Record-Breaking Magnetoresistance at the Edge of a Microflake of Natural Graphite

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Placing several electrodes at the edge of a micrometer-sized Sri Lankan natural graphite sample at distances comparable to the size of the internal crystalline regions, record values for the change of the resistance with magnetic field are found. At low temperatures and at $B \sim 21$ T, the magnetoresistance (MR) reaches $\approx 10^7\%$. The MR values exceed by far all earlier reported ones for graphite and they are comparable or even larger (at $T > 50$ K) than the largest reported in solids including the Weyl semimetals. The origin of this large MR lies in the existence of highly conducting 2D interfaces aligned parallel to the graphene planes.

The electrical transport properties of bulk graphite, multigraphene, and single graphene layers show a variety of interesting phenomena. These phenomena are expected to be of advantage for applications such as solar cells, supercapacitors, flexible transistors, and sensors.1–2 These perspectives in addition to the high carbon abundance in nature still attract the interest of the scientific community. In particular, a detailed understanding of the electronic properties of multigraphene samples is currently of high relevance because of the expected unique properties of the electronic band structure. We refer to stacked graphene layers that can lead to the formation of flat bands, i.e., a region in reciprocal space with a dispersionless relationship, opening the possibility of triggering high-temperature superconductivity or magnetic order.3 This can happen at certain localized regions of twisted graphene layers like in bilayers graphene or at embedded interfaces between twisted Bernal or rhombohedral stacking order regions in graphite or multi-graphene samples.4–7

In this work, we studied graphite samples, which are formed by stacking graphene layers held together by weak Van der Waals forces. The stacking order of the layers occurs naturally in two different ways: the hexagonal one, named Bernal, with the graphene layer order ABAB... (2H) and the rhombohedral ABCABA... (3R). Several scanning transmission electron microscopy (STEM) images of the internal structure of usual graphite samples were published in the last 10 years, showing their inhomogeneity due to the existence of crystalline regions of different thicknesses with different stacking orders or twisted regions at different angles around the common $c$ axis.6

Since the 1980s and partially due to the increasing structural order and quality of the measured graphite samples, the maximum magnetoresistance (MR) found for graphite samples steadily increased.8 Recently published systematic studies of the MR of graphite samples of different thicknesses revealed that this property is directly related to the existence of 2D interfaces between crystalline regions with Bernal or rhombohedral stacking order. These 2D interfaces are also responsible for the metallic-like behavior of the resistance of graphite.9,10 The MR of graphite samples we discuss in this work is always measured at fields normal to the interfaces and the graphene planes. The MR for parallel fields is negligible or related to a normal field component due to misalignment, which can come also from the angle distribution of the internal crystalline regions (finite rocking curve width).11

Attempting to understand the nature behind the internal structure of graphite and to find a way to increase its MR further, we have performed transport measurements under pulsed magnetic fields up to 65 T, placing the voltage electrodes on the sample edge at one side of the sample. This enables the possibility to obtain signals to a greater extent related to the interfaces contribution, at least at temperatures $T < 200$ K where the total conductance of the interfaces exceeds that of the semiconducting 2H and/or 3R matrix.10

From electron back scattering diffraction (EBSD) measurements on graphite samples, we know that the single crystalline regions in the $a,b$ planes of well-ordered bulk graphite samples are $\lesssim 10\, \mu m$, whereas the single stacking order regions along the $c$ axis direction range from a few nm to several 100 nm.6,12

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The distance between grain boundaries within a single interface puts an upper limit to the typical length of the internal interfaces found in graphite samples. Therefore, to decrease the contribution from the grain boundaries, we need to place the voltage electrodes at distances smaller than \( \approx 10 \mu m \). We found that placing the voltage electrodes at the sample edge, contacting as many interface edges as possible and at small enough distances comparable to the extension of a 2D interface region, the obtained MR values are much larger than the ones reported for graphite, multigraphene, or other carbon-based materials before. The obtained MR turned out to be of the order or larger than the largest reported nowadays in solids. The technical simplification we present in this study provides a convenient and relatively easy way to study the response of 2D interfaces with their unconventional properties.

The electrical resistance at different positions along the sample edge was measured using four terminals. The electrodes were made combining electron beam lithography and sputtering of Cr/Au. The aim was to place the voltage electrodes at the edge of the thin sample to directly contact a large number of interface edges present in the sample (see, e.g., the STEM images of different graphite samples in the study of Esquinazi et al.\(^{[6]}\)). Micrometer-sized samples with well-defined interface edges are not easy to prepare. One way is to produce transmission electron microscopy lamellae as studied by Ballestar et al., but their production for transport measurements is very difficult, taking usually several months of preparation to get a single sample.\(^{[13]}\) To overcome these difficulties, we have developed a new method to produce graphite flakes with well-defined edges, avoiding problems of contamination or formation of an amorphous thin layer.

On the top of a 5 \( \times \) 5 \( mm^2 \) silicon substrate with a thickness of 0.525 mm and covered with a 150 nm silicon nitride (Si\(_3\)N\(_4\)) insulating layer, we placed microflakes of Sri Lankan natural graphite (NG). The samples were from the same batch of samples analyzed with STEM, x-ray diffraction, and particle induced x-ray emission published recently.\(^{[14]}\) After selecting flat enough samples, we covered part of the sample surfaces with a 200 nm thick Si\(_3\)N\(_4\) film using electron beam lithography (see the sketch shown in Figure 1a,b).

An Oxford Instruments Plasma Pro NGP80 ICP device was used to etch the sample with inductively coupled plasma (ICP) reactive ion etching (RIE). This process is very effective to remove graphene layers in graphite samples in a controlled way.\(^{[7,15–17]}\) In this way, the area of the sample not covered by the Si\(_3\)N\(_4\) film was completely removed, creating a sharp and well-defined edge. The area protected by Si\(_3\)N\(_4\) remained after RIE (Figure 1c). The parameters used for RIE were 282 V for the applied DC Bias, 50 W HF power, 50 W ICP power, \( \approx 25 \times 10^{-3} \) mbar as chamber pressure, 9 sccm for Ar, and 1 sccm O\(_2\) gas flow rate. Under these parameters, we could completely etch through a 665 nm thick graphite sample in \( \approx 40 \) min.

After etching the sample, electrodes were deposited on the lateral part of the sample (see Figure 1d–f), parallel to the \( c \) axis and contacting the interfaces edges. Electron beam lithography was used to prepare the electrodes, where a chromium thin film with thickness of 5 nm was sputtered first and then a 50 nm gold film on top. The main sample shown here, labeled U11, had in total five electrodes (each \( \approx 2.5 \mu m \) width), allowing electrical transport measurement at different regions of the sample (see Figure 1d–f). The temperature and magnetic field dependence of the resistance to 7 T were measured in a Quantum Design \(^3\)He flow cryostat with a superconducting solenoid, with a high-resolution AC resistance bridge LR-700 at a frequency of 19 Hz and input current of 12 \( \mu A \).

The measurements at high magnetic fields were performed at the pulsed field facility of the NHMFL at Los Alamos National Laboratory in a \(^3\)He + \(^4\)He cryostat with maximum magnetic field of 65 T. Most of the experiments were performed with pulses of 60 T. A down-sweep pulse lasts \( \approx 60 \) ms, wherever the up-sweep peak field is reached at \( \approx 10 \) ms. An AC current

![Figure 1](https://example.com/fig1.png)

**Figure 1.** Sample preparation sketches: a) mesoscopic graphite sample placed on a silicon substrate with a 150 nm Si\(_3\)N\(_4\) insulator layer on the top. b) Part of the graphite sample covered with a 200 nm thick Si\(_3\)N\(_4\) layer. c) During the RIE exposure, only the uncovered area of the sample is removed. d) Sputtered chromium/gold electrodes were placed at different parts of the sample using electron beam lithography. The electrodes labeled “I” were used to apply the electrical current through the sample, and the other three electrodes were selected in pairs to measure the potential difference at different regions of the sample, being \( AB = d_m = 5.7 \mu m \), \( AC = d_l = 13.3 \mu m \), and \( BC = d_l = 4.6 \mu m \). e) 3D sketch of the sample with its electrodes at the edge, parallel to the \( c \) axis of the graphite structure. f) Scanning electron microscopy image of part of the sample U11 with part of its electrodes.
of 12 μA was applied to the sample at a frequency of 50.5 kHz (for further details see the Supporting Information). The voltages were measured with a 20 MHz sampling rate. The field was always applied normal to the graphene planes and 2D interfaces.

To minimize the noise on the measurement, we used copper wires with a diameter of 60 μm, tightly twisted in pairs, with 3–4 windings per mm. We used one pair to apply the current and two other pairs to read the voltages. The wire pairs were glued with GE varnish on the walls of the rod used to insert the sample inside the cryostat, reducing the noise introduced by vibrations due to the pulse. Other source of noise in this kind of measurements is the open loops (untwisted parts of the wires) due to the high dB / dt. To minimize this effect, we fixed the twisted wires as close as possible to the sample. Low-field measurements were performed on three samples. In the main article, we will show and discuss the one with the highest MR. The results of the other samples can be seen in the Supporting Information where we include further details of the samples purity.

The temperature dependence of the normalized resistance at zero field for sample U11 is shown in Figure 2. The data are labeled according to the distance between the voltage reading electrodes, e.g., the data labeled $d_i$ were obtained between the electrodes B and C with the shortest distance (see Figure 1e). The temperature dependence shown in Figure 2 follows the usual metalliclike behavior of well-ordered bulk graphite samples in all temperature range, indicating that the electrodes are sensing regions containing 2D interfaces. The data are similar between $T = 390$ K to $\approx 15$ K. At lower temperatures, the results labeled $d_m$ and $d_l$ tend clearly to saturate due to the contribution of a residual resistance attributed to the scattering of conduction electrons at the grain boundaries, in agreement with a large number of published data. In this temperature region and in contrast to the other two configurations, the curve $d_f$ is remarkably different exhibiting a much lower residual resistance. The resistance ratio $R(390 \text{ K})/R(5 \text{K})$ for $d_i$, $d_m$, and $d_f$ is 29, 13, and 16, respectively. The resistance ratio increases further for the $d_n$ configuration only, reaching a remarkable high value of $R(390 \text{ K})/R(0.48 \text{K}) \approx 100$. The observed behavior implies that the sample is not homogeneous, in agreement with studies realized in the last years on different graphite samples.

The temperature dependence of the resistance is fitted using a phenomenological parallel resistor model as proposed first in the study of Garcia et al. and extended in following years. The model takes explicitly into account the internal structure of real graphite samples, assuming three contributions in parallel. The first one due to the embedded 2D interfaces provides the metalliclike behavior of graphite; the second and third ones are the semiconducting contributions of the hexagonal 2H and rhombohedral 3R stacking orders, see the Supporting Information for details of the model. The metalliclike contribution is composed by a temperature-independent residual resistance, a linear and a thermally activated temperature-dependent term. Such phenomenological model describes with good accuracy the temperature

![Figure 2](image_url)

**Figure 2.** a) Temperature dependence of the normalized resistance for a NG sample from Sri Lanka in logarithmic scale at different voltage–electrodes distances localized at the same sample edge, see Figure 1. The solid lines are fits to the phenomenological parallel resistor model, which includes the contribution of the metalliclike 2D interfaces and the semiconducting crystalline regions with the two stacking orders (see Equation (S1), Supporting Information). b) Same $d_i$ data as in (a) with the different contributions within the parallel model. The linear contribution $R_T$ and the exponential one $R_2 \exp[-E_s/(k_B T)]$ are related to the 2D interfaces, and $R_{2H}$ and $R_{3R}$ are related to the two semiconducting stacking orders. The contributions were normalized to $R(390 \text{ K})$. The normalized value of the residual resistance $R_0$ necessary to fit the low temperature data is shown by the horizontal arrow $R(390 \text{ K}) = 0.1392 \Omega$. 

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dependence of the resistance in the entire investigated temperature range. Note that below \( \approx 200 \text{ K} \), the interface contribution is the most important one, and the fit is not very sensitive to the parameters of the other two contributions, see Figure 2b. A detailed discussion on this issue and on the weight of the parameters in a given temperature range was published recently\cite{ref10}.

The values of the fit parameters can be seen in the Supporting Information. As expected, the residual resistance from the fit at the low temperature of the \( d_i \) data shown in Figure 2a is one order of magnitude smaller than for \( d_m \) and \( d_3 \). From the fits of the data to the parallel resistor model, we find that the linear-in-temperature term of the interface contribution\cite{ref10} is important at \( T < 10 \text{ K} \), whereas the thermally activated exponential term (with an excitation energy of the order of \( \approx 5 \text{ meV} \)) clearly contributes between 15 and \( \approx 200 \text{ K} \). It is interesting to note that the low residual resistance of the \( d_i \) data clearly reveals the linear-in-temperature contribution that holds to the lowest measured temperature. The temperature dependence of the resistance at different constant applied magnetic fields for the three configurations of sample U11, the field-driven metal-insulator transition, and the Shubnikov-de Haas (SdH) oscillations are discussed in the Supporting Information.

We discuss now the field dependence of the resistance and the MR defined as \( \text{MR} = \frac{[R(B) - R(0)]/R(0)}{R(0), \text{ where } R(0) \text{ is the measured resistance at zero applied magnetic field (MR \%}) = 100 \% \times ([R(B) - R(0)]/R(0)). As shown in the Supporting Information, and as example, at \( T = 5 \text{ K} \) and \( B = 7 \text{ T} \), the MR reaches \( 5 \times 10^6 \% \) at the contacts \( d_m \) and \( d_3 \). For the contact configuration \( d_i \), the MR is nearly twice larger reaching \( \approx 10^6 \% \), see Figure 3 for the results obtained with puled fields. Earlier studies in high quality graphite samples show a MR of \( \approx 15 \text{ 000} \% \), 4000\%, and 75\%\cite{ref18-21}. In graphene/boron-nitride heterostructures, a MR of \( \approx 90 \text{ 000} \% \) was measured at similar field and temperature,\cite{ref22} see Figure 4a.

Figure 3 shows the high field results obtained in the \( d_i \) configuration; panel (a) shows the absolute resistance and panel (b) the MR at different constant temperatures. The up and down arrows in (b) indicate the fields at which the commonly reported electronic high field transitions \( \alpha \) and \( \alpha' \) of graphite occur at \( T < 15 \text{ K} \). These transitions as well as the maximum and negative MR above 20 T are related to the electronic 2D systems of some interfaces in the sample and were discussed in detail in a recent publication\cite{ref23}. The MR at \( T = 0.48 \text{ K} \) and \( B \sim 21 \text{ T} \) reaches \( \approx 8 \times 10^6 \% \), exceeding by far all values reported for graphite in literature.

A comparison with the temperature dependence of the MR data reported for different graphite samples in the literature and at fields of 7 and 21 T, is given in Figure 4a,b.\cite{ref24-30} Large MR values were observed for Type-II Weyl semimetal-like WP\(_2\)\cite{ref31} reaching MR \( \approx 2 \times 10^7 \% \) at low temperatures and at 7 T, similar to the MR we obtained at \( d_i \), see Figure 4a. Further data of the semimetals MoP\(_2\)\cite{ref31} and NbP\cite{ref32} the metallic sample \( \alpha \)-galium\cite{ref33} and of the topological insulator Bi\(_2\)Te\(_3\)\cite{ref34} are shown in Figure 4a,b. We note that the MR of graphite at both fields and at the configuration \( d_i \) reaches values comparable or even larger (at \( T > 50 \text{ K} \)) than the largest so far reported.

**Possible origin of the huge MR measured in graphite:** First, we note that the increase of the MR decreasing the distance between the voltage electrodes (the voltage electrode distance in the configuration \( d_i \approx d_i/3 \)) is not related to an increase in the

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**Figure 3.** MR measured at \( d_i \) as a function of magnetic field at different constant temperatures. a) Absolute value of the resistance and b) the normalized MR (in units of \( 10^6 \% \)) versus applied field. The black-down arrows indicate the onset transition \( \alpha \) and the red-up arrows the so-called re-entrant transition \( \alpha' \).
contribution of a ballistic transport. Measurements of the MR in thin graphite samples with no or a low number of interfaces showed that the MR is not only much smaller but decreases with the sample size due to the large mean free path and huge mobility of the carriers within the semiconducting graphene layers in the graphite matrix.[35,36] Experimental studies clearly showed that the large MR as well as the SdH oscillations of graphite are directly related to the response of the 2D interfaces, i.e., they are not intrinsic of the ideal graphite structure and not related to the intrinsic carriers within the graphene layers.[18,23] The values of the MR of graphite samples depend on the thickness of the sample, as one recognizes in the results of the measured samples in this work, see Figure 4a, in agreement with previous studies.[23,30] For small enough sample thickness, the number of interfaces decreases and several features of the MR vanish. For example, the maximum at $B \approx 20$ T, the negative MR and the electronic phase transitions observed at low enough temperatures and above $T \approx 20$ K, completely vanish.[23,30] The semiconductinglike behavior observed in thick samples at high temperatures, see the curves at 200 and 250 K in Figure 3, or even at lower temperatures in much thinner samples,[30] can semiquantitatively be explained with a semiconducting two-band model.[23]

These facts plus the giant magnetic anisotropy clearly indicate that the origin of the huge MR has to be found within the 2D electronic system at the interfaces embedded in the graphite samples. Taking into account that the 3R stacking order remains a minority phase in our samples (less than 15%), the interfaces between twisted 2H stacking regions (type I) and between 2H and 3R regions (type III) are the most probable ones. The possible occurrence of superconductivity at these 2D interfaces has been shown experimentally[6,7,13,14,37,38] and theoretically predicted.[3] The origin of the thermally activated exponential increases with temperature, one part of the interface contribution to the total resistance (see Figure 2b) remains still controversial.[10] We note, however, that it has been already observed in superconducting thin films, granular superconductors, and in artificially grown Josephson-junction arrays.[39-41] For a discussion of the effects of granular superconductivity on the MR of the 2D interfaces of graphite, we refer to a recently published study.[23] We suggest, therefore, that at least part of the observed large MR can be related to the existence of granular superconductivity at certain 2D interfaces embedded in the graphite matrix.[6] Further increase in the MR of graphite samples can be achieved by reducing the residual resistance measured in series with the interface resistance. This should be possible through the

Figure 4. MR of different graphite samples from literature, three Weyl semimetals, α-gallium, and topological insulator Bi2Te3. The legend shows the kind of sample, where HOPG stands for “highly oriented pyrolytic graphite”, and its grade (A or B), KG means “Kish graphite”, and NG “natural graphite”. The number in brackets represents the sample thickness given in the corresponding publication. a) Shows the temperature dependence of the MR at an applied field of $B = 7$ T and b) at $B = 21$ T.
reduction of the electrode distance or trying to contact an interface where superconductivity is less granular. Obviously, in this case, the MR would diverge.

Finally, we compared the MR we obtained in graphite with that of the Weyl semimetals. In Figure 4, one recognizes that the three reported MRs of Weyl semimetals and that of α-gallium show a similar behavior with temperature: below a sample-dependent temperature it tends to saturate, whereas above this temperature it decreases with temperature much more steeply compared to the MR of the graphite samples. This result plus the fact that the MR of graphite thick samples is mainly related to the electronic systems at the 2D interfaces, already suggest that the origin as well as the mechanisms involved in the MR is not the same, despite the expected similarities in the band structure. Moreover, due to the parallel contributions of different electronic systems in the graphite samples, the observed decrease in temperature can be achieved reducing the parallel contribution of the semiconducting regions around the interfaces.

In conclusion, with voltage electrodes separated by few micrometers along the edge of graphite samples, we measured the longitudinal resistance at different regions of the sample edge. This experimental method enables the study of the transport properties of interfaces embedded in the graphite matrix. The obtained results indicate that graphite is an inhomogeneous material at a scale of a few micrometers within the a, b planes. This inhomogeneity is one main factor that affects substantially the measured MR at low temperatures. The MR of the graphite interfaces is very large, exceeding in some temperature and field region, the largest MR values reported for solids.

Supporting Information
Supporting Information is available from the Wiley Online Library or from the author.

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Conflict of Interest
The authors declare no conflict of interest.

Author Contributions
C.E.P. and J.B.-Q. were responsible for the samples preparation. Z.Z. was responsible for SiN deposition. M.S. was responsible for AFM measurements. M.K.C., M.J., J.B.-Q., and C.E.P. conducted the high-field measurements. C.E.P., P.D.E., and J.B.-Q. analyzed the data, whereas P.D.E. and J.B.-Q. contributed equally. M.G. gave the idea to make use of RIE to measure the interfaces. P.D.E. conceived the experiment(s) and took the lead writing of the manuscript. All authors provided critical feedback and helped to shape the research, analysis, and manuscript.

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