Reexamination of thermal transport measurements of a low-thermal conductance nanowire with a suspended micro-device

Annie Weathers,1 Kedong Bi,2 Michael T. Pettes,1 and Li Shi1
1Department of Mechanical Engineering, The University of Texas at Austin, Austin, Texas 78712, USA
2Jiangsu Key Laboratory for Design and Manufacture of Micro-Nano Biomedical Instruments, School of Mechanical Engineering, Southeast University, Nanjing 211189, People’s Republic of China

(Received 12 March 2013; accepted 11 July 2013; published online 12 August 2013)

An increasingly used technique for measuring the thermal conductance of a nanowire is based on a suspended micro-device with built-in resistance thermometers. In the past, the technique has been limited to samples with thermal conductance larger than $1 \times 10^{-6}$ W/K because of temperature fluctuations in the sample environment and the presence of background heat transfer through residual gas molecules and radiation between the two thermometers. In addition, parasitic heat loss from the long supporting beams and asymmetry in the fabricated device results in two additional errors, which have been ignored in previous use of this method. To address these issues, we present a comprehensive measurement approach, where the device asymmetry is determined by conducting thermal measurements with two opposite heat flow directions along the nanowire, the background heat transfer is eliminated by measuring the differential heat transfer signal between the nanowire device and a reference device without a nanowire sample, and the parasitic heat loss from the supporting beams is obtained by measuring the average temperature rise of one of the beams. This technique is demonstrated on a nanofiber sample with a thermal conductance of $3.7 \times 10^{-10}$ W/K, against a background conductance of $8.2 \times 10^{-10}$ W/K at 320 K temperature. The results reveal the need to reduce the background thermal conductance in order to employ the micro-device to measure a nanowire sample with the thermal conductance less than $1 \times 10^{-10}$ W/K. © 2013 AIP Publishing LLC. [http://dx.doi.org/10.1063/1.4816647]

I. INTRODUCTION

Carbon nanotubes (CNTs), graphene, inorganic nanowires, and organic nanofibers have been demonstrated as building blocks of a variety of functional devices.1–4 The performance and reliability of these functional devices are often dictated by the thermal transport properties of the nanoscale building blocks. In addition, these nanostructures provide a platform for investigating low dimensional transport phenomena, including size-dependent thermal and thermoelectric properties.5,6 Hence, as advancement continues in the synthesis of these and other nanostructures, experimental methods for characterization of their thermal properties will remain important.

A number of methods have been developed for probing thermal transport in nanotubes and nanowires,7 including suspended micro-devices with built-in resistance thermometers,8,9 the $3\omega$ method,10 the T-junction method,11 Raman based techniques,12,13 bi-material cantilever sensors,14 doped Si cantilever resistance thermometers,15 and electrothermal transient techniques.16,17 Several designs of suspended micro-devices have been fabricated for thermal transport measurements of nanotubes, graphene, nanowires, nanofibers, and thin films.5 In one design, the nanostructure sample is suspended between two adjacent SiNx membranes each fabricated with a serpentine platinum resistance thermometer (PRT).8 as shown in Figure 1 for the device investigated in the current work. When one PRT is electrically heated, heat conduction through the nanostructure sample results in a temperature rise in the adjacent sensing PRT. The temperature rise of each PRT is obtained from its measured temperature-dependent electrical resistance. Limited by the small temperature rise in the sensing PRT compared to fluctuations in the sample stage temperature, the method has been limited to a sample conductance of $1 \times 10^{-9}$ W/K when the resistance of the PRTs is measured using a lock-in amplifier in a four-probe configuration.18 A recent report has suggested that the sensitivity can be improved to $1 \times 10^{-11}$ W/K with the use of a differential method to minimize the noise caused by sample stage temperature fluctuations, in addition to an increase in the time constant and excitation current used for measuring the resistance of the sensing PRT.19 Such capability would be very useful for thermal measurements of small-cross section and low-thermal conductivity nanostructures.

However, parasitic heat transfer between the two PRTs through radiation, residual gas molecules, and the underlying substrate can result in a background thermal conductance when there is no nanostructure sample bridging the two SiNx membranes. For a vacuum level of the order of $10^{-6}$ torr in the sample space, this background conductance was measured in prior work to be approximately $1 \times 10^{-10}$ W/K, $4 \times 10^{-10}$ W/K, and $8 \times 10^{-10}$ W/K at temperatures 100 K, 300 K, and 500 K, respectively.18 In previous work, the measured background conductance was subtracted from the measured conductance of the device containing the bridged nanostructure, so as to obtain the thermal conductance of the nanostructure itself. However, the background thermal conductance was measured at a different time on a different
reference device without a nanostructure sample bridging the two membranes. The variation in the vacuum level in the sample space between the two measurements can cause error in the as-obtained thermal conductance of the nanostructure, especially when the nanostructure thermal conductance is comparable to or smaller than the background thermal conductance.

Besides the error due to non-negligible background conductance, it has been found by a numerical analysis that radiation loss from the long suspended beams is non-negligible at high and low temperatures. Ignoring this parasitic heat radiation loss from the long suspended beams is non-negligible conductance, it has been found by a numerical analysis that radiation loss from the long suspended beams is non-negligible especially when the nanostructure thermal conductance is comparable to or smaller than the background thermal conductance.

Here we report a comprehensive measurement approach to address these errors in the measurement of nanostructures with low thermal conductance. By switching the heat flow direction between the two membranes, this method can resolve the thermal conductance asymmetry between the supporting beams of the two membranes. Based on the measured asymmetry in both the thermal and electrical resistances of the devices, carefully balanced heating and sensing currents are simultaneously provided to the sample device with the nanostructure and a reference device without a nanostructure, so as to eliminate the error caused by background heat transfer between the two thermometers. In addition, the average temperature rise of a supporting beam of the sensing membrane is measured, and compared with the temperature rise in the sensing PRT to determine and correct for parasitic heat loss from the supporting beams to the environment. This new measurement method is demonstrated with a nanofiber sample with a thermal conductance of $3.7 \times 10^{-10}$ W/K at 320 K temperature.

II. HEAT TRANSFER MODEL OF AN ASYMMETRIC MEASUREMENT DEVICE WITH PARASITIC LOSS

As shown in Figure 1, the measurement device consists of two suspended SiN$_x$ membranes each supported by six long and thin SiN$_x$ beams. For the device tested in this work, the length of the beams is 400 μm. A serpentine PRT is patterned on each of the two SiN$_x$ membranes as well as four electrical contacts to the nanostructure sample. Each supporting beam is patterned with a platinum (Pt) electrode line to allow for four probe electrical measurements of the PRT resistance and nanostructure resistance. A nanowire sample is bridged between the two membranes in the device.

During the thermal conductance measurement, the sample is placed in the evacuated sample space of a cryostat. When a DC current flows through one PRT and two of its Pt leads, the Joule heating raises the serpentine PRT temperature by $\Delta T_b$. Heat transfer between the two membranes increases the adjacent sensing serpentine PRT temperature by $\Delta T_s$. The measurement scheme is illustrated in Figure 2(a). The PRT temperature is assumed to be the same as the temperature of the supporting SiN$_x$ membrane because of the much smaller internal thermal resistance of the membrane compared to the thermal resistance of the supporting beams and the sample thermal resistance, as shown by a numerical heat transfer analysis. The Joule heat is conducted through the supporting beams of the two membranes to the substrate kept at temperature $T_0$, as well as lost via radiation and residual gas molecules from the surface of the long supporting beams to the environment. In general, for a Joule heat $Q_{J,i}$ uniformly dissipated along the $i$th beam, with temperatures $T_0$ and $T_{m,i}$ at the junction with the substrate and membrane, respectively, the temperature distribution can be obtained from a fin heat transfer analysis as

$$\theta_{b,i} = \frac{\theta_{m,i} \sinh mL \cosh mx + (\theta_{0,i} - \theta_{m,i} \cosh mL) \sinh mx}{\sinh mL},$$

where

$$\theta_{b,i}(x) = T_{b,i}(x) - T_0 - \frac{Q_{J,i}}{hPL}$$

$$\theta_{m,i} = T_{m,i} - T_0 - \frac{Q_{J,i}}{hPL}.$$
and that of the sample. $Q_{gap}$ are the thermal resistance of the supporting beams to the two membranes.

FIG. 2. (a) Non-differential measurement setup. (b) Differential measurement setup. (c) Thermal resistance circuit representation of the measurement setup. (c) Thermal resistance circuit representation of the measurement setup.

For each of the six beams on the sensing side ($i = 7–12$) and four of the six beams ($i = 3–6$) connected to the heating membrane with $Q_{h,i} = 0$, the thermal conductance can be found from Eq. (3) as

$g_{b,i} = Q_{b,i}/(T_{m,i} - T_0) = \sqrt{hPkA} \coth mL$.  

A uniform Joule heating is generated in each of the two Pt leads that carry the heating current, $Q_{I} = I^2R_b$, where $R_b$ is the electrical resistance of the Pt lead and $I$ is the DC heating current, in addition to the Joule heat generated uniformly in the serpentine heater, $Q_h = I^2R_s$, where $R_s$ is the electrical resistance of the heating Pt serpentine. Energy conservation for the heating membrane and the sensing membrane yields

$Q_h = \sum_{i=1}^{2} Q_{b,i} + \sum_{i=3}^{6} Q_{b,i} + \sum_{i=7}^{12} Q_{b,i}$,  

in which the first term corresponds to $Q_{i=1–2}$, the second term to $Q_{i=3–6}$, and the third term to $Q_{i=7–12}$ in Figure 3. Equations (3) and (5) can be combined to yield

$Q_{total} \equiv Q_h + \beta Q_I = G_{b1}\Delta T_h + G_{b2}\Delta T_s$,  

where we have defined the total supporting beam thermal conductance for the heater and sensor sides as

$G_{b1} = \sum_{i=1}^{6} g_{b,i}$ and $G_{b2} = \sum_{i=7}^{12} g_{b,i}$.

and

$\beta = \frac{2}{mL} \frac{\cosh mL - 1}{\sinh mL}$.  

The factor $\beta$ reduces to 1 in the limit of vanishing $mL$ for negligible heat loss via radiation and gas molecules. In this limit, Eq. (4) is reduced to $g_{b,i} = kA/l$. With the definition of $\alpha_h = \Delta T_h/Q_{total}$ and $\alpha_s = \Delta T_s/Q_{total}$, Eq. (6) can be rewritten as

$\alpha_h G_{b1} + \alpha_s G_{b2} = 1$.  

In prior works, $G_{b1}$ and $G_{b2}$ have been assumed to be the same. Although the heating and sensing sides of the device are
designed to be identical, slight variation can result in a difference in \( G_{b1} \) and \( G_{b2} \). In order to obtain \( G_{b1} \) and \( G_{b2} \), a similar measurement is made by switching the heating and sensing sides. This measurement obtains an additional equation

\[
\alpha'_h G_{b2} + \alpha'_s G_{b1} = 1, \tag{10}
\]

where \( \alpha'_h \) and \( \alpha'_s \) follow the same definitions as \( \alpha_h \) and \( \alpha_s \), but are measured with the heating and sensing sides switched compared to the measurement of \( \alpha_h \) and \( \alpha_s \), as illustrated in Figure 2(c). Equations (9) and (10) are combined to solve for

\[
G_{b1} = \frac{\alpha'_h - \alpha_s}{\alpha'_h \alpha_h - \alpha'_s \alpha_s}, \tag{11a}
\]

\[
G_{b2} = \frac{\alpha_h - \alpha'_s}{\alpha'_h \alpha_h - \alpha'_s \alpha_s}. \tag{11b}
\]

The thermal conductance across the gap between the two membranes can accordingly be found from the thermal resistance circuit for either of the two heating configurations of Figure 2(c) as

\[
G_s = G_{b2} \frac{\alpha_s}{\alpha_h - \alpha_s}. \tag{12a}
\]

or

\[
G_s = G_{b1} \frac{\alpha'_s}{\alpha_h - \alpha'_s}. \tag{12b}
\]

Measurement errors can result in a small difference between the two \( G_s \) values obtained from Eqs. (12a) and (12b). For a perfectly symmetric device, \( \alpha_h = \alpha'_s, \alpha_s = \alpha'_h, \) and \( G_{b1} = G_{b2} = 1/(\alpha_h + \alpha_s) \), which is the \( G_s \) result obtained in previous works where the device asymmetry has been ignored and only \( \alpha_h \) and \( \alpha_s \) were measured.\(^8\) For a low thermal conductance sample in which \( G_s \ll G_h \) and likewise \( \Delta T_s \ll \Delta T_h \) and \( \alpha_s \ll \alpha_h \), the value of \( G_s = 1/(\alpha_h + \alpha_s) \) is nearly equal to \( 1/\alpha_h \), which is the same as \( G_{b1} \) in Eq. (11a) in the same limit. Therefore, for a low thermal conductance sample, \( G_s \) obtained from the analysis in previous reports\(^8\) is expected to be close to \( G_{b1} \) obtained in the current method. In other words, in the limit of zero heat transfer through the nanowire, all the heat generated in the heater will be dissipated through the beams supporting the heater, and the obtained beam conductance will be that of the heating side beams. As in prior works, substituting this \( G_h \) value in Eq. (12a) is expected to introduce a relative error in the as-obtained \( G_s \) that is as large as the relative difference between \( G_{b1} \) and \( G_{b2} \), which is up to 4% for the devices measured in this work.

III. ELIMINATION OF BACKGROUND THERMAL CONDUCTANCE

In the fin heat transfer analysis of Eq. (1), heat loss from the circumference of each beam to the environment at temperature \( T_h \) has been included. However, there exists an additional heat transfer via radiation and residual gas from the heating PRT and the two beams with heating current \( i = 1–2 \) to the other ten supporting beams without heating current \( i = 3–12 \). This heat source term has not been included in Eq. (1). In addition, there exists heat transfer between the beams carrying heating current \( i = 1–2 \) to adjacent beams on the heating membrane \( i = 3–6 \) through several inter-beam SiN\(_x\) connecting bars, which are used to enhance the structural stability of the device (Figure 1). Consequently, Eqs. (1)–(3) are not strictly valid for the twelve beams. However, the following equation can readily be obtained irrespective of the existence of the inter-beam heat transfer

\[
\frac{d^2}{dx^2} \sum_{i=1}^{12} \theta_{b,i}(x) = m^2 \sum_{i=1}^{12} \theta_{b,i}(x). \tag{13}
\]

The solution to Eq. (13) for \( \sum_{i=1}^{12} \theta_{b,i} \) and thus \( \sum_{i=1}^{12} Q_{b,i} \) is independent of the inter-beam heat transfer. Hence, Eqs. (5)–(11) can be obtained from Eq. (13) even in the presence of inter-beam heat transfer. In addition, we have established the following differential measurement scheme to eliminate the effects of the parasitic heat transfer via radiation and residual gas from the heating side to the sensing membrane and its supporting beams. As such, a modified version of Eq. (12) becomes valid under this differential measurement scheme, as discussed below.

The differential measurement obtains the relative increase in the sensing PRT temperature for the sample device compared to that for a blank reference device. The schematic of the measurement setup is shown in Figure 2(b). The reference device shares the same design as, and is adjacent to, the sample device on the same chip, except that no nanostructure bridges the two membranes of the reference device. During the differential measurement, both the sample and reference heating membranes are simultaneously heated by DC currents controlled by the two resistors, \( R_{DC} \) and \( R_{DC,ref} \) for the sample and reference, respectively, as shown in Figure 2(b). Three SRS 830 lock-in amplifiers are used in the differential measurement. The first lock-in (#1) is used to measure the AC voltage drop \( (v_h) \) across the sample heating PRT, from which the resistance and temperature increase in the heating PRT are obtained according to the same procedure as reported previously for non-differential measurements.\(^8\) The second (#2) and third (#3) lock-in amplifiers are used to measure the AC voltage difference \( v_{i+} - v_i \) and \( v_p \) at the two corresponding nodes between the sample device and reference device, as shown in Figure 2(b). A single AC voltage output from lock-in #2 is applied to both the sample and reference sensing membranes. The reference signal for lock-in #3 is the external, TTL (transistor-transistor logic) square pulse output from lock-in #2, so that lock-ins #2 and #3 are locked in both frequency and phase.

These two measured differential voltages are used to obtain the difference in the AC voltage drop across the two sensing serpentine PRTs, i.e., \( v_i = v_{i+} - v_i \) as the DC heating voltage \( (V_{DC}) \) is swept. Henceforth, the changes in temperature and resistance relative to the corresponding values at zero DC heating voltage \( (V_{DC} = 0) \) will be denoted by \( \Delta \). With this notation, \( v_i \) varies with the applied \( V_{DC} \) because the sensing PRT temperature rise on the sample device \( (\Delta T_s) \) is different from that on the reference device \( (\Delta T_{s,ref}) \), namely,

\[
\Delta v_i = i \frac{d R_s}{d T} \Delta T_s - i_{ref} \frac{d R_{s,ref}}{d T} \Delta T_{s,ref}. \tag{14}
\]
In the experiment, \( v_s = i_R - i_{ref} R_{s,ref} \) is zeroed when \( V_{DC} = 0 \) by adjusting \( R_{AC,s,ref} \). Under this condition, \( \Delta v_s \) would remain zero if \( \delta T_s = \Delta T_s - \Delta T_{s,ref} \) vanishes, and \( \delta T_s = \Delta v_s / (i d R_s / d T) \). In addition, we employ the approach discussed below to adjust the heating to the two membranes so that \( \delta T_s = 0 \) when \( G_s = G_{s,ref} \), where \( G_{s,ref} \) is the thermal conductance across the gap between the two membranes of the reference device. It can be shown that the sensing membrane temperature rise follows the general scaling relation \( \Delta T_s \propto \Delta T_{s,ref}/G_{b2} \propto \int^2 (R_h + \beta R_b) / G_{b1} G_{b2} \). Therefore, when \( G_s = G_{s,ref} \),

\[
\delta T_s = \Delta T_{s,ref} \left( \frac{I_{ref}^2 (R_h + \beta R_b)}{I_{ref}^2 (R_{s,ref} + \beta R_{s,ref}) (G_{b1,ref} G_{b2,ref}) - 1} \right),
\]

which vanishes when \( R_{DC,ref} \) is adjusted until the factor

\[
C_b \equiv \frac{1}{I_{ref}^2} \left[ \frac{G_{b1} G_{b2}}{G_{b2,ref} G_{b1,ref}} \frac{R_{s,ref} + \beta R_{s,ref}}{R_h + \beta R_h} \right]^{1/2}.
\]

This ensures \( \Delta T_s \) equals \( \Delta T_{s,ref} \) when \( G_s \) and \( G_{s,ref} \) are equal.

Under the assumption that the background thermal conductance for the sample device is the same as that of the reference device, \( \delta T_s \) measured under the above balancing condition is the temperature rise due to heat conduction through the nanowire on the sample device. Therefore, Eq. (12) becomes valid if \( \Delta T_s \) is replaced by \( \delta T_s \), to give the thermal conductance of the nanostructure,

\[
G_n = G_{b2} \frac{\delta T_s}{\Delta T_{s} - \delta T_s}.
\]

IV. DETERMINATION OF PARASITIC HEAT LOSS FROM THE BEAMS

Although the differential measurement method can eliminate the background thermal conductance between the heating side and the sensing membrane, it does not eliminate the parasitic heat loss via radiation and residual gas molecules from the supporting beams to the environment at \( T_0 \). Such heat loss from the supporting beam to residual gas molecules and radiation is accounted for in the factor \( \beta \) in Eqs. (6) and (8), which is determined by measuring the ratio between the average temperature rise in one supporting beam of the sensing membrane and the temperature rise in the sensing membrane. When such measurements are conducted with the differential scheme of Sec. III to eliminate the background heat transfer between the heating side and the sensing side, the temperature distribution along each of the six beams supporting the sensing membrane (\( i = 7–12 \)) can be described by a modified version of Eq. (1), with \( Q_L = 0 \), as

\[
\frac{\delta T_{b,i}(x)}{\delta T_s} = \frac{\sinh m(L - x)}{\sinh mL} \quad \text{for} \quad i = 7–12,
\]

where \( \delta T_s \) and \( \delta T_{b,i}(x) \) are the difference in the sensing PRT temperature and local beam temperature between the sample device and reference device, respectively. The spatially averaged temperature rise along one of the six beams can be expressed in a dimensionless form:

\[
\frac{\delta T_{b,i}(x)}{\delta T_s} = \frac{1}{L} \int_0^L \frac{\delta T_{b,i}(x)}{\delta T_s} \, dx = \frac{1}{mL} \cosh mL - 1.
\]

In the limit of vanishing \( mL \), corresponding to negligible parasitic heat loss via radiation and residual gas molecules, Eq. (19) is reduced to \( \delta T_{b,i}/\delta T_s = 0.5 \), corresponding to a linear temperature profile in the beam. By measuring the temperature rise of the sensing PRT \( \delta T_s \) and the average temperature rise on one sensing beam \( \delta T_{b,i} \), the \( mL \) product from Eq. (19) may be obtained, from which the factor \( \beta \), according to Eq. (8), can be calculated. The measurement of \( \delta T_s \) can be carried out with the balancing condition discussed in Sec. III. However, for the measurement of \( \delta T_{b,i} \), the \( R_{AC,s,ref} \) resistor must be adjusted so that \( v_b \) equals zero when \( V_{DC} = 0 \), without changing \( R_{DC,ref} \). This condition follows from a similar analysis as that in Sec. III.

In addition to heat loss from the supporting beams, radiation loss from the circumference of the nanowire can result in an underestimate of the thermal conductance of the sample. However, based on Eq. (4), for a low thermal conductance sample in which \( \Delta T_s \ll \Delta T_{s,ref} \), the thermal resistance of the nanowire can be calculated as \( R_s = (\pi^2 c_0 k T^3 d)^{-1/2} \tanh (mL) \), where \( m = \sqrt{16 \epsilon \sigma T^3 / kd} \), \( d \) and \( \epsilon \) are the diameter and emissivity of the nanowire, and \( \sigma \) is the Stefan-Boltzmann constant. In the limit of \( mL \ll 1 \), this reduces to the expression for diffusive thermal resistance, \( R_s = 4L/\pi kd^2 \). Therefore, the relative error in neglecting radiation loss from the circumference of the nanowire is \( mL \coth (mL) - 1 \). For \( mL \ll 1 \), the relative error is reduced to \( (mL)^2/2 \), which decreases with \( L \) for the same aspect ratio \( L/d \) and \( \epsilon \), the latter of which can also decrease when \( d \) is smaller than the radiation penetration depth. For the nanofiber sample considered in this work with diameter \( \sim 100 \) nm, \( L \approx 8 \mu m \), and \( \epsilon \leq 0.3 \), the relative error in neglecting radiation loss from the circumference of the nanofiber is found to be 0.2%.

V. MEASUREMENT OF A NANOFIBER SAMPLE

As a demonstration of the comprehensive measurement approach to addressing errors caused by device asymmetry, background thermal conductance, and parasitic heat loss from the supporting beams, this method is employed to measure a \( \sim 100 \) nm diameter nanofiber sample suspended across an \( 8 \mu m \) gap between two SiN\(_x\) membranes of a suspended device. At each sample stage temperature, non-differential measurements were first conducted on the sample device and an adjacent reference device with two different heat flow directions. The data were first analyzed using Eq. (11) with the assumption that \( \beta = 1 \), to obtain the \( G_{b1} G_{b2}/G_{b1,ref} G_{b2,ref} \) ratio. The measured electrical resistance of the PRTs and Pt leads were then used to determine the \( R_{DC,ref} \) needed to achieving the balancing condition for the heater side according to Eq. (16), by first assuming \( \beta = 1 \). The \( \delta T_{b,i} \) and \( \delta T_s \) values were then measured in two separate measurements, with \( R_{AC,s,ref} \) adjusted to achieve \( v_b = 0 \) and \( v_s = 0 \) when \( V_{DC} = 0 \), respectively. The obtained \( \delta T_{b,i}/\delta T_s \) ratio was used to determine the \( mL \) and \( \beta \) values according to Eqs. (8) and (19). The
as-obtained $\beta$ was then used to re-analyze the non-differential measurement results and update the $G_{b1}G_{b2}/G_{b2,ref}G_{b1,ref}$ ratio, which is found to be insensitive to $\beta$ because a change in $\beta$ results in a similar change to the four beam conductance values. The updated $G_{b1}G_{b2}/G_{b2,ref}G_{b1,ref}$ ratio and $\beta$ value can be used to fine tune the balancing conditions for the measurement of $\delta T_{b,i}/\delta T_s$, so that an accurate $G_{b1}G_{b2}/G_{b2,ref}G_{b1,ref}$ ratio and $\beta$ can be obtained after one or two iterations.

Figure 4 shows one measurement of $\delta T_{b,i}$ as a function of $\delta T_s$, which yields $\delta T_{b,i}/\delta T_s = 0.49$. The slope is averaged over multiple measurement runs, and $mL$ is found from Eq. (19) to be 0.61, corresponding to $\beta = 0.97 \pm 0.01$ at 320 K. This result suggests that parasitic heat loss from the supporting beams is as small as about 3% at this temperature, although it may increase with increasing temperatures as found by a previous analysis.\textsuperscript{20}

Based on this $\beta$ value and Eq. (11), $G_{b1}$ and $G_{b2}$ were found from the non-differential measurement data to be $131 \times 10^{-9}$ and $126 \times 10^{-9}$ W/K for the sample device with a nanofiber sample, and $129 \times 10^{-9}$ and $130 \times 10^{-9}$ W/K for the reference device without a nanofiber sample at 320 K, representing a maximum of 4% variation. It is worth noting that Eq. (11) can be reduced with about 1% error to $G_{b1} = 1/\alpha_h$ and $G_{b2} = 1/\alpha_h'$ for $G_s < 1 \times 10^{-9}$ W/K, corresponding to a $\alpha_h'/\alpha_h$ ratio of the order of 100.

In addition, Eqs. (12a) and (12b) yield two $G_s$ values which are within 1% difference. These two values are averaged to obtain the conductance of the sample, $G_s$, to be $1.16 \pm 0.02 \times 10^{-9}$ W/K at 320 K, as shown in Figure 5(a). Similarly, the conductance of the blank reference device, $G_{s,ref}$, was found to be $0.82 \pm 0.02 \times 10^{-9}$ W/K at the same temperature, yielding $G_n = G_s - G_{s,ref} = 0.34 \pm 0.03 \times 10^{-9}$ W/K, which suffers from a large uncertainty associated with subtracting two large numbers of comparable magnitude. The measured background conductance of $0.82 \pm 0.02 \times 10^{-9}$ W/K near room temperature is considerably higher than the $0.4 \times 10^{-9}$ W/K value reported previously for a slightly different device geometry.\textsuperscript{18} Several factors can contribute to variations in the background conductance, namely, variations in the gap spacing and beam length of the device, variations in the vacuum level, and differences in radiation shield dimensions and emissivity. For example, a small radiation shield close to the sample with low emissivity can reflect the heater radiation not only back to the heater but also directly to the sensor membrane, contributing to the background signal.\textsuperscript{20} All three of these factors are expected to cause variations in the measured background conductance. This rather large variation in the background thermal conductance measured at different times also suggests a potentially large source of error for the non-differential measurement approach.

Based on the obtained $\beta$ value, the differential measurement was carried out with the correct balancing condition of Eq. (16). The measurement data were used to obtain the thermal conductance ($G_s$) of the nanofiber according to Eq. (17), where the $G_{b2}$ value was obtained from the non-differential measurement. The as-obtained $G_n$ value was $0.37 \pm 0.01 \times 10^{-9}$ W/K at 320 K, which exhibits a reduced uncertainty compared to the value obtained from the non-differential measurement. Figure 5(b) shows that the $G_s$ values obtained from the differential measurement are close to the upper limit of that from the non-differential measurement. The small discrepancy between the two measurement results can be attributed to the errors in one of the two measurements. For the non-differential measurement, one major error source can
be caused by the variation in the vacuum level in the sample space between the two separate measurements of $G_n$ and $G_{n,\text{ref}}$. For the differential measurement, the main error source can be that in the balancing conditions set by $R_{\text{DC,ref}}$ and $R_{\text{AC,ref}}$. For example, Figure 5(b) shows the $G_n$ values measured by the differential method when the $I/I_{\text{ref}}$ ratio, which sets the heating side balancing condition, is increased by 7% and 8% relative to the balancing condition of Eq. (16), which results in nearly 40% error in the obtained $G_n$. Hence, while this comprehensive approach appears to be able to measure the nanofiber sample with $G_n$ well below $1 \times 10^{-9}$ W/K with sufficient accuracy, accurate measurement of a nanowire sample with $G_n$ less than $1 \times 10^{-10}$ W/K requires additional efforts to reduce the background thermal conductance to the order of $G_n$, which may require an ultra high vacuum sample environment as well as radiation shielding between the heating and sensing membranes and beams as demonstrated in a macroscopic version of such steady state measurements.\textsuperscript{23}

Another important factor in the thermal measurement of a low-thermal conductance sample is the signal to noise ratio in the measurement of the temperature rise of the sensing PRT. A recent work has suggested that a differential measurement setup can improve the signal to noise ratio by two orders of magnitude compared to the non-differential measurement. It is worth noting that the lock-in offset and expand function have been used in many of the reported non-differential measurements for nanowire and thin film measurements,\textsuperscript{5} but were not used in the non-differential measurement results reported in that recent work.\textsuperscript{19} Figure 6(a) shows that the temperature resolution is limited to about $50 \times 10^{-3}$ K without using the expand function. In comparison, the offset and expand function can be used to enhance the lock-in resolution by a factor of 100, so that the noise in the as-measured $\Delta T_s$ exhibits the true temperature fluctuation in the sensing PRT, which can be controlled to be as low as about $1 \times 10^{-2}$ K. This noise can be reduced somewhat by using a differential measurement either with or without electrical heating in the heating PRT, because the differential measurement can eliminate the common mode temperature fluctuation in the sensing PRTs of the sample and reference device, as shown in Figure 6(b). However, not all of the temperature fluctuation in the two sensing PRTs, which are thermally isolated from each other, is eliminated. As shown in Figure 6(b), in order to achieve a temperature noise less than $1 \times 10^{-3}$ K, the AC excitation current or the time constant of the lock-in amplifier needs to be increased, which can result in unwanted heating in the sensing membrane or long measurement time and consequently signal drifting.

VI. CONCLUSION

The above analysis and experimental results show that the comprehensive measurement approach consisting of non-differential and differential measurements can allow for the measurement of a nanowire sample with a low thermal conductance of the order of $10^{-10}$ W/K, which is about one order of magnitude improvement compared to that achievable with the previous non-differential measurement method. At each environment temperature and for either the sample device with the nanostructure sample or the blank reference device without the nanostructure, two non-differential measurements with opposite heat flow directions are used to account for the asymmetry in the suspended device. By properly balancing the heating currents and sensing currents in both devices in a differential measurement setup, the background thermal conductance via residual gas molecules and radiation can be eliminated and the thermal conductance of the nanostructure can be obtained directly. Moreover, the parasitic heat loss from the long supporting beams of the device can be determined by measuring the average temperature rise in the sensing PRT and in one of the beams connected to the sensing PRT by using a differential measurement setup. The measurement approach does not suffer from the large uncertainty associated with subtracting the background thermal conductance from the sample thermal conductance, the two of which have a similar magnitude. In order to utilize this comprehensive method for measuring a nanostructure sample with thermal conductance less than $1 \times 10^{-10}$ W/K, it is still crucial to reduce the background thermal conductance to a similar magnitude or smaller, which may require an ultra high vacuum sample environment and a radiation shield between the heating side and the sensing side of the device. Such a radiation shield has been used in a macroscopic measurement.\textsuperscript{23} In addition, the

![Figure 6](image_url)

**FIG. 6.** (a) Sensing PRT temperature rise measured with the non-differential measurement setup with and without using the 100 expand function of the lock-in amplifier. (b) Sensing PRT temperature rise for the non-differential measurement with 0.5 μA AC excitation current and 300 ms time constant, and for the differential measurement for 0.5 μA and 5.0 μA AC excitation current, and 300 ms and 3000 ms time constant. Inset shows noise level near zero heating.
measurement shows that it is important to use the offset and expand function of the lock-in amplifier for improving the temperature resolution in the non-differential measurement.

ACKNOWLEDGMENTS

This work is supported by US National Science Foundation (NSF) Award No. CBET-0933454. A.W. acknowledges support from the NSF Graduate Research Fellowship Program. K.B. would like to thank the support of Natural Science Foundation of China (Award No. 51205061) and Natural Science Foundation of Jiangsu Province (Award No. BK2012340). The nanofiber sample used to demonstrate the measurement method was synthesized by Dr. Virendra Singh and Professor Baratunde Cola at Georgia Institute of Technology, who will report the measurement results of all the nanofiber samples elsewhere.