

Quantitative Mapping of Unmodulated Temperature Fields with Nanometer Resolution

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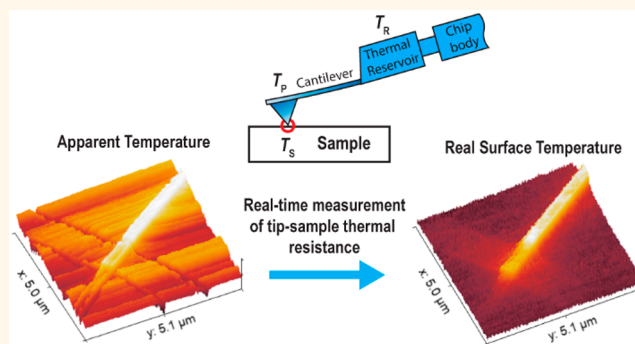
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Supporting Information

ABSTRACT: Quantitative mapping of temperature fields with nanometric resolution is critical in various areas of scientific research and emerging technology, such as nanoelectronics, surface chemistry, plasmonic devices, and quantum systems. A key challenge in achieving quantitative thermal imaging with scanning thermal microscopy (SThM) is the lack of knowledge of the tip–sample thermal resistance (R_{TS}), which varies with local topography and is critical for quantifying the sample temperature. Recent advances in SThM have enabled simultaneous quantification of R_{TS} and topography in situations where the temperature field is modulated enabling quantitative thermometry even when topographical features cause significant variations in R_{TS} . However, such an approach is not applicable to situations where the temperature modulation of the device is not readily possible. Here we show, using custom-fabricated scanning thermal probes (STPs) with a sharp tip (radius ~ 25 nm) and an integrated heater/thermometer, that one can quantitatively map unmodulated temperature fields, in a single scan, with ~ 7 nm spatial resolution and ~ 50 mK temperature resolution in a bandwidth of 1 Hz. This is accomplished by introducing a modulated heat input to the STP and measuring the AC and DC responses of the probe's temperature which allow for simultaneous mapping of the tip–sample thermal resistance and sample surface temperature. The approach presented here—contact resistance resolved scanning thermal microscopy (CR-SThM)—can greatly facilitate temperature mapping of a variety of microdevices under practical operating conditions.

KEYWORDS: scanning thermal microscopy, nanoscale quantitative temperature mapping, quantifying contact resistance, platinum resistance thermometer probes, unmodulated temperature fields



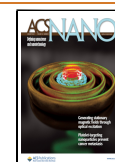
In recent years, probing energy transport and dissipation in nanoscale devices has become critically important. In order to study all these phenomena, it is necessary to probe temperature fields with nanometer resolution. Nanoscale thermometry and thermal microscopy enable such studies and play an important role in various fields, including nanoscale transport,^{1–4} biomedical research,^{5–7} material synthesis and fabrication,^{8–10} electronics,^{11–15} and quantum devices.^{16–18} Specifically, scanning thermal microscopy (SThM), which employs scanning thermal probes (STPs), has enabled temperature mapping with nanometer spatial resolution.^{19–21} In performing quantitative studies in a vacuum environment, STPs are typically used in contact mode, where the heat transfer between the sample and the STP is dominated by conduction via the contact between the STP tip and the sample.^{20,21}

A key challenge for quantitative temperature mapping of a sample using STPs is that the temperature measured by the probe depends on both the sample temperature and the tip–sample thermal contact resistance (R_{TS}). The value of R_{TS} depends on the exact geometry of the sample at the point of contact as well as the local thermal conductivity of the sample. Therefore, it is critical to know the value of R_{TS} when measuring temperature fields from surfaces with variations in topology and thermal conductivity or time-dependent

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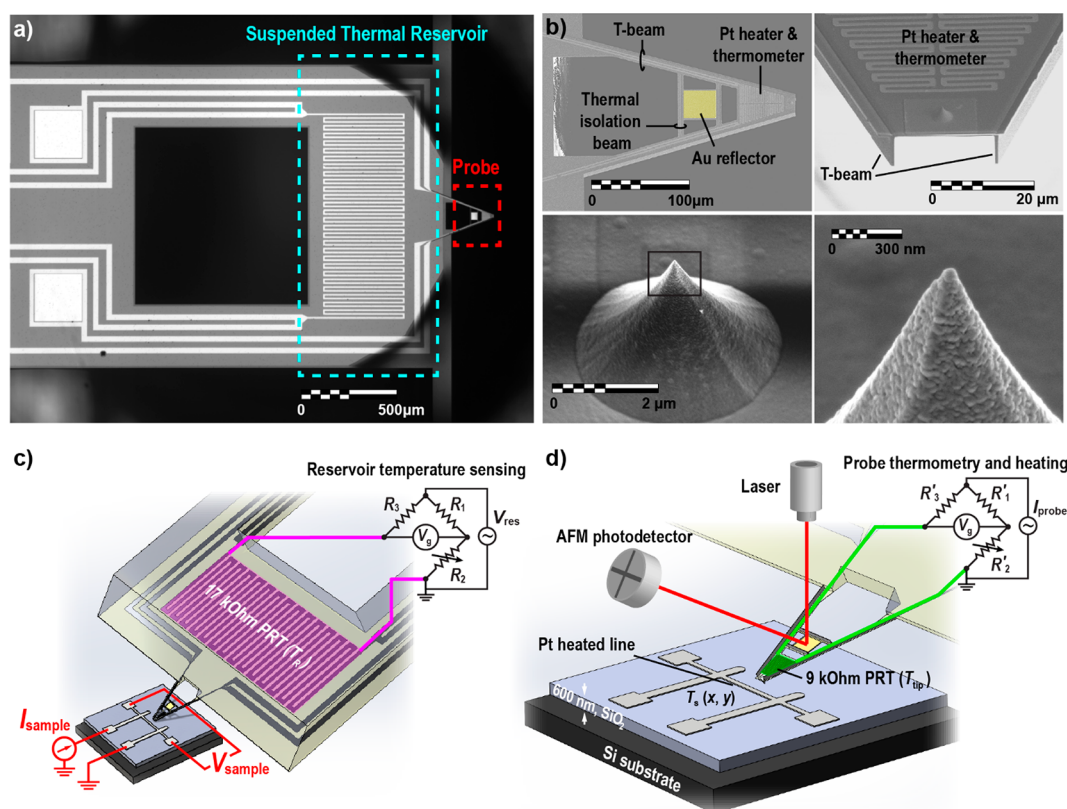


Figure 1. (a) Optical microscopy image of the entire probe showing a suspended region (blue dashed box), which acts as a thermal reservoir, and the suspended scanning thermal probe (red dashed box). (b) Tilted false color SEM micrographs (top left panel) of the scanning thermal probe (STP), along with a zoomed in image (top right panel) showing the Pt serpentine and T-beams. The bottom panels show zoomed in images of the probe tip. (c) Schematic of the probe during measurement. A DC current (I_{sample}) is sourced through the Pt line which generates a steady state temperature field on the sample. A wheatstone bridge driven by an AC voltage is used to read the resistance of the first PRT for sensing the reservoir temperature (T_R). (d) Schematic of the scanning thermal microscope in contact with a heated Pt line. The second PRT integrated into the STP is connected to a Wheatstone bridge, driven by a specific current waveform (I_{probe}) which is used for temperature sensing and modulated heating of the STP (see the main text and the [Methods](#) section).

variations in tip geometry arising from abrasion, plastic deformation, or contamination of the tip while scanning, all of which can lead to spurious signals.^{20,21} Due to these challenges, temperature maps obtained by scanning a sample surface using STPs often include artifacts.^{11,19,22–24} Therefore, to obtain a quantitative map of the sample temperature, one needs to also simultaneously measure the spatial variation of R_{TS} .

Past studies have employed several techniques for quantitative temperature sensing using SThM, such as the null-point (NP) measurement technique^{25,26} that operates by identifying the probe temperature at which the probe-sample heat flow is largely nullified ensuring that the probe temperature equals the local sample temperature. Another method for quantitative temperature sensing is the double-scan measurement,^{27,28} which first determines R_{TS} by comparing the temperature of the probe when it is out-of-contact to that of the probe when it is in contact with the sample at ambient temperature. Subsequently, this map of R_{TS} values is used to determine the sample temperature under operation (*i.e.*, when a temperature gradient is established on the sample). These techniques represent an important step forward, as they quantify the tip-sample contact resistance by combining information obtained from multiple scans or multiple approach-retract cycles. However, they cannot completely

capture the variation of R_{TS} with time for the reasons described above.

A recent technique^{29,30} has aimed to overcome these challenges in quantifying R_{TS} by employing samples whose temperature is periodically modulated. However, this interesting approach cannot be directly applied to temperature mapping of samples or devices which operate under steady state conditions or where modulation of the sample temperature at reasonable frequencies is impractical. Examples include temperature fields generated in biological systems and by surface reactions where the heat source cannot be turned on and off on demand. Other important examples, relevant to this work, involve microdevices where temperature modulation leads to periodic thermal expansion that both mechanically stresses the device and generates additional artifacts during scanning or devices where the amplitude of the modulated temperature field is large and invalidates the assumption of temperature-independence of R_{TS} . Therefore, in many applications, it is essential to have the device operate under steady conditions and perform thermal microscopy without perturbing device operation.

In this work we present an approach—contact resistance resolved scanning thermal microscopy (CR-SThM)—that leverages scanning probes to quantitatively map unmodulated temperature fields even on samples with topography and material variations. Briefly, our approach is based on applying a

modulated heat input to the probe and simultaneously measuring the amplitude of the temperature oscillations as well as the mean temperature of the probe, while the probe scans the sample of interest, in contact mode and in an ultrahigh vacuum environment. Data from these simultaneous measurements contain information on both the sample temperature and R_{TS} , which are extracted using a dynamic, lumped capacitance thermal model. Our approach suppresses artifacts generated by topography variations, STP tip abrasion, and temperature drift, enabling measurements of sample temperature with a temperature resolution of ~ 50 mK (in a bandwidth of 1 Hz), a long-term temperature stability of <10 mK, and a spatial resolution of ~ 7 nm.

RESULTS AND DISCUSSION

In order to quantitatively measure unmodulated temperature fields, we custom-fabricated STPs (see section S1 of the Supporting Information for fabrication details). Images of the fabricated probes, obtained using optical microscopy, are shown in Figure 1a. The probe consists of two suspended regions: The first region, identified by a blue box, shows the suspended thermal reservoir, while the second region, identified by a red box, indicates the scanning thermal probe. The suspended thermal reservoir serves to thermally isolate the suspended scanning probe from the ambient environment and acts as a thermal reservoir for the suspended probe. This thermal reservoir features a 17 k Ω Pt serpentine line which is used as a platinum resistance thermometer (PRT) to sense the reservoir temperature T_R using a Wheatstone bridge and a lock-in measurement technique (see the Methods section). The suspended scanning probe is characterized by both high thermal isolation (quantified below) for the tip and a relatively high mechanical stiffness (~ 7.7 N/m). This large mechanical stiffness (see section S2 of the Supporting Information for an estimate of the stiffness) is accomplished by incorporating T-shaped beams that can be seen in the scanning electron microscope images shown in Figure 1b. Further, a gold-coated region (see Figure 1b) that is used as a reflector for an incident laser beam is also incorporated into the probe and is thermally isolated from the rest of the probe using thin beams to minimize detrimental effects arising from laser heating. Finally, it can be seen that the tip of the probe has a radius of curvature of ~ 25 nm enabling very high spatial resolution imaging as described later.

The suspended scanning probe also features a ~ 9 k Ω PRT that is located close to the tip and is used for heating the probe and for sensing temperatures. In our experiments, this PRT is integrated into a full Wheatstone bridge that is driven by a time (t)-dependent current ($I_{\text{probe}}(t)$) consisting of two superimposed sinusoidal waves, $I_{\text{probe}} = I_1 \sin(2\pi f_1 t) + I_2 \sin(2\pi f_2 t)$. The first current component ($I_1 = 56$ μ A, $f_1 = 4$ Hz) leads to a modulated heat input resulting in sinusoidal oscillations of the temperature of the probe at a frequency of $2f_1$ and the amplitude T_{AC} that is recorded during scanning (see the Methods section). The second AC current component ($I_2 = 14$ μ A, $f_2 = 212$ Hz), which has a smaller amplitude and a higher frequency (the frequency was chosen to be much higher than the thermal cutoff frequency of the suspended scanning probe (~ 8.9 Hz, see Figure 2c), is used for sensing the unmodulated DC temperature of the STP (labeled T_{DC}) via a lock-in technique (see the Methods section). Our approach for quantitatively mapping unmodulated temperature fields via the STP is schematically shown in Figure 1c and d. The figure

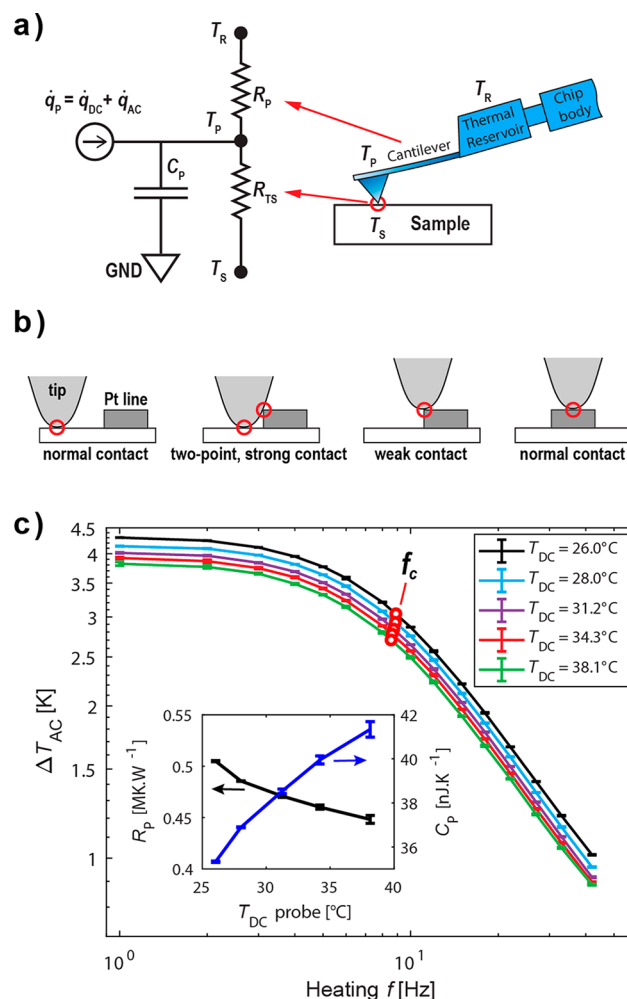


Figure 2. (a) Lumped capacitance thermal network of the STP in contact with a sample. T_R , T_P , and T_S are the thermal reservoir temperature, probe temperature, and sample surface temperature, respectively. R_P and C_P are the thermal resistance and thermal capacitance of the probe, and R_{TS} represents the probe tip–sample thermal contact resistance. Further, $\dot{q}_P$ is the heat deposition rate on the probe by the probe heater and the AFM laser. (b) Schematic demonstration of different contact geometries established during thermal scanning in the perpendicular direction to a heated Pt line sample. (c) Thermal frequency response of the STP to an AC heat input with a peak amplitude of 8.5 μ W that is deposited on the STP heater at various STP temperatures (T_{DC}). The inset shows the variation of the thermal resistance and lumped capacitance of the STP as a function of STP temperature (T_{DC}). The measurements were performed under out-of-contact conditions at a gap-size of ~ 2 μ m from the sample surface.

depicts a STP near a Pt line sample that is heated by passing a DC current (I_{sample}) through it. The resulting steady state surface temperature field $T_S(x, y)$ is quantified using CR-SThM.

THERMAL ANALYSIS FOR QUANTIFICATION OF SAMPLE TEMPERATURE

The thermal behavior of the STP when placed in contact with the sample can be modeled using a lumped capacitance thermal network^{31–33} shown in Figure 2a. In the network, R_P and R_{TS} represent the thermal resistance to heat flow within the probe (defined as the thermal resistance from the PRT

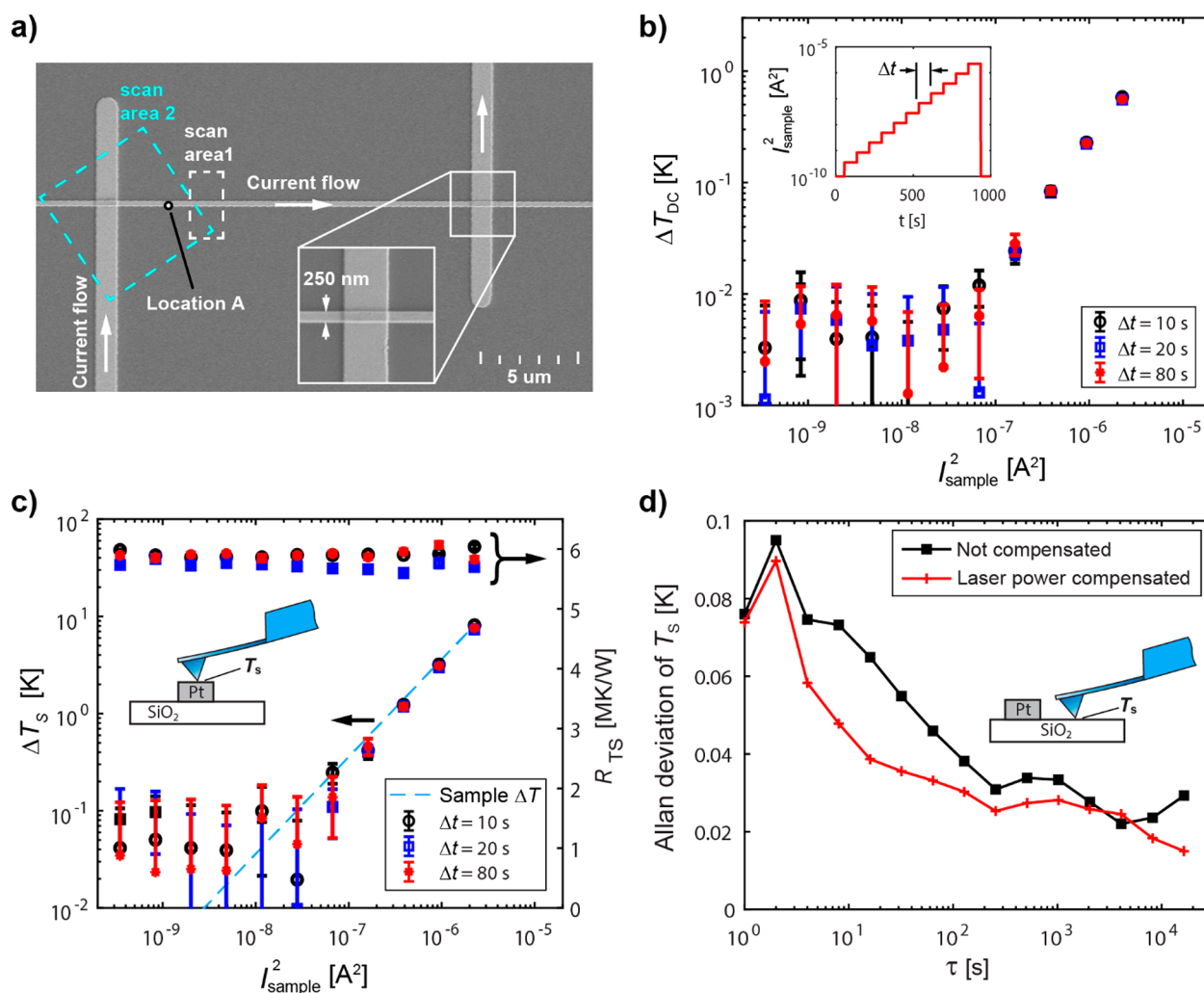


Figure 3. (a) SEM micrograph of the Pt line sample employed in this work. The Pt line is 250 nm wide and 50 nm thick. The white arrows show the path of the unmodulated electrical current (I_{sample}) employed to heat the Pt line via Joule heating, and the dashed squares mark the regions where thermal scanning was performed for the data shown in Figures 4–7. (b) Measured DC temperature rise of the probe when the probe is placed in contact with the Pt line at location A shown in panel a as a function of the square of the heating current sourced to the Pt line sample. The inset shows a stepwise increase of the heating current sourced to the sample during this experiment. (c) The data points corresponding to the left vertical axis represent the rise in sample temperature (Pt-line) estimated using eq 1. To obtain the sample temperature, the value of R_{TS} was extracted using eq 2 and plotted in this panel on the right vertical axis. The dashed line represents the Pt temperature rise as estimated from independent measurements of temperature rise vs heating current described in section 4 of the SI. (d) Allan deviation plot illustrating the stability of the sensed sample temperature (T_{s}) with and without compensation for laser power fluctuations. It is clear from this data that accounting for fluctuations in laser power output improves the measurement stability.

sensing element on the probe to the suspended thermal reservoir) and the probe tip-to-sample thermal resistance (defined as the thermal resistance from the PRT sensing element on the probe to the sample), and C_{p} is the lumped thermal capacitance of the STP. The temperature sensed by the probe PRT (labeled T_{p}) has the components T_{DC} and T_{AC} , as discussed in the previous section, while T_{R} and T_{s} are the temperatures of the probe thermal reservoir and sample, respectively. In addition, $\dot{q}_{\text{p}}$ is the rate of heat deposition on the probe by the integrated probe heater as well as the atomic force microscopy (AFM) laser, which has both DC ($\dot{q}_{\text{DC}}$) and AC ($\dot{q}_{\text{AC}}$) components. By analyzing this network, the temperature of the sample (T_{s}) can be related to other parameters (see section S6 of the Supporting Information for the derivation) via:

$$T_{\text{s}} = \frac{R_{\text{TS}}}{R_{\text{p}}}(T_{\text{DC}} - T_{\text{R}} - R_{\text{p}}\dot{q}_{\text{DC}}) + T_{\text{DC}} \quad (1)$$

As illustrated in Figure 2b, different contact geometries are established when a sample with topographical variations (such as a heated line) is studied, leading to local points of weak or strong thermal contact. It is clear from eq 1 that knowledge of R_{TS} , R_{p} , T_{DC} , T_{R} , and $\dot{q}_{\text{DC}}$ is necessary for evaluating T_{s} . As explained earlier, T_{DC} and T_{R} can be directly measured using the integrated PRTs (see the Methods section for details). We determine R_{p} and C_{p} by placing the STP $\sim 2 \mu\text{m}$ above the sample surface and measuring its thermal frequency response to modulated heat inputs at various frequencies and a peak amplitude of $\dot{q}_{\text{AC}} = 8.5 \mu\text{W}$. These measurements were repeated at various STP temperatures (T_{DC}), and the results from these measurements are shown in Figure 2c. It can be seen that the measured frequency response is consistent with

that of a simple first order system with frequency-independent behavior at low frequencies and a characteristic roll-off frequency (f_c , the -3 dB point), which is indicated by the red circles in Figure 2c (here $f_c \sim 8.9$ Hz, and corresponds to a thermal time constant of ~ 17.9 ms). Using the thermal network described earlier, one can show that the cantilever thermal resistance is given by $R_p = T_{AC} (f = 1 \text{ Hz}) / \dot{q}_{AC}$, while the thermal capacitance of the probe is given by $C_p = (2\pi R_p f_c)^{-1}$. The inset of Figure 2c shows the measured values of R_p and C_p along with their weak dependence on T_{DC} . As can be seen, R_p and C_p decrease and increase as a function of temperature, respectively. This can be attributed to the temperature dependence of the thermal conductivity and the heat capacity of SiN_x films on the probe.³⁴ We note that in this study the maximum variation in the temperature of the sample is <10 K, which corresponds to a <1 K variation of the STP temperature. Therefore, the effect of temperature variation on R_p and C_p is small and thus neglected in our subsequent analysis. However, if the presented technique is employed for measurements where the sample is heated or cooled significantly,³³ the temperature dependence of R_p and C_p must be considered.

Determination of T_s using eq 1 also requires knowledge of the spatiotemporally varying R_{TS} , which can be obtained by monitoring T_{AC} and using it to calculate R_{TS} via the following equations (see section S6 of the Supporting Information for a derivation of eqs 2 and 3):

$$R_{TS} = \frac{R_p}{\sqrt{r^2 + (r^2 - 1)(R_p C_p \omega)^2} - 1} \quad (2)$$

$$r = \frac{T_{AC-o}}{T_{AC}} \quad (3)$$

Here, T_{AC-o} is the AC response of the probe to $\dot{q}_{AC}$ when the STP is positioned away ($\sim 2 \mu\text{m}$) from the sample surface. Given that the STP is sufficiently far away from the sample, the effects of near-field heat transfer are negligible^{1,35} and T_{AC-o} is determined primarily by R_p . Finally, we note that $\dot{q}_{DC}$, which has contributions from both the Joule heating and optical power input from the AFM laser, was quantified by measuring the DC temperature rise of the probe, in response to the power incident by the laser, when the probe is located far away from the sample. Further, small variations in the contributions of the laser power to $\dot{q}_{DC}$ arising from drift in the laser power output are systematically accounted for by monitoring the power output of the laser (see section S6 of the Supporting Information).

CHARACTERIZATION OF THE TEMPERATURE SENSING RESOLUTION AND STABILITY OF CR-STHM

In order to demonstrate our SThM approach, we employed a microfabricated sample with a Pt line (Figure 3a). The 250 nm wide, 50 nm thick, and $18 \mu\text{m}$ long Pt line was deposited on a 600 nm thick SiO_2 layer, which was grown on a Si substrate.²³ We first placed the STP on the 250 nm wide Pt line at a position marked as location A in Figure 3a. Next, we heated this Pt line by supplying a DC current (I_{sample}) whose magnitude was increased as a function of time in a stepwise fashion (with each step lasting $\Delta t = 10$ s), resulting in a corresponding stepwise increase in the heat input (see inset of Figure 3b). This process was repeated for two other durations ($\Delta t = 20$ s and 80 s). The resulting temperature rise of the STP, ΔT_{DC} ,

which represents the time-averaged increase of T_{DC} at each sample heating power compared to the baseline T_{DC} value at zero sample heating power, is shown in Figure 3b as a function of I_{sample}^2 , which is proportional to the supplied heating power. Subsequently, the temperature of the STP was converted to T_s using eq 1 and the R_{TS} values extracted from eq 2, which allowed for quantification of the sample temperature rise as a function of I_{sample}^2 (see Figure 3c). In addition, the average value of R_{TS} corresponding to each T_s data point is plotted on the right vertical axis of Figure 3c. As can be seen, the value of R_{TS} has small fluctuations ($\sim 10\%$) during this specific experiment since the probe is stationary. The dashed line in Figure 3c shows the sample temperature rise as measured independently by using the Pt line as a thermometer (see section S4 of the Supporting Information), which is in good agreement with the STP measurements. The noise floor for sensing T_s in Figure 3c is 41 mK, 55 mK, 37 mK, and 26 mK for the 10 s, 20 s, 40 s, and 80 s heating durations, respectively.

To characterize the intrinsic noise and drift in temperature sensing via CR-SThM, we stabilized the temperature of the sample shown in Figure 3 to 26 ± 0.0001 °C (as shown in section S3 of the Supporting Information) and placed the STP on the SiO_2 region of the sample. The experiment was performed over a duration of 12 h. Since the sample temperature is stabilized, any fluctuation and drift in T_s (sample temperature measured using STP) that is greater than 0.1 mK is due to drift or noise in the temperature measured by the CR-SThM scheme presented here. Figure 3d shows the Allan deviation of the T_s measured by the STP. A plot of the Allan deviation quantifies the fluctuations of the time-series T_s over a range of time scales and contains information about the short-term noise and long-term drift in the measurement.³⁶ The red and black curves demonstrate the measurement stabilities of T_s with and without the effect of fluctuations in the deposited laser power (*i.e.*, variations in $\dot{q}_{DC}$), respectively. To obtain the red curve, the variations of $\dot{q}_{DC}$ due to laser power fluctuations were measured and included in eq 1 for calculation of T_s (see section S6 of the Supporting Information for details of this calculation). As can be seen, compensation for laser power fluctuations improves the measurement stability at all time scales, specifically in the 5–100 s range. In addition, as shown in section S7 of the Supporting Information, compensation for T_R drift improves the stability at time scales >500 s and results in a stability better than 10 mK for time scales >2 h. The amplitude spectrum of the measured T_s signal (see section S7 of the Supporting Information) shows similar trends and confirms the above conclusion regarding the thermal noise and stability of the STP.

THERMAL IMAGING OF STEADY TEMPERATURE FIELDS

In order to establish the thermal and spatial resolution of our approach, when used to quantitatively map the unmodulated temperature fields of a sample with topographical features, we scanned across a heated line sample to measure the topology as well as T_{DC} and T_{AC} using the PRT integrated into the STP. Subsequently, we applied eqs 1–3 to obtain the sample temperature at each pixel. Specifically, we scanned our samples using contact mode AFM at a constant contact normal force of 125 ± 25 nN, stopped at each pixel location for ~ 500 ms, and collected data for T_{DC} , T_{AC} , T_R , as well as the instantaneous laser intensity (I), before moving to the next pixel. In these

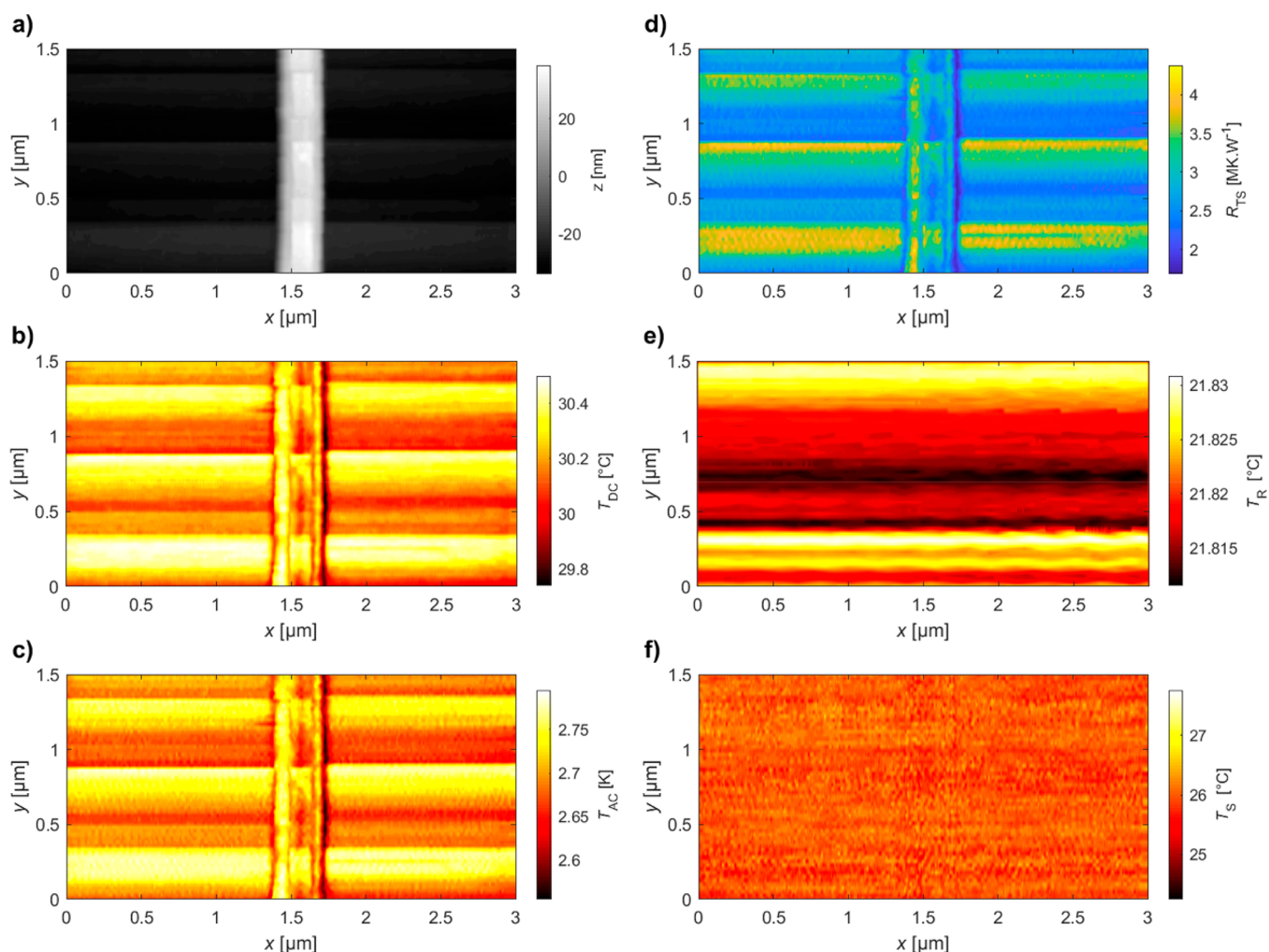


Figure 4. Data obtained from a thermal scan of a Pt line sample at zero heating current. Data shown in all panels were obtained simultaneously in a single scan. (a) Topography map showing the 250 nm wide Pt line on a SiO₂ substrate. (b) DC temperature (T_{DC}) measured by the probe's platinum resistance thermometer (PRT). (c) Amplitude of the AC temperature oscillations (T_{AC}) of the probe measured by the probe's PRT. (d) Estimated tip–sample thermal resistance R_{TS} obtained from eq 2. (e) Thermal reservoir temperature (T_R) measured using an integrated PRT on the probe thermal reservoir. (f) Extracted surface temperature map (T_S) using eq 1, with a pixel-to-pixel standard deviation of 174 mK. Refer to SI, section S10 for the histogram distribution of the pixel values.

measurements we stabilized the bulk sample temperature to 26 °C by employing feedback control (see section S3 of the Supporting Information). Next, we passed a DC current of a chosen magnitude through the Pt line, which resulted in a spatially varying temperature field $T(x, y)$ around the Pt line.

We began our thermal scans by first studying an area of the sample with an unheated Pt line (*i.e.*, a line to which no electrical current is supplied). Specifically, we scanned a $3\ \mu\text{m} \times 1.5\ \mu\text{m}$ section (scan area 1 shown in Figure 3a, 90 deg rotated) and measured the topography, T_{AC} , T_{DC} , and T_R , which are shown in parts a, b, c, and e, respectively, of Figure 4. The raster scans were done in the horizontal direction (*i.e.*, perpendicular to the Pt line), and the image size for Figures 4 and 5 was 512 (horizontal) $\times$ 64 (vertical) pixels, and the scanning rate was $\sim 12\ \text{nm/s}$. Although the entire sample is in thermal equilibrium and nominally at a constant, spatially independent temperature, the temperature map (T_{DC} , Figure 4b) as recorded by the STP probe shows systematic artifacts from the expected uniform distribution that are as large as 0.5–1 K and are aligned with the scan direction and the location of the heater line. Given the average thermal contact

and probe thermal resistances, one would expect that T_{DC} maps as shown in Figure 4b can only arise from local variations in sample temperature as large as 10 K, which is clearly not possible. Further, the T_{AC} data shown in Figure 4c also show features that are very similar to those seen in T_{DC} maps. Next, by using eq 2 along with the measured data, we obtain a spatial map of R_{TS} which is shown in Figure 4d. The data feature local minima at the edges of the Pt line where a two-point contact is presumably made, *i.e.*, both a contact that occurs between the apex of the tip and the sample and a second contact between the edge of the Pt line and the side of the tip (see Figure 2b). Further, one can observe horizontal line-by-line variations in R_{TS} , which are likely due to tip abrasion or tip modification by particle attachment (Figure 4d). In addition, we show in Figure 4e the probe reservoir temperature (T_R), which drifts by $\sim 20\ \text{mK}$ over the $\sim 9.1\ \text{h}$ duration of the thermal scan. Finally, using the measured R_{TS} and T_R maps, we construct the sample temperature T_S using eq 1 in a pixel-by-pixel fashion (see Figure 4f). As can be seen, the measured T_S is largely featureless. Measurement artifacts as revealed in the T_{DC} maps, including scan lines and the heater topology, are basically

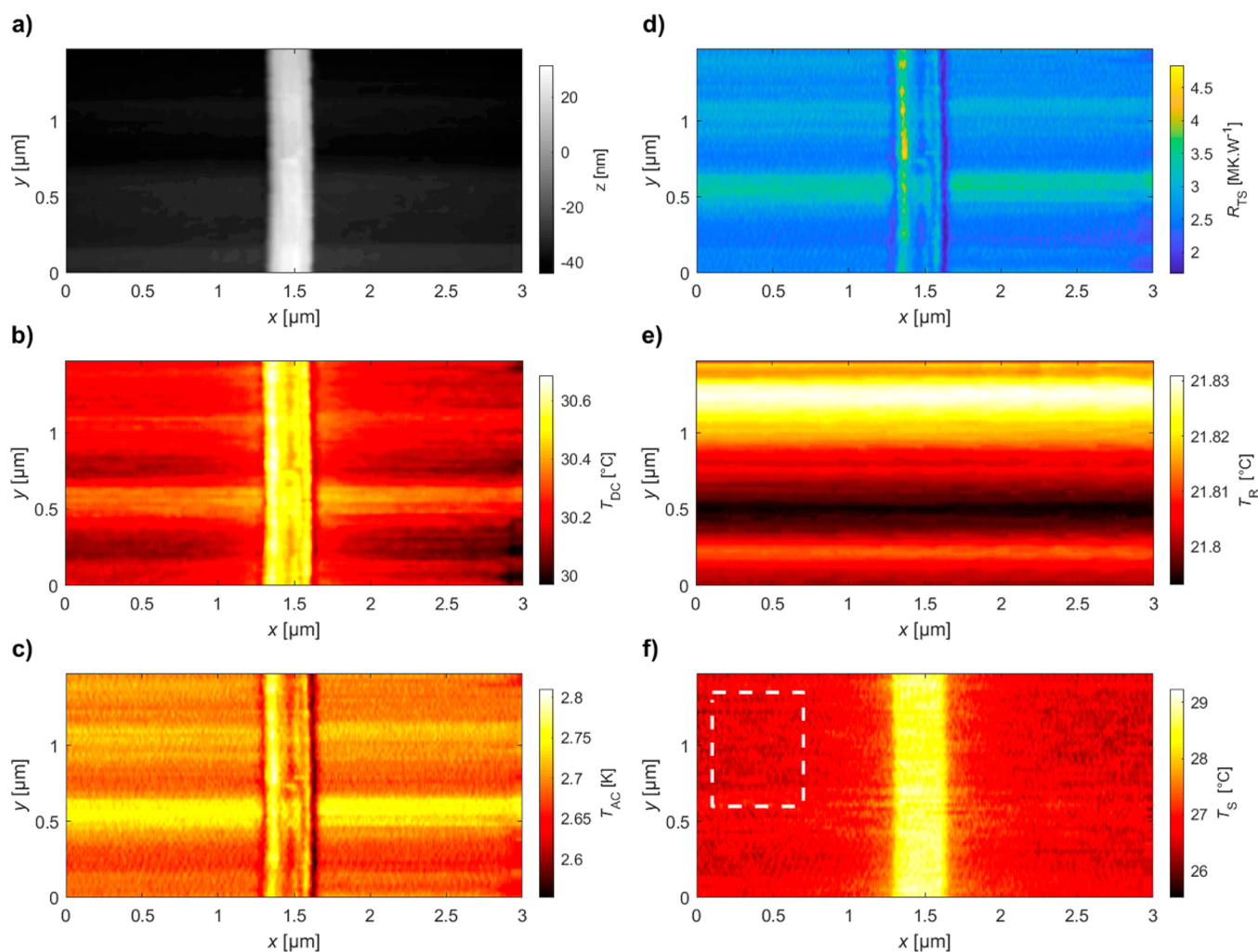


Figure 5. Data obtained from a thermal scan of a Pt line sample at a heating current of $I_{\text{sample}} = 0.5$ mA. Data shown in all panels were obtained simultaneously in a single scan. (a) Topography map showing the 250 nm wide Pt line on a SiO_2 substrate. (b) DC temperature (T_{DC}) measured by the probe platinum resistance thermometer (PRT). (c) Amplitude of the AC temperature oscillations (T_{AC}) of the probe measured by the probe's PRT. (d) Estimated tip-sample thermal resistance R_{TS} obtained from eq 2. (e) Thermal reservoir temperature (T_{R}) measured using an integrated PRT on the probe thermal reservoir. (f) Extracted surface temperature map (T_{S}) using eq 1. The pixels in the dashed box have a standard deviation of 221 mK. Refer to SI section S10 for the histogram distribution of the pixel values.

eliminated. Consequently, the recorded deviations in the T_{S} map (Figure 4f) from the nominal sample temperature of 26 °C arise mainly from pixel-to-pixel variations and exhibit comparatively small temperature noise with a standard deviation of 174 mK. In section S10 of the Supporting Information, we present the corresponding histogram of the distribution of temperature from all pixels, which confirms that the remaining variation in the T_{S} image (Figure 4f) is normally distributed measurement noise and generally devoid of the above-discussed artifacts.

Next, we repeated the same measurements with a heated wire by supplying an electrical current of 0.5 mA to the Pt line (see Figure 5). As can be seen in Figure 5b, which presents the measured T_{DC} , the Pt line is at an elevated temperature. However, there are significant artifacts in the image that represent unphysical features (as explained earlier). In order to reduce these artifacts that are caused by variation of R_{TS} during scanning, we followed the same procedure outlined for the unheated Pt line (Figure 4) and obtained the data necessary to compensate for variation of R_{TS} during scanning. Using these data, we generated a map of R_{TS} (Figure 5d) and used it in

conjunction with the measured T_{R} (Figure 5e) to obtain the surface temperature shown in Figure 5f. The constructed surface temperature map (Figure 5f) demonstrates significantly enhanced image quality with dramatically reduced artifacts. For a statistical analysis of the pixel-by-pixel temperature data, we used the pixels inside the dashed box (see Figure 5f) that is located in a region where the gradient in the sample temperature caused by heating is small. The standard deviation of pixel values in this region is 221 mK, which is slightly higher than the value for the image shown in Figure 4f due to the small background temperature gradient. See section S10 of the Supporting Information for the histogram of the distribution of pixel values in the dashed box of Figure 5f.

In order to confirm that the measured temperature fields are in quantitative agreement with modeling results, we averaged the measured temperature maps in Figure 5 along the y direction and plotted them in Figure 6a–d. As can be seen in Figure 6b, the T_{AC} profile captures key features of the T_{DC} profile that are related to a local change in R_{TS} (shown in Figure 6c). By accounting for these features through the thermal model (eq 1), an almost artifact-free profile of surface

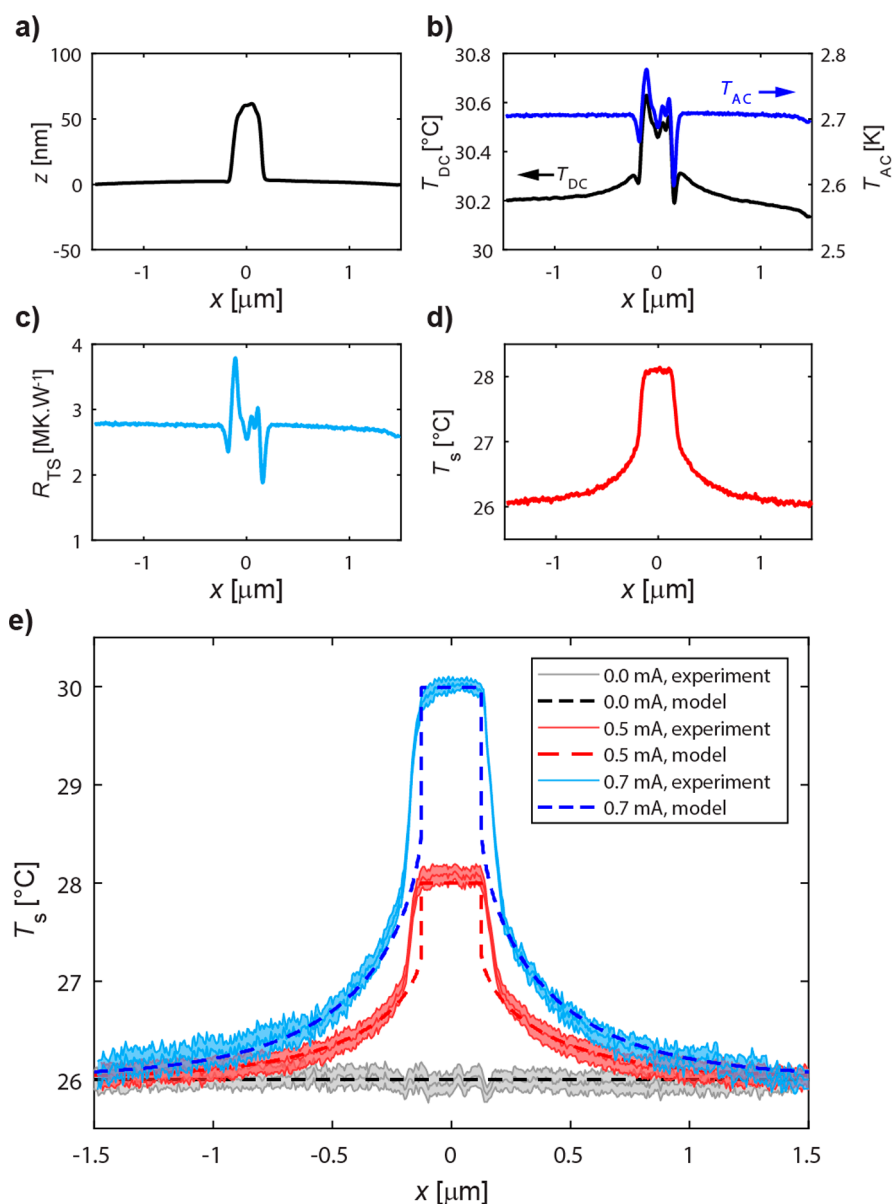


Figure 6. Averaged profiles obtained during thermal scanning of a Pt line such as the scans shown in Figures 4 and 5. To obtain the averaged profiles, the image was averaged in the y -direction (*i.e.*, the line scans were averaged). (a) Topography profile showing the 250 nm wide Pt line. (b) DC and AC temperatures measured by the probe's PRT. (c) Estimated tip-sample thermal resistance R_{TS} obtained from eq 2. (d) Extracted sample surface temperature profile when a DC electrical current of 0.5 mA was supplied to the Pt line sample. (e) Averaged sample surface temperature profiles for three sample heating currents of 0 mA, 0.5 mA, and 0.707 mA. Shaded error bars represent the standard deviation of the measured data.

temperature can be obtained as shown in Figure 6d. We have plotted the surface temperature profiles for three sample currents of 0, 0.5, and 0.707 mA, which are shown in Figure 6e. The shaded error bars represent the standard deviation of the measured temperature. The dashed profiles represent the modeled temperature from a continuum heat transfer model, which includes the interface thermal resistance between the Pt line and the SiO_2 substrate (see section S9 of the Supporting Information), thus generating a discontinuous temperature profile at the edge of the Pt line. The measured T_s profiles are in good agreement with the model. The root-mean-square errors of the model with respect to the experimental profiles are 32 mK, 118 mK, and 224 mK for the 0 mA, 0.5 mA, and 0.707 mA cases, respectively. Further, the coefficient of determination (R^2) values of the model are 0.962 and 0.963

for the 0.5 mA and the 0.707 mA cases, respectively. We note that the experimental profile of the 0.707 mA case demonstrates a maximum slope of $(\partial T/\partial x)$ of ~ 31 mK/nm near the edges of the Pt line.

We estimated the spatial resolution of our probes and the measurement technique as explained below. First, by combining the maximum experimentally observed temperature gradient $(\partial T/\partial x)$ and the temperature sensing resolution (ΔT) , we estimate the spatial resolution (Δx) using $\Delta x = \Delta T/(\partial T/\partial x)$, which results in a resolution of $\Delta x = 50$ [mK]/31 [mK/nm] = 1.6 nm (see section S8 of the Supporting Information for details). We note that a temperature resolution of ~ 50 mK was obtained in stationary probe conditions (from Figure 3c) and is valid for slow scanning rates (such as the 1 nm/s scan rate shown in section S8 of the Supporting

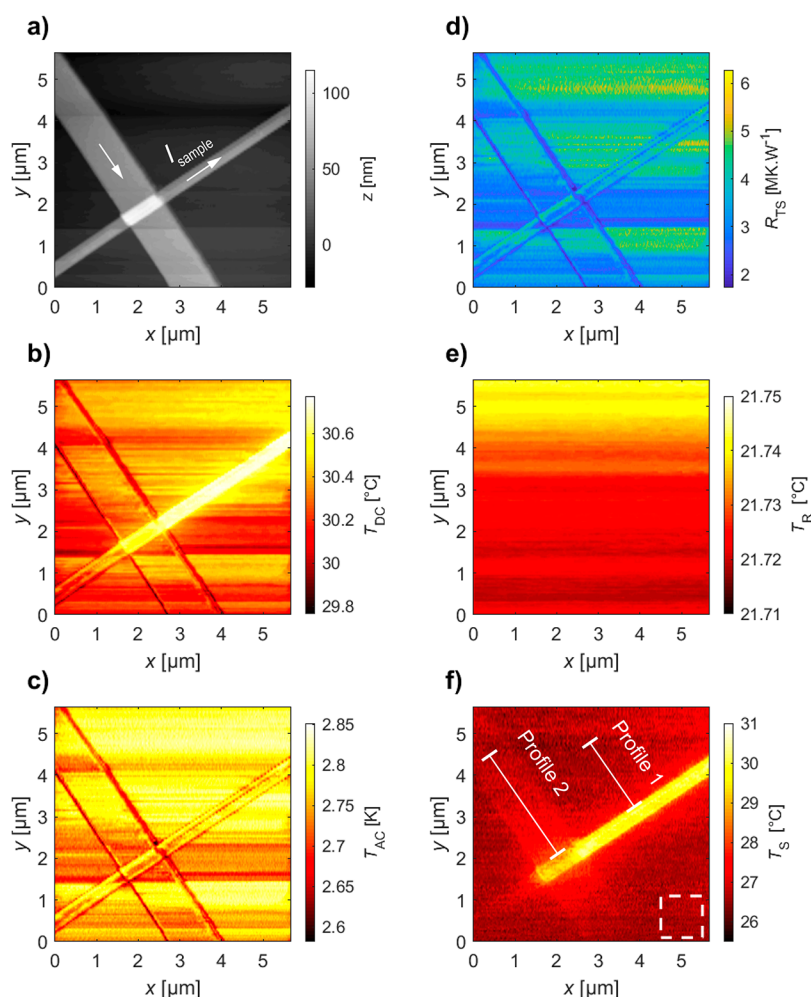


Figure 7. Quantitative mapping of temperature fields from a sample consisting of two overlapping 1 μm wide and 250 nm wide Pt lines that are heated by a DC current of 0.707 mA. Data shown in all panels were obtained simultaneously in a single scan. (a) Topography map showing the electrical current path by arrows. (b) DC temperature (T_{DC}) measured by the probe's PRT. (c) Amplitude of the AC temperature oscillations (T_{AC}) of the probe measured by the probe's PRT. (d) Estimated tip-sample thermal resistance R_{TS} obtained from eq 2. (e) Thermal reservoir temperature (T_{R}) measured using an integrated PRT on the probe thermal reservoir. (f) Extracted surface temperature map T_{S} . The temperature profiles averaged along two lines indicated in the image are shown in section S12 of the [Supporting Information](#). All images were corrected for distortion arising from piezo scanner saturation (see section S11 of the [Supporting Information](#)). The pixel values in the dashed box have a standard deviation of 238 mK.

[Information](#)). However, for faster scan rates (12 nm/s in [Figures 4 and 5](#)) the pixel-to-pixel variation is larger with a standard deviation of ~ 200 mK. Thus, for typical imaging conditions under faster scan rates we obtain a spatial resolution of 6.5 nm. Finally, we estimate the contact diameter of the tip and sample to be ~ 6 nm (see section S8 of the [Supporting Information](#)). Considering all these factors, a conservative estimate of the spatial resolution of our technique is 7 nm.

To evaluate the capabilities of our thermal mapping approach in a thermally and topographically complex environment, we imaged the steady state temperature field at the junction of 250 nm wide and 1 μm wide Pt lines (scan area 2 shown in the dashed box in [Figure 3a](#)). The scan (see [Figure 7](#)) was performed with a sample heating current of $I_{\text{sample}} = 0.707$ mA, the scan direction was horizontal at a rate of ~ 22 nm/s, and the image size was 512 (horizontal) $\times$ 128 (vertical) pixels. The electrical current direction is indicated in the topography scan of [Figure 7a](#), highlighting that only the portion of the 250 nm wide line carrying electrical current is expected to demonstrate significant heating due to a large

current density. The T_{DC} map ([Figure 7b](#)) shows a temperature increase of this portion of the Pt line but also includes significant topography-related (at the edges of the line) and line-by-line tip abrasion/modification artifacts. Similar to previous examples, by applying the thermal model, these artifacts are mostly removed from both heated and unheated parts of the scan, generating the surface temperature map of [Figure 7f](#). This map reveals how portions of the Pt lines are heated in response to current flow and how the deposited heat spreads from the lines to the substrate. For example, the temperature of the narrow Pt line on the SiO_2 substrate decays with a different characteristic length scale (~ 120 nm, corresponding to the length over which the temperature rise decays by 63%, see section S12 of the [Supporting Information](#)) as compared to the larger length (~ 560 nm) along the 1 μm wide Pt line. The exact profiles of these decays (marked as *profile 1* and *profile 2* in [Figure 7f](#) for the SiO_2 and Pt substrates, respectively) are shown in section S12 of the [Supporting Information](#) and relate to the thermal conductivities of the two substrates. It is also apparent that the upper

portion of the 1 μm wide Pt line is only slightly heated due to the lower current density and larger thermal conductance when compared to the 250 nm line. Lastly, the background noise (dashed box in Figure 7f, standard deviation equals 238 mK) is about equally low as compared to the surface temperature maps in Figures 4f and 5f. The slight increase in noise is due to the higher scan rate and the presence of a small gradient in the background temperature. Moreover, we remark that the surface temperature noise is largely determined by the uncertainty in the measured R_{TS} values and can be reduced by decreasing the magnitude of the term $(T_{\text{DC}} - T_{\text{R}} - R_{\text{p}}\dot{q}_{\text{DC}})$ in eq 1 by adjusting T_{R} or $\dot{q}_{\text{DC}}$ prior to scanning.

While we measured steady state temperature fields in this work, one can in principle also probe transient temperature changes using this technique, provided such changes occur sufficiently slowly. Specifically, we note that transient variations in the sample temperature are reflected as variations in the unmodulated temperature (T_{DC}) of the probe, and measurement of such time varying thermal fields is limited by the thermal time constant of the probe (~ 17.9 ms as stated earlier). Therefore, any temperature changes occurring on a time scale slower than this thermal time constant can be measured.

CONCLUSION

To summarize, we have presented a technique—contact resistance resolved scanning thermal microscopy (CR-SThM)—which enables quantitative mapping of unmodulated temperature fields by carefully estimating the effects of variations in the tip–sample thermal resistance. This is accomplished by employing custom-fabricated STPs featuring an integrated platinum heater/thermometer and a sharp tip (radius ~ 25 nm). We simultaneously measured tip–sample thermal resistance during thermal scans by depositing a modulated heat input into the probe and measuring the DC and AC temperature responses of the probe. Further, the probe features a suspended thermal reservoir with temperature sensing capability, which enables compensation for the ambient temperature drift and improves the long-term measurement stability. We established the spatial and temperature resolution of this technique by measuring the steady state temperature field around a 250 nm wide Joule-heated Pt line. This technique demonstrated a temperature sensing resolution of ~ 50 mK for sensing surface temperature in a bandwidth of 1 Hz and a spatial resolution of ~ 7 nm. We expect that the advances presented here will enable studies that aim to directly probe several nanoscale thermal phenomena that arise in active electronic, photonic, and energy conversion devices that are of great current interest.

METHODS

Experimental Setup. The experiments were conducted in an ultrahigh vacuum atomic force microscope (UHV 750, Beetle STM/AFM, RHK Technology) placed in an ultralow noise chamber. The chamber temperature was controlled so that the drift over a 24 h period was <0.1 K. All measurement electronics and instruments were located in this chamber during the measurement to prevent thermal drift. A laser (895 nm wavelength) was used for AFM readout. This relatively long wavelength was used to minimize the absorption of laser light by the gold reflector of the probe (hence minimizing the temperature rise of the probe) while maximizing the responsivity of the AFM photodetector.

Temperature Readout Circuits. Overall, the temperature signals from three different resistive temperature sensors were recorded

during the measurements. All these sensors were read out using balanced Wheatstone bridges driven by either AC voltage or AC current and subsequent measurement of amplitude of bridge output voltage oscillations at driving frequency using a lock-in amplifier as described in refs 32 and 37. The frequency of the bridge excitation was chosen to be different for all three measurements to avoid cross talk during measurements. The bulk sample temperature was measured using a 10 k Ω glass encapsulated thermistor and read out using a Wheatstone bridge driven by a voltage sine wave with a peak amplitude of 200 mV at the frequency 320 Hz. The probe thermal reservoir temperature was measured using a 17 k Ω platinum resistance thermometer (PRT) and read out using a Wheatstone bridge ($R_1 = R_3 = 40$ k Ω shown in Figure 1c) driven by a voltage sine wave with a peak amplitude of 200 mV at a frequency of 388 Hz. The probe tip temperature is measured as explained below.

Scanning Thermal Probe Heating and Temperature Sensing. A 9 k Ω platinum resistance thermometer (PRT) was integrated close to the probe tip which acts as a heater and thermometer. This PRT was connected to a Wheatstone bridge ($R'_1 = R'_3 = 20$ k Ω shown in Figure 1d) driven by a current waveform which was a superposition of two sinusoidal waves: $I_{\text{probe}} = I_1 \sin(2\pi f_1 t) + I_2 \sin(2\pi f_2 t)$. The first sine wave had a low frequency ($f_1 = 4$ Hz) and high amplitude ($I_1 = 56$ μA), which was used for modulated Joule heating of the probe at the frequency $2f_1 = 8$ Hz and the amplitude T_{AC} . The amplitude of the probe temperature modulation (T_{AC}) was measured by the lock-in technique using the $3f_1 = 12$ Hz component of the Wheatstone bridge output voltage.³⁸ To read the unmodulated temperature of the probe (T_{DC}), the second AC component was used which had high frequency ($f_2 = 212$ Hz) and low amplitude ($I_2 = 14$ μA) by locking-in on the f_2 component of the bridge output. Please refer to the SI for the probe fabrication steps and the thermal/mechanical properties of the probe.

ASSOCIATED CONTENT

Supporting Information

The Supporting Information is available free of charge at <https://pubs.acs.org/doi/10.1021/acsnano.1c08513>.

Fabrication of the scanning thermal probe; analysis of the mechanical stiffness and thermal conductance of the scanning thermal probe; feedback control stabilization of the sample temperature; independent temperature rise measurement of the self-heated Pt line sample; analysis of the validity of the lumped capacitance model of the scanning thermal probe; lumped capacitance thermal analysis of the scanning thermal probe; temperature measurement stability and noise; estimation of the spatial resolution; heat transfer model for estimation of the temperature profile on the sample; histogram analysis of the thermal scans; scanning image distortion correction; temperature profiles and characteristic length scales of the sample temperature field (PDF)

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Notes

The authors declare no competing financial interest.

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