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Room temperature picowatt-resolution calorimetry

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Picowatt-resolution calorimetry is necessary for fundamental studies of nanoscale energy transport. Here, we report a microfabricated device capable of <4 pW resolution—an order of magnitude improvement over state-of-the-art room temperature calorimeters. This is achieved by the incorporation of two important features. First, the active area of the device is thermally isolated by thin and long beams with a total thermal conductance (G) of ~600 nW/K. Further, a bimaterial cantilever thermometer capable of a temperature resolution (ΔT_{res}) of ~4 μK is integrated into the microdevice. The small thermal conductance and excellent temperature resolution enable measurements of heat currents ($q = G \times \Delta T_{res}$) with a resolution <4 pW. © *2011 American Institute of Physics*. [doi:10.1063/1.3617473]

Understanding thermal transport at the nanoscale is essential for the development of energy conversion technologies. Towards this challenging goal, several researchers have made critical progress in developing nanoscale thermal measurement techniques,[1–3] including the development of suspended microdevice structures[1] that have enabled ~1 nanowatt (nW) power resolution measurements at room temperature. This technical advance has been applied to a variety of nanoscale phonon transport studies.[1,4] Moreover, a bimaterial cantilever-based calorimeter[3] with a reported resolution[2] of ~40 picowatt (pW) has been used recently in the analysis of nanoscale photon[5] and phonon[6] transport mechanisms. In spite of this important progress, several nanoscale thermal transport phenomena have not been characterized. For example, the effect of surface chemistry on the near field radiative transport properties and heat transport properties of atomic scale point contacts and molecular junctions are of great interest to researchers, but remain largely unexplored.

Elucidation of many of these nanoscale heat transport phenomena requires measurement techniques with single-digit picowatt-resolution. To overcome this challenging technical barrier, we present a technique that can measure, at room temperature, heat currents as small as 4 pW and detect changes in heat flow with a resolution smaller than 4 pW—at least an order of magnitude improvement over previously reported state-of-the-art calorimeters.[2] The basic strategy employed in this work for achieving picowatt-resolution calorimetry is to microfabricate a thermally isolated device from which very precise temperature measurements can be made. The calorimeter (Fig. 1) consists of a thin low stress silicon nitride (SiN_x) membrane that is suspended by thin and long SiN_x beams which have a combined thermal conductance (G) of ~600 nW/K and serve to thermally isolate the suspended membrane. Further, a bimaterial cantilever (BMC) that can detect periodic temperature variations with a resolution (ΔT_{res}) of ~4 μK and a noise floor of ~6.4 μK is integrated into the suspended membrane. When this suspended device is operated in a high vacuum environment ($<10^{-6}$ Torr), thermal conduction via the gas molecules and heat transport by radiation are negligible, ensuring that the total thermal conductance between the suspended region and the environment is ~600 nW/K. The low thermal conductance of the beams and the excellent temperature resolution of the bimaterial cantilever enable single-digit picowatt resolution.

A schematic of the picowatt calorimeter along with a scanning electron micrograph of a fabricated device is shown in Figure 1. The active region of the device is a SiN_x membrane with a thickness of ~0.5 μm. The membrane is suspended by four SiN_x beams, each of which is 50 μm long, 2 μm wide, and 0.5 μm thick. A serpentine gold (Au) line, which is 600 nm wide and 30 nm thick, is integrated into the membrane and serves as both a heater and a thermometer. Further, a bimaterial cantilever made from SiN_x and Au having a length of ~200 μm and a width of ~40 μm is also incorporated into the suspended region. Following previous studies, where bimaterial cantilever sensor sensitivity was optimized, we chose the thickness of Au and SiN_x layers to be ~125 and ~500 nm, respectively.[8]

To evaluate the performance of the picowatt calorimeter described here, we present a theoretical estimate of the

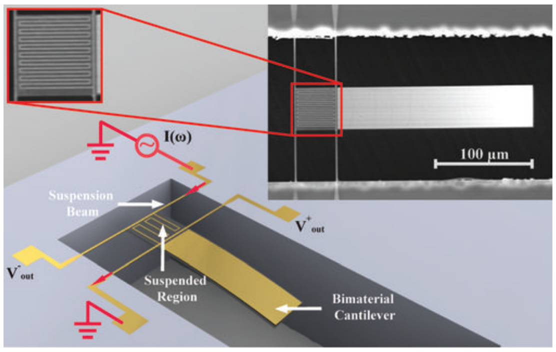

FIG. 1. (Color online) Schematic of a picowatt calorimeter is shown along with a scanning electron micrograph of a microfabricated device (inset). The central area of the device is suspended by thin (~2 μm) and long (~50 μm) beams. A serpentine line that serves as a 4-probe heater/thermometer and a 200 μm long bimaterial cantilever that acts as an ultra-sensitive thermometer are integrated into the suspended region.

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thermal conductance of the beams, the thermal time constant of the device, and the expected temperature resolution of the bimaterial cantilever thermometer. The thermal conductance (G) of the beams, including the Au coating on them, is estimated to be $\sim$0.5 μW/K ($G = 4(k_{SiN_x}A_{SiN_x} + k_{Au}A_{Au})/L_b$), where k_{SiN_x} ($\sim$2 W/m·K) and k_{Au} ($<$100 W/m·K) are the thermal conductivities of thin film SiN$_x$ (Ref. 9) and thin film Au (Ref. 10), respectively, A_{SiN_x},A_{Au} are the areas of cross section of silicon nitride and gold in the beams, and L_b is the length of the beams. Another parameter of interest is the thermal time constant (τ) of the device, which limits the temporal response of the device. A lumped capacitance approach[7] suggests that τ is related to the thermal conductance of the beams (G) and the total heat capacity of the suspended region by $\tau = (\textit{heat capacity of suspended region})/G$. Given the device dimensions, this implies that the time constant is $\sim$30 ms. This estimate suggests that sinusoidal heat currents at a frequency much smaller than the cutoff frequency f_o ($1/2\pi\tau = 5.3$ Hz) result in a full thermal response, whereas heat inputs at frequencies much larger than the cutoff frequency result in an attenuated thermal response.

Bimaterial cantilevers are well suited for detecting extremely small temperature changes.[3] A temperature change, ΔT, of a bimaterial cantilever results in deflections due to the differential expansion of SiN$_x$ and Au. The deflection sensitivity of the cantilever can be obtained from[2] $\Delta z/\Delta T = -3(\gamma_1 - \gamma_2)(t_1 + t_2)l^2/Kt_2^2$, where $K = 4 + 6(t_1/t_2) + 4(t_1/t_2)^2 + (E_1t_1/E_2t_2)^3 + (E_2t_2/E_1t_1)$. Here, Δz is the deflection of the tip of the cantilever and subscripts 1 and 2 refer to Au and SiN$_\mathrm{x}$, respectively. The symbols γ_1 and γ_2 are the coefficients of thermal expansion, E_1 and E_2 are the Young's moduli, t_1 and t_2 are the thicknesses, and l is the length of the cantilever. From the above equation the deflection sensitivity of the bimaterial cantilever used in this work is estimated to be $\sim$257 nm/K. Therefore, if tip deflections are detected with a resolution of 0.01–0.001 nm, temperature changes of $\sim 4 \times 10^{-5}$ K to 4×10^{-6} K can be measured. Measurement of such small deflections can be accomplished by optical means and is implemented in this work.[7]

In order to experimentally characterize the thermal conductance of the beams (G) a sinusoidal current of known amplitude (I_o) and frequency (f) is supplied through the serpentine heater (Fig. 1) resulting in joule heating, $q = I_o^2R/2 \times [1 - \cos(2\pi \times 2f \times t)]$, where R is the resistance of the portion of the heater line embedded between the voltage measuring probes of the serpentine heater line (Fig. 1). This heat input q has a second harmonic ($2f$) component, which results in temperature oscillations with amplitude ΔT_{2f} at a frequency $2f$. We note that when a sinusoidal current at a sufficiently low frequency is passed through the serpentine heater line, the thermal gradients within the suspended region are negligible.[7] This is because the internal resistance to heat flow within the suspended region is much smaller than the thermal resistance within the beams that suspend the central region. Under these conditions, the amplitude of the temperature oscillations (ΔT_{2f}) of the island can be related to the amplitude of the voltage oscillations at $3f$ (V_{3f}) across the four-probe serpentine resistor integrated into the microdevice by $\Delta T_{2f} = 2V_{3f}/(I_oR\alpha)$, where α is the temperature coefficient of resistance (TCR) for the Au line.[7]

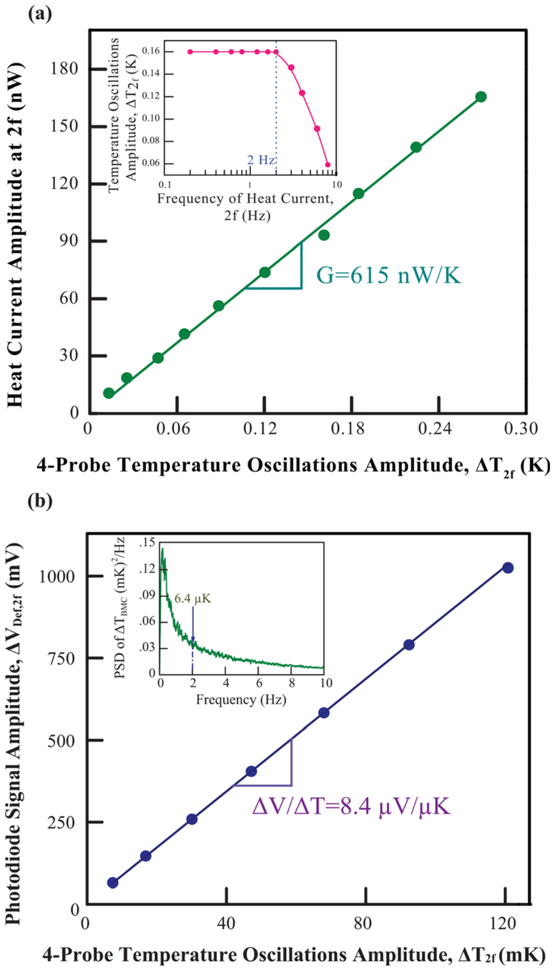

FIG. 2. (Color online) (a) The measured amplitude of temperature oscillations of the suspended region (x-axis) for known inputs of heat currents (y-axis) at 2 Hz is shown. The inset shows the thermal response of the microdevice as a function of the frequency ($2f$) of the input sinusoidal heat current. (b) The response of the bimaterial cantilever thermometer is calibrated by inputting heat currents of known amplitudes at 2 Hz ($2f$) while simultaneously recording the bimaterial cantilever oscillations ($\Delta V_{Def,\,2f}$) and the temperature oscillations of the suspended region. The inset shows the power spectral density (PSD) of the noise in the bimaterial cantilever deflections.

The measured amplitude of temperature oscillations for heat currents of fixed amplitude ($\sim$100 nW) at various frequencies are shown in Figure 2(a) inset. The amplitude of temperature oscillations of the suspended region are invariant for frequencies less than or equal to 2 Hz (cut-off frequency $\sim$5 Hz). However, for frequencies larger than the cut-off frequency the temperature response is attenuated as expected for a first order system.[7] The thermal conductance of the beams is experimentally determined from the measured amplitude of temperature oscillations for known sinusoidal heat inputs using $G = q(2f = 2\mathrm{Hz})/\Delta T_{2f}$, where $q(2f = 2$ Hz) is the amplitude of the sinusoidal heat input at 2 Hz and is well approximated[7] by $I_o^2R/2$. The obtained temperature oscillations for various known heat inputs at 2 Hz are shown in Figure 2(a), the slope of which shows that the thermal conductance of the beams is (0.615 ± 0.04) μW/K. The uncertainty ($\sim$7%) arises primarily from the joule heating in the beams.[7]

Measuring heat currents with picowatt sensitivity requires excellent temperature resolution. To achieve this, a bimaterial cantilever sensor capable of $\sim$4 μK resolution[2] was integrated into the microdevice. The deflection of the bimaterial cantilever is proportional to the temperature change and is measured using an optical detection scheme[7] that outputs a voltage signal (V_{Def}) proportional to the deflection. The temperature induced deflection response of the bimaterial cantilever was calibrated by using the four-probe thermometer (serpentine line) integrated into the device. Specifically, sinusoidal heat currents at 2 Hz ($2f$) (frequency chosen based on signal to noise ratio considerations described below) were input into the serpentine heater of the microdevice resulting in sinusoidal temperature oscillations. The temperature oscillations were monitored by using the four-probe thermometer, while the amplitude of the oscillations of the bimaterial ($\Delta V_{Def,\ 2f}$) at $2f$ was detected by the optical scheme and recorded using a lock-in amplifier (bandwidth of 1 mHz). The data obtained in these measurements (Fig. 2(b)) show that the deflection sensitivity is $\sim$8.4 μV/μK.

To determine the frequency corresponding to optimal performance of the device, the signal to noise ratio was characterized at various frequencies. The noise in the measured voltage ($\Delta V_{Def,\ 2f}$) arises due to thermal fluctuations, random vibration sources that excite the cantilever, thermal drift, and electronic noise. To minimize the thermal drift and acoustic/seismic vibrations, all the studies were performed in a temperature controlled stage (305 K ± 1 mK), in a high vacuum chamber ($<10^{-6}$ Torr) located on an isolated optical table. The combined effect of all noise sources is quantified by the power spectral density (PSD) of V_{Def} in the absence of any heat input to the device. The measured PSD (Fig. 2(b), inset) shows that noise is larger at lower frequencies. Such a behavior is expected due to the *1/f* noise arising from thermal and electronic drift in the system. From the measured frequency dependence of the signal (Fig. 2(a), inset) and the measured PSD, the signal to noise ratio is determined to have a maximum at $\sim$2 Hz.[7]

To experimentally determine the ultimate sensitivity of the device, picowatt-level sinusoidal thermal excitations were applied via the serpentine heater and temperature oscillations ($\Delta T_{BMC,\ 2f}$) were detected from the bimaterial cantilever (Fig. 3). Each data point in Figure 3 corresponds to the mean of 10 individual measurements, and the error bar represents the standard deviation. These data show that even when no power is input, the drift in the bimaterial cantilever corresponds to a temperature oscillation ΔT_{noise} of $\sim$6.4 μK. This shows that the noise equivalent power input ($q_{NEP} = G \times \Delta T_{noise}$) of the device is $\sim$4 pW. To determine the resolution of the device, the power input to the device was increased from 0 to 40 pW in steps of 2 pW. The measured mean values of $\Delta T_{BMC,\ 2f}$ are found to increase directly proportionally with the thermal loading, demonstrating that changes in heat currents smaller than 4 pW can be resolved.

In order to determine the dynamic range of the device, the amplitude of sinusoidal heat input was varied from 10 pW to 100 nW, while the temperature oscillations were measured by monitoring the deflections of the bimaterial cantilever (Fig. 3, inset). Remarkably, the response of the device is linear over the entire range suggesting that the dynamic range of the device spans at least *three* orders of magnitude. Although the device response is linear for this large range, the response for low heat inputs ($\sim$0–10 pW) is non-linear (Fig. 3). This behavior can be understood by noting that the amplitude of the bimaterial cantilever oscillation is determined by: (1) the amplitude of sinusoidal heat inputs, which results in bimaterial cantilever oscillations with a mean square value of $\langle x_S^2 \rangle$ and (2) random forces arising from thermal noise and ambient mechanical vibrations, which in the absence of any other forces results in a displacement with a mean square value of $\langle x_{random}^2 \rangle$. It can be shown[7] that the combined effect of these two forces is to give a total cantilever deflection whose RMS value $\langle x_{deflection}^2 \rangle^{1/2}$ is equal to $[\langle x_S^2 \rangle + \langle x_{random}^2 \rangle]^{1/2}$. The solid line in Figure 3 accounts for the effect described by the above equation and is seen to agree well with the experimentally obtained data.

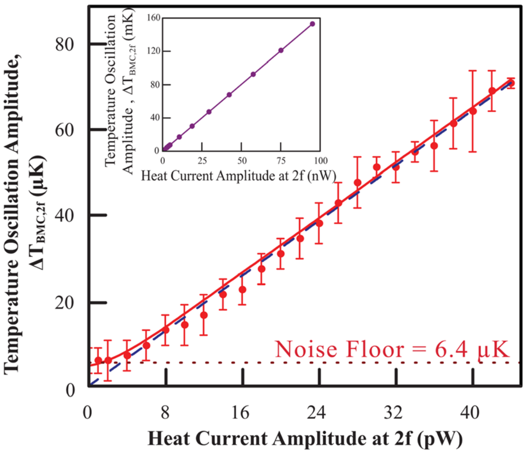

FIG. 3. (Color online) The measured temperature oscillations ($\Delta T_{BMC,\ 2f}$ determined using the bimaterial cantilever) for various inputs of sinusoidal heat currents (0–44 pW, increased in steps of 2 pW) at 2 Hz. The noise floor of the device ($\sim$6.4 μK) corresponds to <4 pW heat inputs. In the inset the response of the device to heat inputs of up to 100 nW is shown, demonstrating a dynamic range exceeding three orders of magnitude.

To summarize, we have demonstrated a calorimetric technique capable of a resolution better than 4 pW at room temperature—an order of magnitude improvement over state-of-the-art methods. This is achieved by integrating an ultra-sensitive bimaterial cantilever based thermometer into a suspended microdevice. This device is well suited for the study of nanoscale thermal transport and is expected to be an important tool for nanoscale heat transport studies where heat flows on the order of a few picowatts are expected to occur.

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