Integer Quantum Hall Effect in Graphite

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Abstract

We present Hall effect measurements on highly oriented pyrolytic graphite that indicate the occurrence of the integer quantum-Hall-effect. The evidence is given by the observation of regular plateau-like structures in the field dependence of the transverse conductivity obtained in van der Pauw configuration. Measurements with the Corbino-disk configuration support this result and indicate that the quasi-linear and non-saturating longitudinal magnetoresistance in graphite is governed by the Hall effect in agreement with a recent theoretical model for disordered semiconductors.

Key words: A. metals, semiconductors D. Galvanomagnetic effects, Quantum Hall effect

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Recent experimental\cite{1,2,3,4,5,6} and theoretical\cite{7,8,9} work demonstrates the occurrence of correlated phenomena in the electron system of graphite, indicating that the classical view of the physics of its magnetotransport properties is not adequate. Besides, the quantum Hall effect (QHE)\cite{1} as well as evidence for Dirac fermions (massless particles due to the linear dispersion relation)\cite{5} have been reported for bulk samples of highly oriented pyrolytic graphite of high quality. Recently published results obtained on bulk graphite\cite{10,11}, a few layers thick graphite\cite{12}, and in graphene (single graphite layer)\cite{13,14} have confirmed both observations and showed that in graphene the QHE is anomalous in that the contribution of the Dirac fermions makes the plateaus to occur at half-integer filling factors. Certainly, the understanding of the electronic properties of graphite is necessary for the development of advanced technology of the graphite-related nanomaterials.

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The experimental work we report here is based partially on data presented in an international meeting [15]. These data clearly demonstrate the quantization of the Hall effect and in particular the existence of an even QHE in graphite, i.e. a transverse voltage that is symmetric on magnetic field reversal. The occurrence of the QHE in bulk graphite indicates that the coupling between graphene layers is much smaller than the one assumed by the Slonczewski-Weiss-McClure theory [16] but perfectly agrees with the results obtained by Haering and Wallace [17]. Also, the experimental results we present here indicate a possible solution to the longstanding problem of the quasi-linear and non-saturating magnetoresistance (MR)[18] providing evidence that the MR at high enough fields is governed by the Hall resistance, in agreement with a recent theoretical model [19].

The occurrence of the QHE is not restricted to perfectly two-dimensional (2D) systems, as measurements in highly anisotropic layered systems [20], organic Bechgaard salts [21], Molybdenum bronzes [22], layered crystals of the type \((\text{Bi}_{0.25}\text{Sb}_{0.75})_2\text{Te}_3\) [23], \((\text{TMSTF})_2\text{AsF}_6\) system [24], as well as in 200-layer quantum-well structures [25] indicate. Nevertheless, it is surprising that for the paradigm of strongly anisotropic systems, graphite, no clear evidence for the QHE has been published till 2003 [1,2,3]. The main obstacle to obtain clear evidence for the QHE in graphite is the sample quality, i.e. the internal short circuits between graphene planes caused by defects and impurities. As a characterization of the sample quality and orientation of the samples one may use the full width at half maximum (FWHM) of the rocking curves. In general we observed that the smaller the FWHM and the distance between voltage electrodes, the clearer are the plateaus in the Hall effect. The samples measured in this work are highly oriented pyrolytic graphite (HOPG) obtained from Advanced Ceramics (AC sample) and from the research institute "Graphite" in Moscow (sample HOPG-3), with FWHM equal to 0.40° and 0.64°, respectively.

Angle dependent magnetoresistance (MR) oscillations as well as a ratio of the order of \(10^4\) between \(c\)-axis and in-plane resistivities in highly oriented graphite samples[2] indicate a much larger anisotropy, a much smaller coupling between the graphene layers and a greater influence of the two-dimensionality of the sample on the transport properties than earlier studies have reported. Among these findings is the magnetic-field induced metal-insulator-transition (MIT), which shows significant similarities to the one discovered in dilute 2D electron systems [26]. As in graphite [1], in some of these 2D systems the MITs have been shown to be connected to quantum-Hall-insulator-transitions at high magnetic fields [27]. After these experimental observations and the first theoretical prediction for the QHE in graphite [28], which was followed by further developed theoretical studies [29,30], the occurrence of quantum-Hall-states in graphite was already expected.
The longitudinal resistance as a function of magnetic field of all samples has been measured with the conventional ac-method, where four electrodes made of silver paint are placed on top of a surface parallel to the graphene layers. To calculate from this measurement the absolute resistivity (longitudinal as well as transverse) it is necessary to know the effective penetration depth of the current \( \lambda \). Independent measurements at room temperature using electrodes at different positions of the same sample reveal that for macroscopic, bulk samples \( \lambda \) is in the range \( 1 \mu m \lesssim \lambda \lesssim 10 \mu m \). With a conventional electrode geometry a good estimate of the resistivity is given by \( \rho \sim b \lambda V / I d \) where \( b \) is the width of the sample, \( I \) the applied electrical current, and \( V \) and \( d \) are the voltage and distance between the inner contacts. However, part of the data presented here were obtained using a van der Pauw configuration consisting of four point-like electrodes (1 . . . 4) placed at the corners of the main sample surface. In the van der Pauw configuration the typical surface between contacts fixed at the corners of a parallelepiped was \( \sim 10 \text{ mm}^2 \).

If we apply a current \( I_{12} \) between contacts 1 and 2 and we measured the voltage \( V_{34} \) between contacts 3 and 4, the longitudinal resistivity is given by the equation \( \rho_{xx} = 4.53 \lambda V_{34} / I_{12} \) [31]. The transverse resistivity is obtained from this arrangement measuring the voltages \( V_{42} \) and \( V_{13} \) with the input currents \( I_{13} \) and \( I_{42} \), respectively, using \( \rho_{xy} = 0.5 \lambda (V_{42}/I_{13} - V_{13}/I_{42}) \) [31]. The longitudinal and transverse conductivities \( \sigma_{xx} \) and \( \sigma_{xy} \) are calculated from the measured resistivities inverting the two dimensional matrix, i.e.

\[
\sigma_{xx} = \frac{\rho_{xx}}{\rho_{xx}^2 + \rho_{xy}^2}, \quad \sigma_{xy} = \frac{\rho_{xy}}{\rho_{xx}^2 + \rho_{xy}^2}.
\]

(1)

The magnetic field was always applied perpendicular to the graphene layers (the main sample surfaces). For all geometries the applied current was of the order of 1 mA, which is in the ohmic regime and for which no heating effects are observed. The resistance was measured with a LR-700 resistance bridge from Linear Research Inc., which works at low ac-frequency.

The essential signature of the QHE is the occurrence of plateaus in the Hall conductivity \( \sigma_{xy}(H) \) or resistivity \( \rho_{xy}(H) \) isotherms. The transverse conductivity value at the position of the apparent first plateau-like feature at \( H \sim 5T/\mu_0 \) is for this sample (HOPG-3) [1,2,3] \( \sigma_{xy}(H \sim 5T/\mu_0, T \leq 2 \text{ K}) \approx 0.27/\lambda \Omega^{-1}\text{m}^{-1} \), where \( \lambda \) is given in meters. Assuming an exponential decay of the current with the distance from the surface and taking into account that the current penetration depth \( \lambda \gg c/2 \), the distance between the graphene layers \( c/2 = 3.35 \text{ Å} \), the contribution of a single graphene layer can be written as

\[
\sigma_{xy}^{\square} = \sigma_{xy} \lambda \left( 1 - \exp \left( -\frac{c}{2\lambda} \right) \right) \approx \sigma_{xy} c / 2.
\]

(2)
If we assume that the first plateau occurs when \( \sigma_{xy} = \sigma_{xy}(c/2) = e^2/h = 3.86 \times 10^{-5} \Omega^{-1} \), using our data we estimate a penetration depth \( \lambda = 2.35 \mu m \), which is within the expected range. Figure 1 shows both conductivities for the HOPG-3 sample obtained from the measured resistivities and calculated using \( \lambda = 2.35 \mu m \). This figure shows that regular plateaus are clearly observable in the \( \sigma_{xy}(H) \) (or in \( \sigma_{xy}(H) \)) measured in the van der Pauw configuration and plotted as a function of the inverse applied field. From the data in Fig.1 we obtain that the transverse conductivity for a single layer at the apparent first plateau agrees within experimental error (±50% due to the uncertainty in the absolute value of \( \lambda \)) with the separation between the plateaus \( \Delta \sigma_{xy} \approx 3 \times 10^{-5} \Omega^{-1} \).

Considering the simplicity of the model and the geometrical errors involved in the determination of the absolute values the quantitative agreement with the expected value for an integer-like QHE seems reasonable. Within experimental error, the value obtained for \( \sigma_{xy} \) depends on sample and also on the electrode arrangement, indicating that the disorder and current distribution may have an influence. From the sign of the Hall signal we conclude that the carriers have the same electronic charge as the electrons.

A vanishing longitudinal resistivity \( \rho_{xx}(H) \) in the vicinity of the fields where the Landau levels are filled is a concomitant of the QHE, which appears in 2D systems at very low temperatures [32]. However, \( \rho_{xx}(H) \) (or \( \sigma_{xx}(H) \)) of graphite does not show any minima or a decreasing behavior with field, see Fig.1. The positive MR of graphite is very large in clear contrast to the relatively small MR in most of the 2D systems showing the QHE. Moreover, it is nearly linear in field without any sign of saturation (see Fig.1). High resolution measurements of the MR of HOPG samples indicate a quasi-linear field dependence at fields as low as \( \sim 10^{-3} T \) with an anomaly in the field exponent at the MIT at \( \mu_0 H \sim 0.1 T \) [33]. Note that the magnetoconductance at high fields, where the Shubnikov - de Haas (SdH) oscillations due to the Landau level quantization occur, shows a striking similarity with the transverse conductivity, see Fig. 1.

The origin for the linear MR (LMR) in graphite as well as in other semimetals is a matter of current discussion [18,19,34]. The simple two band model [35] does not explain the quasi-LMR observed at low and high fields, unless one assumes ad-hoc field dependences for the electron and hole mobility. Abrikosov proposed that the LMR takes place in the Landau level quantization regime and above the field \( H_{QL} \) that pulls carriers into the lowest Landau level. In order to account for the low values of the characteristic field (\( H_{QL} \sim 10^{-3} T \)), the graphite-like energy spectrum with a linear dependence of energy on momentum has been assumed [18,34]. The theory clearly states that LMR should be a generic property of graphene, which is the model system. The evidence we provide for an integer-like QHE casts doubts whether the transport properties
Fig. 1. The components of the conductivity tensor per graphene layer vs. inverse field for the sample HOPG-3 measured in van der Pauw-configuration at 0.1 K (○) and 2 K (●). The conductivities were calculated using Eqs. (1) and (2) and assuming a penetration depth $\lambda = 2.35 \, \mu m$. The absolute values of the conductivities $\sigma_{xx}$ and $\sigma_{xy}$ can be easily obtained dividing the plotted values by $3.35 \times 10^{-10} m$. Smaller (larger) values of the penetration depth $\lambda$ increase (decrease) the absolute values of the conductivities. In the off-diagonal element regular plateaus are seen, indicative of the quantum-Hall-effect. We note that in this configuration the transverse Hall voltage changes sign when the field is reversed.

Experimental evidence based on a large number of transport measurements in different HOPG samples indicates that the internal disorder of a graphite sample influences the field and temperature dependence of the transport properties in such an extent that the QHE can in some cases transform in a linear field of graphite can be appropriately described by this “quantum magnetoresistance” model added to the fact that a quasi-linear MR is observed also at low fields.
dependent Hall effect with minima at specific fields [1]. Moreover, different contact distributions on the same sample show also a small but striking influence on the transport properties. Therefore these samples should be considered as disordered semimetals. Regarding the quasi-linear and non-saturating MR in disordered semiconductors, Parish and Littlewood showed recently that this behavior might be provided internally by the Hall effect [19]. In what follows we demonstrate experimentally that the Hall-like signal can be measured in a graphite sample at large enough fields in a voltage contact configuration where for the homogeneous case this signal is not expected.

The sample for a Corbino-disk experiment has been prepared by means of e-beam lithography using a 200 nm gold layer sputtered on the sample (AC) main surface, as shown in the inset of Fig. 2. In the Corbino disk geometry the diameter of the outer ring was 1.4 mm and 0.1 mm its width as well as the diameter of the electrodes. In this configuration, the current is applied between the inner point-like electrode and four contacts along the outer ring-like electrode to assure homogenous current input. The voltage is measured between any of the other contacts. In a homogenous, isotropic (along the planes) 2D sample, the application of a constant current $I$ would result in a two-dimensional radial current density $j_r = I/2\pi r$ at a point located at distance $r$ from the center, which is deflected by the magnetic field by the Hall-angle $\theta$ with $\tan(\theta) = \sigma_{xy}/\sigma_{xx}$. The result is a current path along logarithmic spirals, consisting of the radial component $j_r$ and an azimuthal (circulating) component given by $|j_\phi| = |j_r\sigma_{xy}/\sigma_{xx}|$, see the inset in Fig. 2. If we neglect any influence of the Hall component, the voltage between any two contacts at the distances $r_1$ and $r_2$ from the center is then given by

$$V = \frac{1}{\sigma_\square} \int_{r_1}^{r_2} j_r \, dr = \frac{I}{2\pi \sigma_\square} \ln \frac{r_2}{r_1}.$$  (3)

Here $\sigma_\square$ is the longitudinal in-plane conductivity. As expected, the voltage vanishes between contacts located at the same distance from the center. The circulating current component is caused by the Lorentz force perpendicular to the electric field and therefore it is a non-dissipative current, which does not contribute to the voltage. The aim of the experiment is to show that when for a given pair of electrodes inside the Corbino disk the “longitudinal”-like signal is minimized (i.e. $r_1 \simeq r_2$, see Eq. (3)), still a voltage is measured and this resembles the Hall signal.

Figure 2 shows the field dependence of the inverse conductivity obtained from voltage measurements between various contact pairs. Two of them (A-C and G-F) provide the longitudinal magnetoconductivity and three pairs at nominally the same distance from center (B-C, E-F and C-D) should show no voltage. In contrast to the expectations for a homogeneous sample, the volt-
Fig. 2. Inverse diagonal component of the conductivity tensor vs. applied field for the AC sample measured in Corbino-configuration at 2 K. The conductance $g \sim \sigma$ is calculated from Eq. (3) assuming a relation of the distances of the voltage contacts $x = r_2/r_1$ which leads to the best scaling onto the curve for the contact pair A-C (see below). The current is applied between the inner electrode and the outer ring; voltage is measured between pairs of the other electrodes. The curves were obtained from the voltage contacts: (+) A-C, $x = 1.5$; (X) G-F, $x = 1.957$; (□) B-C, $x = 1.111$; (O) E-F, $x = 0.915$; (●) C-D, $x = 1.009$. The electrode designations correspond to the ones in the inset. Inset: View from top onto the Corbino-configuration with designations of the electrodes as used in the text. The current path along a logarithmic spiral is schematically shown together with the radial component $j_r$, the azimuthal component $j_\phi$ and the Hall-angle $\theta$.

The first explanation for this observation would be that this is due to a misplacement of the contacts giving rise to a radial distance between them. To check this, the data in Fig. 2 is presented as follows. For the contact pair A-C (see inset in Fig. 2) the conductance $g \sim \sigma$ is calculated from (3) assuming the nominal value for $x = r_2/r_1 = 0.45 \text{ mm}/0.30 \text{ mm} = 1.5$. For the other pairs, $x$ is chosen in such a way, that the curves scale onto the one for the pair A-C.

As can be seen in Fig. 2 the curve for the pair G-F scales very well assuming $x = 1.957$, corresponding to a shift of the inner contact from the nominal one by 70 $\mu$m, caused by the limited accuracy of the preparation (nominal electrode diameter is 100 $\mu$m). The same holds for the pairs B-C and E-F,
Fig. 3. Data obtained from the contact pair C-D in the Corbino-configuration. Top: Curve taken at 2 K. Bottom: Curve taken at 5 K at positive (●) and negative (○) field directions. Note that the voltage does not change sign when the field is reversed.

and therefore we conclude that those signals are mainly determined by the "nominal" longitudinal MR. The pair C-D, however, has the smallest nominal misplacement (x = 1.009), but shows the most significant deviation from the assumable purely longitudinal MR curve A-C. Interestingly, C-D is the contact pair where we observe the clearest plateau-like structures similar to the ones measured in the van der Pauw configuration, as shown in the upper part of Fig. 3. The periodicity of the plateaus agrees very well with that obtained from the van der Pauw configuration. The inverse-field period of the plateaus is ∆(1/μ₀H) ≃ 0.17 T⁻¹. Using the Onsager relation [37] for a 2D electron system ∆(1/μ₀H) = e/πℏn_{2D} we obtain a 2D electron density for our HOPG samples n_{2D} ≃ 2.85 × 10^{15} m⁻². This value is comparable with literature values for the 3D electron density n_{3D} ≃ 3 × 10^{24} m⁻³ ≃ n_{2D}²/c. From this evidence
we conclude therefore, that the measured voltage between the electrodes C-D at large enough fields is not related to the longitudinal MR.

The origin for the unexpected Hall signal at the pair C-D is compatible with the disordered semiconductor model of Ref.[19]. In this model the LMR is governed by the Hall resistance of the current paths. Note that due to the influence of $\rho_{xy}$ in $\rho_{xx}, \sigma_{xy} \sim 1/\rho_{xy}$. However, this signal is independent of the field direction (switching the field direction will also switch the transverse direction of the current within the disordered network). That means that the measured MR at C-D should be field direction independent in case the Hall current paths are perfectly symmetric against field inversion. To check this we performed measurements at the two field directions, i.e. $+z$ and $-z$. The results shown in Fig. 2 and in Fig. 3 indicate that the observed voltage does not change sign and does depend only slightly on the field direction probably due to non-identical current paths for both field directions as expected for a real disordered system. The voltage at the pair C-D at fields below $\sim 0.3$ T indicates that a longitudinal MR signal mixes into the overall signal showing an anomaly at the MIT at $\sim 0.1$ T [33].

We would like to note that an even Hall effect that occurs in inhomogeneous materials is a well known phenomenon. This effect has been observed in the mixed state of strongly inhomogeneous type-II superconductors [38,39,40] as well as in inhomogeneous semiconductors [41,42].

In summary, transport measurements performed in different configurations on HOPG samples provide evidence for an integer-like QHE in agreement with theoretical expectations. Our experiments indicate that the absence of minima in the longitudinal MR (that usually accompany the plateaus in the Hall resistivity) as well as the non-saturating LMR observed in all graphite samples is due to the contribution of a Hall resistance generated by perpendicular current paths in the disordered media. This appears to be the origin for the new phenomenon reported here, namely the even QHE.

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