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Nikesh Koirala, Maryam Salehi, Jisoo Moon, and Seongshik Oh Phys. Rev. B **100**, 085404 — Published 5 August 2019 DOI: 10.1103/PhysRevB.100.085404

1 Gate tunable quantum Hall effects in defect-suppressed Bi₂Se₃ films

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Despite many years of efforts, attempts to reach the quantum regime of topological surface 10 states (TSS) on an electrically tunable topological insulator (TI) platform have so far failed 11 12 on binary TI compounds such as Bi₂Se₃ due to high density of interfacial defects. Here, utilizing an optimal buffer layer on a gatable substrate, we demonstrate the first 13 electrically tunable quantum Hall effects (QHE) on TSS of Bi₂Se₃. On the *n*-side, well-14 defined QHE shows up, but it diminishes near the charge neutrality point (CNP) and 15 completely disappears on the *p*-side. Furthermore, around the CNP the system transitions 16 from a metallic to a highly resistive state as the magnetic field is increased, whose 17 temperature dependence indicates presence of an insulating ground state at high magnetic 18 fields. 19

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Key words: Bi₂Se₃, quantum Hall effect, topological insulator, electric field effect, charge
neutrality point

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24 TSS provides a rich playground for a number of topological quantum effects such as topological magnetoelectric effect, Majorana fermions and QHE[1-8]. However, due to high level of surface 25 Fermi level originating from unintended native dopants, it has been challenging to access the 26 quantum regime of TSS. In particular, even if QHE is one of the most intensely studied 27 phenomena in 2D systems, gate tuned studies of QHE of TIs has been so far limited only to 28 ternary or quaternary compounds such as Bi_{2-x}Sb_xTe₃ and (Bi_xSb_{2-x}Se_yTe_{3-y})[4,5,9]. On the other 29 hand, binary compounds can potentially provide cleaner platforms compared with quaternary or 30 tertiary solid solutions, allowing better access to the Dirac point, due to increased carrier 31 mobilities and reduced electron-hole puddles. For the binary compounds such as the prototypical 32 TI Bi₂Se₃, tracking evolution of QHE as a function of gate voltage has not been possible due to 33 high density of bulk and interfacial defects[10,11]. Recently, however, utilizing a structurally 34 and chemically-matched buffer layer that solves the defect problem, QHE was observed in 35 Bi₂Se₃ thin films[12,13]. Here, by adapting this buffer layer scheme to a gatable SrTiO₃(111) 36 substrate[14], we present the first gate-dependent study of QHE in Bi₂Se₃. 37

Low-carrier density Bi_2Se_3 thin films were grown on an electrically insulating buffer layer, which comprises a heterostructure of 5 QL $In_2Se_3 - 4$ QL $(Bi_{0.5}In_{0.5})_2Se_3$ grown on SrTiO₃ (111) substrate following the recipe of ref. 9 [15]. The films were then capped *in situ* either by a 100 nm Se or a 50 nm MoO₃/50 nm Se layer to protect against ambient contamination[16]. Then, a ~100 nm thick Cu layer was deposited *ex situ* on the back surface of SrTiO₃ substrate as a back gate. The films were then scratched into millimeter sized Hall bars using a metal mask and a tweezer, and indium leads were used to make electrical contacts [15].

45 On these Hall bar patterns, Hall (R_H) and sheet resistance (R_S) were initially measured at 46 magnetic field (B) up to ± 0.6 T in a cryostat at T = 5 K. The measured raw data were 47 symmetrized or anti-symmetrized to eliminate mixing of longitudinal and Hall resistances due to imperfection in the measurement geometry [15]. $[e \cdot (dR_H/dB)]^{-1}$, which corresponds to sheet 48 carrier density (n_S) for single-carrier species transport, and mobility $(\mu) = (R_o \cdot n_S \cdot e)^{-1}$ were then 49 calculated, where dR_H/dB is the slope of low field linear part of Hall resistance (unless otherwise 50 stated), e is the electronic charge and R_o is the zero-field sheet resistance. These films have n-51 type carriers with $n_S \approx 5 \times 10^{12}$ cm⁻² and $\mu \approx 1,000 - 3,000$ cm²V⁻¹s⁻¹ [15]. Compared to the films 52 grown directly on SrTiO₃(111), where n_S is typically 4×10^{13} cm⁻², the buffer-layer grown 53 54 films exhibit an order of magnitude decrease in the defect density[17], which is consistent with 55 our previous report[12]. This low sheet carrier density obtained with the buffer layer was essential for reaching the quantum regime of TSS via gating as we present below. 56

In the rest of the paper, we focus on the gate voltage dependence of R_H and R_S in MoO₃/Se-capped films. As reported previously, MoO₃ capping further reduces the *n*-type Fermi level of Bi₂Se₃ films toward the charge neutrality point (CNP)[12,18]. We measured films of three different thicknesses: 8, 10 and 15 QL. 8 QL film was measured at T = 1.5 K and *B* up to \pm 9 T and 10 QL and 15 QL films were measured at T = 5 K and *B* up to \pm 0.6 T.



resistance (R_o , upper panel) and ($e \cdot dR_H / dB$)⁻¹ (lower panel), which corresponds to sheet carrier density (n_S) for single carrier species transport as a function of back-gate voltage, V_G , for 8, 10 and 15 QL films respectively. Solid black lines are a guide to the eye. The insets show magnetoresistance (R_S , upper panel) and corresponding Hall resistance (R_H , lower panel) as a function of magnetic field, B, taken at several representative back-gate voltage values from which R_o and n_S were extracted. Note the different magnetic field range and temperature for 8 QL compared to 10 and 15 QL films. Ambipolar behavior is observed in (a-b).

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Figures 1 (a)-(c) show R_o (upper panel) and $(e \cdot dR_H/dB)^{-1}$ (lower panel; for single species transport it is equivalent to n_S) as a function of back-gate voltage (V_G) of 8, 10 and 15 QL films, respectively. In all three samples, the *n*-type carrier density is less than 3×10^{-12} cm⁻² at $V_G = 0$ V, which is well below the maximum carrier density ($\sim 1 \times 10^{-13}$ cm⁻²) required to make the bulk state of TIs insulating[19]. For the 15 QL film, we were able to modulate n_S from 2.8×10^{-12} cm⁻² to 1.5×10^{-12} cm⁻² and R_o from ~ 1.5 kΩ/sq to ~ 2.8 kΩ/sq, as V_G is tuned from 0 to ~ 20 V.

77 However, ambipolar transport was not observed in this film, presumably because 15 QL is too thick for its top surface to be electrostatically modulated by the bottom gating. For 8 and 10 QL 78 films, R_o increases with V_G , reaches a maximum value (for example at $V_G \approx -37$ V for 10 QL 79 film) and then decreases with further increase in V_G . Concurrently, (*n*-type) n_S decreases with V_G 80 and eventually changes to p-type (for example at $V_G \approx -45$ V for 10 QL film). Such an ambipolar 81 behavior not only confirms the TSS conduction, but also the tunability of chemical potential 82 across the CNP[20]. Therefore, we focus on 10 QL films below and carry out more in-depth 83 measurements up to much higher magnetic fields (34.5 T). 84



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Figure 2 | QHE as a function of magnetic field at several gate voltage values for a 10 QL film. (a)-(e) sheet resistance (R_s , upper panel) and Hall resistance (R_H , lower panel) up to |B| = 34.5 T at $V_G = 0$, -30, -40, -70 and -100 V respectively. Change in the sign of Hall effect and corresponding maxima in (f) zero field sheet resistance R_o at $V_G \approx$ -70 V indicates that *p*-type carriers dominate the transport for V_G -70 V. For *n*-type carriers, v = 1 QHE is clearly observed at high magnetic fields. (g) Evolution of QHE

91 with carrier density, where QHE disappears when carriers change from *n*- to *p*-type. *p*-type carrier density 92 for $V_G = -100$ V was estimated from the average slope of Hall effect in (e).

Figures 2(a)-(e) show R_S (upper panel) and R_H (lower panel) of another identically 93 prepared 10 QL film as a function of B up to \pm 34.5 T for various V_G values at T = 0.35 K [15]. 94 At low magnetic fields, magnitude of the negative slope of R_H increases as V_G changes from 0 to 95 -40 V, indicating decreasing *n*-type carrier density from 1.5×10^{12} cm⁻² to 5.9×10^{11} cm⁻². At V_G 96 = -70 V, R_H fluctuates strongly around zero indicating mixed transport from electrons and holes 97 and at $V_G = -100$ V the slope becomes positive albeit non-linear. The nonlinear Hall effect 98 possibly indicates multi-carrier transport likely due to the effect of electron-hole puddles or due 99 to residual *n*-type carriers on the top surface [5,21]. However, the overall positive slope of R_H 100 indicates that conduction is now dominated by *p*-type carriers. Figure 2(f) shows corresponding 101 change in R_o , where it increases till $V_G = -55$ V, shows a maximum at -70 V $\leq V_G \leq -55$ V and 102 then decreases below $V_G = -70$ V. This indicates that the film goes through CNP at $V_G \approx -70$ V. 103

At high magnetic fields, we observe increasingly developed dips in R_S and plateaus at 104 h/e^2 in R_H for -30 V $\leq V_G \leq 0$ V indicating v = 1 QH state. Additionally, we observe developing 105 plateau-like features at $R_H \approx h/3e^2$ in the same voltage range, which is consistent with top and 106 bottom surfaces having similar carrier density in this gate voltage range [15]. At $V_G = -40$ V both 107 the dip in R_S and the plateau in R_H are less well developed than at $V_G = -30$ V indicating that the 108 v = 1 QH state weakens as the Fermi level is lowered toward CNP. For $V_G = -70$ V (-100 V), QH 109 signature is completely gone and R_S increases monotonically with B and reaches ~878 k Ω /sq 110 111 (~90 k Ω /sq) at B = 25 T (32 T), corresponding to ~10,000% (~1000%) of magnetoresistance as defined by MR% = $\frac{R_S(B) - R_S(0)}{R_S(0)}$ × 100 %. In Fig. 2(g), we summarize our observation, where v = 112 1 QHE emerges with decreasing *n*-type carrier density until $V_G \approx -30$ V, then diminishes while 113

approaching CNP and gives way to a highly resistive state in the *p*-regime. The color plot in Fig. 2(g) was obtained by smoothly interpolating between the carrier densities measured at $V_G = 0$ V and at $V_G = -100$ V. We note that the *p*-type carrier density at $V_G = -100$ V in Fig. 2(g) was estimated by taking the average, rather than the low-field, slope of the Hall resistance.

In order to observe continuous evolution of transport with V_G , we have measured R_S and 118 R_H as a function of V_G at various magnetic fields B from 0 to 44.5 T and various temperatures T 119 from 0.35 K to 9 K on another 10 QL thick film. Figures 3(a) and 3(b) show R_S and R_H , 120 respectively, at different magnetic fields and at T = 0.35 K [15]. For all magnetic fields we 121 observe a peak in R_S and a change in the sign of R_H from negative to positive at -30 V $\leq V_G \leq$ -122 26 V, indicating that *p*-type carriers dominate the transport below these gate voltages. For higher 123 124 magnetic fields (B > 23 T), we observe a developing dip in R_S and a plateau in $R_H \approx -25.8$ k Ω for -20 V $\lesssim V_G \lesssim$ -15 V indicative of v = 1 QH state for *n*-type carriers. Near CNP and for *p*-type 125 carriers, we observe neither the dips in R_S nor the plateaus in R_H in magnetic field up to 44.5 T. 126 Inset of Fig. 3(a) shows the magnetic field dependence of R_S at CNP (R_{CNP}) and at $V_G = -76$ V (R_{CNP}) 127 $_{76V}$; where p-type carriers dominate) indicating that R_S increases monotonically with B for both 128 CNP and *p*-type carriers, reaching as high as 250 k Ω (44 k Ω) and ~44 k Ω (18 k Ω) at B = 44.5129 3,000% (600%) and 650% (100%) of T (11.5 T), respectively, which correspond to 130 magnetoresistance. 131



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In order to get an additional perspective, we plot sheet conductance $\sigma_{XX} = R_S / (R_S^2 + R_H^2)$ 138 and Hall conductance $\sigma_{XY} = R_H / (R_S^2 + R_H^2)$ in Fig. 3(c) and 3(d), respectively. Consistent with 139 resistance plots, we observe a σ_{XY} plateau at $\sim h/e^2$ and a minimum in σ_{XX} at -20 V $\lesssim V_G \lesssim$ -15 V 140 indicative of v = 1 QHE. Apart from a plateau-like feature at $\sigma_{XY} \approx 0$ and corresponding 141 minimum in σ_{XX} for -35 V $\lesssim V_G \lesssim$ -30 V, which can possibly indicate the v = 0 state, no features 142 resembling QHE are observed in conductance plots for $V_G \leq -30$ V[4,5,9,22], implying that QHE 143 is lost when *p*-type carriers dominate the transport. The lack of QHE on the *p*-side is consistent 144 145 with both the recent transport result on compensation-doped Bi₂Se₃ films and the lack of Landau levels (LL) on the *p*-side of Bi₂Se₃ in scanning tunneling spectroscopy measurements[13,23,24]. 146

147 It can be explained by the proximity of the Dirac point to the bulk valence band and the much 148 broader surface band on the *p*-side of $Bi_2Se_3[25,26]$. This is in marked contrast with Sb-based 149 TIs, which exhibit QHE and LLs for both *n*- and *p*-sides due to relatively symmetric surface 150 bands with a well-exposed Dirac point[4,27,28,27].

Next, we discuss temperature dependence of R_S versus V_G at different magnetic fields in 151 order to understand the behavior of p-type carries in Bi₂Se₃. As shown in Fig. 4(a), zero-field R_S 152 is $< 7.5 \text{ k}\Omega/\text{sq}$, which is much lower than the quantum resistance (25.8 k Ω), and increases only 153 slightly (~4% at CNP) at T = 0.35 K compared to T = 2 K for all V_G . At higher temperatures ($T \approx$ 154 6 to 12 K), we observe similarly small variation in R_S with temperature in a different but 155 identically prepared 10 QL film [15]. In the inset of Fig. 4(a) we show R_S versus T at $V_G = 0$ V, 156 157 where small upturn at low temperatures is observed, consistent with previous studies on TI films. In addition, we observe weak anti-localization at low fields for all V_G values and in all samples 158 (see Fig. 2 and 3). Both of these observations are consistent with gapless Dirac band transport in 159 160 the presence of disorder[4,29].



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Figure 4 Temperature dependence of resistance at high magnetic field. Sheet resistance (R_S) at B =(a) 0 T (b) 11.5 T (c) 20 T and (d) 44.5 T as a function of V_G at several different temperatures. Note that the film was not measured at 9 K in (c). Inset in (a) shows semi-log plot of R_S versus T at $V_G = 0$ V indicating a small upturn at low temperatures, which fits $\log(T)$ dependence as indicated by the blue line. Insets in (b-d) show behavior of R_S around $V_G = 0$ V in greater detail. (e) 2D plot of ratio

167 plotted as a function of V_G and B. Metallic-like behavior is observed for $V_G > -21$ V while, for V_G 168 ≤ -21 V insulating-like behavior is observed. (f) Semi-log plot of temperature dependence of normalized 169 R_{CNP} at B = 11.5 T and 44.5 T, where larger slope at 44.5 T indicates stronger insulating tendency: the 170 solid lines are least-square fits using $R_{CNP} \sim \log(T)$.

In order to see how transport changes at higher fields, we plot R_S versus V_G at 11.5 T, 20 171 T and 44.5 T for T = 0.35 K – 9 K in Fig. 4 (b)-(d), respectively [15]. A slight shift in V_G 172 corresponding to CNP at T = 9 K compared to lower temperatures is observed, but does not 173 affect our analysis. In Fig. 4 (b)-(d), $R_S(T_1) \approx R_S(T_2)$ for $V_G > -21$ V indicating metallic 174 behavior, while for $V_G \lesssim -21$ V, $R_S(T_1) >> R_S(T_2)$ suggesting an insulating behavior for these 175 gate voltages for all three magnetic fields, where $T_1 < T_2$ are temperatures. Figure 4(e) 176 177 summarizes this observation, where we show a 2D plot of the ratio of R_S at T = 0.3 K to that at T = 1 K $(R_{0.3 \text{ K}}/R_{1 \text{ K}})$ as a function of V_G and B. n-type carrier region shows metallic-like behavior 178 (i.e. $R_{0.3 \text{ K}}/R_{1 \text{ K}} \approx 1$) along with QHE at high magnetic fields, while an insulator-like highly 179 resistive state (i.e. $R_{0.3 \text{ K}}/R_{1 \text{ K}} > 1$) is observed near CNP and for *p*-type carrier region at high 180 fields. 181

In order to further understand the nature of the highly-resistive state, we have plotted 182 R_{CNP} (T)/ R_{CNP} (9 K) as a function of temperature at B = 11.5 T and 44.5 T in Fig. 4(f). R_{CNP} 183 $(T)/R_{CNP}$ (9 K) increases logarithmically with decreasing temperature for both B = 11.5 T and 184 44.5 T, with stronger insulating behavior observed at higher field as indicated by greater slope of 185 R_{CNP} vs. log(T) for B = 44.5 T. For comparison, insets of Fig. 4 (b)-(d) show an enlarged view of 186 R_S versus V_G at $V_G \approx 0$ V, where R_S either decreases or does not increase significantly with 187 decreasing temperature indicating a metallic behavior. Such an increasingly insulating behavior 188 near CNP at higher magnetic fields indicates presence of a magnetic-field-induced insulator 189 phase, whose origin remains unknown at present. Local and non-local measurements at lower 190

temperatures and higher magnetic fields could elucidate the nature of this ground state, which weleave for future work.

In conclusion, we have studied gate-dependent QHE on low-carrier density Bi₂Se₃ thin films for the first time by employing a novel buffer layer growth method on gate-amenable SrTiO₃ substrates. At low fields we observe ambipolar transport for thinner films, and at high fields we observe v = 1 QHE for *n*-type carriers. On the other hand, for CNP and *p*-type carriers we observe non-saturating magnetoresistance up to B = 44.5 T, whose temperature dependence point to the existence of a magnetic-field-induced insulating state. Further experimental and theoretical efforts are necessary to clarify its origin.

200 Acknowledgements:

This work is supported by the Gordon and Betty Moore Foundation's EPiQS Initiative (GBMF4418) and National Science Foundation (NSF) grant EFMA-1542798. A portion of this work was performed at the National High Magnetic Field Laboratory which is supported by NSF Cooperative Agreement No. DMR-1644779 and the State of Florida.

205 Author contributions:

N.K. and S.O. conceived the experiment. N.K., M.S. and J.M. synthesized the samples and performed transport measurements. N.K. and S.O. wrote the manuscript with inputs from all the authors. All authors contributed to the scientific analysis and manuscript revisions.

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