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Aharonov-Bohm effect on columnar defects in thin graphite and graphene

Yu.I. Latyshev¹, A.P. Orlov¹, E.G. Shustin², N.V. Isaev²,
W. Escoffier³, P. Monceau⁴, C.J. van der Beek⁵, M. Konczykowski⁵, I. Monnet⁶

¹Kotelnikov Institute of Radio-Engineering and Electronics RAS, Mokhovaya 11-7,
125009 Moscow, Russia

²Kotelnikov Institute of Radio-Engineering and Electronics RAS, Fryazino 141190,
Moscow region, Russia

³Laboratoire National des Champs Magnétiques Intenses (LNCMI-T), 143 Av. de
Rangueil, 31400, Toulouse, France

⁴Institut Néel, CNRS, 25 Rue des Martyrs, 38042 Grenoble, France

⁵Laboratoire des Solides Irradiés, Ecole Polytechnique, Palaiseau, 91128 France

⁶Centre de Recherche sur les Ions, les Matériaux et la Photonique (CIMAP, GANIL),
Caen, 14050 France

E-mail: yurilatyshev@yahoo.com

Abstract. We studied Aharonov-Bohm effect on thin graphite single crystals containing columnar defects (nanoholes) produced by irradiation with heavy ions. The thickness of irradiated single crystals has been varied from 50 nm down to less than 1 nm by thinning the thicker samples with beam-plasma etching. For irradiated samples we found the existence of oscillating, field-periodic contribution of magnetoresistance with a periodicity corresponding to the flux quantum hc/e per area of nanohole independently of sample thickness. The results point to the significant contribution of Dirac fermions to the oscillating part of magnetoresistance and also to the existence of the edge states located near the nanohole perimeter.

1. Introduction

Shortly after a discovery of Dirac fermions in graphene [1, 2], they were also found in thin single crystals of graphite by far infrared (FIR) magneto-transmission measurements [3], ARPES [4], STM [5] and interlayer tunneling [6] techniques. The specific features of Dirac fermions: (1) conic spectrum $E(k) = \pm v_F \hbar |k|$, where v_F is the Fermi velocity, and (2) specific Landau quantization with the energy of Landau levels E_n proportional to $(n H)^{1/2}$, have been used for their identification in graphite. The

presence of Dirac fermions in graphite was attributed either to the linear dispersion at the H-point of the graphite Brillouin zone [3] or to the existence of graphene layer at the surface of graphite [7, 8].

Another interesting property of Dirac fermions is the absence of backscattering [9, 10] and the related absence of the weak localization effects. That feature was confirmed on graphene mesoscopic ring samples, where oscillations of magnetoresistance with a period, corresponding to flux quantum $\Phi_0 = hc/e$ per area of the hole have been observed, while a contribution of $hc/2e$ oscillations, due to weak localization, was strongly suppressed [11].

In a present paper we study A-B effect on columnar defects produced by irradiation of thin single crystals of graphite and graphene with heavy ions. Columnar defects serve as nanoholes (antidots) for studies of magnetic flux quantization. We found the same periodicity of oscillating part of magnetoresistance of irradiated sample hc/e , as for graphene ring indicating contribution of Dirac fermions [11]. We show that this periodicity does not depend on graphite thickness with its variation from about 50 graphene layers down to bi-layer graphene. That points to the surface contribution of Dirac fermions in graphite.

2. Experimental

We used in experiments high quality single crystals of natural graphite. The in-plane conductivity was 30–50 $\mu\Omega$ cm and conductivity anisotropy along and across the layers was $4 \cdot 10^3$ at room temperature. At liquid helium temperatures it reached $3 \cdot 10^4$. The high quality of the crystals was also confirmed by studies the FIR magneto-transmission [13] and Shubnikov–de Haas oscillations of magnetoresistance [14].

Thin graphite single crystals with thickness of 30-100 nm and with lateral sizes of several hundred microns have been detached out of fixed thick graphite single crystal using adhesive tape. The glue layer of the tape has been solved in acetone leaving thin crystal to float freely in a solvent and then it has been picked up onto the substrate.

At the second stage thin graphite crystal has been thinned by soft plasma etching [15]. We used original beam-plasma reactor [16] to provide low energy ions. Electron beam excites Ar-plasma in a reactor and induces a flow of low energetic monochromatic ions in a direction perpendicular to the beam direction. This flow has been used for etching. The energy of ions was 60 - 80 eV providing soft defectless etching with a rate of about 10 nm per hour. A thickness of the thinned crystal has been controlled by measuring of its resistance *in situ*. The etching was terminated when the sheet resistance of the sample achieved few k Ω corresponding to 1-2 layer graphene.

Thin graphite/graphene crystals have then been masked and patterned in oxygen discharge to get Hall bar geometry. The width of the patterned part of the crystals was 10-20 μm and the length between potential probes was about 100 μm (Fig.1). Low ohmic electric contacts have been made by pressing of In-stubs into the crystal, as shown in Fig.1.

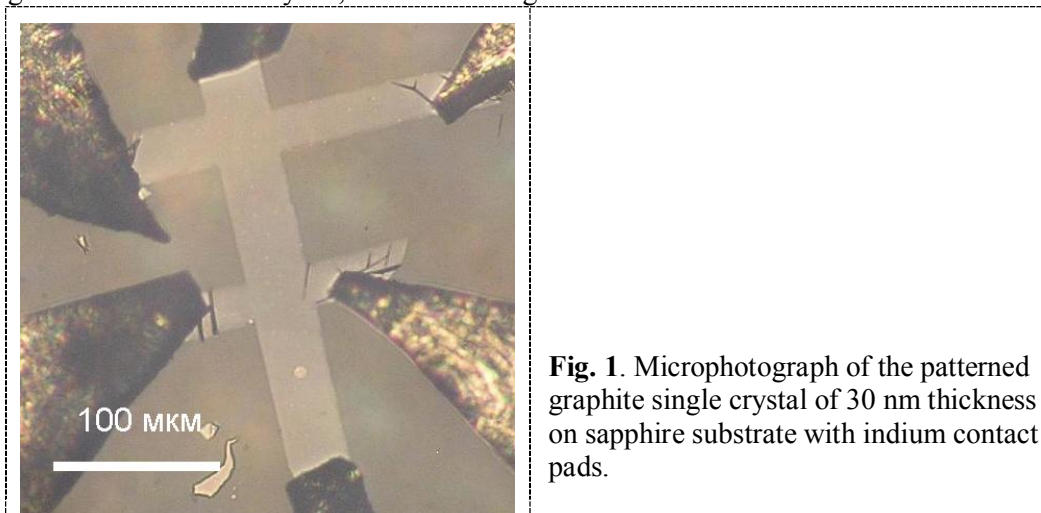


Fig. 1. Microphotograph of the patterned graphite single crystal of 30 nm thickness on sapphire substrate with indium contact pads.

We measured 8 samples of graphite single crystals irradiated with Xe-ions with energy of 90 MeV along the c -axis with doses corresponding to $5 \cdot 10^8 - 10^{10}$ def/cm². Two of them have been preliminary thinned by soft plasma etching. We analyzed the size of columnar defects using scanning AFM, as a size of hillocks of the amorphous substance squeezed out of them [17] (Fig. 2). This effect is known and is attributed to the relaxation of force stresses accompanying the formation of columnar defects. The hillock density corresponds to the radiation dose. The columnar defect diameter D over the hillock flat part is estimated as $D \leq 24$ nm at its height of about 6 nm [12]. Magnetoresistance of graphite crystals has been measured in resistive magnet (up to 32 T) in Grenoble National High Magnetic Field Lab. (LNCMI-G) or in pulsed fields (up to 55 T) in LNCMI-T, Toulouse, using standard techniques. The lowest achievable temperature was 1.2 K.

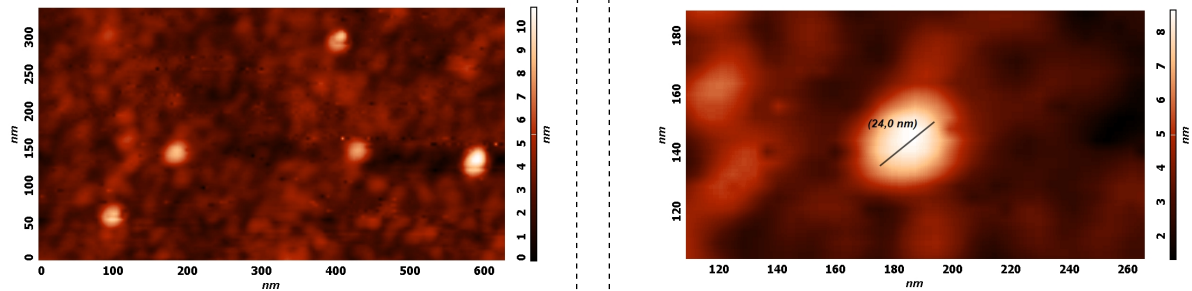


Fig. 2. AFM images of (a) a group of columnar defects and (b) a single columnar defect in sample # 3 irradiated by Xe-ions with energy of 90 MeV with a dose corresponding to 10^9 def/cm². The columnar defects are AFM recognized as hillocks of an amorphous substance squeezed out of them (see the main text).

3. Experimental results

Fig.3 (left panel) shows the full scale dependence of resistance of low dose irradiated sample #1 on magnetic field $H//c$ at several specified temperatures. In the region below 8 T, the Shubnikov–de Haas oscillations are clearly seen (right panel of Fig. 3). Their period is inversely proportional to the field and equals to 0.22 T^{-1} , in agreement with previous data [14, 18]. At higher fields new, field periodic oscillations appear. Three oscillations with the minima at 15, 22.5, and 30 T are pronounced in the region of high magnetic fields exceeding 10 T. The period of these oscillations corresponds to 7.5 T and a much weaker minimum at 7.5 T is also seen. The $R(H)$ curves almost coincided in the field of increasing and decreasing regimes as well as in reversing the field direction, $H \rightarrow -H$. Specific features of these oscillations are their big amplitude (up to 15%) and their existence up to quite high temperatures. Fig. 2a shows the $R(H)$ curves at 1.2, 4.2, 10, 20, and 32 K. It is seen that the oscillation amplitude decreases with increasing temperature, however, they are still observable up to temperatures of 30 K.

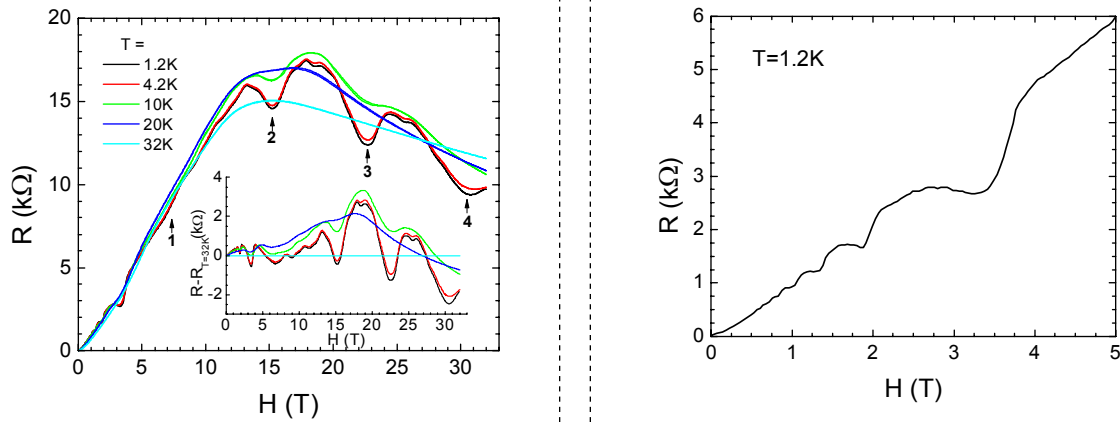
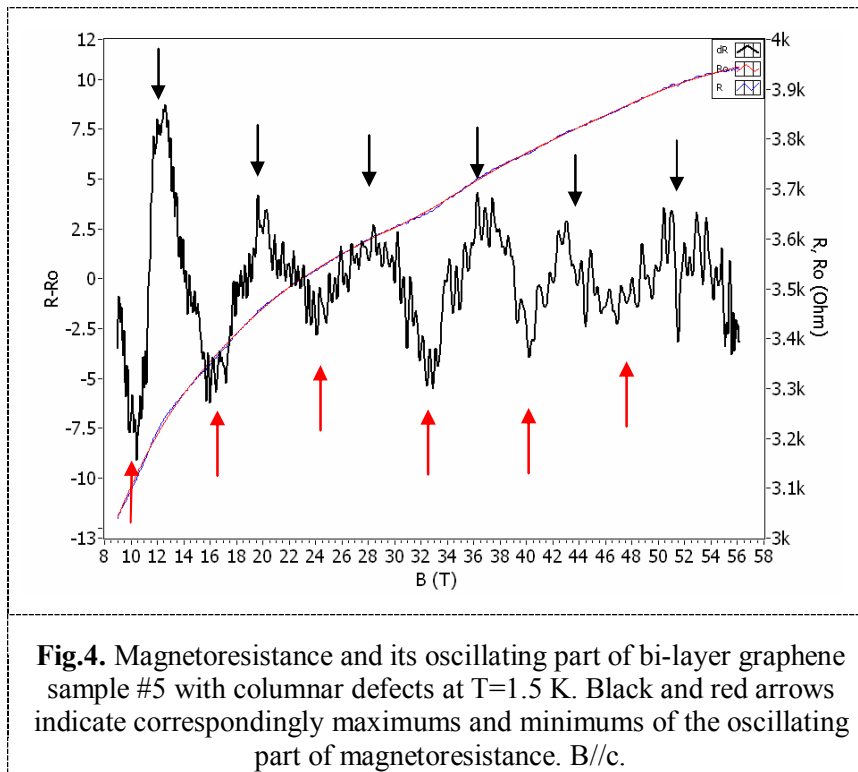


Fig. 3. The in-plane resistance of the irradiated graphite single crystal (sample # 3) versus magnetic field $H \parallel c$ at 1.2, 4.2, 10, 20, and 32 K. The sample is 50-nm thick, the columnar defect density is $c = 10^9$ defects/cm². The arrows mark the group of minima $R(H_n)$, where $H_n = nH_0$ (n is an integer, $H_0 = 7.5$ T). The right panel shows the enlarged fragment of the $R(H)$ curve up to 5 T at $T = 1.2$ K, which illustrates the Shubnikov–de Haas magnetoresistance oscillations.

Similar large scale, field-periodic oscillations were observed in the samples at the radiation doses of $3 \cdot 10^9$ and 10^{10} defects/cm², but with lower amplitudes. The reference measurements of the unexposed samples demonstrate the absence of such oscillations. On average, the $R(H)$ curves of the unexposed samples and low dose exposed samples are similar in their shape to those of bulk samples of high quality graphite single crystals [19]. They have a wide maximum near 20 T, then slowly droop to a minimum at 28–30 T. Near the maximum, the normalized magnetoresistance $R(H_{\max})/R(0)$ reaches $5 \cdot 10^3$. At a defect density of 10^9 defects/cm², it is almost invariable; however, with further increase of defect density c the magnetoresistance at $H = 15$ –20 T decreases (by an order of magnitude at $c = 10^{10}$ defects/cm²), and the $R(H)$ curves become monotonic with saturation at $H > 20$ T.

To extend the range of effect observation we used pulsed magnetic field up to 55 T. Fig. 4 shows $R(H)$ dependence and its oscillating part of our thinnest sample #5 obtained by soft plasma etching. This sample was characterized by local Raman spectroscopy as a bi-layer graphene [15]. Fig. 3 clearly shows 6 oscillations of magnetoresistance above 10 T observed on this thinnest sample. The period of oscillations, 7.6 ± 0.3 T, is very close to what was observed in sample #3 of 50 nm thick under the same irradiation conditions.



4. Data analysis and discussion.

We begin analysis of the experimental data with an estimation of the magnetic flux through the area of columnar defect corresponding to the period of magnetoresistance oscillations. The field period for one oscillation, ΔH , obtained from experiment, is $\Delta H = 7.5 - 7.6$ T. Under the assumption that the paths close to the columnar defect perimeter make the main contribution, the corresponding magnetic flux is $\Delta\Phi = \Delta H \pi D^2/4 = 3.5 \pm 0.8 \cdot 10^{-8} \text{ mT cm}^2$. Despite a large uncertainty in the columnar defect size, this value is much closer to $hc/e = 4.14 \cdot 10^{-8} \text{ mT cm}^2$ than to $hc/2e = 2.07 \cdot 10^{-8} \text{ mT cm}^2$. We should also take into account that the effective diameter of the orbit of carriers around a columnar defect is larger than its geometric diameter. This shifts the estimate of $\Delta\Phi$ closer to hc/e . Thus we can conclude that the period of magnetoresistance oscillations corresponds to the flux quantum per area of columnar defect. This result agrees well with the experiments on graphene rings [7] and implies that for both objects, thin graphite crystals and bi-layer graphene with columnar defect, we observed Aharonov-Bohm interference of Dirac fermions.

The fact that effect does not depend on sample thickness indicates that effect is very likely related with the surface layers of graphite similarly as it was recently observed for topological insulators [20]. That conclusion is supported by recent STM [7] and FIR magneto-transmission [8] measurements which showed that the surface layer of graphite is often represents as a graphene layer lying on graphite substrate.

There is another interesting point following from our experiment. Normally, Aharonov-Bohm effect is observed in ring geometry samples. This geometry lets fix the quantized orbits between inner and outer boundary of the ring. In our experiment A-B type effect was observed on non-ring geometry and as shown is determined by the orbits located close to the hole. We consider that the restricted orbits near the hole can exist due to the edge states located near graphene boundary. Otherwise, the averaging contribution of all possible orbits around hole would smear out interference effect. Existence of the edge states in graphene has been recently predicted theoretically [21]. Our observations of Aharonov-Bohm effect on nanoholes (antidots) strongly support the existence of the edge states in graphene.

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