Single-dopant band bending fluctuations in MoSe₂ measured with electrostatic force microscopy

Megan Cowie^(D),¹ Rikke Plougmann^(D),² Zeno Schumacher,³ and Peter Grütter^(D)

¹Department of Physics, McGill University, Montrèal, Quèbec H3A 2T8, Canada

²Department of Physics, Technical University of Denmark, Lyngby 2800, Denmark

³Department of Physics, Institute of Quantum Electronics, ETH Zurich, 8093 Zürich, Switzerland

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In this paper, we experimentally demonstrate two-state fluctuations in a metal-insulator-semiconductor device formed out of a metallic atomic force microscopy tip, vacuum gap, and multilayer MoSe₂ sample. We show that noise in this device is intrinsically bias dependent due to the bias-dependent surface potential and does not require that the frequency or magnitude of individual dopant fluctuations are themselves bias dependent. Finally, we measure spatial nonhomogeneities in band bending (charge reorganization) timescales.

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I. INTRODUCTION

Individual charge state fluctuations have been observed in a variety of electrically isolated systems such as adatoms [1], quantum dots [2,3], and molecules [4] on insulators. Understanding these systems is critical for the study of single-electron physics, and two-state systems are of particular relevance for emerging quantum information technology. In semiconducting devices, individual charge states such as dangling bonds [5], individual dopants [6], and defects [7] are not electrically isolated from their environment, and it is necessary to understand their effects on the global electronic structure, in particular, device efficiency and noise. In this paper, we measure single-dopant fluctuations which give rise to variations in the surface potential of a mesoscopic metal-insulator-semiconductor (MIS) capacitor device. The MIS device is composed of a metallic frequency-modulated atomic force microscopy (fm-AFM) tip, a vacuum gap, and a multilayer MoSe₂ semiconducting sample.

The energy of a classical capacitor U_{ts} is found by summing the energies of the charge distributions of each capacitor plate:

$$U_{ts} = \frac{1}{2} \left(\int_{t} \rho_t(z) V_t(z) \partial \tau_t + \int_{s} \rho_s(z) V_s(z) \partial \tau_s \right), \quad (1)$$

where the first term is the energy of the top electrode (in this case, the fm-AFM tip) and the second term is the energy of the bottom electrode (MoSe₂ sample). $\rho_{t,s}(z)$ is the volume charge density, $\partial \tau_{t,s}$ is an infinitesimal volume element, and $V_{t,s}(z)$ is the potential of the tip or sample. For a metal-insulator-metal capacitor, ρ and V are spatially invariant, and the expression simplifies to $U_{ts} = \frac{1}{2}Q_{ts}V_{ts} = \frac{1}{2}C_{ts}V_{ts}^2$. The total charge Q_{ts} and potential difference V_{ts} are linear with applied bias, and the capacitance C_{ts} is solely geometric. Deviations from a purely metallic system, such as upon introduction of static charges [8], sample or tip polarizability [9,10], and surface or interface dipoles [11,12] can cause ρ and V to vary spatially and have a nonlinear bias dependence. This is the case for

semiconducting samples, where the surface potential V_S that is established by the charge Q_S inside a semiconductor is bias dependent and spatially nonuniform (thus the potential that arises due to band bending). In this paper, V_S has been calculated by numerically solving the following equation [13]:

$$V_S = V_{\text{bias}} + V_{\Phi} - \frac{Q_S}{C_I},\tag{2}$$

where $Q_S = Q_S(V_S)$ and C_I is the (geometric) capacitance per unit area, found by solving the Poisson equation (see Sec. I of the Supplemental Material [14] for this derivation). V_{bias} is the applied bias between the tip and sample, and V_{Φ} is the difference in the tip and sample Fermi levels ($V_{\Phi} = E_{f,t} - E_{f,s}$). The total charge density is composed of thermal carriers, ionized dopants, and "effective dopants," which are generically any ionizable states such as surface states, interface traps [15], point vacancies [16], contaminants, adatoms, interstitial atoms [17], etc. This incorporation of ionized effective dopant states into the total charge density means that even an undoped sample can behave as though it were doped if other contributing effective dopant densities are appreciable. The occupation of dopant states can vary due to charge transfer from other sample locations, such as the substrate or interstitial contamination layers [18]. The MIS force F_{ts} per unit area a_{tip} , derived by Hudlet *et al.* [13], is

$$\frac{F_{ts}}{a_{\rm tip}} = \frac{Q_s^2}{2\epsilon}.$$
(3)

II. SINGLE-DOPANT FLUCTUATIONS

Figures 1(a) and 1(b) show the bias dependence of V_S and F_{ts} for two different ionized acceptor concentrations, where each data point is a numerical solution to Eqs. (2) and (3). The nonlinear surface potential V_S leads to a nonparabolicity in the force F_{ts} .

In fm-AFM, a tip mounted on a cantilever (spring constant, k; Q factor, Q) oscillates sinusoidally above a sample surface



FIG. 1. Modeled bias dependence of the MIS (a) surface potential [Eq. (2)] and (b) force [Eq. (3)] for two ionized acceptor concentrations: 6.3×10^{17} cm⁻³ (blue) and 7.1×10^{17} cm⁻³ (red). (c) Modeled fm-AFM frequency shift [blue and red, Eq. (4a)] and data (black) on multilayer MoSe₂. Inset: A zoom-in of the frequency shift for which fluctuations in the data (black) were observed. The experimental and model parameters for (a)–(c) are given in this paper. The sweep was acquired over approximately 10 s, and the bandwidth of the phase-locked loop was 305 Hz.

 $[z = A \sin(\omega t)]$. Tip-sample force F_{ts} contributions which are in phase with the cantilever motion lead to shifts $(\Delta \omega)$ in the cantilever resonant frequency ω_o , and out-of-phase force contributions lead to variations in the cantilever drive F_d , or excitation, required to maintain constant oscillation amplitude A:

$$\Delta \omega = \omega - \omega_o = \frac{-\omega_o}{2kA} \frac{\omega_o}{\pi} \int_0^{2\pi/\omega} F_{ts}(t) \sin(\omega t) \partial t, \qquad (4a)$$

$$F_d = \frac{kA}{Q} - \frac{\omega_o}{\pi} \int_0^{2\pi/\omega} F_{ts}(t) \cos(\omega t) \partial t$$
 (4b)

[see Sec. II of the Supplemental Material [14] for a derivation of Eqs. (4a) and (4b)]. The nonparabolicity in the force F_{ts} which arises due to the nonlinearity in surface potential V_S leads to a nonparabolic fm-AFM frequency shift above MoSe₂ [Fig. 1(c)]. Recently, similar nonparabolicities have been reported in other systems measured with fm-AFM, including dangling bonds on Si(111) [5] and pentacene on KBr [19]. Reference measurements on SiO_2 , in contrast, show a parabolic frequency shift as a function of bias. (See Sec. III of the Supplemental Material [14] for these measurements.)

The inset of Fig. 1(c) shows a zoom-in of the kink in the frequency shift parabola. At these biases, the measured frequency shift (black) fluctuates between two states. This is due to individual dopant fluctuations which cause variations in Q_{S} . Notably, what is being measured here is the change in the global electrostatic environment (band bending, $V_{\rm S}$) due to this single-dopant fluctuation, and not a localized changing Coulomb interaction due to the fluctuating occupation of an isolated charge state. The former leads to the changes in nonparabolicity observed in this paper, whereas the latter leads to parabola shifts, such as those shown in Ref. [1]. This is supported by modeling: The two model fits shown differ only in their acceptor concentrations, which are 6.3×10^{17} cm⁻³ (blue) and 7.1×10^{17} cm⁻³ (red). This is a dopant concentration difference of 0.8×10^{17} cm⁻³, which corresponds to approximately one charge within the estimated 2.8×10^{-18} cm³ tip probing volume, assuming a probe volume equal to the effective tip area ($\pi \times 10^2$ nm²) times the sample thickness (9 nm). This fluctuation effect does not manifest homogenously over the sample surface; rather, it is only present at certain locations and vanishes when the tip is moved slightly (50 nm).

The measured frequency shift fluctuation is maximized at biases corresponding to the kink in the surface potential (approximately -1.4 V). This is demonstrated in Fig. 2, which shows the surface potential and force as a function of tipsample separation (z_{ins}) for two acceptor concentrations at -2.2 V [Figs. 2(a) and 2(d)], -1.4 V [Figs. 2(b) and 2(e)], and 1.0 V [Figs. 2(c) and 2(f)]. Equation (4a) indicates that the closest z_{ins} force gives the largest contribution to the fm-AFM frequency shift, due to the multiplication of F_{ts} by $\sin(\omega t)$. The difference in this integrand for the two acceptor concentrations is shown over two oscillation cycles in Fig. 2(g). Integrating Fig. 2(g) according to Eq. (4a) gives Fig. 2(h). Figure 2(h) is effectively a "noise sensitivity function" for these two-state fluctuations.

Highlighted by horizontal lines in Figs. 2(a)–2(c) and markers with insets in Figs. 2(d)–2(f) are the surface potential and force at the closest tip-sample separation. At -1.4 V, the two acceptor concentrations have a comparatively large difference in surface potential and force at the closest z_{ins} , and consequently the difference of $F_{ts} \times \sin(\omega t)$ [the integrand of Eq. (4a)] is largest. In comparison, at -2.2 V there is a large difference in surface potential at the top of the oscillation cycle, but since it is not maximally amplified by $\sin(\omega t)$, the resulting frequency shift difference is small.

This model therefore shows that the bias dependence of the frequency shift fluctuation amplitude [Fig. 1(c)] is *not* due to a bias dependence of the dopant state (which has nowhere been incorporated into this model). Rather, it is due to an amplification from the intrinsic nonlinear surface potential: At biases corresponding to the parabola kink, where the surface potential has the largest nonlinearity, the difference between the ionized state force and the un-ionized state force is maximized at the closest tip-sample position, leading to the largest difference in frequency shift.



FIG. 2. (a)–(f) The surface potential and force as a function of tip-sample separation z_{ins} at -2.2 V [(a) and (d)], -1.4 V [(b) and (e)], and 1.0 V [(c) and (f)]. Two different acceptor concentrations are shown: 6.3×10^{17} cm⁻³ (blue) and 7.1×10^{17} cm⁻³ (red). Vertical black lines indicate the region of the curves over which the cantilever oscillates with an amplitude of 6 nm and closest z_{ins} of 5 nm [a demonstrative sinusoid is shown in (a)]. In (a)–(c), horizontal lines are drawn to highlight the surface potential at the closest tip-sample separation for each acceptor concentration [in (c), they are overlapping]. In (d)–(f), an inset shows the closest z_{ins} force at the position indicated by the marker. (g) The difference between the two acceptor concentrations of Eq. (4a) [$F_{ts} \times \sin(\omega t)$] over two oscillation cycles at -2.2, -1.4, and 1.0 V. (h) The frequency shift difference between the two acceptor concentrations, with representative biases indicated with markers.

III. BAND BENDING TIMESCALE

Figure 3 shows the excitation signal measured simultaneously with the frequency shift data shown in Fig. 1(c). The nonconstant excitation signifies an out-of-phase tip-sample force F_{ts} , according to Eq. (4b). This out-of-phase component arises from charge reorganization inside the sample, or in



FIG. 3. Modeled excitation signal as a function of bias with (a) constant Q factor (18 000) and (b) constant lag (30 ns). The other parameters match those defined in this paper. Experimental results taken simultaneously with those shown in Fig. 1 are shown in black.

potential. This lag time is modeled by incorporating a phase offset between the closest tip-sample position and the maximum tip-sample force. Modeled lag times ranging from 0 to 50 ns are shown in Fig. 3(a), and the best estimate of this band bending timescale is 30 ns. This timescale is effectively a resistance-capacitance (RC) time constant for the tip-vacuumsample MIS system. An order-of-magnitude estimate of the time constant $\tau = RC$, assuming a simple parallel-plate capacitance $C = \frac{\epsilon \epsilon_{oa}}{z_{ins}}$ and resistivity $\rho = \frac{Ra}{z_{ins}}$ (where ϵ and ϵ_o are the relative and free permittivity and a and z_{ins} are the plate area and separation distance), agrees with this model: Using the best-fit relative permittivity given in this paper $(\epsilon = 5.9)$ and a resistivity of 500 Ω m (which is within the wide reported resistivity range of $0.1-1000 \ \Omega m$ for MoSe₂ [20]) gives $\tau = 26$ ns, which is consistent with the measured 30 ns. The model appears to be simplistic as the fit misses many details of the data. This could be due to a bias-dependent lag time, which has not been accounted for here. Nonetheless, this explanation can be useful for an order-of-magnitude estimate of the band bending timescale. Note that when the lag is zero, the excitation signal is flat, and this is independent of Q. Furthermore, Fig. 3(b) demonstrates that this excitation signal does not arise from variations in Q as a function of bias, since large variations in Q at a constant lag time (30 ns) do not reproduce the measured phenomenon. This confirms that the measured excitation signal is not due to "dissipation" (i.e., a nonconservative force), but rather a time-delayed conservative force. In comparison, reference measurements on SiO_2 , shown in Sec. III of the Supplemental Material [14] show constant excitation as a function of bias.

other words, it is an equilibration timescale for the surface



FIG. 4. Multipass fm-AFM image (i.e., constant-frequency-shift image) of a region of an exfoliated $MoSe_2$ island. (a)–(d) Topography (a), oscillation amplitude (b), frequency shift (c), and excitation (d) channels recorded during the first pass, at 0 V and -2 Hz set point. The topography shown in (a) is retraced for each subsequent pass. The frequency shift [(e), (g), and (i)] and excitation [(f), (h), and (j)] channels are shown for the subsequent passes, which are at +1 V [(e) and (f)], -2 V [(g) and (h)], and -4 V [(i) and (j)]. The horizontal scale bars correspond to 500 nm.

The excitation channel can be measured spatially to map variations in band bending timescales over a sample surface. This is demonstrated in Fig. 4, which shows multipass frequency shift and excitation images at varying bias. The highest layer of the island, susceptible geometrically to the largest surface potential since it is not so spatially limited in the z direction, exhibits the most appreciable bias dependence in both frequency shift and excitation. At -2 V, bright spots appear in the excitation channel, indicating a spatial nonhomogeneity in the charge equilibration time. The charge reorganization timescales at these sites are largest near -1.4 V because this bias corresponds to the largest change in surface potential over every oscillation cycle (as demonstrated in Fig. 2). At larger negative voltages, rings appear in the excitation channel. These rings further indicate the spatial and bias dependence of the charge reorganization time: When the tip is laterally offset from a long-reorganization-timescale site, there is an additional potential drop. These rings, visible in Fig. 4(j) at -4 V, therefore correspond to the same phenomenon shown in Fig. 4(h) at -2 V.

We note that this observation of bias-dependent excitation is not a piezoacoustic excitation system transfer function artifact such as described in Ref. [21], since by changing the position slightly on the sample, the frequency shift and excitation vary independently (this is shown in the Supplemental Material [14]). This excitation signal is also not due to tunneling charge transfer between the sample and tip, since the effect was observed at tip-sample separations of >5 nm (also see Supplemental Material [14]).

IV. MODEL DESCRIPTION

The surface potential, force, frequency shift, and excitation were modeled according to Eqs. (2), (3), (4a), and (4b). Over every oscillation cycle, the tip-sample separation changes, the potential drop across the vacuum gap changes, and the potential drop inside the semiconductor changes, resulting in a time-dependent surface potential and force. This time-dependent force is integrated to determine the frequency shift and excitation, where the excitation signal is only nonzero when there is a band bending timescale, and consequently a phase offset between the closest tip-sample position and the maximum tip-sample force. See Sec. IV of the Supplemental Material [14] for a more detailed explanation of this process and a video description. Each model point shown in Figs. 1(a)–(c) and 3 is a numerical solution to Eqs. (2), (3), (4a), and (4b) at a different bias.

Model parameters. Equations (2), (3), (4a), and (4b) involve 14 experimental parameters. The impact each has on the overall parabola shape is distinct: The band gap, acceptor concentration, permittivity, and temperature introduce nonparabolicity; the tip work function and sample electron affinity introduce lateral shifts and leave the shape unchanged; and the tip-sample separation, tip radius, oscillation amplitude and frequency, spring constant, and Q factor are all fm-AFM scaling factors that are multiplicative prefactors which do not change the overall shape. See Sec. V of the Supplemental Material [14] for a further explanation of the sensitivity of each of these parameters and their impact on the shape. Optimal fit values were found by exploring a large parameter space $(>120\,000$ curves) and finding the fit that minimizes residuals. The few-layer MoSe₂ band gap is 1.55 eV [21,22] (best fit, 1.5 eV), the electron affinity is 3.5 eV [23] (no fit against this parameter), and the relative (dielectric) permittivity is 5.7 [24] (best fit, 5.9). A rigorous interpretation of permittivity is challenging because there is likely water in between the thin flaked MoSe₂ sample and the SiO₂ substrate. However, taking this fitted permittivity as an "effective permittivity" of the net system does not affect the overall interpretation. The effective electron and hole masses were assumed to be 1.0, and the temperature was assumed to be 300 K. The spring constant of 42 N/m estimated by the tip manufacturer agreed with the best fit. The Q factor of 18000 and resonant frequency of 330 kHz were measured experimentally by performing a frequency sweep. The oscillation amplitude set point, maintained with a phase-locked loop and a feedback circuit, was 6 nm. The work function of Si/SiO₂ is 4.4 eV, and given that the substrate parabola peak (i.e., V_{Φ} ; see Sec. III of the Supplemental Material [14]) is -250 mV and V_{Φ} is the difference in the tip and sample work functions ($V_{\Phi} = \Phi_{\text{tip}} - \Phi_{\text{sample}}$), Φ_{tip} is 4.15 eV (best fit, 4.1 eV). The effective tip radius was taken to be 10 nm, which is consistent with the tip manufacturer's estimate of an effective radius "better than 25 nm." The optimal closest tip-sample separation z_{ins} was 5.2 nm. The position of the experimental parabola kink at negative biases indicates that the effective dopants are of p type. The dopant concentration, the final fitting parameter, is optimized at 6.3×10^{17} cm⁻³ (blue curves in Fig. 1) and 7.1×10^{17} cm⁻³ (red curves in Fig. 1). The deviation at large negative biases in Fig. 1(c) could be due to the emergence of stray capacitances as the applied electric field increases.

V. EXPERIMENTAL DETAILS

The sample of MoSe₂ on SiO₂ measured in this paper was prepared by all-dry viscoelastic stamping [25]. The top layer of the micrometer-scale multilayer island of MoSe₂ is 9.0 ± 0.4 nm above the silicon substrate (see Ref. [26] for height measurement methodology). All fm-AFM measurements were taken using Nanosensors platinum-iridium-coated silicon tips (PPP-NCHPt) with 330 kHz resonant frequency, a spring constant of 42 N/m, and *Q* factors of approximately 18 000 in a JEOL JSPM-4500A ultrahigh-vacuum AFM with a Nanonis control system. All measurements were performed in ultrahigh vacuum (base pressure $<3 \times 10^{-10}$ mbar) at room temperature. The sample was annealed at $120 \,^{\circ}$ C for 8 h each time it was introduced into the vacuum and was grounded during all measurements.

VI. CONCLUSION

In conclusion, we have experimentally demonstrated the direct relationship between a single-fluctuating-dopant state and its effect on the global band structure (band bending) in a mesoscopic MIS device. The bias dependence of these fluctuations does not depend on the bias dependence of the dopant state occupation, but is, rather, inherently due to the bias dependence of the surface potential. This has important ramifications for MIS-like device functionality and noise: It indicates that even in the absence of bias-dependent dopant or defect states, device noise is bias dependent. The fluctuating two-state effect is demonstrated here for the wellcharacterized two-dimensional MoSe₂ system, but it has also been observed other systems, such as pentacene [19]. This indicates that this is not a sample-specific phenomenon, but rather is relevant for a thorough understanding of noise in any semiconductor device. Additionally, we have demonstrated that band bending equilibration timescales may be measured using the fm-AFM excitation signal. Given that fm-AFM affords high spatial resolution, this approach may be used to directly measure band bending timescales of different types of defects.

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