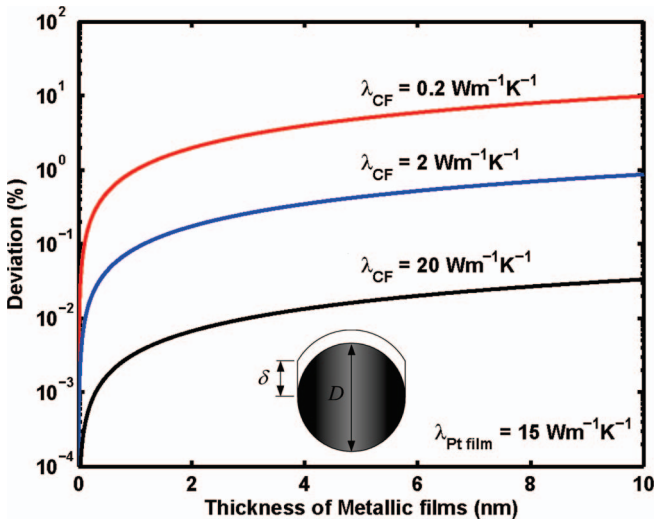


FIG. 5. (Color online) The deviations of thermal conductivity of carbon fibers due to depositing additional sensing metallic layers. The thermal conductivity of metallic layer and fiber thickness are assumed to be 15 Wm$^{-1}$K$^{-1}$ and 10 $\mu$m. The insert is a cross-sectional schematic of Pt-coated fiber.

conductivity resulting from the coating. The total thermal conductivity of the fiber and the Pt layer can be determined as $\lambda_{tot} = \beta\lambda_{Ptfilm} + (1 - \beta)\lambda_{CF}$, where the cross-sectional area ratio $\beta = (\pi D\delta/2)/(\pi D^2/4)$, $D$ is the fiber thickness and $\delta$ is the thickness of the Pt coating. The deviation is defined as $(\lambda_{tot} - \lambda_{CF})/\lambda_{tot}$ shown in Fig. 5. The Pt film is so thin that there exists a number of grain boundaries, as well as a restricted film surface, which dramatically reduces the mean free path of the conduction electrons and thermal conductivity.[19] According to the experimental data,[20] a safe value of 15 Wm$^{-1}$K$^{-1}$ is used for a 10 nm thick Pt film fabricated by electron beam evaporation. For thermally conductive fibers, i.e., $\lambda_{CF} > 2$Wm$^{-1}$K$^{-1}$, the effect of Pt coating was found to be negligible; for fibers with a poor thermal conductivity, i.e., $\lambda_{CF} > 0.2$Wm$^{-1}$K$^{-1}$, the error introduced by the Pt coating was less than 10%. For the fibers with ultralow thermal conductivity, i.e., $\lambda_{CF} < 0.1$Wm$^{-1}$K$^{-1}$, the effective thermal conductivity can be determined by subtracting the contribution of the metallic coating after the measurements.

In conclusion, the steady-state dc thermal bridge method has been successfully utilized to measure the thermal conductivity of individual PEK/CNT composite fibers over a temperature range of 295–400 K. This approach can be utilized to evaluate both conductive and non-conductive fibers. When compared with the existing techniques, the present approach appears to present a reasonable alternative due to its simplicity and a high degree of reliability.

TABLE I. Specifications of the samples used.[a]

| Sample | Thickness ($\mu$m) | Length (mm) | Resistance (298 K, Ω) |
|---|---|---|---|
| Pt-1 | 26.70 | 7.32 | 1.56 |
| Au-1 | 25.38 | 12.50 | 0.62 |
| CF-28 | 23.11 | 7.95 | $7.38 \times 10^4$ |
| CF-28-Pt-1 | 27.35 | 10.32 | $6.02 \times 10^4$ |
| CF-28-Pt-2 | 25.60 | 11.35 | $6.90 \times 10^4$ |
| CF-20-Pt | 24.31 | 7.32 | $1.61 \times 10^4$ |
| CF-10-Pt | 25.87 | 5.99 | $1.95 \times 10^4$ |
| CF-5-Pt | 24.97 | 6.91 | $6.13 \times 10^4$ |

[a]Resistance variation of fibers with Pt coatings mainly comes from the differences in Pt film thickness and annealing time.

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[1]M. L. Minus and S. Kumar, JOM **57**, 52 (2005).
[2]H. G. Chae and S. Kumar, Science **319**, 908 (2008).
[3]X. Zhang, S. Fujiwara, and M. Fujii, Int. J. Thermophys. **21**, 965 (2000).
[4]X. Zhang and M. Fujii, Polym. Eng. Sci. **43**, 1755 (2003).
[5]M. T. Demko, Z. T. Dai, H. Yan, W. P. King, M. Cakmak, and A. R. Abramson, Rev. Sci. Instrum. **80**, 036103 (2009).
[6]J. B. Hou, X. W. Wang, and L. J. Zhang, Appl. Phys. Lett. **89**, 152504 (2006).
[7]J. Q. Guo, X. W. Wang, and T. Wang, J. Appl. Phys. **101**, 063537 (2007).
[8]J. Q. Guo, X. W. Wang, D. B. Geohegan, G. Eres, and C. Vincent, J. Appl. Phys. **103**, 113505 (2008).
[9]B. Feng, W. G. Ma, Z. X. Li, and X. Zhang, Rev. Sci. Instrum. **80**, 064901 (2009).
[10]X. P. Huang, J. M. Wang, G. Eres, and X. W. Wang, Carbon **49**, 1680 (2011).
[11]L. Lu, W. Yi, and D. L. Zhang, Rev. Sci. Instrum. **72**, 2996 (2001).
[12]C. Dames and G. Chen, Rev. Sci. Instrum. **76**, 124902 (2005).
[13]B. Feng, Z. X. Li, X. Zhang, and G. P. Peterson, J. Vac. Sci. Technol. B **27**, 2280 (2009).
[14]F. Volklein and E. Kessler, Phys. Status Solidi A **81**, 585 (1984).
[15]Y. Yang, R. M. White, and M. Asheghi, ASME Trans. J. Heat Transfer **128**, 113 (2006).
[16]X. Zhang, H. Q. Xie, M. Fujii, H. Ago, K. Takahashi, T. Ikuta, H. Abe, and T. Shimizu, Appl. Phys. Lett. **86**, 171912 (2005).
[17]R. W. Powell, C. Y. Ho, and P. E. Liley, *Thermal Conductivity of Selected Materials (NSRDS-NBS 8)*, National Bureau of Standards Reference Data Series (U.S. Govt. Print. Off., Washington, D.C., 1966).
[18]R. Jain, Y. H. Choi, Y. Liu, M. L. Minus, H. G. Chae, S. Kumar, and J.-B. Baek, Polymer **51**, 3940 (2010).
[19]B. Feng, Z. X. Li, and X. Zhang, Thin Solid Films **517**, 2803 (2009).
[20]Q. G. Zhang, B. Y. Cao, X. Zhang, M. Fujii, and K. Takahashi, J. Phys.: Condens. Matter **18**, 7937 (2006).