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Scanning Gate Spectroscopy and its Application to Carbon Nanotube Defects

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Abstract

A variation of scanning gate microscopy (SGM) is demonstrated in which this imaging mode is extended into an electrostatic spectroscopy. Continuous variation of the SGM probe's electrostatic potential is used to directly resolve the energy spectrum of localized electronic scattering in functioning, molecular scale devices. The technique is applied to the energy-dependent carrier scattering that occurs at defect sites in carbon nanotube transistors, and fitting energy-resolved experimental data to a simple transmission model determines the electronic character of each defect site. For example, a phenolic type of covalent defect is revealed to produce a tunnel barrier 0.1 eV high and 0.5 nm wide.

Keywords

Carbon nanotube; scanning gate microscopy; defect; molecular electronics; electronic spectroscopy

Scanning probe microscopies are enabling tools of nanoscience, providing a range of information to characterize topography as well as electronic, magnetic, chemical, and other physical properties.¹ In some modes, particularly tunneling current and magnetic force measurements, the standard implementation scans over an energy range in order to achieve spectroscopic detail.¹ Tunneling spectroscopy is not, however, particularly applicable to molecular scale electronic devices fabricated on gate oxides. For such devices, transport spectroscopy² can identify the presence of discrete electronic levels, but ideally one also desires spatial interrogation of these electronic features. Here, we combine the powerful principle of energy spectroscopy with the scanning probe technique of scanning gate microscopy (SGM). SGM is ideal for imaging local, electronic inhomogeneities, and converting this imaging mode into an electrostatic spectroscopy produces a tool well suited to examining functioning molecular devices and their local inhomogeneities.

In conventional SGM, the scanning probe is a noncontact atomic force microscopy (AFM) cantilever, doped or coated to be conductive at its apex, that serves as a movable source of electric fields.¹ The long range interaction of these fields with a surface both probes and redistributes carriers and currents, making the technique particularly relevant to the characterization of operational electronic devices. An SGM image, which is usually acquired simultaneously with surface topography, represents changes in device conductance that accompany the probe movement for each location on a two-dimensional surface.

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SUPPORTING INFORMATION. Detailed schematics, implementation details, device fabrication, and additional control measurements mentioned in the text.

While straightforward in principle, surfaces often exhibit dramatically different SGM images depending on the exact experimental conditions,³⁻¹⁰ and this apparent irreproducibility has traditionally been perceived as a significant weakness of the experimental technique. Furthermore, even a relatively simple three terminal device has a conductance G that depends upon three relevant biases: the drain-source bias V_{source} , the drain-gate bias $V_{backgate}$, and the drain-tip bias V_{tip} of the moveable probe. Any single SGM image is, by necessity, only a small slice of the complex parameter space $G(V_{source}, V_{backgate}, V_{tip}(x,y))$. An electronic device that is nonlinear or otherwise interesting requires a multitude of SGM images taken at different biases to separate and meaningfully understand the functional and spatial G variations. Essentially, one desires to acquire many SGM images over a continuum of bias values.

The purpose of this letter is to describe an implementation of this technique, and to demonstrate its usefulness in explaining complex device behavior. By measuring the entire function $G(V_{source}, V_{backgate}, V_{tip})$ at every position on a surface, a very detailed map of electronic response can be acquired. Because V_{source} determines the energy of injected carriers, and $V_{backgate}$ and V_{tip} modulate the chemical potentials of the current carrying states, the map comprises an energy spectroscopy and we term the technique scanning gate spectroscopy (SGS).

The general experimental setup is identical to that found in the existing SGM literature. The SGS technique itself is relatively straightforward to realize, because sweeping one or more bias parameters is easily implemented on modern scanning probe instruments with adaptable control software, and it is already the norm for current imaging tunneling spectroscopy (CITS).¹ Our instrument is a high vacuum AFM with a liquid nitrogen cold finger (JEOL JSPM-5200), controlled by a custom LabVIEW application. V_{source} , $V_{backgate}$, and V_{tip} are independently controlled during scanning via a multifunctional data acquisition board (National Instruments NI-6289), and the drain current I_d is measured by a homebuilt, floating transimpedance amplifier. A more extensive description with schematics is provided in the Supporting Information. The scanning of the charged probe over an insulating oxide propels the motion of contaminants and populates shallow charge traps, leading to various hysteretic electrostatic effects on the sample surface.^{7, 11} To minimize these effects and greatly improve reproducibility, a combination of sinusoidal V_{source} excitation and cryogenic cooling to $T = 130$ K is used. G is computed from I_d or determined differentially with a lock-in amplifier (Stanford Research Systems SRS 830). Geometric capacitances between the probe and the device substrate dominate the out-of-phase response, and in practice only the in-phase component exhibits bias or spatial dependence at low frequencies.¹²⁻¹⁵ In practice, SGS imaging is greatly simplified by working in the low bias regime where dG/dV_{source} is constant, and by acquiring a subset of $G(V_{backgate}, V_{tip})$ at preselected pixels of interest. Nevertheless, the resulting data set remains a multidimensional vector at each point in space, combining the bias and spatial dependencies of G .

To demonstrate the SGS technique, we have performed measurements on a large number of devices composed of individual, single-walled carbon nanotubes (SWCNTs). These devices are fabricated using standard techniques in the field, namely chemical vapor deposition of SWCNTs on SiO_2 substrates, electrically contacted by lithographically-defined Ti electrodes (see Supporting Information). SWCNTs are an excellent test system because these geometrically simple, one-dimensional devices respond to electrostatic gates through at least three, spatially distinguishable mechanisms. First, a semiconducting SWCNT has a bandstructure that can be locally modulated at every position. Second, contact or Schottky barriers exist at the source and drain electrode interfaces of small diameter semiconducting SWCNTs. Third, SWCNTs can have local defects that are highly sensitive to gating.^{4-7, 16, 17} In general, these three mechanisms have different electrostatics and transconductances.¹⁸

Thus, distinguishing between the three gate sensitivities in a single device provides an excellent test of the motivation behind SGS. In addition, the same SWCNT can be interrogated before and after introduction of a defect by using the “point functionalization” method of dilute electrochemical oxidation.¹⁷ The combination of point functionalization with SGS allows us to clearly investigate the conductance properties of added defects, and potentially provides an avenue for differentiating between different defect types. At present, characterization techniques for SWCNT point defects are severely limited and simple SGM has been able to locate such sites⁴⁻⁷ but not probe their properties.

The three different gating mechanisms are demonstrated in Fig. 1 using standard SGM imaging on a typical SWCNT device (Fig. 1a). At one value of V_{tip} , a defect site introduced into the SWCNT is the only part of the device sensitive to gating (Fig. 1b), while at a different bias the defect contrast is reversed and a Schottky barrier becomes visible (Fig. 1c). In a third bias range, more uniform contrast can result from local band bending and carrier depletion, and this is depicted for a different, defect-free semiconducting SWCNT in Fig. 1d. Individual, fixed-bias SGM images such as these convey limited insight into the transport mechanisms at work, even with fortuitous tuning of the relevant biases.

SGS maps provide much more information than static SGM images, as demonstrated in Fig. 2 for two semiconducting SWCNT devices. In SWCNT and other one-dimensional devices, the quantity of data acquisition is greatly reduced by limiting attention to a line cut of pixels along the conduction path.⁹ Accordingly, the images in Fig. 2 only show the maximal G response that occurs when the probe is directly over one position along the SWCNT length. This data reduction allows the SGS y-axis to represent a non-spatial, independent variable like V_{tip} , variation of which determines the degree of perturbation by the probe. A horizontal slice through an SGS map is therefore equivalent to a static SGM measurement at fixed V_{tip} . A vertical cut, on the other hand, specifies the bias dependence $G(V_{tip})$ of local gate sensitivity at each SWCNT position.

Fig. 2a is an easily interpreted example of an SGS map for a pristine, 2.0 nm diameter semiconducting SWCNT. G shows no appreciable response to negative potentials $V_{tip} < -1$ V, because the local accumulation of additional hole carriers does not substantially increase conduction in a p-type SWCNT. At the other extreme, large, positive potentials $V_{tip} > 1.5$ V are sufficient to deplete these carriers and pinch the device off at any position along the SWCNT. At intermediate potentials $-1 \text{ V} < V_{tip} < 1.5 \text{ V}$, the response of the SWCNT is relatively uniform from source to drain electrode, with small modulations due to contaminants and inhomogeneities of the supporting SiO_2 .¹⁹⁻²¹ The SGS image clearly exposes the spatial positions and relative potential offsets of these individual sites, which often dominate low bias, low temperature device behaviors. By comparison, a static SGM measurement at $V_{tip} = 2.0 \text{ V}$, such as the one shown in Fig. 1d, is equivalent to one strip of the SGS image and often excludes many such sites.^{4, 5} The full SGS image furthermore indicates that no particular features are observed at the source or drain contacts at any gate bias. It is typical for Schottky barrier effects to be negligible for SWCNTs of this diameter.^{14, 22, 23}

Fig. 2b depicts very different results from a smaller, 1.3 nm diameter semiconducting SWCNT containing a defect. In this device, G is more sensitive to specific combinations of probe position and potential. Most of the SGS map, including areas extending over the source and drain electrodes, are uniformly colored, indicating a lack of local gate sensitivity ($dG/dV_{tip} = 0$). Two spatially-localized, gate sensitive regions sit on top of this background. A Schottky barrier exists at the left (source) electrode, where minority carriers are injected. The middle feature centered at $x = 0.8 \mu\text{m}$ is associated with a defect intentionally introduced by point functionalization; this defect resulted from electrochemical oxidation in

H₂O, so it is likely to be one or more phenolic adducts.¹⁷ SGS mapping of the same device before point functionalization observed the Schottky barrier but no other gate sensitivity (see Supporting Information). Fig. 2c depicts a second device, for comparison, which has been modified by a longer duration oxidation treatment. This device has been oxidized for 45 seconds, resulting in a substantially wider area of SWCNT gate sensitivity that effects the SGS map at most biases and tip positions.

In both examples, SGS imaging helps to clearly distinguish the Schottky barrier from the oxidation-induced damage and to quantitatively compare them. For example, Fig. 2b clearly differentiates the potentials at which each site becomes electronically transparent. At the Schottky barrier, a G maximum occurs for $V_{tip} = -1.5 \pm 0.2$ V, whereas the defect site is transparent at $V_{tip} = -0.5 \pm 0.1$ V. The maximum of the Schottky barrier at $V_{tip} = -1.5$ V corresponds to the flat band condition for the SWCNT-Ti interface. In Fig. 2b, the spatial extents of both the defect and the Schottky barrier are broadened by the long-range electrostatics between the probe and SWCNT, but experimental refinements such as those used in Kelvin force imaging¹ may improve the spatial resolution to < 50 nm. In Fig. 2c, on the other hand, it is evident that the SWCNT damage extends beyond the instrumental resolution, even in the present implementation.

In addition to their detailed, graphical information, SGS maps can be made quantitatively instructive by converting changes in V_{tip} to changes in the local carrier potential or energy. In general, this conversion requires an accurate modeling of the device capacitances, but in special cases only the relative capacitances need to be known. For example, an SGS map as uniform as Fig. 2a indicates that $G(V_{tip})$ at every position is identical to the nonlocal, backgated characteristic $G(V_{backgate})$, except for a scaling factor to account for the capacitive ratio $C_{tip}/C_{backgate}$. By comparing the two curves, this ratio can be directly extracted. For typical noncontact AFM scanning with a vibrating probe, we find that the time-averaged capacitance between a segment of SWCNT and probe tip C_{tip} is quite similar to its coupling to the back gate $C_{backgate}$, so that $C_{tip}/C_{backgate}$ is of order unity. A comparison of $G(V_{tip})$ with $G(V_{backgate})$ for the device in Fig. 2a leads to a ratio of approximately 1.1.

This ratio is particularly useful for nanoscale and one-dimensional devices where accurate modeling of the capacitances to the channel may be difficult. The geometric capacitance between the whole SWCNT device and the backgate is calculated²¹ to be $C_{backgate} = 66$ aF. Knowing $C_{backgate}$ and $C_{tip}/C_{backgate}$ provides an effective capacitance for the portion of SWCNT that is affected by the local tip gating, and thus calibrates the effects of V_{tip} . We calculate the energy modulation of the SWCNT under the tip to be approximately 0.2 eV per Volt applied by V_{tip} .

While straightforward, this ratiometric calibration method is limited in practice to devices having a single barrier. When devices have multiple gate-sensitive regions, as in Figs. 2b and 2c, it is impossible to accurately extract $C_{tip}/C_{backgate}$ from a comparison to the $V_{backgate}$ dependence. To illustrate this fact, Fig. 2d reproduces the data of Fig. 2c in a quasi-three-dimensional representation of $G(V_{backgate}, V_{tip})$ along a SWCNT. Two independent SGS maps, one varying $V_{backgate}$ and the other V_{tip} , show that the two imaging conditions are similar but not identical: the central oxidized region matches well at the intersection of the two images, but the Schottky barrier does not. The discrepancy occurs because the backgate affects the entire device: $V_{backgate}$ probes multiple barriers in series and is unable to separate the effect of any one barrier on G . The oxidized region in Fig. 2c has a lower gating threshold than the Schottky barrier does, so that the SWCNT pinches off before the Schottky barrier becomes apparent in the $G(V_{backgate})$ map. Along the V_{tip} axis, on the other hand, each barrier is probed individually to clearly reveal the Schottky barrier's transmission and

transconductance. These differences complicate the determination of C_{tip} , but they also clarify the device's electronic behavior and enable more accurate modeling. In fact, both types of SGS mapping provide much more information than a static SGM image. Fig. 2e shows the optimum SGM image that can be obtained on this device, using potentials that maximize the device response and clarify the relative effects of different SWCNT sections. Finding the ideal operating point for Fig. 2e is normally very time consuming, but the necessary conditions can be directly read from an SGS map like Fig. 2d.

Returning to the discussion of a calibrated SGS energy scale, we note that a more sophisticated and accurate approach will use all of the spatial information in the SGS map to empirically determine C_{tip} . In the vicinity of any spatially-isolated SGS feature, contours of constant G are equivalent to curves of constant potential at the gate-sensitive site. Thus, the electrostatic potential of a defect site V_d is given by the expression

$$V_d = \frac{C_{tg}}{4\pi\epsilon_0 \sqrt{x^2 + z^2}} (V_{tip} + V_g), \quad \text{Eq. 1}$$

where x and z are the lateral distance and height between the tip and the defect site, and C_{tg} is the calculable²⁴ geometric tip-backgate capacitance. Fig. 3a depicts a portion of the SGS data in Fig. 2b, overlaid by a family of constant V_d contours. A simple fit of Eq. 1 to each contour, as shown in Fig. 3b, precisely determines the desired energy calibration $d(eV_d)/dV_{tip} = 0.17 \pm 0.01$ eV/V, in good agreement with the estimate provided above. Past work has applied a similar analysis to multiple SGM images,^{4,5} and the SGS technique provides a much larger, nearly continuous data set for most accurate fitting.

Once the energy scale of a site has been thus calibrated, SGS mapping enables further quantitative analysis of the gate dependence. For example, the Schottky barrier in Fig. 2b has a maximum transmission at $V_{tip} = -1.5$ V. The depth of the Schottky barrier is therefore 0.24 eV, or a little less than half of the SWCNT band gap, in agreement with measurements based on traditional, transport characterization.^{14, 25} The depth of the defect site on the same device is smaller, at 0.08 eV. Both values vary with $V_{backgate}$, but the difference between them does not. In other words, the defect potential is approximately 0.16 eV higher than the flat band condition of the Schottky barrier, regardless of $V_{backgate}$. The flat band condition of the Schottky barrier proves to be an especially useful reference point for subsequent modeling and for the comparison of SGS maps acquired at different $V_{backgate}$, on different days, or from different samples. In the devices studied here, SGM features associated with SWCNT oxidation defects (Fig. 2b) or extensive damage (Fig. 2c) are generally observed close in energy to this reference point. Consequently, it is facile to image a Schottky barrier device having a defect and to compare that defect to the flat band reference point, as has been done here for Fig. 2b. Alternatively, the conduction band edge is not a useful reference energy because it is not accessed in either SGS image, being located higher in energy by half of the SWCNT band gap.

In addition to defining the energetics of a gate-dependent scattering site, SGS information can be used to build and test models of that site's conduction. For example, a vertical line cut extracted from Fig. 2b at the position $x = 0.8 \mu\text{m}$ directly gives $G(V_{tip})$ for the SWCNT defect site (Fig. 4a). Various models have been proposed for scattering by a SWCNT defect,²⁶ but here we compare the observed gate dependence against the simplest possible model consisting of a tunneling barrier²⁷ having energetic height E_b and geometric width a_b . Calculating the transmission T_b through this model barrier in the WKB approximation yields the result

$$T_b(E_b) = \left[1 + \frac{E_b^2}{4E_h(E_h - E_b)} \sin^2 \sqrt{\frac{2m_h a_b^2}{\hbar^2} (E_h - E_b)} \right]^{-1}, \quad \text{Eq. 2}$$

where m_h and E_h are the effective mass and energy of the hole carriers, respectively. The independent variable E_b is taken to be the electrostatic potential of the defect site, and is given by the transformation $E_b = eV_d + E_o$, where E_o is a parameter to match the barrier height observed when $V_{tip} = 0$. Fig. 4b depicts Eq. 2 over the experimentally relevant range of E_b , along with the corresponding values for V_{tip} . The transmission T_b decreases exponentially on either side of $E_b = 0$, and it is asymmetric because holes see a repulsive barrier for $E_b > 0$ but an attractive one when $E_b < 0$. For very negative E_b , T_b oscillates due to bound states in the potential well (Fig. 4b, inset), but this occurs outside of the experimentally accessible energy range $C_{tip} V_{tip}^2$.

To compare the SGS data to the calculated transmission T_b , the experimentally measured $G(V_{tip})$ curve is converted to a defect transmission T_{defect} using the Büttiker-Landauer formalism. This calculation requires correctly accounting for other resistances in the device, such as the Schottky barrier and contact resistance at the drain electrode. Fortunately, the sum of these resistances are constant when the probe is located over the defect site, so they can be lumped together as a “contact” transmission factor T_c in series with the defect. Formally, the measured conductance is expressed in terms of the two series transmission factors as $G(V_{tip}) = (4e^2/h) T = (4e^2/h) [1/T_{defect} + 1/T_c - 1]^{-1}$.²⁸ Taking the maximum in $G(V_{tip})$ to correspond to a transparent defect with $T_{defect} = 1$, all other contributions to the resistance are equivalent to $T_c = 0.1\%$.

Fig. 4b shows the resulting experimental curve $T_{defect}(V_{tip})$ at the defect site and the result of fitting this data using Eq. 2. A simple tunnel barrier characterized by width $a = 0.48 \pm 0.05$ nm and height $E_o = 110 \pm 20$ meV has a transmission T_b that adequately reproduces the data around the T_{defect} maximum.²⁹ Far from this maximum, disagreement between the data and model arises from experimental limitations, namely the conductance noise baseline and the response of the AFM feedback loop to increasing electrostatic forces. The latter error can be reduced by disabling feedback during the $G(V_{tip})$ acquisition, but with other negative experimental consequences. Instead, the data in Fig. 2 is acquired with feedback engaged and with continuous monitoring of any vertical probe motion that results. Fig. 4c shows the vertical displacement of the cantilever during acquisition of the data for Fig. 4b, confirming that the disagreement between the data and model is wholly due to a retracting AFM probe.

In conclusion, by extending SGM with continuous sweeps of V_{tip} , we have interrogated the energy landscape of scattering by a SWCNT point defect. The information content of the SGS imaging technique is high enough to allow quantitative determination of local conductance characteristics. In the case of the SWCNT defect, the spatial dependence in the SGS image allows precise determination of the electrostatic alignment between the defect and the SWCNT bands, and the conductance data provides a measure of the defect’s effective barrier height and width. These results suggest that SGS will be able to distinguish between SWCNT defects having differing chemistries. The experimental technique is readily applied to not just SWCNTs, but also quantum point contacts or other nanoscale electronic devices, and may serve as a useful mapping technique complementary to traditional tunneling spectroscopy. The results are particularly promising for furthering the understanding of defects in one-dimensional conductors on insulating gate oxides, where tunneling techniques are extremely limited.

Supplementary Material

Refer to Web version on PubMed Central for supplementary material.

Acknowledgments

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27. Note that defects added to a pristine SWCNT by oxidation are not chirality changing, so the left- and right-hand sides of the SWCNT have identical bandstructures and work functions.
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29. The stated error bars are determined by the estimated error in determining α for the energy scale. At a given value of α , the standard deviation in the curve fitting to Eq. 1 is 1% or less.

FIGURE 1.

Topography and scanning gate microscopy. (a) Topography of a typical 1.3 nm diameter SWCNT device connected to source and drain electrodes. (b) Device conductance G measured concurrently highlights a defect site ($V_{tip} = 0$ V, $V_{backgate} = -7$ V). (c) At a positive bias $V_{tip} = 2$ V, the defect contrast changes and a Schottky barrier becomes visible at the source electrode. (d) A different, semiconducting SWCNT with transparent contacts and no defect, in which G varies uniformly near the valence band edge. In (b-d), dashed lines indicate contact electrodes, separated by $2\ \mu\text{m}$ in each case. Color scale depicts ΔG modulation on a scale of 0% (white) to 40% (black).

FIGURE 2.

Scanning gate spectroscopy of three different devices, each showing local gate sensitivity $G(V_{tip})$ as a function of SWCNT position. (a) A pristine, semiconducting SWCNT is spatially uniform in its response to local gating. In this device, the dominant gating mechanism is depletion of carriers, or local band bending, wherever the probe travels. (b) A smaller, semiconducting SWCNT exhibits spatially localized sensitivities including a Schottky barrier at $x = 0$ and a weakly scattering defect site added by electrochemical oxidation at $x = 0.8 \mu\text{m}$. (c) Extensive oxidation of a similar device results in spatially extended regions characterized by stronger tip-gate sensitivity. The same data is replotted in (d) in a 3-D composite intersection that shows $G(V_{backgate})$ SGS mapping of the same device. (e) Corresponding SGM map acquired at $V_{tip} = 4 \text{ V}$, $V_{backgate} = -6 \text{ V}$. In each image (a-e), dashed lines indicate the position of contact electrodes, and a rainbow color scale is used to enhance image contrast.

FIGURE 3.

Modeling the defect electrostatics. (a) A section of SGS data from Fig. 2b in the vicinity of the defect, overlaid by curves of constant potential at the defect site. From bottom to top, the lines correspond to potentials V_d from -0.3 to $+0.3$ V in 0.1 eV steps. (b) An example equipotential contour extracted from the left image. Least-squares fitting to the pair of curves yields values $|V_d| = 150 \pm 5$ meV, $E_o = -0.57 \pm 0.02$ eV, and $z = 123 \pm 8$ nm.

FIGURE 4.

Modeling the defect conductance. (a) $G(V_{tip})$ directly over the defect site in Fig. 2b. (b) Defect transmission probability T_{defect} extracted from $G(V_{tip})$ (black), overlaid on the theoretical transmission for a tunnel barrier (red) having width $a = 0.5$ nm and height $E_o = -0.11$ eV. Inset shows T_b over a wider energy range, outside the relevant experimental conditions. (c) Retraction of the AFM probe occurs at large V_{tip} , causing $G(V_{tip})$ and T_{defect} to plateau as higher applied potentials no longer effect stronger gating at the SWCNT.