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Contact potential measurement using a heated atomic force microscope tip

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This letter reports contact potential measurement between the tip of a heated atomic force microscope cantilever and a biased gold film. Force-distance experiments were performed with tip temperature, tip potential, and substrate potential independently controlled. Experiments were conducted for probe temperatures of 23 to 200 °C and tip potentials of −1 V to 1 V. The measured contact potential was a function of temperature, due to the thermoelectric properties of the tip and substrate. The Seebeck coefficient for the combined system was close to −4.30 mV/K, consistent with the tip and substrate materials. The technique is scalable to arrays suitable for large area imaging. © *2007 American Institute of Physics*. [DOI: 10.1063/1.2789927]

Electrical scanning probe techniques rely on a conductive tip to map surface responses along with the physical topography. In such methods, a bias voltage is often applied between the tip and substrate to measure electrical variants of their interaction, such as current, resistance, or capacitance.[1] If a temperature gradient exists near the tip-sample contact, a thermoelectric potential may emerge. The thermoelectric potential can produce non-negligible error in surface potential measurements,[2] but can be exploited to characterize the substrate in other cases—for example, to measure dopant concentrations or molecular charge transport at semiconductor junctions. A few papers outlining this approach have employed scanning tunneling microscopy[3] in which the tip was at ambient temperature and the substrate was heated.[4,5] Others have shown by scanning Kelvin probe microscopy[6] that the temperature dependence of ferroelectric polarization states[7] and field-effect mobility in thin-film transistors[8] can be revealed through surface potential mapping. However, no published paper has used a heated atomic force microscope (AFM) probe tip to measure the contact potential. Tip-side heating would offer advantages in that the probe temperature could be rapidly changed over a large temperature range.

This letter demonstrates the feasibility of acquiring local contact potentials with a heated AFM cantilever tip. The experiments used single-crystal silicon cantilevers having integrated heater thermometers, such as were originally developed for data storage.[9,10] Similar heated cantilever probes have been used in nanometer-scale manufacturing,[11,12] for thermal property measurement,[13–15] and for small-scale fluid[16] measurements. The thermal, mechanical, and electrical properties of these cantilevers and their methods of calibration are well understood[9,17,18] and not described here in depth. Figure 1(a) shows a probe similar to the one used in this experiment, which has a silicon tip with a near 20 nm radius of curvature below the outlined heater. The cantilever is "U" shaped such that a voltage drop across the legs drives current through the resistive heater near the tip.

The cantilever was mounted in our MFP-3D AFM system (Asylum Research) above a glass substrate coated with a 100-nm-thick gold layer. The substrate served as an electrode that could be biased independently from either cantilever leg. Potential offsets to the tip and substrate permitted investigation of a wide combination of probe temperatures and electrostatic field strengths. Figure 1(b) illustrates the experimental setup, in which the tip bias was assumed to be an average of the driving voltages. The contact potential was recovered by analyzing the electrostatic force between cantilever and substrate.

During each trial, the cantilever was held approximately 6 μm from the surface as the substrate bias was varied between −9 and +9 V in random order. In contrast, potentials applied to and localized heating of the cantilever free end by assembly of various external circuits were not manually adjusted. Temperature calibration near the surface yielded target cantilever temperatures of 23, 70, 130, and 200 °C based on prior measure of the Stokes peak position with cantilever power via Raman spectroscopy.[17] The spring constant was calculated on gold for each probe condition by the thermal noise method[19] and ranged from 0.15 N/m at 23 °C to 0.10 N/m at 200 °C. The tip was slowly brought into contact with the gold substrate under open-loop feed-

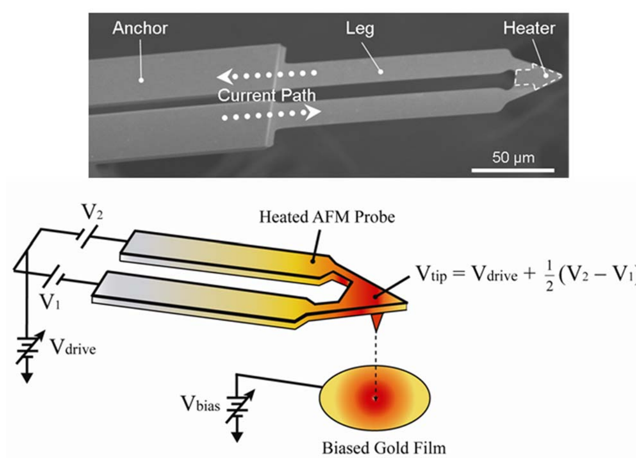

FIG. 1. (Color online) Scanning electron microscope image of the heatable silicon cantilever. Electrical contact is made on the silicon chip to which the anchors are attached. (b) The heated cantilever approaches a biased gold surface. Driving voltages on the legs generate resistive heating and/or bias the free end near the tip. Not shown is the contact potential V_C, which represents the substrate bias that could be applied to zero the net electrostatic field.

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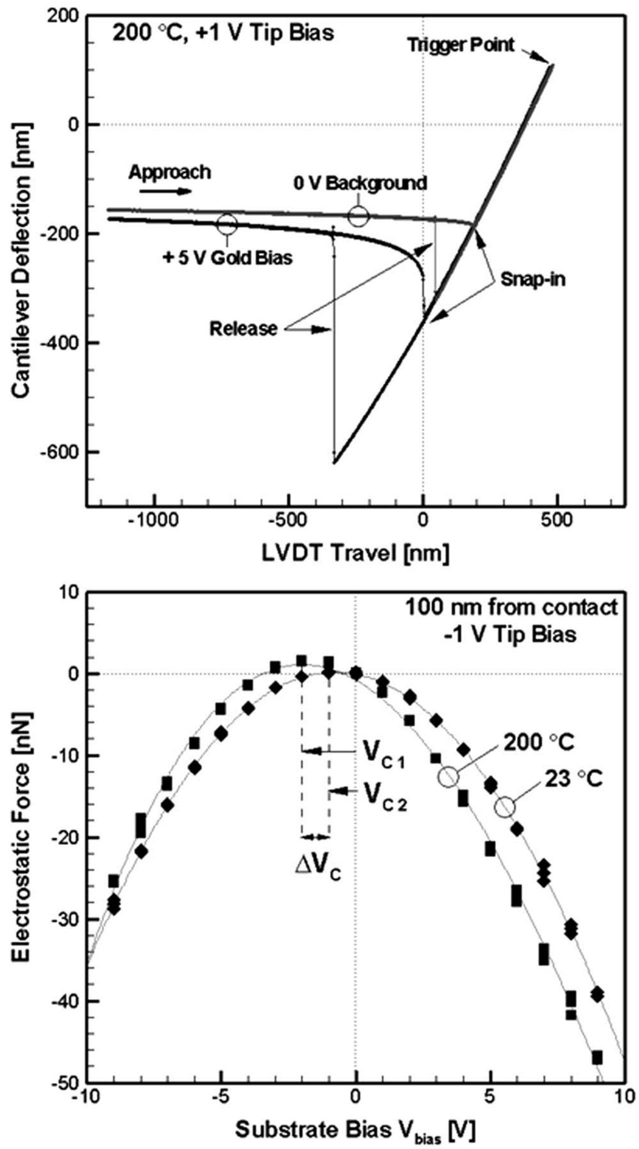

FIG. 2. Example raw data for a probe at 200 °C with +1 V bias on the tip. Introducing a sample bias (+5 V) increases cantilever deflection near the snap-in point relative to the unbiased background. Data were normalized and interpolated before conversion to a force-distance curve. (b) Comparison of force vs substrate bias for a probe at room temperature and 200 °C with −1 V on the tip and 100 nm from contact. The gold bias that minimizes the attractive force is offset from the unbiased case by the measured contact potential.

back at constant velocity, and was retracted upon reaching target deflection at a mean contact force of 40 nN.

The cantilever deflection during the force-distance experiment indicates the electrostatic force between the tip and substrate, which is due to a material work function difference and the applied potentials. The deflection was matched to the output of a linear variable differential transformer (LVDT) sensor that operates in parallel with the piezoactuator on the driving stage and tracks the probe vertical displacement. Three deflection-LVDT curves were captured per substrate bias, each in conjunction with an unbiased curve to account for background forces independent of the source voltages.

Figure 2(a) compares raw data for the gold substrate at 0 and +5 V, cantilever temperature of 200 °C, and tip potential of +1 V. The cantilever deflection near "snap in" scales with the magnitude of the gold bias. The analysis constructs force-distance curves based on the assumption that maximum negative deflection corresponds to a point of zero tip-sample separation.[20,21] The raw data were interpolated to within 20 nm of contact. The data were normalized by the slope of each linear contact region to correct for drift between measured deflection and piezoresponse. The net deflection (bias minus background) was then multiplied by the precalibrated spring constant to obtain the force measurement and added to the LVDT output to calculate the distance traveled by the tip. For the data presented here, a constant approach velocity of 1.98 μm/s was maintained; velocities of 200 nm/s and 20.8 μm/s yielded nearly identical results. Figure 2(b) shows measured force as a function of substrate bias voltage at 23 and 200 °C. The tip voltage was −1 V and the force was measured 100 nm before snap in occurred. Force measurements 20 nm from snap in yielded nearly identical results.

Figure 3(a) shows the measured contact potential as a function of cantilever temperature and applied voltage. By definition, the contact potential V_C is a measure of the bias required to zero the net field, and corresponds to the voltage offset between unbiased gold and the value that minimizes the attractive force. The total tip-sample interaction F can be described as a function of the substrate bias V_{bias} according to $F=F_{\mathrm{bg}}+f(d)(V_{\mathrm{bias}}-V_C)^2$, where F_{bg} represents the aforementioned background forces and $f(d)$ is a capacitive coupling that depends on tip geometry and separation distance.[22] V_C was extracted by removing F_{bg} at fixed distance such that $F=F(V_{\mathrm{bias}})-F(0)=f(d)(V_{\mathrm{bias}}^2-2V_{\mathrm{bias}}V_c)$ and comparing the result to a parabolic fit of the measured force versus substrate bias, $F=p_1V_{\mathrm{bias}}^2+p_2V_{\mathrm{bias}}$. A least squares fit determined the trial results in Fig. 3(a) to within a 95% confidence interval, where error was calculated as a function of the regressed parameters p_1 and p_2. At 100 nm before snap-in, increasing cantilever temperature shifted the contact potential to increasingly negative offsets. A nearly linear dependence of V_C on temperature became evident with potential removed from the tip, as in Fig. 3(b). The regressed intercept values reflect multiple measurements across the gold surface, where the probe temperature was varied randomly at each location. Results were consistent over the range of contact forces to suggest that systematic contamination of the tip with gold particles did not occur.

It is possible to estimate a Seebeck coefficient[23] from the measurements. The contact potential can be expressed in terms of a generalized Seebeck coefficient $S(T)$ in the form $V_C=V_S+(W_{\mathrm{Au}}-W_{\mathrm{Si}})/e+V_{\mathrm{tip}}+\int_{T_0}^{T}S(T)dT$, where V_{tip} is the tip bias voltage, V_S is the surface potential of the silicon tip, W_{Au} and W_{Si} are the work functions for gold and silicon, e is the electron charge, and T is the temperature. The estimate assumes that V_S and the work function difference do not vary significantly with temperature such that the thermoelectric potential $\int_{T_0}^{T}S(T)dT$ is given by $V_C(T)-V_C(23\ ^\circ\mathrm{C})$. To arrive at this relation, V_S was regarded as the difference between V_C measured by an unbiased tip at 23 °C and $(W_{\mathrm{Au}}-W_{\mathrm{Si}})/e$ evaluated at 23 °C. The slope of the thermoelectric potential versus temperature plot corresponds to a combined Seebeck coefficient of −4.30±2.52 mV/K. The expected coefficient may be calculated from theory for uniformly doped bulk silicon of undefined geometry with weighting factors to account for phonon scattering and drag.[23–25] The calculated thermal voltage for a doping level comparable to the silicon

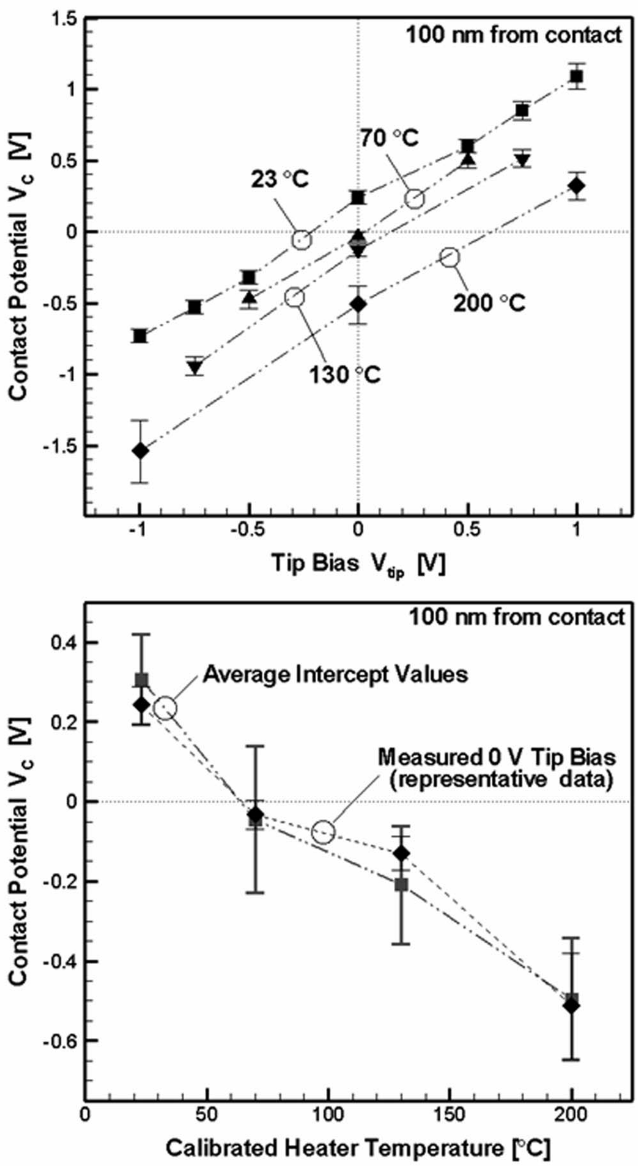

FIG. 3. Contact potential vs applied tip bias 100 nm before snap in at the four temperatures tested (representative data). As the temperature gradient increases, the Seebeck effect shifts the contact potential to increasingly negative offsets. Error was calculated by parabolic regression of the measured electrostatic force. (b) Effect of heat on the contact potential for the unbiased tip, also 100 nm from contact. The 0 V tip data in 3(a) are compared against average intercept values of each linear temperature fit. Uncertainty is based on a minimum of six contact potential measurements taken at multiple locations across the gold surface.

tip (1×10^{17} cm^{-3}) and a weighting factor of 2 yields a Seebeck coefficient of -0.69 ± 0.027 mV/K over the same temperature range. The extracted coefficient therefore has the same sign and order of magnitude as expected for *n*-type silicon,[26,27] with a discrepancy that might be attributed to the nonlinear temperature distribution and nonuniform doping in the cantilever, sensitivity of the work function[28] and surface potential[29] to adsorption of ambient gas molecules, and thermal contact resistance between the cantilever tip and gold substrate. We stress that the temperature drop is not uniform over the doped silicon tip, and so a departure from the theoretical value for the cantilever alone is expected. Gold has a reported Seebeck coefficient of $+1.94$ μV/K,[27] and so the gold has a small effect on the measured Seebeck coefficient.

This paper demonstrates the feasibility of using a heated AFM cantilever tip to perform nanometer-scale measurement of temperature-dependent contact potential. These results suggest that it may be possible to perform dopant profiling or thermoelectric measurements with a heated probe tip. The technique is highly scalable to large probe arrays, which could be used to perform imaging over large areas, such as would be suitable for semiconductor wafers or biological tissue.

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