Quantum corrections to the conductivity of disordered graphene on SiC (000\overline{1}): weak localization and current-bias dependent electron–electron interactions

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Abstract
The properties of epitaxial graphene grown via thermal decomposition of silicon carbide are extremely sensitive to annealing conditions. Here we show how the surface morphologies resulting from a range of UHV growth protocols affect the electron scattering rates associated with various quantum corrections to the conductivity. Detailed analysis of magnetotransport data provides insight into the degree of disorder via fits to weak localization and weak antilocalization models, while additional fitting is used to identify more subtle contributions from electron–electron (e–e) interaction effects. This second contribution is found to be current-bias dependent, and is seen only for more disordered samples, which is attributed to the shorter mean free path in these materials.

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1. Introduction

The extraordinary linear band structure of graphene was calculated as long ago as 1947 by Wallace [1]. Nevertheless, it was many years before the material was isolated in freestanding form by micromechanical cleavage and measured [2], whereupon graphene flakes were quickly shown to possess an exceptional and fascinating array of electronic properties [3]. These include electric field effects [4], unusual quantum Hall effects [5, 6], extremely high mobility [7], Klein tunneling [8, 9] and a remarkably long spin diffusion length [10].

Nevertheless, small, randomly placed flakes are not suitable for a manufacturable technology, and wafer-scale graphene is required for applications [11]. Growth of graphene on the surface of SiC wafers can be achieved by simply heating the substrate under appropriate conditions [12, 13]; the surface characteristics of the graphitic material grown are a consequence of both the substrate surface prior to graphitization [14], and the details of the annealing conditions used [15, 16]. Epitaxial growth from an insulating substrate is advantageous for transport measurements, and also as a platform for incorporating graphene into technological applications [17]; with no external source of carbon required, the available variables through which to influence the growth dynamics are simply temperature, pressure and duration of anneal.

Here we use low-temperature magnetoconductance (MC) measurements to determine the electron scattering mechanisms for samples annealed under a variety of conditions. The use of MC data as an indication of the degree of disorder in carbon films has a long history, and predates transport measurements on graphene [18, 19]. Detailed analysis of magnetotransport and surface measurements are complementary techniques for the investigation of growth dynamics with a view to optimizing growth conditions. Here we focus on quantum interference corrections to the conductance of our graphitized SiC wafers, and their dependence on magnetic field. Such MC measurements allow time-of-flight measurements of the carriers [20], and have been studied widely in graphene, both in the form of weak localization (WL) and antilocalization, as well as e–e interactions [21–28]. Data for the latter are more sparse in the literature. As well as giving rise to quantum corrections to the conductivity, e–e interactions can renormalize the Dirac spectrum [29, 30], and give rise to the fractional quantum Hall effect [31, 32].

Our graphene layers were analyzed using low-energy electron microscopy (LEEM) [15, 33, 34], with lateral grain size found to increase with anneal temperature. The MC data are analyzed by fitting to conventional two-dimensional WL expressions, and to graphene-specific WL theory [35]; the electronic properties are seen to evolve as the graphene becomes more ordered, with fewer grain boundaries. For highly disordered samples with ~30 nm grain size, we report a contribution to MC from e–e interaction effects [18], which has the remarkable feature that its strength depends on the current-bias applied to the sample.

2. Sample preparation and surface characterization

The samples discussed here are graphene grown on the (000\bar{1}) face of 4H SiC in ultrahigh vacuum (UHV) (base pressure $10^{-10}$ mbar) using radiative heating. Annealing at 1300 $^\circ$C for 60 min resulted in surface graphitization that is continuous over the entire substrate; higher temperatures require shorter durations, and resulted in different surface morphologies. These annealing conditions result in low-energy electron diffraction patterns (not shown) consisting of substrate spots (a first indication of graphene thickness being only a few layers) combined with
Figure 1. Sample characterization. Panels (a) and (b) show LEEM images of two samples, annealed as indicated. The field of view in each case is of 10 µm diameter. In panel (b) cyan indicates a monolayer, blue a bilayer and red a trilayer of graphene on the surface. (c) Raman spectra for the two samples with spectra normalized to the 1580 cm$^{-1}$ $G$ peak intensity and offset for clarity. (The image shown in panel (a) was previously published in [15].)

a dashed ring structure from the graphene (due to rotational disorder, with preferred orientation of graphene layers) [36]. Samples were also characterized using Raman micro-spectroscopy and LEEM, images from which provide a succinct summary of the surfaces resulting from different annealing conditions.

The temperatures used in pioneering work on epitaxial graphene (∼1250°C) were influenced by those known to cause (at the time unwanted) graphitization of SiC during studies in the 1970s [37]; if the issues of carbon supply from SiC substrate and bond formation are considered independently, the literature for nanographitic material would suggest a choice of temperature (based on sp$^2$ bond formation alone) higher than 1600°C [38], and anneal temperatures used in work on epitaxial growth have converged to these values [34]. We have previously reported on the types of graphene layer that are obtained for various annealing conditions in the 1300–1400°C range [15], here we recap the main features for some of the samples in which we have now studied the quantum corrections to the conductivity.

Anneals for long durations are required at 1300°C to form a complete graphitized surface coverage, which comprises clusters of very small grains of a few tens of nm lateral size (as seen in the LEEM image displayed in figure 1(a) for the outcome of 60 min anneal at that temperature), whilst annealing at 1300°C and subsequently at 1400°C causes individual grains (30 nm for a 1300°C anneal) to coalesce into larger single grains [15]. Graphene layer
thicknesses can be found using the reflected intensity ($I$) of the material, measured in the LEEM apparatus while varying the energy of the incident electrons ($V$) [33, 34]. This technique was used to produce the image seen in figure 1(b), where the $I(V)$ is processed for each pixel in the set of LEEM micrographs taken, and a color assigned based on the number of layers present, enabling both lateral size and thickness to be represented. The image indicates growth of just over a monolayer for a 1400 °C, 30 min anneal, with some of the monolayer areas extending over more than 100 nm. For the more disordered sample shown in figure 1(a) it is necessary to average over several pixels, due to the grain size and resolution of the apparatus; the average number of graphene layers from LEEM $I(V)$ is two. Approximate grain sizes determined for $D$ to $G$ peak ratios by Raman spectroscopy [39] (spectra are shown in figure 1(c)) are consistent with the lateral sizes seen in the LEEM images, with disordered samples yielding grain sizes of 20 nm and more ordered samples values of over 40 nm. The irregular shape of the regions means that there is a large contribution from edges; it is also possible that there are domains of the same thickness but different orientation [40].

3. Magnetotransport measurements and analysis

Sensitivity to annealing conditions, seen in the increased lateral grain size achieved using higher temperatures, is also evident in previous transport measurements, where we observed a crossover from a variable range hopping to WL-like behavior in the temperature dependence at zero field [15]. The electronic properties of pristine graphene prepared via exfoliation are routinely probed using MC measurements [41–43] and this technique can also give insight into the evolution of electronic coherence in these samples as structural disorder decreases due to improvements made to annealing protocols.

All data presented here are (unless otherwise stated) dc measurements of the conductance $G$ of sheet material that were carried out in a constant-flow cryostat with a temperature range from 300 to 1.4 K, using a four-probe geometry with a voltage–probe separation of 5 μm and a bias current of 300 nA, made in the same manner as in our previous report [15]. The current direction was switched, and each data point is the mean of five repeated measurements. This simple measurement geometry precludes carrier density measurements, although Hall measurements on a related sample showing weaker disorder (annealed at 1450 °C for 20 min) gave a carrier density of $\sim 10^{12}$ cm$^{-2}$, typical for epitaxial graphene. Current–voltage ($I$–$V$) characteristic curves are shown in figure 2 for a selection of samples, showing the curves becoming steeper and more Ohmic in our measured range as the material becomes more ordered through hotter and longer anneals.

3.1. Disorder

Figure 3 shows data from low-temperature MC measurements (with the MC defined as $(G(B) - G(0))/G(0) = \Delta G/G$), with fields to ±8 T applied perpendicular to the plane of the sample for three samples, representative of all those measured to date. They show an evolution through several regimes, distinguished by which of several scattering mechanisms predominates. Samples annealed at 1300 °C show broad positive MC out to 8 T, indicative of a WL contribution with a small coherence length $l_c$. (WL effects have previously been measured to fields as high as a few teslas [44–46], or even a few tens of teslas [47, 48], in materials with...
Figure 2. Current–voltage characteristics measured at 1.4 K for samples annealed under various conditions, displayed as (anneal temperature in °C, duration in minutes).

Figure 3. MC measured at 1.4 K for samples annealed under various conditions, displayed as (anneal temperature in °C, duration in minutes). Greater coherence is indicated by a narrowed dip, while for situations where $\tau_i > \tau_\phi$ the gradient is negative at higher fields. Fits to the data using the expressions discussed in the main text are shown as solid cyan lines.

sufficiently strong disorder.) The measured resistance is $\sim 10 \, \Omega$ at 1.5 K, and the dependence of resistance on temperature $R(T)$ on cooling from room temperature was previously shown to follow a variable range hopping behavior [15]. Samples prepared with a subsequent anneal at 1400 °C show a sharper dip in the positive MC, corresponding to the coalescence of grains into the larger, faceted domains (as reported in [15]) and hence giving rise to a longer dephasing length $l_\phi$. A single stage anneal at 1400 °C results in MC which is positive at low fields, but which becomes negative at higher fields: an indication of a weak anti-localization (WAL) contribution being able to manifest itself due to fewer crystallite boundaries [49]. For both
samples annealed at 1400 °C the resistance is lower (around 1 kΩ), and cooling \( R(T) \) data obey a WL-like behavior [15]. This indicates that the Fermi wavevector-mean free path product \( k_F l \) that is greater than unity, as a result of fewer scattering events, associated with the greater structural coherence.

When the diffusion constant \( D \) of the material is known (for instance if the samples are gated, or measurements are on devices with Hall geometry defined via lithography), values can be directly assigned to the scattering rates \( 1/\tau \). This is not possible for the measurements of sheet material presented here, and comparisons instead use representative magnetic fields \( B_n \), which are proportional to the various scattering rates. In the work of Wang and Santiago-Avilés [18], the scattering due to inelastic and elastic collisions are represented by \( B_1 \) and \( B_2 \), respectively, in the expression for the MC

\[
\frac{\Delta G}{G} = \eta(T) \left[ \Psi \left( \frac{1}{2} + \frac{B_2}{B} \right) - \Psi \left( \frac{1}{2} + \frac{B_1}{B} \right) - \ln \frac{B_2}{B_1} \right], \tag{1}
\]

where \( \Psi \) is the digamma function and a prefactor \( \eta \) is introduced in order to scale a fit of this equation to the MC data. Once the characteristic fields \( B_n \) are known from the fit it is straightforward to determine the relevant scattering lengths using

\[
l_n = \sqrt{\frac{\hbar}{4eB_n}}, \tag{2}
\]

but determination of the scattering lifetimes requires knowledge of the diffusion constant \( D \) in order to employ the relationship

\[
\tau_n = \frac{\hbar}{4eDB_n}. \tag{3}
\]

When intervalley scattering becomes less significant so that electrons maintain their valley index during scattering events, then WAL takes place [35]. In this case, the model is cast in terms of three scattering rates, those for intervalley scattering, \( 1/\tau_i \), scattering from atomically sharp defects, \( 1/\tau_\phi \), and scattering associated with trigonal warping, \( 1/\tau_* \). It is possible to define a lengthscale and characteristic field associated with each of these, as above, through the relationships given by equations (2) and (3). In this case the expression for the MC becomes [35]

\[
\frac{\Delta G}{G} = -\frac{e^2}{\pi \hbar} \left[ F \left( \frac{B}{B_\phi} \right) - F \left( \frac{B}{B_\phi + 2B_1} \right) - 2F \left( \frac{B}{B_\phi + B_*} \right) \right], \tag{4}
\]

where

\[
F(z) = \ln z + \Psi \left( \frac{1}{2} + \frac{1}{z} \right). \tag{5}
\]

The data in figure 3 can be best fitted to different models. The most disordered sample (1300 °C anneal for 60 min) has been fitted to a standard WL model (as used by Wu et al [22], effectively equation (1) with \( \eta = 1 \)). A scaled version of this fit was used for the data from the two-stage anneal sample (equation (1)). Meanwhile, a full weak antilocalization fit (equation (4)) was required for the MC with a negative component seen in the sample annealed at 1400 °C anneal for 30 min. These three regimes agree qualitatively with the literature [18, 22, 24], and the values for \( l_\phi \) obtained from the fits at 1.4 K are 23, 59 and 106 nm, in good agreement with feature sizes from the surface data discussed above.
Figure 4. Trend of characteristic fields $B$ ($\propto$ scattering rate) with temperature for various samples. The data points represented with solid symbols are the values of $B_\phi$ arise from fits to equation (4) and are plotted on the left hand ordinate axis. The data points represented with open symbols are values of the field $B_2$ arising from fits to equation (6) and correspond to the right hand ordinate axis. The annealing conditions of the various samples are indicated as ($^\circ$C, minutes), and the error bars are based upon two measurements of sample (1300,60) made five months apart. Lines are guides to the eye.

3.2. Temperature dependence

Fits to MC data taken at various temperatures $T$ using equation (4) were used to extract values for $B_\phi$ as a function of $T$, which are shown as solid symbols in figure 4. As might be expected, the scattering rates are lower for samples which show structural coherence on a longer length-scale using surface measurement techniques (i.e. those annealed at higher temperatures). For the more ordered samples there is also the expected linear dependence of dephasing rate on temperature, as reported previously by Tikhonenko et al. [24].

There are, however, two samples that show clear exceptions to this expected behavior. Below $\sim$25 K the scattering rate ($\propto B_\phi$) increases and then diverges at very low temperatures, a situation which is clearly unphysical. These samples were annealed under UHV conditions, at 1300°C for 45 and 60 min, respectively, i.e. under the same conditions which produced the sample characterized in figure 1(a), and which were previously shown to be in a variable range hopping regime [15]. These are thus much more highly disordered than the other samples studied here. It is evident that equation (4) does not capture all the relevant physics for these two samples, and further terms describing the MC need to be introduced.

3.3. Electron–electron interaction

A set of ac measurements at 1.4 K on one of these heavily disordered samples using considerably higher current biases provides an explanation for this discrepancy in terms of e–e interactions. Representative MC plots from this set of data are shown in figure 5(a). At low bias the MC shape is conventional, and can be fit using the WL expression with very high scattering rates, while at higher current bias the MC shows the same overall negative shape, indicative of a WL regime, but has an additional non-monotonic feature at low fields, which is more prominent for
Figure 5. Current-bias dependent data. (a) MC data taken at 1.4 K (crosses), with fits to basic WL theory (red) and WL theory with e–e interaction included (light blue) for sample (1300,45) measured at ac bias of (from top) 1, 3, 5 and 10 µA. At 1 µA the two fits both overlay the data, corresponding to a low value for $A_e$ in the fit expression. For clarity, curves are offset and only every fifth data point is plotted, in order that the fitted curve can be seen. The fit was to the full data set. (b) $I$–$V$ characteristic of this sample at 1.4 K. Note that the sample was current-biased, although $I$ is plotted on the ordinate. The data as a log–log plot, revealing power law behavior. Circles represent forward bias data, squares reverse bias. The same data are shown in the inset on linear–linear axes.

higher current bias. This behavior can arise through strong spin–orbit scattering [50], but this mechanism can be discounted for carbon-based samples, since no heavy atoms are present.

Fitting the data (shown as crosses in figure 5(a)) to equation (1) results in the red curves, which do not reproduce the non-monotonic low-field feature seen in the data for bias currents of 3 µA or greater. The data are very reminiscent of those measured by Wang and Santiago-Avilés in highly disordered electrospun carbon nanofibers, both in overall functional form and also the experimental field scales [18]. In order to fit the full range of our data, we modified equation (1) following the precedent set in that work, by including a term describing the effects of e–e interactions. The MC is now written as

$$\frac{\Delta G}{G} = \eta(T) \left[ \Psi \left( \frac{1}{2} + \frac{B_2}{B} \right) - \frac{1}{2} + \frac{B_1}{B} \right] - \ln \left( \frac{B_2}{B_1} \right) - A_e \frac{B^2}{B^2 + B^2_e},$$

(6)
where $B_e$ is a characteristic field for spin alignment, and $A_e$ is a prefactor for the contribution arising from e–e effects. The results of this fit for each data set are plotted in figure 5(a), and show that fitting using equation (6) results in a very good agreement with the data, with the remarkable feature that the e–e interaction term is current bias dependent.

A possible explanation for this arises when the $I$–$V$ characteristic of this sample is considered, which is shown in the inset of figure 5(b), measured at the same temperature of 1.4 K. It is highly non-Ohmic, and is well described by a power law $I \propto V^\gamma$, with the exponent $\gamma = 1.599 \pm 0.005$ when determined from fitting the slope of a log $I$–log$V$ plot (see figure 5(b)). Power laws of this type are expected for space-charge limited currents, where $\gamma - 1 = E_C/kT$ [51]. In that model, the energy scale $E_C$ is that on which charge traps are distributed below the bottom of the conduction band. Our fit yields $E_C \approx 72 \mu$eV, this energy scale being consistent with the small size of the gaps in bilayer graphene. The presence of space charges suggests the intriguing possibility of enhanced e–e interactions for increased charge density in the disordered graphene layer.

The full expression of equation (6) can then be used to fit the 300 nA bias MC data at various temperatures for the two samples in figure 4 where $B_e$ deviates from the expected linear dependence on $T$. The results are shown using open symbols in figure 4. The temperatures at which the additional term becomes significant (indicated by values of $A_e$) match those at which the values for $B_e$ extracted using the WAL expression diverge from the expected trend. Although only one sample shows the low-field feature seen in figure 5, the anomalous temperature dependence of the scattering rate seen for both disordered samples in figure 4 can be corrected to the expected quasilinear dependence when equation (6) is used to fit the data. This is in agreement with contributions from the e–e interaction reported elsewhere [25]. It is important to note that these changes are not due to Joule heating by the higher current density: a purely WL-like MC signal was observed when $T$ was deliberately raised. Thus it is clear they must arise from some non-equilibrium effect.

Following the identification of e–e interaction from MC data, the $R(T)$ data we previously presented in reference [15] can be revisited. In a variable range hopping regime in the absence of the e–e interaction, $\ln R \sim T^{-1/(n+1)}$, where $n$ is the dimensionality of the sample. In systems where the e–e interaction is present, the exponent is always $-\frac{1}{2}$, irrespective of dimensionality [52]. While plotting using an exponent of $-\frac{1}{3}$ produces a close to linear plot, fitting the data using the exponent as a free parameter yields values of between $-\frac{1}{2}$ and $-\frac{1}{3}$, providing further indication of the presence of e–e interactions in these samples.

4. Conclusions

The previously observed evolution of structural coherence of epitaxial graphene on SiC [15] is shown here to result in distinct behavior of the MC, due to changes in the type and frequency of scattering events experienced by electrons. While MC data measured in more ordered samples can be fitted using weak (anti)localization expressions, and coherence lengths extracted which agree with the structural coherence from surface measurements, in highly disordered samples there is a significant contribution from the e–e interaction; when this is included in the expression used the MC data can again be fit accurately and the linear dependence of scattering rate on temperature is recovered. The size of the MC contribution due to the e–e interaction has the unusual feature that is dependent on the current-bias used.
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