

# Ultrathin epitaxial graphite: 2D electron gas properties and a route toward graphene-based nanoelectronics.

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We have produced ultrathin epitaxial graphite films which show remarkable 2D electron gas (2DEG) behavior. The films, composed of typically 3 graphene sheets, were grown by thermal decomposition on the (0001) surface of 6H-SiC, and characterized by surface-science techniques. The low-temperature conductance spans a range of localization regimes according to the structural state (square resistance 1.5 k $\Omega$  to 225 k $\Omega$  at 4 K, with positive magnetoconductance). Low resistance samples show characteristics of weak-localization in two dimensions, from which we estimate elastic and inelastic mean free paths. At low field, the Hall resistance is linear up to 4.5 T, which is well-explained by *n*-type carriers of density 10<sup>12</sup> cm<sup>-2</sup> per graphene sheet. The most highly-ordered sample exhibits Shubnikov - de Haas oscillations which correspond to nonlinearities observed in the Hall resistance, indicating a potential new quantum Hall system. We show that the high-mobility films can be patterned via conventional lithographic techniques, and we demonstrate modulation of the film conductance using a top-gate electrode. These key elements suggest electronic device applications based on nano-patterned epitaxial graphene (NPEG), with the potential for large-scale integration.

The exceptional electronic transport properties of low-dimensional graphitic structures have been amply demonstrated in carbon nanotubes and nanotube-based transistors. Ballistic transport has been observed up to room temperature [1, 2, 3], and quantum interference effects at cryogenic temperatures [4, 5, 6]. Simple nanotube transistors [7, 8], and interconnected logic gates [9] have been demonstrated, which rely on the ability to control the nanotube conductance via an electrostatic gate. The basic transport parameters of these devices are so compelling that nanotubes are considered to be a candidate material system to eventually supplant silicon in many electronic devices.

An under-appreciated fact is that most electronic properties of carbon nanotubes are shared by other low-dimensional graphitic structures. For example, planar nanoscopic graphene ribbons (i.e. ribbons of a single sheet of graphite) have been studied theoretically [10, 11], and they exhibit properties that are similar to nanotubes. Graphene ribbons with either metallic or semiconducting electronic structure are possible, depending on the crystallographic direction of the ribbon axis [10]. Thus, if suitable methods were developed to support and align graphene sheets, it would be possible to combine the advantages of nanotube-like electronic properties with high-resolution planar lithography to achieve large-scale integration of ballistic devices. An essential difference between nanotubes and planar graphene ribbons is the presence of dangling bonds at the edges. Normally these would be hydrogen-terminated, with little influence on the valence electronic properties. However, edge atoms could be passivated with donor or acceptor molecules, thus tuning the electronic properties without affecting the graphitic backbone of the device.

This Letter presents recent results [12] that show the two-dimensional nature of electrical transport in ultrathin graphite (multilayered graphene) grown epitaxially on SiC(0001). 6H-SiC is a large bandgap (3 eV) semiconductor, which provides

an insulating substrate at temperatures below 50 K for the *n*-type (nitrogen) doping employed here. We use magnetoconductance measurements and the physics of weak-localization to determine transport parameters of the graphite 2D electron gas (2DEG), and we show that the character of the magnetotransport/localization spans a wide range of behaviors, depending on the amount of disorder in the film or substrate. Quantum oscillations in the magnetoconductance and in the Hall resistance are found for the most ordered sample. The character of these features suggests that the quantum Hall effect could be observed at lower temperatures, higher fields, or in ultrathin graphite films of only slightly higher mobility. To our knowledge, these are the first transport measurements on oriented and patterned graphite films of only a few monolayers thickness (hence “graphene” films), although related transport experiments have been done on thicker (65–100 graphene layers) free-standing graphite microdisks, which were nano-patterned by focused-ion-beam (FIB) lithography [13].

Given the large mean free paths measured in high-quality graphites [14], the unusual electronic dispersion of graphene, and the fact that the carriers lie near an air-exposed surface, this unique 2DEG system holds great scientific potential. Furthermore, with sufficiently high-quality material, ballistic and coherent devices analogous to nanotube designs [15] would be possible. This goal requires that the epitaxial graphene can survive the processing necessary for creation of submicron ribbons [10, 11], and that the 2DEG can be gated electrostatically. Below we also demonstrate these critical elements for the realization of electronic devices based on nano-patterned epitaxial graphene (NPEG).

Ultrathin epitaxial graphite films were produced on the Si-terminated (0001) face of single-crystal 6H-SiC by thermal desorption of Si [16, 17, 18, 19]. After surface preparation by oxidation [20] or H<sub>2</sub> etching [21], samples were heated by electron bombardment in ultrahigh vacuum (UHV; base pressure 1  $\times$  10<sup>-10</sup> Torr) to  $\sim$ 1000  $^{\circ}$ C in order to remove the ox-

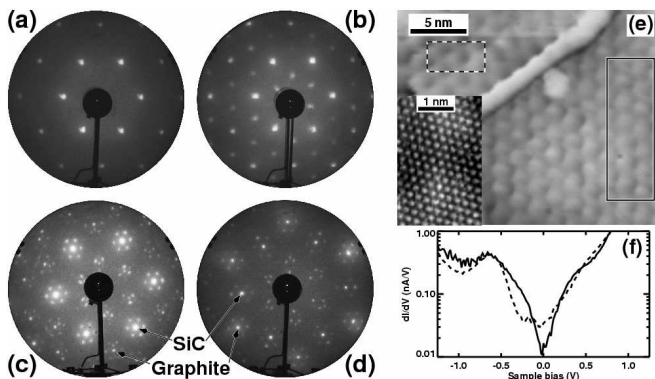


FIG. 1: (a)-(d) LEED patterns from Graphite/SiC(0001). The sample was heated several times to successively higher temperatures. (a) 1050 °C for 10 min. Immediately after oxide removal, showing SiC  $1 \times 1$  pattern at 177 eV. AES C:Si ratio 1:2. (b) 1100 °C, 3 min. The  $\sqrt{3} \times \sqrt{3}$  reconstruction is seen at 171 eV. AES ratio 1:1.9. (c) 1250 °C, 20 min. 109 eV pattern showing diffracted beams from the  $6\sqrt{3} \times 6\sqrt{3}$  unit cell. Examples of first-order SiC and graphite spots are marked. Note the surrounding hexagons of “ $6 \times 6$ ” spots. AES C:Si ratio 2:1 ( $\sim 1$  ML graphite). (d) 1400 °C, 8 min. 98 eV LEED pattern. AES ratio 7.5:1 ( $\sim 2.5$  ML graphite). (e) STM image of a surface region of the sample described in Fig. 1d. Inset: Atomically-resolved region (different sample, similar preparation). (f):  $dI/dV$  spectra (log scale) acquired from the regions marked with corresponding line types in the image at top. The solid line is an average of 396 spectra at different positions, the dashed line an average of 105. With a few “glitchy” exceptions, individual spectra in each region showed negligible variation from the average  $dI/dV$  shown.

ide (some samples were oxidized/de-oxidized several times to improve the surface quality). Scanning force microscopy images showed that the best initial surface quality was obtained with  $H_2$  etching (sample A). After verifying by Auger electron spectroscopy (AES) that the oxide was removed, samples were heated to temperatures ranging from 1250 °C to 1450 °C for 1–20 min. Under these conditions, thin graphite layers are formed [16, 17, 18], with the layer thickness determined predominantly by the temperature. Multilayered-graphene film thicknesses were estimated by modeling the ratio of measured intensities in the Si 92-eV and C 271-eV Auger peaks (3 keV incident energy) [22, 23, 24].

Figures 1 (a)–(d) show low energy electron diffraction patterns (LEED) at different stages during the growth of a 2.5-monolayer (ML) graphite film grown in-situ. Figure 1e displays an STM image from the sample obtained after stage (d). The image reveals a distinct  $6 \times 6$  corrugation of the overlayer [25] and a raised region along a step on the surface. This modulation has been previously attributed to variations of the interlayer interaction arising from Moiré coincidences between the graphite and SiC lattices within a fundamental  $6\sqrt{3} \times 6\sqrt{3}$  surface unit cell [16, 25, 26, 27]. LEED confirms that the graphene sheets register epitaxially with the underlying SiC, as shown in Figs. 1c and 1d. The mean height difference between terraces (0.25 nm), indicates that the step in Fig. 1e is a bilayer step in the SiC substrate. Terrace sizes

Sample	C:Si	Thickness	$R_{4K}$	Mobility
A	10	3 ML	1.5 k $\Omega$	1100 cm <sup>2</sup> /Vs
B	$\infty$	> 5	2.2	
C	9	3	22	
D	10	3	33	15
E	9	3	225	
F	7	2.5		

TABLE I: Sample properties. Ratio of intensities in the C(271 eV) and Si(92 eV) AES peaks, calculated thickness in graphene monolayers, square resistance at 4 K, and mobility (where measured).

(corresponding to a single  $6 \times 6$  domain) are found by STM to be up to several hundred nanometers in extent. Preliminary high-resolution LEED studies indicate that the graphene layers are strained in-plane by 0.3–0.5%, with a mean structural coherence length of greater than 20 nm.

Also shown in Fig. 1f are derivative tunneling spectra ( $dI/dV$  vs.  $V$ ) acquired within the respective boxed regions of the image. The  $dI/dV$  spectrum obtained from the lower terrace (solid line) is consistent with that of a zero-gap semiconductor, as found typically for bulk graphite. On the upper terrace, the  $6 \times 6$  domain images somewhat differently, and the  $dI/dV$  spectrum (dashed line) displays a region of constant, finite conductance around the Fermi energy (zero bias). Spectral shapes are very uniform within each  $6 \times 6$  domain. The  $dI/dV$  curves show that the electronic properties of the film are not entirely homogeneous. This may relate to differing lateral registry (i.e. not orientational) of the graphite on the SiC substrate, or electron confinement within  $6 \times 6$  domains.  $dI/dV$  spectra acquired over the buckled region at the step edge are nearly identical to those found on the upper terrace, suggesting that the graphite layer remains continuous over the step.

DC and low-frequency AC conductance measurements were made for temperatures  $T = 0.3$ –50]K, and for magnetic fields  $H$  from 0–8 T on graphite films with thicknesses of typically 3 graphene sheets (see Table I). For Hall-effect measurements, samples were defined using standard optical lithography (photoresist coating, plasma etching, photoresist removal via solvents). Four contacts were painted with silver paste directly on the surface or on evaporated Pd-Au pads on mm-size samples. For samples B, C and E, the voltage probe distance  $d_V$  is 2 mm. For the Hall bar samples A and D  $d_V = 600$  and  $300 \mu\text{m}$  respectively (see photo inset, Fig. 2a). Reported values below are the square conductance  $G$ .

The 2D nature of electrical transport in the film is vividly demonstrated in Fig. 2a by the large anisotropy in the magnetoconductance [ $MC = G(H)$ ]: For  $\mathbf{H}$  perpendicular to the graphene plane the differential magnetoconductance ( $dMC = dG/dH$ ) is large and positive ( $3.0 \mu\text{S/T}$  at 4 K), whereas there is essentially no response when  $\mathbf{H}$  lies in the plane (in-plane MC was measured with  $\mathbf{H}$  transverse to the current direction). This anisotropy is found in all of our samples, and indicates

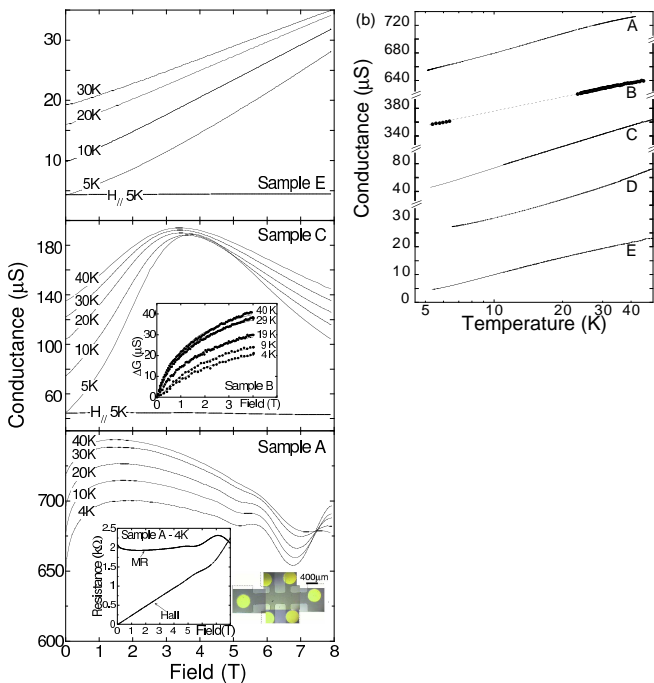


FIG. 2: (a) Magnetoconductance  $G(H)$  for samples E, C and A in perpendicular field for temperatures as indicated. Also shown for samples E and C, the in-plane magnetoconductance at 5 K. Note the strong anisotropy between the perpendicular and in-plane field configuration. In sample C, the conductance goes through a maximum at about the same value of field and conductance for all  $T$ . In sample A Shubnikov-de Haas oscillations develop at high magnetic field. Center-panel inset :  $G(H) - G(H = 0)$  for sample B. Solid curve are fits to the weak localization theory with no spin-orbit, yielding elastic scattering length  $\ell_e \approx 15$  nm and inelastic scattering length  $\ell_i(4K) \approx 100$  nm. Lower-panel inset : Hall resistance  $R_{xy} = V_{xy}/i$  and magnetoresistance  $R_{xx}$  as a function of magnetic field at 4K for sample A. Below 4.5 T,  $R_{xy}$  is remarkably linear, indicative of  $n$ -type carriers. At high magnetic field, the oscillations coincide with the oscillations in the magnetoresistance. (b) Square conductance as a function of temperature for samples of various conductance (samples A, B, C, D and E from top to bottom). Note that the conductance scale is  $\times 4$  for samples D and E, compared to the scale for sample A-C. Samples A, B and C show a  $\ln T$  form in this decade of temperature, characteristic of 2D weak localization.

that the motion of charge carriers is confined to the graphene planes. The observed positive dMC is in contrast to bulk graphite [28], which shows negative dMC for well-ordered single-crystals, and also a large anisotropy. However, carbon foils fabricated from exfoliated graphite [29] and partially graphitic carbons [30] have positive MCs initially, which become negative at large fields. This behavior is a consequence of disorder-induced localization in the sample.

Figure 2a shows systematic changes in the perpendicular MC for samples of successively larger zero-field conductance (i.e. decreasing disorder). For sample C the initial 4 K slope is larger, at  $30 \mu\text{S}/\text{T}$ , and the MC attains an approximately temperature-independent maximum of  $185 \mu\text{S}$  at  $H \approx 3.5$  T. Sample A shows even more structure. Following

a large initial dMC ( $500 \mu\text{S}/\text{T}$  at 4 K), the MC maximizes near 1.5 T, then decreases, followed by a series of (Shubnikov-de Haas) oscillations. A temperature-independent “fixed point” at  $G = 680 \mu\text{S}$  and  $H = 7.4$  T is also observed.

As a function of temperature, the conductance increases proportional to  $\ln T$  at low  $T$ , as shown in Fig. 2b for samples A–C. This is quite characteristic of a 2D electron gas in the weak localization regime [31, 32]. The least conductive samples (D and E;  $G < e^2/h = 38.8 \mu\text{S}$ ) deviate slightly from the  $\ln T$  dependence, which is indicative of a transition to strong localization [see for instance Ref. 33].

In a 2D system, carriers will localize at low temperature due to constructive quantum interference of time-reversed paths for carriers scattered elastically from static disorder [31]. The interference is reduced (conductance increased) by breaking time-reversal symmetry through the application of a magnetic field, or by increasing temperature. These coherent effects are manifest when the elastic mean free path  $\ell_e$  is smaller than the inelastic mean free path  $\ell_i(T)$ . For  $k_F \ell_e \gg 1$  ( $k_F$  is the Fermi wave vector), the system is in the weak localization regime. Strong localization occurs for smaller  $k_F \ell_e$  [33, 34].

Shown in the center-panel inset of Fig. 2a is the perpendicular MC of sample B for five different temperatures (circles), and fits to the data according to 2D weak-localization theory (lines) [32]. The entire family of MC curves is fit by a single temperature-dependent parameter,  $\ell_i(T)$ . Note that for transport in two dimensions, the mean free paths are obtained from the modeling without knowledge of either the Fermi velocity or the carrier effective mass. For sample B, we find  $\ell_e = 15$  nm and  $\ell_i = 100$  nm at  $T = 4$  K. Weak localization effects are observed over a much smaller range of magnetic field for sample A, but a similar estimate gives  $\ell_e = 20$ –30 nm, and a much larger inelastic mean free path  $\ell_i(4K) \sim 300$  nm. From the carrier density  $n = 10^{12} \text{ cm}^{-2}$  per graphene sheet (see below), we find for sample A  $k_F \ell_e \sim 5$ , in agreement with the weak localization regime.

In two cases we have observed a reversal of the dMC. At the maximum MC for sample C (Fig. 2a), the conductance per graphene sheet (Table I) is  $1.5e^2/h$ , i.e. comparable to the conductance quantum. This behavior, and the large change in MC of this sample (550%) are reminiscent of disordered 2DEGs, which have been explained in terms of a transition from an Anderson insulator to a quantum Hall liquid [35]. The second case is that of sample A, which underwent an improved substrate preparation. For this sample, we also observe an initial maximum in the MC, but at much lower field (1.5 T). Apparently weak-localization dominates the MC behavior of sample A in the low-field region, but the longer scattering paths are dephased by a smaller magnetic field. The subsequent appearance of Shubnikov-de Haas oscillations indicates quantization of the electron energy spectrum, and wave function coherence on a scale comparable to the cyclotron radius ( $\sim 30$  nm at 5 T), which is consistent with the elastic scattering lengths estimated above. The significance of the quasi-temperature-independent fixed point at  $680 \mu\text{S}$  and a field of 7.4 T is not yet fully understood. Note that  $1/H$  at the crossing point is

equal to the mobility obtained from Hall measurements (see below). This correspondence would be an expected consequence of electron-electron interactions under weak localization [33, 36], but the observed quantum oscillations show that at high fields the system is beyond this regime.

The Hall resistance  $R_{xy}$  was measured for samples D and A in the Hall bar configuration (photo inset, Fig. 2a) at a bias current of 100 nA. For sample D at 4 K,  $R_{xy}$  versus  $H$  is linear from 0 to 8 T, with the slope corresponding to a density  $n = 10^{13} \text{ cm}^{-2}$   $n$ -type charge carriers, and a mobility of  $15 \text{ cm}^2/\text{Vs}$ . The Hall voltage is also linear up to 4.5 T in sample A, from which we determine  $n = 3.6 \times 10^{12} \text{ cm}^{-2}$  ( $n$ -type), and an enhanced mobility of  $1100 \text{ cm}^2/\text{Vs}$ . The observation of a linear Hall effect is particularly remarkable, since single-crystal graphite samples display a substantial quadratic component at small fields [29, 37, 38, 39], due to three sub-bands (one electron, two hole). Apparently, our samples cannot be thus described. The carrier densities found here are comparable to those of other 2DEG systems, although the density per graphene sheet ( $\sim 10^{12} \text{ cm}^{-2}$ ) is higher than that found in high-quality graphites [28, 38, 39]. It remains to be determined what effect substrate doping has on the carrier density and mobility in the graphite film.

For the case of sample A, a carrier density can be obtained from the period (in  $1/H$ ) of the Shubnikov-de Haas oscillations. Assuming a circular 2D Fermi surface, we estimate  $n = 10^{12} \text{ cm}^{-2}$  ( $k_F = 2.5 \times 10^6 \text{ cm}^{-1}$ ). The carrier density determined by the Hall effect is very nearly a factor 3 larger, which is the number of graphene layers measured via AES. Clearly, the simplest explanation would be that each graphene sheet supports a 2D electron gas which remains confined within the sheet. This would be consistent with the large anisotropy in conductivity for bulk graphite [28]. The temperature dependences of samples A–C also support this interpretation:  $dG/d(\ln T)$  falls between  $2.5e^2/\pi h$  and  $3.5e^2/\pi h$ , about a factor 3 larger than the predicted weak-localization contribution to the conductance,  $3.5(e^2/\pi h) \ln T$  [32].

Above 4.5 T in sample A, we observe nonlinearities in  $R_{xy}$  vs.  $H$ , as shown in Fig. 2a (lower-panel inset) for  $T = 4 \text{ K}$ . These coincide with the Shubnikov-de Haas oscillations in the magnetoconductance, showing that they have the same origin: Either broadened quantum Hall plateaus, or bulk magneto-quantum oscillations in a metallic system. If the Hall conductance at the location of the local maximum ( $\approx 5.5 \text{ T}$ ) is normalized with respect to the number of sheets, one obtains a conductance  $\approx 4e^2/h$  which suggests a quantum Hall effect (see also predictions in Ref. 40). Experiments at lower temperatures and higher fields will be necessary to verify this conjecture.

At 0.3 K, we also observe a pronounced zero-bias anomaly in the highly resistive sample D. The conductance is found to increase by about a factor 10 as the bias voltage is increased from 0 to 25 mV [41]. For weak electron-electron interactions it can be understood in terms of enhanced scattering of carriers near the Fermi energy: the wavelengths of these carriers are commensurate with the Friedel oscillations surrounding

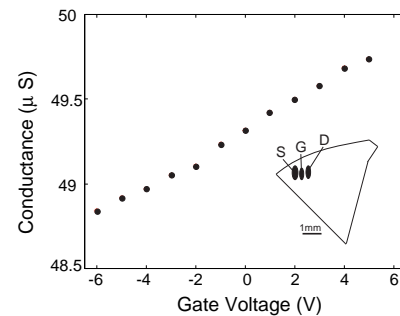


FIG. 3: Conductance as a function of gate voltage for sample F at 4 K. Inset, sketch of the sample showing the contacts and top-gate geometry (S, D, G = source, drain, gate). The top-gate is only partially effective due to the open geometry. Nevertheless, a 2% change in conductance is observed.

impurities, thus they scatter strongly. The coherence is lost at higher bias (higher kinetic energies). Zero-bias anomalies have also been observed in carbon nanotubes [42, 43, 44].

It should be noted that the samples are remarkably stable over time. For instance, measurements in Figs. 2a and 2b were made 4 months earlier than those in the lower-panel inset of Fig. 2a, with no particular storage precautions. The features observed are essentially the same, except for a slight decrease in conductance and carrier density. The results presented above for sample A also show that the multi-sheet epitaxial graphene film survives conventional lithographic processing extraordinarily well.

Finally, as a preliminary demonstration of the device potential of this new 2DEG system, a large-area gated graphite-channel field-effect transistor (FET) structure was assembled. A schematic of the “device” is shown in Fig. 3, as well as the measured source-drain resistance as a function of gate voltage. The top-gate structure consisted of a conductive coating on a 100-nm-thick insulating aluminum oxide layer. The gate covered only a portion of the graphite film between the source and drain electrodes, leaving large ungated leakage paths (see inset, Fig. 3). Consequently, the resistance modulation is rather small (2%), but these results show clearly that multi-sheet epitaxial graphene films can be gated, in distinct contrast to thicker samples [45]. Thus we anticipate that FET-type devices will be possible, particularly when the channel electronic structure is controlled by patterning the graphite into a narrow strip [10, 13, 46].

The experimental results presented here demonstrate the rich scientific promise of ultrathin epitaxial graphite (“graphene”) films. Several points should be appreciated: First, the production method allows graphitic films to be grown epitaxially, as evidenced by LEED and STM measurements. From Auger spectroscopy we further conclude that the layers involve only a few graphene sheets. Remarkably, the films are electrically continuous over several mm. Magnetoconductance measurements clearly reveal 2D electron gas properties, including large anisotropy, high mobility, and 2D localization, in samples patterned by conventional lithogra-

phy. Quantum oscillations observed in both the magneto-conductance and the Hall resistance indicate a potential new quantum Hall system. Finally, control of the 2D electron gas carrier density via electrostatic gating was also demonstrated.

Considered with prior research in graphitic systems, these results provide ample evidence that the graphite/SiC system could provide a platform for a new breed of seamlessly-integrated ballistic-carrier devices based on nano-patterned epitaxial graphene. Such an architecture could have many advantages for nanoelectronics, including potentially coherent devices, energy efficiency, and facile integration with molecular devices.

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