Probing defect states in few-layer MoS$_2$ by conductance fluctuation spectroscopy

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Despite the concerted effort of several research groups, a detailed experimental account of defect dynamics in high-quality single- and few-layer transition metal dichalcogenides remain elusive. In this paper we report an experimental study of the temperature dependence of conductance and conductance-fluctuations on several few-layer MoS$_2$ exfoliated on hexagonal boron nitride and covered by a capping layer of high-$\kappa$ dielectric HfO$_2$. The presence of the high-$\kappa$ dielectric made the device extremely stable against environmental degradation as well as resistant to changes in device characteristics upon repeated thermal cycling enabling us to obtain reproducible data on the same device over a time-scale of more than one year. Our device architecture helped bring down the conductance fluctuations of the MoS$_2$ channel by orders of magnitude compared to previous reports. The extremely low noise levels in our devices made in possible to detect the generation-recombination noise arising from charge fluctuation between the sulphur-vacancy levels in the band gap and energy-levels at the conductance band-edge. Our work establishes conductance fluctuation spectroscopy as a viable route to quantitatively probe in-gap defect levels in low-dimensional semiconductors.

Following the discovery of graphene$^1$, the exploration of the basic physics and technological implications of two-dimensional (2D) materials has gained tremendous importance. Though graphene is a system rich in novel physics, the lack of band-gap limits its applications in transistor technology. Transition metal dichalcogenides (TMD) like MoS$_2$ and MoSe$_2$, on the other hand, have band-gaps of the order of eV in the few-layer limit$^2$, making them ideal for opto-electronic applications$^3-5$. On the flip-side, the reported mobilities of these TMD based field effect transistor (FET) devices are very low$^4,6$ and the quoted values vary widely between samples. It is now understood that defect-levels (primarily arising from chalcogenide vacancies) adversely affect the mobility and optical properties of these TMD-based devices$^7-10$.

Despite extensive research, there is no clear understanding of the underlying defect dynamics in this system. Traditional transport measurements like current-voltage characteristics and the temperature dependence of the resistance, while providing indications of the existence of defect states, cannot directly probe their energetics$^9,11$. Photoluminescence measurements report the appearance, at low temperatures of an additional peak in the spectrum which is tentatively attributed to transitions from a 'defect'-level$^{12,13}$, but no direct evidence of this level has been found from optical studies. Transmission electron microscopy (TEM)$^7,14-17$ and scanning tunnelling microscopy (STM)$^{18-23}$ have shown that the primary point-defects are S-vacancies although other types of defects like interstitials, dislocations, dopants and grain boundaries were also seen. These two techniques come with their own sets of limitations. While TEM imaging is believed to induce additional defects in MoS$_2$$^{24,25}$, atomic-resolution imaging of few-layer TMD using STM has proved challenging$^{11,26-28}$. Thus, although theoretical studies predict the presence of prominent defect-levels in these materials$^{29-33}$, probing them experimentally has proved to be challenging.

In this paper, we present conductance fluctuation spectroscopy$^{34}$ as a viable technique to identify these defect states and their characteristic energy levels. Conductance fluctuations (noise) in TMD-based devices has been studied by several groups$^{35-39}$. In different studies, the observed conductance fluctuations have been variously attributed to charge-carrier number density fluctuations due to trapping at the interface$^{36}$, to mobility fluctuations$^{37,38}$ or to contact noise$^{39}$. In general, in the high doping regime, carrier-number density fluctuation model could explain the measured noise behavior while in the low doping regime mobility fluctuation models seemed to better fit the experimental observations$^{40}$. Thus there is a lack of consensus in the community as to the origin of the observed large conductance fluctuations in this system. The problem is aggravated by the fact that ultra-thin layers of TMD degrade extremely fast when exposed to the ambient$^{39,41-43}$. This makes repeated, reliable measurements on the same device challenging while at the same time severely limiting the scope of practical applications.

We have performed detailed measurements of temperature $T$ dependence of conductance and conductance-fluctuations on several few-layer MoS$_2$ exfoliated on hexagonal boron nitride (hBN) and covered by a film of high-$\kappa$ dielectric HfO$_2$. We find that over a large range of $T$, the noise in the system is dominated by generation-recombination processes caused by random charge fluctuations via transitions between the S-vacancy impurity band and the conduction band of MoS$_2$. The presence of
Figure 1: (a) Optical image of device D1. The bottom hBN is defined with a solid green line and the few-layer (FL) MoS$_2$ is outlined by a red dashed line. The top HfO$_2$ is outlined by a dashed black line. (b) Gate-voltage $V_g$ dependence of the resistance $R$ of device D1 at a few representative temperatures ranging from 115 to 300 K in steps of 15 K. The inset plots the on-set voltage $V_{th}$ versus $T$ for devices D1 (red open circles) and D2 (green open squares).

In Fig. 2(a) we plot the sheet resistance $R$ of the device D1 in a semi-logarithmic scale versus inverse temperature for few representative values of $V_g$. The linearity of the plots indicates that, at least in the high $T$ limit, electrical transport is dominated by thermal activation of the charge carriers. More specifically, as we go higher in $V_g$, the range of $T$ where this linearity holds extends down to lower temperatures. The activation energy, $\varepsilon$, extracted from the slope of the $\ln(R)$ versus $1/T$ plots is plotted in Fig. 2(b). One can see that $\varepsilon$ increases as one decreases the gate bias and it varies from 20 meV at high $V_g$ to an order of magnitude higher $\sim$200 meV, close to the off-state of the device. The activation energy for device D2, extracted in a similar fashion is, as expected, higher than that of D1 at all values of $V_g$.

The field-effect mobility $\mu$ of the devices can be obtained from the relation $\mu = \frac{dV_{th}}{dV_g} \frac{1}{wC}$. Here $L$ is the length of the channel, $w$ its width and $C$ is the gate capacitance per unit area. In Fig. 2(c) we show plots of $I_{ds}$ versus $V_g$, the slope of this curve gives the mobility of the device. A plot of the $T$ dependence of the mobility is shown in Fig. 2(d). We find that $\mu$ for device D1 is $\sim 20$ cm$^2$V$^{-1}$s$^{-1}$ at 100 K. With increasing $T$, $\mu$ increases monotonically till about 225 K beyond which it begins to fall with increasing $T$. To understand the measured $T$ dependence of $\mu$ we note that in 2D semiconductors, the mobility of the charge carriers is affected by Coulomb scattering, acoustic and optical phonon scattering, scattering by the interface phonon and roughness due to the surface. At high $T$, scattering due to phonons is dominant which causes the mobility to have a $T^{-3/2}$ dependence. On the other hand, scattering from charge impurities located randomly in the sample is the dominant factor limiting $\mu$ at low temperatures causing the mobility to depend on temperature as $T^{3/2}$.
Figure 2: (a) Scatter plot of resistance $R$ of device D1 plotted on a semi-logarithmic scale versus $1/T$ at several representative values of $V_g$ from 10 V to 90 V in steps of 5 V. The solid lines are the linear fits of $1/T$ vs $\ln(R)$. (b) Plots of activation energy $\varepsilon$ versus $V_g$ extracted from the $\ln(R)$ versus $1/T$ plots for the two devices, D1 (red open circles) and D2 (green open squares). The lines are guides to the eye. (c) Plots of $I_{ds}$ versus $V_g$ at different $T$ ranging from 115 K to 300 K for device D1. (d) Plots of mobility $\mu$ versus $T$ for the two devices, D1 (red open circles) and D2 (green open squares). The blue line is fit to the data for D1 using Eqn. 1.

\[ \frac{1}{\mu} = \frac{1}{M_p T^{-3/2}} + \frac{1}{M_i T^{3/2}} \]  (1)

where $M_p$ and $M_i$ represent the relative contributions of the phonon-scattering and impurity-scattering mechanisms respectively. These coefficients are not independent, but are related by $(M_p/M_i)^{1/3} = T_{max}$, where $T_{max}$ is the temperature at which $\mu$ has a maxima. In Fig. 2(d) we show a fit of the $T$ dependence of the mobility of D1 to Eqn. 1. The mobility of D2, on the other hand, monotonically increases with $T$ showing that over the range of $T$ studied, impurity-scattering dominates the transport in on-SiO$_2$ substrate devices.

The presence of both bulk- and surface-transport channels complicates the charge transport in these systems. To understand the charge-carrier dynamics arising from the surface- and bulk-states in this system, we studied the low-frequency conductance fluctuations over the temperature range 70 K-300 K using a 2-probe ac digital-signal-processing technique$^{51}$. As established in several previous reports, $1/f$ noise is an excellent parameter to probe inter-band scattering of charge-carriers in systems with multiple conduction channels$^{52-54}$. We used an SR830 dual-channel digital LIA to voltage-bias the sample at a carrier frequency of $f_0 \sim 228$ Hz. The current $I_{ds}$ through the device was amplified by the low-noise current preamplifier and detected by the LIA. The data were acquired at every $T$ and $V_g$ for 32 minutes at a sampling rate of 2048 points/s using a fast 16-bit data acquisition card. This time-series of current fluctuations $\delta I_{ds}(t)$ was digitally anti-alias filtered and decimated. The power spectral density (PSD) of current-fluctuations, $S(f)$ was calculated from this filtered time-series using the method of Welch-periodogram$^{51,56}$. The system was calibrated by measuring the thermal (Johnson-Nyquist) noise of standard resistors. The time-series of $I_{ds}$ measured for device D1 at a few representative temperatures at $V_g = 90$ V are shown in Fig. 3. We find that over the $T$ range $\sim$140-190 K, the measured $I_{ds}(t)$ (and consequently the conductance $g(t) = I_{ds}(t)/V_{ds}$) for D1 fluctuates between two well-defined levels$^{27,58}$. This ‘Random telegraphic noise’ (RTN)$^{39}$ usually signifies that the system has access to two (or more) different states separated by an energy barrier. We come back later in this article to a discussion of the detailed statistics of the RTN and the physical origin of these states.

In Fig. 4(a) we plot the PSD of current fluctuations at a few representative values of $T$ and $V_g = 90$ V for the device D1. We find that over the $T$ range where RTN were present in the time-series $I_{ds}(t)$, the PSD deviates significantly from the $1/f$ dependence (shown in the plot by a gray line). This can be appreciated better from
Figure 4: (a) Plots of $S(f)$ versus $f$ at a few representative temperatures for device D1. The data were measured for $V_g = 90\, \text{V}$. (b) Plots of $fS(f)$ versus $f$ for the same values of $T$ as in (a). The dotted purple lines are fits using Eqn. 2 to the data at 165 K, 175 K and 185 K. (c) Plot of $fS(f)$ as a function of $f$ over the temperature range 145 K (purple data points) –195 K (red data points) in steps of 5 K. The arrow indicates the evolution of $f_C$ to higher values with increasing $T$. The solid lines are guides to the eye. (d) Plot of the logarithm of $f_C$ versus $1/T$. The solid line is a linearized fit to the Arrhenius relation $f_C = f_0\exp(-E_a/k_BT)$. The inset shows a plot of $f_C$ versus $T$. The two dotted lines are the upper (28 Hz) and lower (31.25 mHz) limits of our measurement bandwidth.

(a) Plots of $S(f)$ versus $f$ at a few representative temperatures for device D1. The data were measured for $V_g = 90\, \text{V}$. (b) Plots of $fS(f)$ versus $f$ for the same values of $T$ as in (a). The dotted purple lines are fits using Eqn. 2 to the data at 165 K, 175 K and 185 K. (c) Plot of $fS(f)$ as a function of $f$ over the temperature range 145 K (purple data points) –195 K (red data points) in steps of 5 K. The arrow indicates the evolution of $f_C$ to higher values with increasing $T$. The solid lines are guides to the eye. (d) Plot of the logarithm of $f_C$ versus $1/T$. The solid line is a linearized fit to the Arrhenius relation $f_C = f_0\exp(-E_a/k_BT)$. The inset shows a plot of $f_C$ versus $T$. The two dotted lines are the upper (28 Hz) and lower (31.25 mHz) limits of our measurement bandwidth.

Figure 5: (a) Plot of $fS(f)$ as a function of $f$ over the temperature range 156 K (purple data points) – 207 K (red data points) in steps of 4 K for the device D1b. (b) Plot of the logarithm of $f_C$ versus $1/T$. The solid line is a linearized fit to the Arrhenius relation $f_C = f_0\exp(-E_a/k_BT)$. The inset shows a plot of $f_C$ versus $T$. The measurements were done at $V_g = 90\, \text{V}$.

Figure 6: (a) Plot of $I_d$ versus time at a few representative values of $T$ for the device D2. (b) PSD $S_I(f)$ corresponding to the time-series shown in (a). The gray line shows a representative $1/f$ curve. The measurements were done at $V_g = 72\, \text{V}$.

levels. This motivated us to fit the measured PSD of current fluctuations to an equation which contains both $1/f$ and Lorentzian components:

$$
\frac{S_I(f)}{f^2} = \frac{A_1}{f} + \frac{A_2 f_C}{f^2 + f_C^2}
$$

(2)

$A_1$ and $A_2$ are fit parameters that denote the relative contributions of the random and RTN fluctuations respectively to the total PSD. The dotted purple lines are
fits to the data at 165 K, 175 K and 185 K using Eqn. 2. In Fig. 4(c) we show plots of $fS(f)$ versus $f$ over an extensive range of $T$. We find that as $T$ increases, the peak position evolves from a few mHz to few tens of Hz [see the inset of Fig. 4(d)]. Beyond this $T$ range, the value of $f_C$ goes beyond our measurement frequency bandwidth (31.25 mHz–28 Hz). The value of $f_C$ is thermally activated and follows the Arrhenius relation: $f_C = f_0 \exp(-E_a/k_BT)$. Figure 4(d) shows a plot of ln($f_C$) versus $1/T$, the red-line is a fit to the activated behaviour. The value of activation energy $E_a$ extracted from the fit is 370 meV. These measurements were repeated on three such devices (MoS$_2$ encapsulated between hBN and HfO$_2$); we find that the activation energy-scale in all of them lie in the range $370 \pm 30$ meV. In Fig. 5, we show data for another device, D1b, for which we obtain $E_a=353$ meV. We come back to the physical implications of this energy-scale later in this article.

The time-series $\delta I(t)$ for device D2, on the other hand, did not have any RTN component (Fig. 6(a)) and the PSD had a $1/f^\alpha$ (with $0.9 < \alpha < 1.1$) dependence on $f$ over the entire $T$ and $V_g$ range studied (Fig. 6(b)). The $I_{ds}(t)$ data were obtained for the device D2 at 72 V. We have attempted to compare the data in the two sets of devices at similar values of number-densities. Due to the presence of the hBN layer, the effective thickness of the dielectric layer in D1 was higher than that of D2 — requiring a higher gate-voltage for D1 than that for D2 to achieve similar carrier number density. On the other hand, D1 had a lower threshold voltage than D2. Taking both these factors into account, we have estimated the $V_g$ at which the induced number densities are similar for both D1 and D2. Thus, for D1, the data are presented for $V_g=90$ V while for the device D2, the data are presented for $V_g=72$ V.

In Fig. 7(a) we present the $V_{ds}$ dependence of the quantity $fS(f)$ measured at $T=175$ K and $V_g=90$ V for the device D1. We see that the form of the PSD is independent of $V_{ds}$. To make this observation quantitative, we plot in Fig. 7(b) the dependence of $f_C$ on $V_{ds}$ extracted from these plots using Eqn. 2. The fact that $f_C$ is independent of $V_{ds}$ within experimental uncertainties shows that this time-scale is intrinsic to the sample.

The PSD, $S_I(f)$ can be integrated over the frequency bandwidth of measurement to obtain the relative variance of conductance fluctuations, $G_{\text{var}}$ at a fixed $T$ and $V_g$:

$$G_{\text{var}} \equiv \frac{\langle \delta g^2 \rangle}{\langle g \rangle^2} = \frac{\langle \delta I_{ds}^2 \rangle}{\langle I_{ds} \rangle^2} = \frac{1}{(I_{ds})^2} \int_{0.03125}^{28} S_I(f)df.$$  

(3)

The relative variance of conductance fluctuations, $G_{\text{var}}$ was found to be independent of $V_{ds}$ at all $T$ and $V_g$ confirming that the noise arises from conductance fluctuations in the MoS$_2$ channel and not from the contacts [for representative data, see Fig. 7(c)].

We measured the noise as a function of gate-bias voltage, $V_g$ — the results obtained at $T=170$ K for the device D1 are plotted in Fig. 8(a). We find $f_C$ to be independent of $V_g$ (Fig. 8(b)) with-in experimental uncertainties. In Fig. 8(c) we have plotted $G_{\text{var}}$ as a function of $V_g-V_{\text{th}}$. The total noise has been separated into its $1/f$-component and the RTN-component. At low values of $V_g-V_{\text{th}}$, the $1/f$-component noise contribution is comparable to that of the RTN-component while at higher $V_g-V_{\text{th}}$, the RTN-component dominates the measured conductance fluctuations. This motivated us to perform our noise measurements at high $V_g$ (90 V) so that the RTN component of the noise is easily resolvable.

In Fig. 9 we show a plot of $G_{\text{var}}$ versus $T$ for the two devices. The noise for device D1 (plotted in green open circles) has a prominent hump over the $T$ range (~140–190 K) coinciding with the regime where we observed RTN. To appreciate this, we plot on the same graph the relative variance of conductance fluctuations arising from the $1/f$ component (red filled circles) as well as the Lorentzian component (blue open circles). It can be seen that the increase in noise over the 140–190 K temperature range is entirely due to two-level conductance fluctuations in the system. For comparison, we also add a plot of $G_{\text{var}}$ versus $T$ for the unencapsulated device prepared on SiO$_2$-substrate, D2. The noise on SiO$_2$ substrate devices, is more than two orders of magnitude larger than that of D1 and matches with previous reports of measured noise in MoS$_2$ by various groups. Our work thus shows that encapsulation helps in significantly improving the signal to noise ratio.
A careful study of Fig. 9 provides clues to the origin of the observed noise in this system. The temperature dependence of the relative variance of conductance fluctuations $\varphi_{\text{var}}(T)$ measured for the on-SiO$_2$ substrate device D2 closely resembles the $T$-dependence of the $1/f$ component of $\varphi_{\text{var}}$ measured on D1. This indicates that these two noises have similar origins. The primary source of the $T$-dependence of noise in many semiconductor devices is generation-recombination (GR) noise due to trapping-detrappping of charges at the gate dielectric-channel interfaces. This process can be quantified by the McWhorter model$^{40,61,62}$:

$$N_{\text{im}} = f S_T(f) \langle R \rangle^2 \frac{W L C^2}{e^2 k_B} \frac{1}{T}$$

(4)

where $N_{\text{im}}$ is the areal-density of trapped charges per unit energy, $W$ and $L$ are respectively the width and the length of the device-channel, $C$ is the gate-capacitance per unit area, $k_B$ is the Boltzmann constant and $e$ is the charge of the electron. Equation 4 predicts the linear dependence of $f S_T(f)$ on the temperature. Fig. 10 shows a plot of $f S_T(f)$ versus $T$ for both D1 and D2. The plots are linear to within experimental uncertainties. From the slopes of these plots, the value of $N_{\text{im}}$ for device D2 was extracted to be $3.5 \times 10^{12}$ cm$^{-2}$ eV$^{-1}$ which agrees with previously reported values for MoS$_2$ devices prepared on SiO$_2$ substrates$^{63}$. On the other hand, for the HfO$_2$ covered, on-hBN device D1, $N_{\text{im}} = 1.8 \times 10^{10}$ cm$^{-2}$ eV$^{-1}$, more than two orders of magnitude lower than that in the on-SiO$_2$ substrate device D2.

The non-$1/f$ seen only in the encapsulated device has a different origin. The presence of RTN in the time-series of conductance fluctuations and the associated Lorentzian component in the PSD indicates that the noise originates from random charge fluctuations via transitions between two well-defined energy states separated by an energy barrier. We propose that in this case, these two levels correspond to the S-vacancy impurity band and the conduction band. This is supported by the fact that the value of the activation energy, $E_a = 370$ meV extracted from the temperature dependence of the corner-frequency $f_c$ of the Lorentzian component of the current fluctuations matches closely with the estimated position of the S-vacancy impurity band with respect to the conduction band edge$^{64}$. Note that it was possible for us to detect this fluctuation-component only because of the two orders of noise reduction made possible by the introduction of hBN between the MoS$_2$ and SiO$_2$ substrate.

The HfO$_2$ layer has a two-fold effect on the noise. Firstly, being a high-k dielectric with a dielectric constant value of about 25, its presence screens the device from Coulomb scattering, and reduces the $1/f$ noise by orders of magnitude, enabling us to detect the RTN. Secondly, it acts as a capping layer that shields the MoS$_2$ from the ambient. We believe that this prevents the S-vacancies from getting saturated by adsorbates, thus preserving the RTN. With the current data, we cannot distinguish between these two effects. Preliminary results obtained on devices fabricated on hBN without the HfO$_2$ capping layer had higher on-off ratios, higher-mobilities and lower noise levels as compared to MoS$_2$ devices fabric-
Figure 10: Plot of $fS_I(f)$ versus $T$ for the (a) on-SiO$_2$ substrate device D2, and (b) on-hBN HfO$_2$-encapsulated device D1. The red dashed line in both the plots are fits to Eqn. 4.

cated on SiO$_2$ without the HfO$_2$ capping layer - however, we did not find any RTN in these devices. From these results, one can tentatively conclude that both the top- and bottom-layers are necessary to preserve the RTN. This issue is currently under detailed investigation.

Finally, coming to the question of stability of the devices, we have compared the $R$ versus $T$, $R$ versus $V_g$ and the noise measurements on device D1 immediately after fabrication and after a gap of several months. The sample was thermally cycled several times during this period between 300 K and 77 K. As shown in Fig. 11, the temperature and $V_g$ dependence of the resistance of the device were quite reproducible. This is in sharp contrast to un-encapsulated on-SiO$_2$ devices like D2 in which after a few days the channel and contacts both degrade drastically making further measurements impossible.

Similarly, thermal cycling alters the characteristics of such devices and makes the channel resistance unstable. The stability of the resistance of D1 over time period of months confirms that encapsulation between hBN and HfO$_2$ makes the device robust to thermal cycling and against degradation with time.

To conclude, in this paper we reported on detailed conductance fluctuation spectroscopy of high-quality MoS$_2$ devices encapsulated between hBN and HfO$_2$. The presence of the high-$\kappa$ dielectric made the device extremely stable against environmental degradation enabling us to obtain reproducible data on the same device for over 1 year. The hBN substrate helped bring down the conductance fluctuations by over two orders of magnitude as compared to similar devices on bare SiO$_2$ substrates. The low noise levels in our devices made it possible to detect the generation-recombination noise arising from charge fluctuation between the S-vacancy levels in the MoS$_2$ band-gap and states at its conductance band edge. Our work establishes conduction fluctuation spectroscopy as a viable route to detect in-gap defect levels in low-dimensional semiconductors.

Figure 11: (a) Comparison between the $R$ versus $T$ data at $V_g = 90$ V for device D1 right after fabrication (red data points) and several months as well as several thermal cycles later (green data points). (b) Similar comparison of $R$ versus $V_g$ data measured at $T = 270$ K for D1 in the pristine state and several months (and thermal cycles) later.

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