Applied Physics Letters

Direct measurement of forces during scanning tunneling microscopy imaging of silicon pn junctions

Jeong Young Park, R. J. Phaneuf, D. F. Ogletree, and M. Salmeron

Citation: Appl. Phys. Lett. **86**, 172105 (2005); doi: 10.1063/1.1906297
View online: http://dx.doi.org/10.1063/1.1906297
View Table of Contents: http://apl.aip.org/resource/1/APPLAB/v86/i17
Published by the American Institute of Physics.

Direct measurement of forces during scanning tunneling microscopy imaging of silicon pn junctions

Jeong Young Park
Materials Sciences Division, Lawrence Berkeley National Laboratory, University of California, Berkeley, California 94720

R. J. Phaneuf
Department of Materials Science and Engineering, University of Maryland, College Park, Maryland 20740

D. F. Ogletree and M. Salmeron[a)]
Materials Sciences Division, Lawrence Berkeley National Laboratory, University of California, Berkeley California, 94720

(Received 20 August 2004; accepted 7 March 2005; published online 20 April 2005)

We investigated the forces acting between tip and surface during scanning tunneling microscopy (STM) imaging of a silicon *pn* junction. Using a conductive and stiff atomic force microscopy (AFM) cantilever, the current between the tip and sample, and the normal force (or lever bending) were measured independently. This method allows us to use either AFM or STM, depending on the feedback signal. By comparing topographic images of the *pn* junction acquired in contact AFM mode with the STM images, large variations of STM topography and normal force across the junction could be observed. We find that at reverse bias the tip presses against the surface to draw the set-point current, while it is in noncontact tunneling regime at the forward bias. The current measured as a function of tip-sample distance shows a strong dependence on polarity of the bias in the *p*, *n*, and inverted regions, consistent with the force measurements during constant current STM mode. © *2005 American Institute of Physics*. [DOI: 10.1063/1.1906297]

When two surfaces are brought into close proximity, forces of various origins appear as a result of physical and chemical interactions. At subnanometer, separation, forces of chemical origin can result in the formation and rupture of chemical bonds. These strong forces decay exponentially so that above one nanometer only electrostatic forces due to contact potential differences, or induced by charges and electrical dipoles from adsorbates, step edges, etc., and the weaker van der Waals forces remain. During imaging heterogeneous electronic structure in scanning probe microscopy, the variation of tip-sample force have a significant influence on image contrast. Measuring and understanding the force between tip and surface in these electrically distinct regions is therefore very important for accurate image analysis and characterization of semiconductor devices.

Many efforts have been made to characterize the tip-sample force during scanning tunneling microscopy (STM) study.[1–5] On metals and highly doped semiconductors the forces during non contact tunneling are attractive. Due to poor electrical conductivity in lightly doped semiconductors or in insulating layers however, the tip might come into direct contact with the surface, giving rise to a strong repulsive force. Repulsive forces indicative of contact during STM imaging have been observed on graphite.[3–6] In electrically heterogeneous regions, variation of forces during noncontact atomic force microscopy (AFM) imaging leads to the strong bias dependence in the topographical images.[7]

Previously, we have reported on the lateral distribution,[8] time dependence of the carrier density, and potential distribution across the *pn* junction device[9] using STM and conductance mapping. Bias dependent images showed "dips" within the junction with an apparent depth that was much larger than the tip-sample separation in the tunneling regime. We assigned the contrast in conductance maps with the variation of the electrical character of the metal-insulator-semiconductor (MIS) junction formed by the STM tip and the *pn* junction.

In this article, we present results of a study using simultaneous topographic and force measurements with a conductive cantilever in an ultrahigh vacuum AFM system using a Si sample patterned with stripes of three electrically different regions: *n*, *p*, and inverted regions.

The experiments were performed with a commercial RHK-technology STM/AFM system mounted in a chamber with base pressure of 1.0×10^{-10} torr.[10] We used cantilevers coated with approximately 20–30 nm of TiN, and with spring constants of 48 N/m.[11] The device studied in this experiment has been described elsewhere.[8,9,12] It consists of an array of stripes of *p*-type with a doping level of 10^{18} /cm^3, within a lightly $(1.6\times10^{14}\ \mathrm{cm}^{-3})$ *n*-type Si(100) substrate. Heavily doped *p* stripes are spaced at 30 μm intervals. The surface is terminated by a wet chemical oxide,[13] prepared using the Shiraki procedure, and transferred through a load lock into the ultrahigh vacuum chamber.

Figure 1 shows a contact AFM topographic image of the *pn* junction. The spacing between two arrows in the plot, which represents the separation of the outer dips is 3.3 μm, consistent with the previous AFM and scanning photoelectron microscopy measurements.[12] Height corrugation for the inner grooves is approximately 6 Å, while that of the outer grooves is ~10 Å. This depth is much smaller than the apparent value reported in the earlier STM profiles of filled states,[8] confirming the assumption that the STM tip was in fact being loaded against the surface in the regions where the

[a)]Author to whom correspondence should be addressed; electronic mail: salmeron@stm.lbl.gov

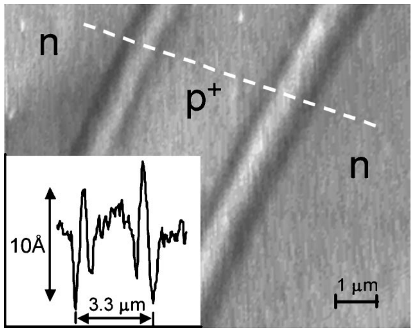

FIG. 1. 7.2 μm $\times$ 5.0 μm topographic AFM contact image of a *pn* junction patterned on a Si wafer, under an applied load of 260 nN. A height profile across the junction is shown in the inset. At the boundary between the *p* stripes and the *n* region two sets of grooves are visible. They are caused by the etching procedures used in fabricating the ion implantation window.

tip-sample junction was in a state of electrical inversion.

With our simultaneous STM and force measurement experiments we studied this in more detail, using tunneling current feedback while measuring the bending of the cantilever. An example is shown in Figs. 2(a)–2(c) for a sample bias of −4 V and tunneling current of 1 nA. The STM image is a plot of the variation of the length of the piezoactuator supporting the sample during scanning. In normal STM, where the tip is rigid, this reproduces the topography of the sample. In the present case, the lever is bending due to variations in normal force. Therefore, the raw topography in the STM image needs to be corrected for the lever bending to obtain the real surface topography. Figure 2(c) shows the compensated STM topography, which is simply the sum of the STM raw image [Fig. 2(a)], and the lever displacement [Fig. 2(b)]. The prominent dark features in the STM image of Fig. 2(c) at the edges of the *p*-doped stripe are the same "electrical dips" which we earlier related to the MIS junction behavior across a *pn* junction.[8] At negative sample bias (−2 V), the *p* stripe is in weak inversion, the *n* region is in

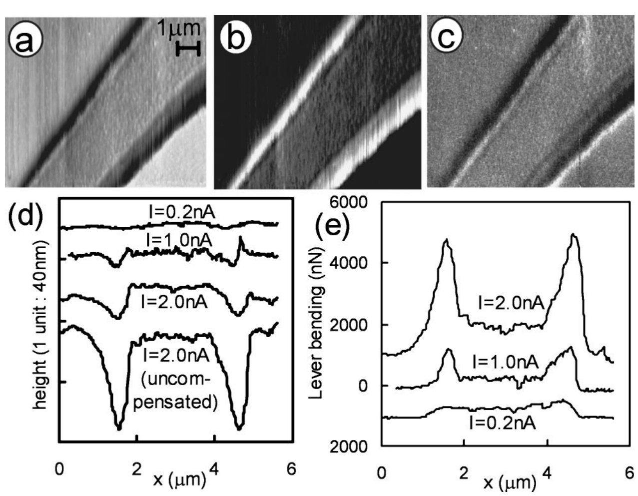

FIG. 2. (a) 6.6 μm $\times$ 5.1 μm raw STM image and (b) simultaneously measured lever deflection (normal force) at a sample bias of −4 V and for a tunneling current of 1 nA. (c) STM topography after compensation of the lever deflection by addition of the vertical STM piezodisplacement and the cantilever deflection. (d) Height profiles across the junction in (c) for three STM current setpoints (0.2, 1, and 2 nA). The bottom trace is a line profile of the raw STM topography [from (a)] for a set point of 2 nA; (e) line profiles of the force mapping (b). The position of each curve is shifted by 1000 nN for clarity.

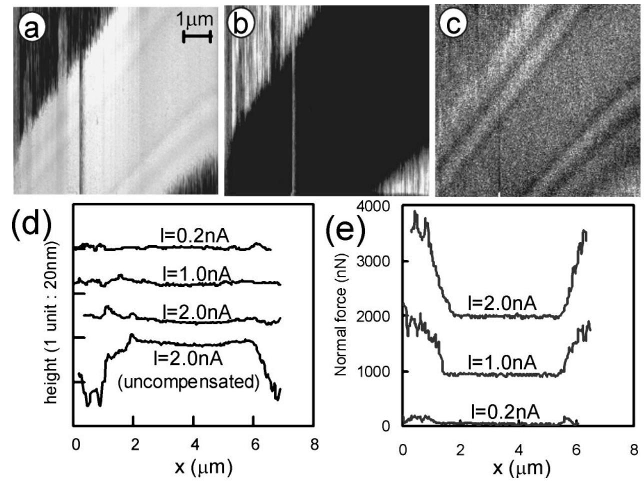

FIG. 3. (a) 6.0 μm $\times$ 5.2 μm raw STM image and (b) normal force simultaneously taken at a sample bias of +4 V and tunneling current of 1 nA. (c) STM topography after compensation for the lever deflection, obtained by adding (a) and (b). (d) Line profiles of STM compensated topographical image for three STM current setpoints (0.2, 1, and 2 nA). The bottom trace is a line profile of the raw STM topography [from (a)] for a setpoint of 2 nA. (e) Line profiles of the forces across the junctions. Positions of each curve are shifted by 1000 nN.

accumulation, and the region between them in strong inversion.[8,14] In this study, we find that the electrical dips become discernable at a voltage higher than that of previous study, −4 V compared to −2 V, which we believe is due to a smaller electrical conductance through the 20–30 nm thick metal coating of the conductive cantilever, compared to that for an STM tip.

Figures 2(d) and 2(e) show the line profiles of STM topography image (after compensation) and force mapping at three different setpoint currents: 0.2, 1, and 2 nA. The corresponding apparent depths of the dips in the inverted region are 1, 8, 15 nm, respectively, equal or larger than the $<\sim 1$ nm tip-sample separation typical in tunneling, and in the latter two cases much larger than the measured corrugation in AFM maps. The line profiles across the force maps [Fig. 2(e)] show repulsive force at the inverted region, demonstrating that indeed the tip is loading against the sample, strongly in the inverted region and to a lesser degree in the less strongly inverted *p* stripe.

For positive sample bias, positioning the tip above the *n* regions reverse biases the tip-sample MIS junction, while positioning it above either the *p* stripe or inverted region results in forward bias (accumulation).[8,14] Figures 3(a) and 3(b) show STM uncorrected images and the corresponding normal force maps. Figure 3(c) shows the tip-deflection compensated STM image obtained from the images in Figs. 3(a) and 3(b). While Fig. 3(c) shows a similar feature with AFM topography [Fig. 1(a)], the force mapping is noticeably different from that recorded with negative sample bias. Here, the force map shows a bending of the cantilever corresponding to a repulsion, which increases as the set-point current is increased from 0.2 to 1 to 2 nA.

The normal force and current were measured as a function of tip-sample distance across the *pn* junction, as shown in Fig. 4. Figure 4(a) shows the force-distance curves measured in the *n* region with a sample bias of 0, +4, and −4 V. The pull-off force with a sample bias of 0 V is 60 nN. The observed increase of pull-off force with the sample bias is

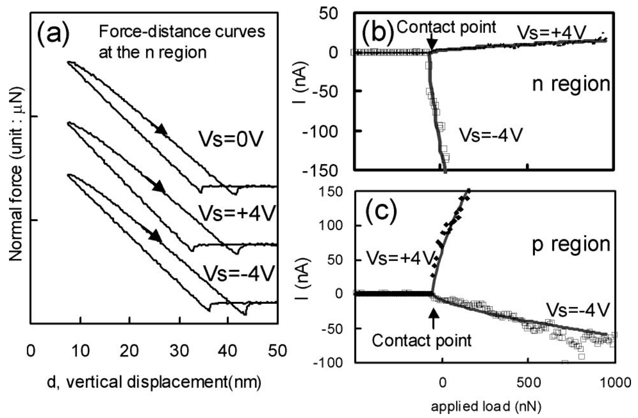

FIG. 4. (a) Force-distance curves measured at the n region with sample bias of 0, +4, and −4 V. (b) Plot of the current between the tip and surface in the n region as a function of applied load, measured during acquisition of the force-distance curves. Solid lines are DMT curve fits obtained using a tip radius of 100 nm, and a pull-off force of 110 nN ($Vs=-4$ V), and 80 nN ($Vs=+4$ V). (c) Plot of the current vs applied load measured at the center of the p stripe and corresponding DMT curve fits. Pull-off forces are 100 nN ($Vs=+4$ V), and 80 nN ($Vs=-4$ V).

mainly due to the increasing electrostatic force. The effect is asymmetric (80 nN at +4 V, and 110 nN at −4 V) mainly because of the difference in contact potential between TiN tip and Si surface.

Figure 4(b) shows a plot of current between the tip and n region as a function of applied load. Because of adhesion force, the current increases from the negative applied load. As shown in the plot, the current at the forward bias (−4 V) is much larger than that of reverse bias (+4 V) at the same applied load, as expected. Figure 4(c) shows the corresponding result for the p stripe region. The lines drawn over the points are the Derjaguin–Muller–Toporov (DMT) fittings.[15] The agreement with DMT fitting is quite good and consistent with the high hardness of TiN and silicon.

In the DMT approximation,[16] the current (I) is given by the equation, $I=J_eA=J_e\pi(R/E^*)^{2/3}\times(L+2\pi\gamma R)^{2/3}$, where L is the applied load, R is the tip radius, γ is the work of adhesion, E^* is the combined elastic modulus of the two materials, and J_e is the effective current density. Pull-off forces (L_c) measured at each bias were used to estimate the work of adhesion $[\gamma=L_c/(2\pi\mathrm{R})]$.

In the n region, effective current density at the sample bias of −4 V (forward bias) is 2.24×10^5 (A/cm^2), larger than that of +4 V[5.45×10^3 (A/cm^2)] by a factor of 40. In the p region, J_e is 2.06×10^4 (A/cm^2) at −4 V, smaller than that of +4 V[1.5×10^5 (A/cm^2)] by a factor of 7. The good agreement between the current and the DMT fit indicates that the assumption of the current being proportional to the contact area is correct, at least in accumulation or inversion. However during depletion, the existence of tunneling through the Schottky barrier caused by tip-induced band bending, may require modification of the equation.

In conclusion, we confirm directly that the anomalously deep features measured at the edges of a pn junction in STM images result from mechanical tip-sample interaction during reverse-biased MIS junction tunneling. We have observed and measured the normal forces across the junction and find that it varies consistently with features in STM across the junction, which are electronic rather than topographical in nature. Since the experiment was performed in ultrahigh vacuum, the effect of forces due to capillary condensation of water is ruled out, and only electrostatic and van der Waals forces dominate the force variation in the pn junction. An intriguing possibility is to use the variation of force along STM images to analyze the charge character of the underlying device.

This work has been supported by the Director, Office of Energy Research, Office of Basic Energy Sciences, Materials Sciences Division, of the U.S. Department of Energy under Contract No. DE-AC03-76SF00098, and in part by the Laboratory for Physical Sciences and the University of Maryland/Rutgers NSF-MRSEC under Grant No. DMR-00-80008.

[1]M. Guggisberg, M. Bammerlin, R. Lüthi, Ch. Loppacher, F. Battiston, J. Lü, A. Baratoff, E. Meyer, and H.-J. Güntherodt, Appl. Phys. A: Mater. Sci. Process. **66**, S245 (1998); S. Schär, R. Bennewitz, T. Eguchi, E. Gnecco, O. Pfeiffer, L. Nony, and E. Meyer, Appl. Surf. Sci. **210**, 43 (2003).
[2]T. Arai and M. Tomitori, Appl. Phys. A: Mater. Sci. Process. **72**, S51 (2001).
[3]U. Durig, O. Zuger, and D. W. Pohl, Phys. Rev. Lett. **65**, 349 (1990).
[4]D. A. Grigg, P. E. Russell, and J. E. Griffith, J. Vac. Sci. Technol. A **10**, 680 (1992).
[5]D. Anselmetti, A. Baratoff, H. J. Guntherodt, Ch. Gerber, B. Michel, and H. Rohrer, J. Vac. Sci. Technol. B **12**, 1677 (1994).
[6]M. Salmeron, D. F. Ogletree, C. Ocal, H. C. Wang, G. Neubauer, and W. Kolbe, J. Vac. Sci. Technol. B **9**, 1347 (1991).
[7]S. Sadewasser and M. Ch. Lux-Steiner, Phys. Rev. Lett. **91**, 266101 (2003).
[8]M. L. Hildner, R. J. Phaneuf, and E. D. Williams, Appl. Phys. Lett. **72**, 3314 (1998); J. Y. Park, E. D. Williams, and R. J. Phaneuf, J. Appl. Phys. **91**, 3745 (2002).
[9]J. Y. Park and R. J. Phaneuf, Appl. Phys. Lett. **82**, 64 (2003); J. Y. Park and R. J. Phaneuf, J. Appl. Phys. **94**, 6883 (2003).
[10]J. Y. Park, D. F. Ogletree, M. Salmeron, C. J. Jenks, and P. A. Thiel, Tribol. Lett., **17**, 625 (2004).
[11]NT-MDT Co., Zelenograd Research Institute of Physical Problems, Moscow, Russia.
[12]R. J. Phaneuf, H.-C. Kan, M. Marsi, L. Gregoratti, S. Günther, and M. Kiskinova, J. Appl. Phys. **88**, 863 (2000).
[13]A. Ishizaki and Y. Shiraki, J. Electrochem. Soc. **133**, 666 (1986); J. Y. Park, and R. J. Phaneuf, J. Vac. Sci. Technol. B **21**, 1254 (2003).
[14]S. M. Sze, *Physics of Semiconductor Devices* (Wiley, New York, 1981).
[15]If the tip is in contact with the conductive surface, the current is proportional to the contact area, which can be described by the Derjaguin–Muller–Toporov (DMT) or the Johnson–Kendall–Roberts (JKR) models, depending on the adhesion force and hardness of the sample. Generally, the JKR model is valid for large tip radius and large adhesion, while DMT applies best in the case of small radius and low adhesion.
[16]M. Enachescu, R. J. A. van den Oetelaar, R. W. Carpick, D. F. Ogletree, C. F. J. Flipse, and M. Salmeron, Phys. Rev. Lett. **81**, 1877(1998); In the DMT fitting, the tip radius of 100 nm, $E^*_{\mathrm{TiN-Si}}$ of 130 GPa are used.