Bilayer-induced asymmetric quantum Hall effect in epitaxial graphene

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Abstract
The transport properties of epitaxial graphene on SiC(0001) at quantizing magnetic fields are investigated. Devices patterned perpendicularly to SiC terraces clearly exhibit bilayer inclusions distributed along the substrate step edges. We show that the transport properties in the quantum Hall regime are heavily affected by the presence of bilayer inclusions, and observe a significant departure from the conventional quantum Hall characteristics. In particular, we observe anomalous values of the quantized resistance and a peculiar asymmetry with magnetic field which was not observed before for graphene on SiC. A quantitative model involving enhanced inter-channel scattering mediated by the presence of bilayer inclusions is presented that successfully explains the observed symmetry properties.

Keywords: Epitaxial graphene on SiC, quantum Hall effect, resistance standard, monolayer graphene, bilayer graphene, metrology

(Some figures may appear in colour only in the online journal)

1. Introduction

The quantum Hall (QH) effect, discovered by Klaus von Klitzing et al in 1980 \cite{1}, occurs when a high-mobility two-dimensional electron system is subject to a strong perpendicular magnetic field at low temperatures. In these conditions, the Hall resistance $R_H$ is observed to be quantized at rational fractions of the universal quantum $h/e^2$, where $h$ is the Planck constant and $e$ the electron charge \cite{2}. In clean GaAs/Al\textsubscript{1−x}Ga\textsubscript{x}As \cite{3} and Si \cite{4, 5} samples, the resistance quantization can have a relative uncertainty as low as a few ppb \cite{6, 7}, and since 1990 the QH effect has been used as the primary resistance standard \cite{8, 9}. Graphene is recently emerging as a new ideal material for QH metrology \cite{10–14} and has the potential to outperform existing alternatives. Indeed, thanks to its particular linear dispersion relation, graphene carries a much larger energy spacing between the first Landau levels compared to GaAs-based systems \cite{15}. As a consequence, magnetic field and temperature requirements for the observation of the QH effect are less demanding in graphene and have already allowed the observation of resistance quantization at room temperature \cite{16}.

While the first devices produced from exfoliated graphene suffered from poor contact resistances and comparatively high relative uncertainties (15 ppm) \cite{10}, more recently devices based on epitaxial graphene on SiC(0001) reached values better than 1 ppb and could offer a route for scalable applications \cite{11}. Epitaxial graphene on SiC is obtained by annealing SiC wafers at high temperature (above 1200 °C) \cite{17–19} which induces a sublimation of Si atoms and the formation of a graphene monolayer. The unavoidable
presence of a slight wafer miss-cut causes a number of atomically sharp step edges separated by flat terraces across the crystal surface [20] where nucleation of multilayer graphene domains is favored [21]. This results in the formation of narrow multilayer inclusions—typically bilayers [22–25]—which run along the SiC step edges and can have a detrimental impact on the transport characteristics.

From the experimental side, the relation between magnetotransport properties and orientation of SiC step edges is still under investigation [26–29]. In particular, focusing on the QH regime, recent works have shown that in devices in which the bilayer patches did not cross even the narrowest part of the Hall-bar, the expected half-integer QH effect was observed irrespective of the orientation between the device and the substrate [28, 30]. Differently, anomalous QH traces were observed in samples where bilayer domains cross the Hall-bar and induce shunting of edge channels [31–33]. Transverse transport channels were put forward as a possible mechanism, but a quantitative comparison with theory is still lacking.

In our work, we demonstrate that bilayer inclusions can cause deviations in the $R_{xx}$ and $R_{xy}$ resistance characteristics which—differently from previously reported—can even be asymmetric with respect to the magnetic field direction. Thanks to a combined analysis based on micro-Raman mapping and transport analysis, we quantitatively demonstrate that these anomalies can be fully explained within the Landauer–Büttiker picture, and we correlate transport data with the orientation of the multilayer inclusions.

3. Results and discussion

3.1. Micro-Raman layer topography

Raman spectroscopy is a powerful tool to differentiate between mono- and multilayer graphene [35]. In the case of SiC substrates, most studies concentrate on the so-called 2D peak at around 2700 cm$^{-1}$ caused by a double resonant scattering process involving two phonons. It is the signal of choice since its frequency lies far away from the excitation structures of the underlying SiC substrate that make the detection of other graphene signatures more difficult.

The intensity, width, and frequency of the 2D peak are sensitive to the number of layers [35]. However, the 2D peak shows also a great sensitivity to strain and charge inhomogeneity: this makes the assessment of layer number not conclusive. The ultimate signature of monolayer graphene is considered to be the line-shape of the 2D peak [36, 37]. In particular, there is general agreement that the peak of monolayer graphene can be fitted with a single Lorentzian, while a decomposition into four Lorentzians is necessary for bilayer graphene [38].

As a first step, we produced a fine Raman-scattering map to determine the layer topography of the entire device. Figure 2(a) shows our result for a step size of 0.5 μm with integration times as long as 10 s for low-noise spectrum detection. As discussed in the following, for our device we were able to detect the number of graphene layers directly by integrating the scattering intensity in suitable spectral ranges.

The device investigated in this work is a Hall-bar (length × width = 300 μm × 50 μm) fabricated by standard optical lithography from an epitaxial graphene layer grown on a 6 H–SiC(0001) substrate. The epitaxial graphene was grown by annealing 6 H–SiC(0001) in Ar atmosphere at 100 Torr. The annealing temperature was approximately 1820 °C. A cross-section and a schematic of the device is displayed in figures 1(a) and (b), respectively. The orientation of the Hall-bar was deliberately chosen to be perpendicular to the SiC step edges, in order to have the terraces running across the device from one side to the other. Ohmic contacts were made by a Cr/Au (5/250 nm) metallic layer. As can be seen in figure 1(b), the Ohmic contacts were not recessed as is commonly done, but jotted out into the Hall-bar. A bilayer stack (140 nm of hydrogen silsequioxane (HSQ) and 40 nm of SiO$_2$) [34] was used as a dielectric. On top of this dielectric, Cr/Au (10/30 nm) electrodes in split-gate geometry were defined by e-beam lithography (not used in the experiments discussed here). We obtained similar results from another, nominally identical device.

Transport measurements were performed in a Heliox $^3$He cryostat with a base temperature of 250 mK. Longitudinal and transverse resistances $R_{xx} = V_{xx}/I_{SD}$ ($xx = 4 - 6, 1 - 3$) and $R_{xy} = V_{xy}/I_{SD}$ ($xy = 4 - 1, 6 - 3$) were measured by standard lock-in technique in a 4-point configuration using small bias currents $I_{SD} \sim 10$ nA to avoid heating. Magnetic fields in the 0–9 T range were used to characterize the device in the QH regime. The measured values of the carrier density and mobility are $n = 3.4 \times 10^{11}$ cm$^{-2}$ and $\mu = 4660$ cm$^2$ V$^{-1}$ s$^{-1}$, respectively.

Raman spectra were collected at room temperature, using a Renishaw Micro-Raman spectrometer employing a 532 nm laser excitation and a typical spot diameter of <1 μm.
A systematic analysis of the spectra acquired at several points across the device gives us confidence that we can identify two integration ranges corresponding to monolayer and bilayer graphene.

In order to obtain a Raman map, we first acquired the whole spectrum (1300–2800 cm\(^{-1}\)) at each point and corrected it for the SiC contribution by subtracting a reference spectrum of the bare substrate. The resulting spectral data were then integrated over the ranges indicated in figures 2(b–d), where three typical spectra acquired at different positions are shown, each one exhibiting different and peculiar characteristics. At point (b), the shape of the 2D peak is perfectly fitted by a single Lorentzian, thus demonstrating that monolayer graphene is present at this position. At point (c) the peak has a complex shape, and it is significantly red-shifted. Most importantly, the peak can be satisfactorily fitted only by using four Lorentzians, a clear signature of bilayer graphene. Point (d) shows the intermediate situation, where the laser spot covers an area where mostly monolayer graphene is present, but the bilayer contribution cannot be neglected. By exploiting the red-shift occurring for bilayer graphene, we can identify two separate integration ranges 2680–2720 cm\(^{-1}\) (range I) and 2720–2760 cm\(^{-1}\) (range II) for the detection of the number of graphene layers. We carefully checked that red-shifted peaks could be fitted by four Lorentzians only by analyzing the spectra at several locations, and we could assign range I and range II to monolayer and bilayer graphene, respectively. As an example, the points where the spectra in figures 2(b–d) were acquired are shown in figure 2(e), which is a low-noise Raman intensity map, integrated over the 2720–2760 cm\(^{-1}\) range (bilayer), of a portion of the device marked by the red rectangle in (a). Monolayer domains (b) can be fitted with a single Lorentzian, while bilayer domains (c) require a four-Lorentzian fit. At the same time, the 2D peak of bilayer domains is red-shifted, which allows identifying the number of layers easily from the integrated intensity. The dashed lines in (c) and (d) repeat the monolayer curve from (b), to visualize the red shift, while the horizontal bars in (c) indicate range I (light green) and range II (violet). The RGB composite map in (a) contains range I in the G channel, range II in the B channel, and the sum of ranges I and II in the R channel.

Figure 2. Raman topography of the device. (a) Composite map of Raman intensity integrated over the intervals 2680–2720 cm\(^{-1}\) (range I) and 2720–2760 cm\(^{-1}\) (range II). Large stripes of monolayer graphene (light green) lie on the SiC terraces, and are partially intersected by narrower bilayer domains (violet). An overlay indicating the Ohmic contacts and the split-gates is also shown. (b)–(d) 2D peak extracted from the Raman spectra acquired at three different points of the device. Their positions are indicated in (e), which is a low-noise Raman intensity map, integrated over the 2720–2760 cm\(^{-1}\) range (bilayer), of a portion of the device marked by the red rectangle in (a). Monolayer domains (b) can be fitted with a single Lorentzian, while bilayer domains (c) require a four-Lorentzian fit. At the same time, the 2D peak of bilayer domains is red-shifted, which allows identifying the number of layers easily from the integrated intensity. The dashed lines in (c) and (d) repeat the monolayer curve from (b), to visualize the red shift, while the horizontal bars in (c) indicate range I (light green) and range II (violet). The RGB composite map in (a) contains range I in the G channel, range II in the B channel, and the sum of ranges I and II in the R channel.

3.2. Quantum Hall regime

Magnetoresistance traces of our device are shown in figure 3. The traces of longitudinal (figure 3(a)) and transverse (figure 3(b)) resistance were measured using different contact pairs. For \(|B| < 5\) T, the traces of the longitudinal resistance
are similar, and both show the typical behavior expected for clean graphene monolayer Hall-bars, comprising a weak localization peak around zero-field, and the developing of magneto-oscillations precursory to the Shubnikov–de Haas oscillations [39]. In the same field range, both traces of transverse resistance $R_{xy}$ show a monotonic dependence and display kinks at $|B| \approx 3$ T. These kinks coincide with the most pronounced minima in $R_{xx}$ and correspond to a filling factor $\nu = 6$.

For $|B| > 5$ T, the curves measured using different contact pairs are strongly asymmetric, and deviate significantly from the quantized values expected for homogeneous monolayer graphene in the QH regime. Considering the longitudinal resistance, both $R_{1-3}$ and $R_{4-6}$ saturate at a value $\approx 10$ k$\Omega$ for $B > 0$. For $B < 0$, $R_{1-3}$ saturates at the same value $\approx 10$ k$\Omega$, whereas $R_{4-6}$ displays a vanishing value. Correspondingly, the transverse resistance $R_{3-6}$ displays a $0.5 \times h/e^2$-plateau for both field signs, whereas $R_{1-4}$ reaches the $0.5 \times h/e^2$-plateau only for $B > 0$, and $R_{1-4} \approx -1.6$ k$\Omega$ for $B < 0$.

A magnetic-field dependence similar to $R_{1-3}$ was already observed for Hall-bars intersected by bilayer inclusions which are believed to shunt the transport channels at opposite sides of the bar [31, 33, 40]. In these structures, $R_{xx}$ was insensitive to an inversion of the direction of circulation of the channels, and it thus exhibited the same saturation values regardless of the sign of magnetic field. In contrast to these previous findings, however, here we show that $R_{1-6}$ has a remarkably asymmetric field-dependence, and is not invariant under inversion of the magnetic field. Analogously, in the transverse direction, a symmetric behavior is displayed only by $R_{3-6}$, with clear plateaus corresponding to filling factor $\nu = 2$, as expected for transverse contact pairs connected by a continuous monolayer region. On the other hand, $R_{1-4}$ is markedly asymmetric, thus implying the presence of an effect which depends on the chirality of the edge-channels. We note that for $|B| > 5$ T, all magnetoresistance curves are flat, suggesting that their values are pinned to quantized resistance plateaus.

In order to analyze our results, in the following we develop a model based on the information provided by the Raman data shown in figure 2. In particular, we start with the presence of bilayer stripes which connect one side of the device to the other. In agreement with previous publications [31, 33, 40] we assume that additional edge channels are present in the bilayer regions that cause a shunting of the edge states circulating in the monolayer graphene. Our quantitative analysis exploits the Büttiker–Landauer formalism [41], in an approach similar to the one applied to monolayer–bilayer planar junctions on exfoliated graphene [42–45]. For the sake of simplicity we model our device as consisting of monolayer graphene intersected by one bilayer region. Of course in the real device more than one stripe might contribute to the transport, but as we will show, this simple model already explains the main features of our data. The direction of the bilayer stripe is chosen so as to be consistent with the Raman map of figure 2(a). Furthermore, as motivated in the previous paragraph, we assume that both monolayer and bilayer graphene are in a quantized QH state, possibly hosting different numbers of edge states. If we assume that the bilayer stripe connects the two sides of the Hall-bar without being in direct contact with any of the Ohmic contacts, we obtain deviations from the quantized values of monolayer graphene, but the curves are symmetric upon inversion of the direction of the magnetic field. In order to obtain asymmetric curves it turns out to be necessary that the stripe connects an Ohmic contact with a region between two contacts on the other side of the device. Finally, in order to reproduce the observed peculiar asymmetry of the curves in figure 3, it is necessary to impose that the bilayer connection runs between contact 4 and the other side of the device between contacts 1 and 2, as shown in figure 4. Any other configuration fails to reproduce the observed symmetries while simultaneously remaining consistent with the Raman data. The fact that in our device the Ohmic contacts stick out into the Hall-bar makes this

Figure 3. Quantum Hall effect in the device. (a) Longitudinal and (b) Hall resistance as a function of magnetic field at $T = 250$ mK. The traces were measured using different contact pairs. The values of the filling factor $\nu$ are indicated. Resistance values as predicted by the model in figure 4, calculated using the formulae given in table 1, are included as short horizontal lines.
configuration more likely than for a Hall-bar with recessed Ohmic contacts and is probably the reason why this asymmetry was not observed before.

The arrangement considered is depicted in figure 4 for clockwise (CW) channel chirality ($B > 0$) and filling factors in the monolayer ($\nu_M$) and in the bilayer ($\nu_B$) such that $\nu_B > \nu_M$. In such a case, the channels exiting contact 1 and the channels from contact 4 undergo a full equilibration while co-propagating along the upper device edge, and emerge at point A at the same potential. By making use of current conservation relations, it is possible to write down the equation at each node, obtaining

$$V_{4-6} = V_{1-3} = \frac{\nu_B - \nu_M}{2\nu_B - \nu_M} \times V_D$$

for the longitudinal direction, and

$$V_{1-4} = V_{3-6} = \frac{\nu_B}{2\nu_B - \nu_M} \times V_D$$

for the transverse direction. We note that for positive sign of the magnetic field, longitudinal voltages have identical values for opposite sides of the devices, as in the conventional quantum Hall regime. However, their value is considerably larger due to the presence of shunting channels in the bilayer.

For $B < 0$, a counter-clockwise (CCW) propagation is expected and the corresponding values for the longitudinal voltage drops are

$$V_{4-6} = 0, \quad V_{1-3} = \frac{\nu_B - \nu_M}{2\nu_B - \nu_M} \times V_D$$

while in the transverse direction we obtain

$$V_{1-4} = -\frac{\nu_M}{2\nu_B - \nu_M} \times V_D, \quad V_{3-6} = -\frac{\nu_B}{2\nu_B - \nu_M} \times V_D.$$  

The values of the resistance, calculated using the source-drain current $I_{SD} = \nu_M \times I_D / \left(2\nu_B - \nu_M\right)$, are summarized in table 1 in units of $\hbar/e^2$. In the following we shall focus on the case $|B| > 5$ T, in which $\nu_M = 2$ channels propagate along the device, as inferred from the high-field values of $R_{3-6}$. By setting $\nu_M = 2$, and using the measured value $R_{1-3} |_{-7T} = 0.393 \times \hbar/e^2$, we obtain the filling factor in the bilayer region $\nu_B = 9.33$. The nearest Hall plateau for bilayer graphene is at $\nu_B = 8$ [46]. We can estimate the variation in our data by considering the difference in $R_{4-6}$ and $R_{1-3}$ at $B = +7T$, which should be identical according to our model (see table 1). We obtain $R_{1-3} |_{+7T} = 0.397 \times \hbar/e^2$ and $R_{4-6} |_{+7T} = 0.346 \times \hbar/e^2$, i.e., a variation in $R$ of 0.051 $\times \hbar/e^2$. This slight difference in the traces measured with different contacts is presumably caused by an inhomogeneity of the charge density and a mixing between resistance components due to geometrical effects. Using the filling factor values $\nu_M = 2$ and $\nu_B = 8$, we calculate a transverse resistance value $R_{1-4} = -0.125 \times \hbar/e^2$, which is roughly consistent with the measured value $R_{1-4} |_{-7T} = -0.064 \times \hbar/e^2$ within the variation. Using the formulae given in table 1, we have calculated all quantum Hall resistance values both for $\nu_M = 2$ and $\nu_B = 8$ as well as for $\nu_M = 6$ and $\nu_B = 24$ and included them as thin horizontal lines in figure 3. The excellent agreement between experiment and model further strengthens our interpretation. In summary, this simple model explains the main features of our data. In particular, it fully accounts for the peculiar asymmetry of the magnetoresistance curves. Our results imply the coexistence of QH states on both monolayer and bilayer graphene grown on the same substrate.

This topic was addressed in a very recent work [40], where an electrostatic model describing the domain of coexistence of QH in both monolayer graphene and bilayer inclusions was proposed. It was stated that QH conditions in mono- and bilayer regions at large carrier density are not expected to be simultaneously present, so that the bilayer inclusions act as dissipative shunts when the monolayer graphene is pinned at a certain filling factor. However, we argue that if monolayer and bilayer regions have different carrier densities, it is possible to have the conditions for dissipationless transport in both graphene domains. A density of $3.4 \times 10^{11} \text{ cm}^{-2}$ in monolayer graphene and $\nu_M = 2$ would require a 4 times higher carrier density in bilayer graphene to reach $\nu_B = 8$ at the same magnetic field $B$, i.e., a carrier density of $1.4 \times 10^{12} \text{ cm}^{-2}$. These values are in excellent agreement with values reported in literature for similar mono- and bilayer samples [47].

### Table 1. Calculated quantum Hall resistances (in units of $\hbar/e^2$) obtained from the model in figure 4.

| $R_{1-3}$ | $R_{4-6}$ | $R_{1-4}$ | $R_{3-6}$ |
|-----------|-----------|-----------|-----------|
| $B > 0$ (CW) | $\frac{\nu_B - \nu_M}{2\nu_B - \nu_M}$ | $\frac{\nu_B - \nu_M}{2\nu_B - \nu_M}$ | $\frac{\nu_B - \nu_M}{2\nu_B - \nu_M}$ |
| $B < 0$ (CCW) | $0$ | $-\frac{1}{\nu_B} - \frac{1}{\nu_M}$ | $\frac{\nu_B}{2\nu_B - \nu_M} \times V_D$ |

4. Conclusion

We observed an asymmetric dependence of the magnetoresistance of graphene in a Hall-bar oriented perpendicularly to the SiC(0001) step edges, which we attribute to the presence of continuous bilayer graphene stripes crossing the device. We propose a quantitative model involving the simultaneous coexistence of QH conditions in the monolayer and bilayer regions, at different filling factors. The transport channels in the bilayer inclusions are responsible for inter-channel scattering, which results in mixing of the edge-channels in the monolayer and deviations from the conventional quantum
Hall effect. These effects should be carefully considered in future resistance standard Hall-bar devices based on epitaxial graphene.

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