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Thermal analog to AFM force-displacement measurements for nanoscale interfacial contact resistance

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Abstract

Thermal diffusion measurements on PMMA-coated Si substrates using heated AFM tips were performed to determine the contact resistance between an organic thin film and Si. The measurement methodology presented demonstrates how the thermal contrast signal obtained during a force-displacement ramp is used to quantify the resistance to heat transfer through an internal interface. The results also delineate the interrogation thickness beyond which thermal diffusion in the organic thin film is not affected appreciably by the underlying substrate.

Interfaces between mating parts or dissimilar materials introduce thermal resistance to heat flow due to partial surface contact. One-dimensional reference bar experiments are commonly used to quantify area-averaged contact resistance across interfaces.\textsuperscript{1} However, this averaging does not provide local information for individual asperities and is generally limited to thick samples in order to accommodate temperature measurement. Organic materials are

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commonly used as a filler to increase the contact area between mating surfaces and often serve as a matrix for dispersion of higher-conductivity materials (such as metal particulates, carbon nanotubes, etc.) to further decrease contact resistance. While thermal conductivity of thin-film resists has been studied, contact resistance between these thin films and substrates is a lingering challenge.  

In order to characterize local thermal resistance across thin films and interfaces, the atomic force microscopy (AFM) calibration technique known as a force-displacement ramp is used with heated AFM tips that can simultaneously detect changes in temperature. Heated cantilever tips are commonly used in scanning thermal microscopy (SThM) or local thermal analysis (LTA) and provide resolution on the order of the tip, typically tens of nanometers.  

Calibration of the thermal behavior of heated cantilevers has been studied, allowing these sensing tools to be used for material-property measurement and characterization. Early application of these tools includes sub-surface detection. Models of scanning force microscopy resolution depth have also been presented. Force-displacement experiments have been used as a method for quantifying contact potential between a cantilever and a surface as a function of temperature. Of particular note, Park et al. demonstrated that bringing a heated cantilever tip in and out of contact with a surface provides a measurable change in tip thermal diffusion. We exploit this observed change in thermal diffusion to estimate contact resistance at Si/organic interfaces using thin-film polymethylmethacralate (PMMA).  

During force-displacement curve calibration, the cantilever experiences multiple stages as the cantilever ramps toward the surface, snaps into contact, bends and then straightens again as the cantilever moves away from the surface until it suddenly releases from surface contact (see Figure 1 and Figure 2a). The AFM cantilevers used in this work incorporate a Wheatstone
bridge for heat generation at the tip and temperature sensing (Anasys Instruments model GLA). One resistor of the bridge is a metal strip along the cantilever tip (40 nm thick Pd trace, shown in Figure 1), which heats as current flows and also provides a temperature-dependent resistance used for sensing thermal changes as the tip comes in contact with surfaces. With a heated cantilever tip and a cool substrate, imbalance in the Wheatstone bridge is detected in the thermal-displacement curve (Figure 2b). First, the voltage decreases due to a decrease in resistance to heat flow through a thinning air gap between cantilever and substrate. Once snap-in occurs, a significant change in voltage imbalance is detected as conduction from tip to substrate increases and heat transfer is no longer hindered by a low-conductivity air gap. This abrupt change in voltage is dependent on the thermal diffusivity of the substrate in addition to tip heating conditions (Figure 2b) and is the primary measurement used in quantifying the resistance to thermal diffusion in this work. Thermal diffusion is considered steady since the time the tip \( D \approx 150 \text{ nm diameter} \) is in contact with the sample is determined to be more than six orders of magnitude larger than the characteristic time \( (D^2/\alpha) \) appearing in the Fourier number for heat conduction. Figure 2c illustrates the resistances to heat transfer between the cantilever and a Si substrate.

With the ability to distinguish thermal properties of a probed surface, an organic layer was included in an effort to measure the contact resistance between PMMA and Si \( (R_{Si/O}, \text{ Figure 2d}) \). However, resistance associated with thermal diffusion through the organic thin film \( (R_O) \), would also be present. By using thinned PMMA layers that approach zero thickness, the difference between the sum of the resistances shown in Figure 2c and d approaches the interface contact resistance, \( R_{Si/O} \). A series of Si substrates with PMMA films of varying thickness were prepared by spinning Microchem 950 (PMMA A4) on polished Si (5 nm roughness). In order
to span a range of thicknesses, Microchem 950 was diluted with Anisole in ratios of 1:1 and 1:1.5 and stirred for 90 min at 600 rpm. The solutions were spun on at varying speeds (typically 500-5000 rpm) for 40 s yielding thicknesses from 40-720 nm, as measured using an AFM. The PMMA films were then heated in air at 180 °C for 90 s, and then at 140 °C for 1 h.

Using the heated AFM tip, a series of thermal force-displacement measurements at various applied tip voltages ($V_{app}$) were performed. Thermal contrast measurements, obtained as a change in Wheatstone bridge voltage for the tip in and out of contact with the substrates during the approach ramp ($\Delta V$, Figure 2b), were obtained for each PMMA-coated sample. Similar experiments were performed on uncoated Si. The resulting $\Delta V$ is plotted in Figure 3a as a function of PMMA thickness. First, it is apparent that for PMMA thicknesses above ~300 nm there is little change in the thermal contrast. In this range, the heat transferred from tip to substrate approaches the behavior of bulk PMMA and provides an upper limit to the interrogation thickness for measuring resistance to diffusion under the PMMA layer. For higher thermal-conductivity films, we anticipate larger interrogation thicknesses. Second, a dramatic increase in the thermal signal is observed with reducing PMMA thickness (below the maximum interrogation thickness) for higher applied voltages. For lower applied voltages (0.8 V and 1.0 V), the amount of heat available for dissipation is reduced, and hence the maximum interrogation thickness is approached at smaller film thicknesses, making this $\Delta V$ less pronounced.

However, there is a marked difference between the measurements for the thinnest PMMA layers (Figure 3a) and the $\Delta V$ measured for uncoated Si. This difference results from the interface contact resistance, $R_{Si/O}$. The data obtained for bridge voltages of 1.2-1.6 V was fitted in order to obtain the y-intercept for the limiting PMMA thickness, as shown in Figure 3b. The $\Delta V$ values reduce to a single line by scaling the measurement by the bridge voltage and the
inverse of the film thickness. The resulting curve-fit equation and constants are provided in Figure 3b and is plotted as the dashed line on Figure 3a and b. The curve fit has an average error of 2.9% and a maximum error of 9.7%.

The intercept for reducing PMMA thickness ($AV_{\text{Intercept}}$, Figure 3b) and the $AV$ measured on uncoated Si ($AV_{\text{Si}}$) is plotted in Figure 4a as a function of the applied voltage. The difference between $AV_{\text{Si}}$ and $AV_{\text{Intercept}}$ represents the interfacial contribution to thermal resistance, $R_{\text{Si/O}}$.

The $AV_{\text{Si}}$ data points for 0.8, 1.2 and 1.6 V in Figure 4a are an average of 8 data points taken at 500 nm spacing increments on Si. The standard deviation of these measurements divided by the average of the measurements is 6.4%, 2.9% and 1.4%, respectively. This highlights the measurement repeatability and indicates that measurement error in $AV$ is less than 10%.

The interfacial resistance was calibrated by relating $AV$ to the temperature change of the tip. A heated stage with a thin-film polyimide heater (Minco) was mounted between Si (500 nm thick) and an insulating foam pad (~3 mm). The stack was bonded using Duralco 132 thermal epoxy (Cotronics) with a thermocouple attached to the top surface of the Si. Thermal force-displacement curves were measured over a temperature range from 25~150 °C. Figure 4b illustrates the measured thermal contrast, normalized by the $AV$ obtained at room temperature ($AV_0$), as a function of the heated Si surface temperature for three applied voltages. The $AV$ decreases linearly with increase in temperature with a slope of 0.36, 0.58 and 1.01 for 0.8, 1.2 and 1.6 V, respectively.

The tip heating is a function of the temperature-dependent resistance and the voltage drop across the tip resistor. The tip resistance was recorded for every measurement of Figure 3; average values are provided in Table 1 and vary less than 1% from the measured value. The
calculated Joule heating for the applied voltages considered here is in the range of 0.12-0.50 mW (Table 1).

To estimate the contact resistance between the PMMA and Si, all the heat transferred from tip to substrate is assumed to cross the Si/organic interface. In this manner, the resistance can be calculated as \( R_{Si/O}^2 = \Delta T / q^". \) Park et al. showed that approximately 25% of the heat generated in the cantilever tip is transferred to the substrate through the tip contact.\(^{13}\) Despite differences in cantilever geometry, we assume the same fractional amount of heat transfer from tip to substrate to provide a quantitative estimate of contact resistance. Analysis of heat transferred across the gap and through the tip is the subject of future work to be performed through modeling and experimental analysis. A 150 nm tip diameter is assumed to approximate the contact area. Converting the measured \( \Delta V_{Si} - \Delta V_{intercept} \) (Si/organic contact resistance contribution to the thermal contrast) to \( \Delta T \) using the slope of the curve fits in Figure 4b (and estimating the slope for 1.0 and 1.4 V using a linear fit), we can calculate the interface contact resistance in terms of a temperature difference. Using 25% of the generated heat, the contact resistance for the Si/organic contact resistance is calculated to be \( 1.8 \times 10^{-8} \) to \( 4.0 \times 10^{-8} \) m\(^2\)K/W (Table 1). This is 2-5 times smaller than the lower range of contact resistances reported for an Al/Al interface with a Pb coating.\(^{14}\)

This paper demonstrates the feasibility of using heated AFM tips to quantify the contact resistance between thin films and underlying substrates. The thermal contrast signal measured during force-displacement ramping is demonstrated to be a useful thermal measurement technique. Further, we show that there is a limit to the thickness of the film for characterization using thermal diffusion above which the impact of the underlying substrate diffusivity is not
discernible. This technique is capable of spatial resolution on the order of the AFM tip radius, making small-scale defect detection possible.
References


Tables

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Figure 2. Representative force-displacement measurement illustrating the (a) force and corresponding (b) thermal response of the cantilever during a displacement ramp for three substrates of different thermal conductivity (numbering corresponds with Figure 1). Resistance to heat transfer between heated cantilever and (c) Si substrate, and (d) Si substrate coated with an organic thin film.

Figure 3. Thermal contrast during force-displacement ramp (a) as a function of varying PMMA thicknesses for various applied voltages, and (b) reduced data for 1.2, 1.4 and 1.6 V with curve fit parameters.

Figure 4. (a) Thermal contrast during force-displacement ramp as a function of the applied voltage for experiments on Si and the curve-fit y-intercept from Figure 3 (filled markers represent an average of eight measurements at 500 nm spacing increments) and (b) thermal contrast during force-displacement ramp as a function of the Si surface temperature.
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