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## Searching for the Fractional Quantum Hall Effect in Graphite

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Measurements of basal plane longitudinal  $\rho_b(B)$  and Hall  $\rho_H(B)$  resistivities were performed on highly oriented pyrolytic graphite samples in a pulsed magnetic field up to  $B = 50$  T applied perpendicular to graphene planes, and temperatures  $1.5 \text{ K} \leq T \leq 4.2 \text{ K}$ . At  $B > 30$  T and for all studied samples, we observed a sign change in  $\rho_H(B)$  from electron- to holelike. For our best quality sample, the measurements revealed the enhancement in  $\rho_b(B)$  for  $B > 34$  T ( $T = 1.8$  K), presumably associated with the field-driven charge density wave or Wigner crystallization transition. In addition, well-defined plateaus in  $\rho_H(B)$  were detected in the ultraquantum limit revealing possible signatures of the fractional quantum Hall effect in graphite.

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The behavior of matter in a very strong magnetic field ( $B$ ) continuously attracts the interest of physicists working in various fields ranging from astrophysics [1,2] to semiconductors [3]. The field-induced Landau level quantization in (semi)conductors leads to a variety of spectacular phenomena such as, e.g., integer and fractional quantum Hall effects (IQHE and FQHE) in two-dimensional (2D) systems [3]. In three-dimensional (3D) samples, a strong enough field localizes the electron (hole) motion in the plane perpendicular to  $B$ , while the motion along  $B$  remains intact [4]; this can be viewed as the field-induced  $3\text{D} \rightarrow 1\text{D}$  dimensional crossover. The reduced dimensionality in the electron system becomes pronounced for  $B > B_{\text{QL}}$  (QL stands for quantum limit) that pulls all carriers into the lowest Landau level (LLL). In this limit, competing charge density wave (CDW) and superconducting correlations [5], or excitonic [6] instabilities driven by the field are expected. In addition, a field-induced Luttinger liquid state has been proposed [7]. Low carrier density 3D semimetals such as bismuth and graphite have been considered [5–7] as promising materials for the experimental observations of above phenomena. Very recently, 3D FQHE in both bismuth and graphite has been theoretically proposed [8,9], corroborating the experimental results obtained for bismuth [10]. In contrast to bismuth, graphite is extremely anisotropic material with weakly coupled graphene layers, in which exciting physics of one, two, or few layers can be revealed [11]. The present work reports the first experimental results showing that FQHE may occur in graphite, pointing out on its quasi-2D nature.

It has been known for a long time that magnetic field  $B > 20 \text{ T} > B_{\text{QL}} = 7\text{--}8 \text{ T}$ , applied along the hexagonal  $c$

axis, induces in graphite an anomalous high-resistance state (HRS) that can be detected using either basal-plane  $\rho_b(B, T)$  or out-of-plane  $\rho_c(B, T)$  resistivity measurements [12–18]. The boundaries that trace the HRS domain on the  $B$ - $T$  plane [15,17] are in qualitative agreement with theoretical expectations [19] for the Landau-level-quantization-induced normal metal—charge density wave (CDW) as well as the reentrant CDW-normal metal transitions. However, while the CDW is predicted to occur in the direction of magnetic field [19], the experimental results [13,14] indicate the in-plane character of CDW, or formation of 2D Wigner crystal (WC) state(s) [20,21]. Supporting either CDW- or WC-based scenarios, the non-Ohmic electrical transport was measured in HRS [14,18]. Typically, for  $T = 2 \text{ K}$ , the HRS emerges in the field interval  $25 \text{ T} < B < 52 \text{ T}$ , and the HRS does not occur for  $T > 10 \text{ K}$  [17].

So far, all the HRS studies [12–18] were performed on artificially grown Kish or natural single crystalline graphite samples. To the best of our knowledge, no measurements above 28 T [12] were performed for HOPG, and no Hall resistivity  $\rho_H(B)$  measurements above 30 T [16] were made on any type of graphite.

Recent magnetoresistance [22,23] and scanning tunneling spectroscopy (STS) [24] experiments revealed the integer quantum Hall effect (IQHE) in graphite. The IQHE takes place only in strongly anisotropic (quasi-2D) HOPG samples with the room temperature out-of-plane/basal-plane resistivity ratio  $\rho_c/\rho_b > 10^4$ , and mosaicity  $\leq 0.5^\circ$  (FWHM obtained from x-ray rocking curves). Together with the high electron mobility  $\mu \sim 10^6 \text{ cm}^2/\text{Vs}$  [25], this makes HOPG a promising system for the FQHE occurrence.

In the present work, we studied magnetoresistance in HOPG in pulsed magnetic field up to  $B = 50$  T and  $1.5 \text{ K} \leq T \leq 4.2 \text{ K}$ . The measurements were performed in Ohmic regime with 300 ms for the total pulse length in ac configuration, at LNCMPI (Toulouse, France). Additional measurements were made using Janis 9T-magnet He-4 cryostat.

Commercially available HOPG samples ZYA and SPI-3 were measured. The sample parameters are: FWHM =  $0.4^\circ$ ,  $\rho_c/\rho_b = 4 \times 10^4$  ( $\rho_b = 5 \mu\Omega \text{ cm}$  and  $\rho_c = 0.2 \Omega \text{ cm}$ ) for ZYA, and FWHM =  $3.5^\circ$ ,  $\rho_c/\rho_b = 3.8 \times 10^3$  ( $\rho_b = 40 \mu\Omega \text{ cm}$  and  $\rho_c = 0.15 \Omega \text{ cm}$ ) for SPI-3 HOPG samples (the resistivity data were obtained for  $B = 0$  and  $T = 300 \text{ K}$ ). X-ray diffraction ( $\Theta$ - $2\Theta$ ) measurements revealed a characteristic hexagonal graphite structure in the Bernal ( $ABAB\dots$ ) stacking configuration, with no signature for the rhombohedral phase and the following unit cell parameters:  $a = 2.48 \text{ \AA}$  and  $c = 6.71 \text{ \AA}$ .

Here, we report the results obtained on the ZYA sample of dimensions  $l \times w \times t = 2.5 \times 2.5 \times 0.5 \text{ mm}^3$ . The magnetic field was applied parallel to the hexagonal  $c$  axis ( $B \parallel c \parallel t$ ), and  $\rho_b(B)$ ,  $\rho_H(B)$  were recorded using the van der Pauw method, sweeping the field between  $-50$  and  $+50$  T.

From the data presented in Figs. 1(a) and 1(b), one observes that  $\rho_b(B)$  goes through the maximum at  $B_{m1} = 18$  T, develops two local minima at  $B_\alpha = 30$  T and  $B_{\beta1} = 34$  T, and passes through the second maximum at  $B_{m2} \approx$

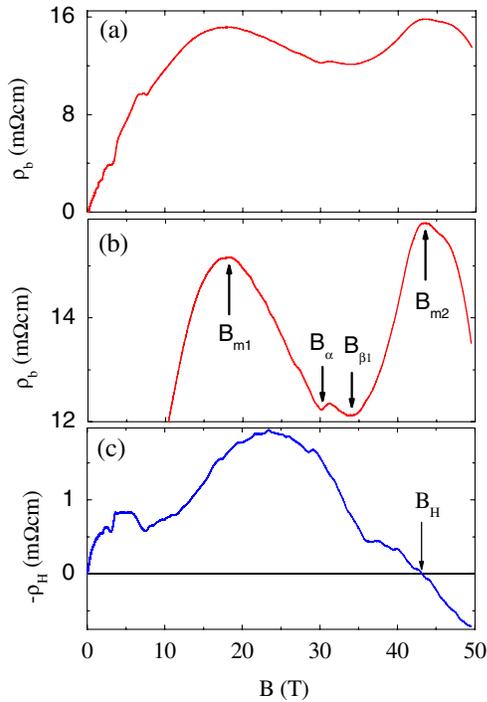


FIG. 1 (color online). (a) Basal-plane resistivity  $\rho_b(B)$  measured up to  $B = 50$  T at  $T = 1.8 \text{ K}$ ; (b) A detailed view of high-field nonmonotonic behavior of  $\rho_b(B)$  discussed in the text; (c) Hall resistivity  $\rho_H(B)$  demonstrating the sign change at  $B_H = 43$  T.

43 T. Thus,  $\rho_b(B)$  represents all characteristic features reported for Kish graphite [17], where, e.g., the resistivity minima at  $B_\alpha = 28$  T and  $B_{\beta1} = 33$  T, attributed to multiple field-induced CDW phases, were measured at  $T = 1.7 \text{ K}$ .

The onset of HRS in Kish graphite is accompanied by a rapid decrease of  $\rho_H(B) \sim \sigma_H(B) = -e(n_e - n_h)/B$  [16], where  $n_e$  and  $n_h$  are majority electron and hole carrier densities, respectively. At low enough temperatures,  $\rho_H(B)$  tends to zero as  $B$  approaches  $\sim 30$  T, suggesting that  $\rho_H(B)$  may change its sign from “minus” to “plus” with a further field increasing [16]. Our results [Fig. 1(c)] give the experimental proof that the sign of  $\rho_H(B)$  changes at  $B_H = 43$  T. The straightforward explanation of this effect would be the carrier density imbalance change from  $n_e > n_h$  ( $B < B_H$ ) to  $n_h > n_e$  ( $B > B_H$ ). This provides us with a new insight on the resistivity drop taking place at  $B > B_{m2}$ . Namely, one assumes that decrease of both  $\rho_b(B)$  and  $-\rho_H(B)$  at  $B > B_{m1}$  originates from the hole density increase [12], whereas the HRS is due to the field-induced Wigner crystallization of electrons or CDW formation. Then, nonmonotonic  $\rho_b(B)$  can be simply understood using the equation for parallel resistors  $\rho_b = \rho_{be}\rho_{bh}/(\rho_{be} + \rho_{bh})$ , [ $\rho_{be}(B)$  and  $\rho_{bh}(B)$  are electron and hole basal-plane resistivities, respectively], without invoking any reentrant transition in the electronic state (noting,  $\rho_b \gg \rho_H$ ).

We also measured the similar sign reversal in  $\rho_H(B)$  at  $B \sim 30$ – $35$  T for two SPI-3 samples. However, due to a poorer quality of those samples, neither negative magnetoresistance nor HRS were detected. Instead,  $\rho_b(B)$  saturates for  $B > 18$  T.

Next, we focus our attention on plateaulike and oscillatory features in  $\rho_H(B)$  and  $\rho_b(B)$ , seen in Figs. 1–4 as a fine structure.

In Fig. 2, we plotted  $\Delta\rho_b(B)$  vs  $1/B$  for  $B < 5$  T, where  $\Delta\rho_b(B)$  is obtained after subtraction of the monotonic background resistivity  $\rho_b^{bg}(B)$ : the data clearly demonstrate that the fine structure is due to Shubnikov–de Haas (SdH) oscillations. The obtained period of SdH oscillations  $\Delta(B^{-1}) = 0.208 \pm 0.004 \text{ T}^{-1}$  (the frequency  $B_0 = 4.8 \pm 0.1 \text{ T}$ ) corresponds to the extremal cross section of the Fermi surface of the majority electrons [26].

The analysis of experimental results obtained for  $B < B_{QL}$  [22,27] showed that electrons mainly contribute to the measured  $\rho_H(B)$ , whereas the contribution from Dirac-like majority holes is tiny [27]. Thus, the measured IQHE staircase is consistent with either conventional massive electrons with Berry’s phase 0, or chiral massive electrons having Berry’s phase  $2\pi$ , as in graphene bilayer [28,29]. We stress that IQHE staircases measured for HOPG [22,27] and graphene bilayer samples [29] overlap when plotted as a function of the filling factor  $\nu = B_0/B$  [27], testifying on the quasi-2D nature of HOPG. The inset in Fig. 2 illustrates the QH plateau occurrence at  $\nu = 1$  and  $\nu = 2$ . In the same figure, we show  $\rho_H(\nu)$  measured [22] for HOPG-UC (Union Carbide Co.) sample. It can be

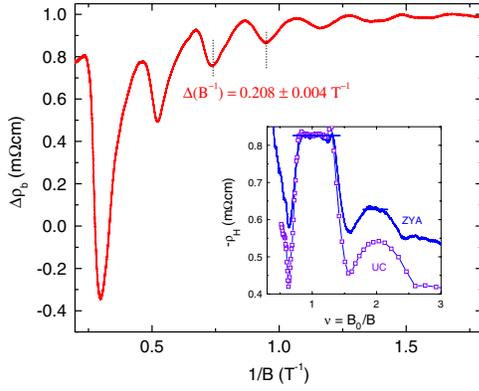


FIG. 2 (color online). Shubnikov de Haas resistivity oscillations with the period  $\Delta(B^{-1}) = 0.208 \pm 0.004 \text{ T}^{-1}$  corresponding to the majority electrons;  $\Delta\rho_b$  is obtained subtracting the monotonic background resistivity  $\rho_b^{bg}(B)$ . The inset demonstrates quantum Hall plateaus measured for HOPG-ZYA and HOPG-UC [ $\rho_H(B)/3.6$ ] samples;  $\nu = B_0/B$  ( $B_0 = 4.68 \text{ T}$  for HOPG-UC, and  $B_0 = 4.8 \pm 0.1 \text{ T}$  for HOPG-ZYA).

readily seen that the main IQHE plateau is centered at  $B = B_0$  for both ZYA and UC HOPG samples, demonstrating the universality in the behavior of these strongly anisotropic samples. The Hall resistivity  $\rho_H(\nu = 1) = 0.82 \text{ m}\Omega \text{ cm}$  for ZYA HOPG was obtained from the measured Hall resistance  $R_H(\nu = 1) = 16.5 \text{ m}\Omega$ , assuming the uniform current distribution through the sample thickness  $t = 0.5 \text{ mm}$ . Taking the distance between neighboring graphene planes  $d = c/2 = 3.355 \text{ \AA}$ , one gets  $N \approx 1.5 \times 10^6$  independent graphene (bi)layers contributing to the measured signal. This gives the Hall resistance per (bi) layer  $R_H^\square(\nu = 1) = N \cdot R_H(\nu = 1) \approx 24.8 \text{ k}\Omega$ , that practically coincides with the Klitzing fundamental Hall resistance  $h/e^2 \approx 25.8 \text{ k}\Omega$ . However, because of the strong sample anisotropy ( $\rho_c/\rho_b = 4 \times 10^4$ ), the measuring current can be concentrated within the effective sample thickness  $t_{\text{eff}} < t$  [30], implying that the actual value of  $R_H^\square(\nu = 1)$  can be smaller. Taking the QH plateau sequence  $R_H^\square = h/4\nu e^2$  as predicted (and measured) for graphene bilayer [28,29], and the measured difference  $\Delta\rho_H(\nu) = \rho_H(\nu = 1) - \rho_H(\nu = 2) \approx 0.2 \text{ m}\Omega \text{ cm}$  (Fig. 2, inset), one estimates the effective thickness of the electron “layers”  $l_{\text{eff}} \approx 6.2 \text{ \AA}$ , responsible for IQHE. Interestingly, the obtained value of  $l_{\text{eff}}$  agrees well with the  $c$  axis lattice parameter  $c = 6.71 \text{ \AA}$ , resembling the theoretical result for IQHE in bulk graphite [31]. Whether this is an accidental coincidence or it has a deeper reason, remains to be seen. The sample resistivity ratio  $\rho_c/\rho_b = 4 \times 10^4$  implies a very small tunneling amplitude in the  $c$  axis direction  $t_\perp \sim 3\text{--}5 \text{ meV}$  [11]  $< \hbar\omega_c$  for  $B > 1\text{--}2 \text{ T}$ , allowing to consider independent QH states in each “bi-layer,” see also [32].

The data presented in Fig. 3 demonstrate that plateaus in  $\rho_H(B)$  also take place for  $\nu \ll 1$ . As Figs. 3(a)–3(c) exemplify, plateaus and quasiplateaus are centered quite accurately (within the error bar for  $B_0 = 4.8 \pm 0.1 \text{ T}$ ) at

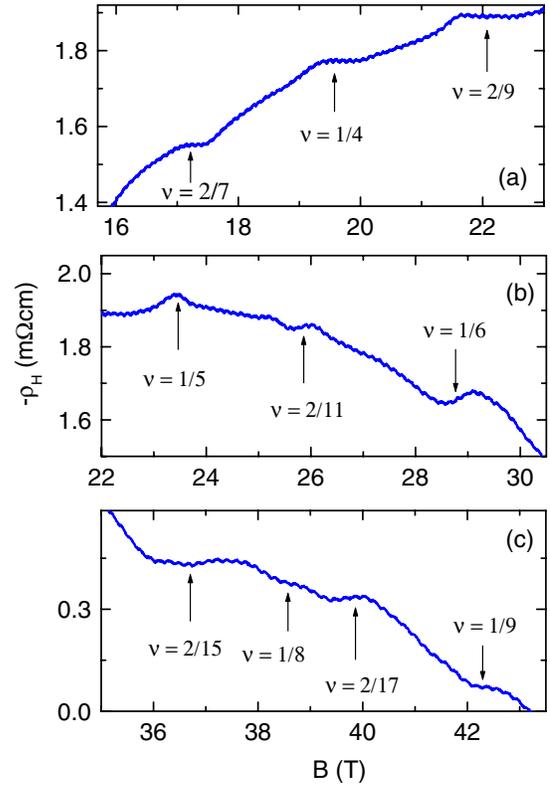


FIG. 3 (color online). Quantum Hall plateaus observed for various fractional filling factors  $\nu = B_0/B$  ( $B_0 = 4.8 \pm 0.1 \text{ T}$ ).

$\nu = 2/7, 1/4, 2/9, 1/5, 2/11, 1/6, 2/15, 1/8, 2/17, 1/9$  [33]. It appears that all these numbers correspond to the filling factors  $\nu = 2/m$  ( $m = 1, 2, 3, \dots$ ) proposed by Halperin [34] for the case of bound electron pairs, i.e.,  $2e$ -charge bosons. In principle, the existence of  $2e$  bosons in the ultraquantum limit can be justified assuming the electron pairing driven by the Landau level quantization [5].  $\Delta\rho_H(B)$  steps between neighboring plateaus agree with the FQHE scenario, as well. For instance,  $\Delta\rho_H(B) \approx 0.22 \text{ m}\Omega \text{ cm}$  measured between  $\nu = 1/4$  and  $\nu = 2/7$

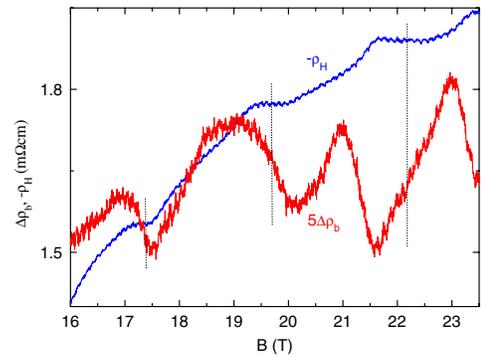


FIG. 4 (color online). Plateaus in the Hall resistivity  $\rho_H(B)$  correlate with the minima in  $\Delta\rho_b$  (multiplied by factor 5 and arbitrary shifted along the vertical axis); dotted lines mark centers of QH plateaus.

[Fig. 3(a)] plateaus, coincides with the expected value  $\Delta\rho_H(B) = (h/8e^2) \cdot c \approx 0.216 \text{ m}\Omega \text{ cm}$ .

It is worth noting that  $l_{\text{eff}} \approx c = 6.71 \text{ \AA}$  is much smaller than the magnetic length  $l_B[\text{\AA}] = (\hbar/eB)^{1/2} = 250/B^{1/2}[T^{1/2}]$  in the whole studied field range. Thus, recent 3D models for FQHE [8,9], probably relevant to bulk bismuth [10], do not apply to highly anisotropic graphite.

On the other hand, one may argue against the QHE in both bulk graphite and bismuth because  $\rho_b(B)$  does not vanish in the plateau region, and  $\rho_b(B) > \rho_H(B)$ . However, small dips and not vanishing of the longitudinal resistivity  $\rho_{xx}(B) > \rho_{xy}(B)$  were measured, e.g., for Bechgaard salt  $(\text{TMTSF})_2\text{PF}_6$  [35],  $\text{Bi}_{2-x}\text{Sn}_x\text{Te}_3$  and  $\text{Sb}_{2-x}\text{Sn}_x\text{Te}_3$  [36],  $\eta\text{-Mo}_4\text{O}_{11}$  [37] layered crystals, as well as for GaAs/AlGaAs 2DES [38,39], in both IQHE [35–37] and FQHE [38,39] regimes. In particular, in Ref. [39], FQH states resulting from the melted Wigner crystal were detected at very high global longitudinal resistance level of  $R_{xx} \sim 1 \text{ M}\Omega$ .

Figure 4 illustrates the correlation between QH plateaus and dips in  $\Delta\rho_b(B)$  measured in the present work. Figure 4 also demonstrates that minima in  $\Delta\rho_b(B)$  are somewhat shifted from the plateau centers which is the characteristic feature of QHE in bulk materials [36,37]. Thus, it is legitimate to treat the data obtained on graphite in a similar way. For  $\nu > 2/7$ , no correlation between dips in  $\Delta\rho_b(B)$  and plateaulike features in  $\rho_H(B)$  is found, and no plateaus corresponding to Halperin's  $\nu = 2/m$  fractional filling factors can be unambiguously identified. Further studies should clarify this observation.

In summary, we report the results of basal-plane Hall resistivity  $\rho_H(B)$  and longitudinal resistivity  $\rho_b(B)$  measurements performed on HOPG samples up to  $B = 50 \text{ T}$ . The sign change in the Hall resistivity from electron- to hole-type in ultraquantum limit is reported for graphite for the first time. For our best quality samples, FQHE associated with majority electrons is detected for filling factors  $\nu \ll 1$ , and ascribed to a quantum liquid of  $2e$  bosons [34]. The obtained results provide evidence that strongly anisotropic graphite can be considered as a system of quasi-2D layers of the thickness  $l_{\text{eff}} \approx c = 6.71 \text{ \AA}$  that exhibit independent integer ( $\nu \geq 1$ ) or fractional ( $\nu < 1$ ) quantum Hall states.

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