

**Magnetothermal conductivity of highly oriented pyrolytic graphite in the quantum limit**

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We report on the magnetic field ( $0 \text{ T} \leq B \leq 9 \text{ T}$ ) dependence of the longitudinal thermal conductivity  $\kappa(T, B)$  of highly oriented pyrolytic graphite in the temperature range  $5 \text{ K} \leq T \leq 20 \text{ K}$  for fields parallel to the  $c$  axis. We show that  $\kappa(T, B)$  shows large oscillations in the high-field region ( $B > 2 \text{ T}$ ) where clear signs of the quantum-hall effect are observed in the Hall resistance. With the measured longitudinal electrical resistivity we show that the Wiedemann-Franz law is violated in the high-field regime.

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**I. INTRODUCTION**

Over the last 60 years the literature contains a substantial number of measurements and theoretical work on the electrical and thermal transport properties of the semimetal graphite.<sup>1,2</sup> In contrast to the common believe, however, several transport properties of graphite are not understood and some theoretical assumptions done in the past seem now less plausible. These doubts have their origin in the relatively new and controversial physics of a two-dimensional (2D) electron gas. Graphite has been tacitly assumed to be a 2D material, but the relatively low quality of the samples prevented clear measurements of its true 2D transport properties. The number of open questions regarding the transport properties of graphite is significant. Already early work noted that the magnetic field dependence of the electrical longitudinal resistivity  $\rho_{xx}$  and Hall resistance  $R_H$ , even including *ad hoc* dispersion relation and trigonal warping of the constant energy surfaces, was (and still remains) basically unexplained.<sup>3</sup>

Recent measurements of the longitudinal resistivity of highly oriented pyrolytic graphite (HOPG) samples show a clear magnetic field driven metal-insulator transition (MIT) with a giant magnetoresistance at low fields and at low temperatures.<sup>4</sup> For example, the longitudinal resistivity at 4.2 K can increase by more than one order of magnitude with a field  $B \approx 0.2 \text{ T}$ . This MIT shows a scaling as found for 2D electron systems with similar scaling exponent but with a critical field  $\sim 0.1 \text{ T}$  applied perpendicular to the graphene layers.<sup>5,6</sup> Possible origins for the MIT in graphite are being discussed nowadays in the literature in terms of superconducting fluctuations,<sup>4</sup> excitonic insulator state triggered by a magnetic catalysis phenomenon,<sup>7</sup> and/or a Bose metal transition.<sup>6,8</sup> High-resolution angle dependence of the magnetoresistance along the  $c$  axis in HOPG samples indicates that the transport between layers gets incoherent the better the sample quality—characterized by the full width at half maximum (FWHM) of the rocking curve—and suggests that the coupling between planes is much less than the commonly assumed  $0.3 \text{ eV}$ .<sup>9</sup>

In the quantum limit (QL) when only the lowest Landau

levels of graphite are occupied ( $B > 2 \text{ T}$ ), the longitudinal electrical resistivity shows a reentrance to a metallic-like state below a field-dependent temperature  $T_m(B)$ .<sup>10,11</sup> The function  $T_m(B)$  depends on the dimensionality of the graphite sample and it oscillates as a function of field for quasi-2D samples. This behavior might be an evidence for field-induced superconductivity at the QL in graphite, discussed in Refs. 12,13. In the QL the Hall resistance  $R_H$  shows clear signs of the quantum Hall effect for samples with small FWHM.<sup>11</sup>

For ideal graphite the conduction electrons are expected to follow a Dirac dispersion relation. These quasiparticles (QP) should have some similarities with the nodal QP of the  $d$ -density wave (DDW) state, which applies also to high-temperature superconductors. For a not superconducting DDW QP the violation of the Wiedemann-Franz (WF) law<sup>14</sup> has been predicted<sup>15</sup> as a characteristic of the relativistic spectrum of the QP. Briefly, in the DDW state the QP can carry electrical current much more effectively than thermal current in the limit of very small QP density, a situation that applies to graphite (Fermi energy  $E_F \sim 200 \text{ K}$ ). The theoretical work of Ref. 16, however, goes a step further taking into account the presence of a magnetic field within the DDW state in the calculations. This work finds that the WF law is restored at low enough temperatures (compared with  $E_F$ ) independently of the applied field. We note that in none of these references quantum Hall states have been taken into account in the calculations. Taking into account recently published experimental evidence,<sup>11</sup> these theories cannot be applied straightforwardly to graphite. The aim of our work is to check whether the WF law applies in graphite in particular in the QL regime.

The magnetic field dependence of the thermal conductivity of strongly anisotropic HOPG samples was already measured in the past but one does not find curves in the published literature with the necessary resolution in the QL regime. There are at least two unpublished studies which deserve our attention. In his Ph.D. thesis, Ayache<sup>17</sup> measured  $\kappa(T, B)$  and observed well defined oscillations of  $\kappa(B)$  at fields above  $2 \text{ T}$  and at constant temperatures below  $10 \text{ K}$ . This behavior was apparently also observed by Woollam<sup>18</sup>

but the curves were not included in the corresponding paper. Interestingly, both authors emphasized that the oscillations in  $\kappa(B > 2 \text{ T})$  as a function of field were apparently in phase with the electrical resistivity oscillations, although the electronic contribution to  $\kappa$  at high fields freezes out according to the Wiedemann-Franz law. In spite of this striking result neither the curves nor any analysis of the data was published to our knowledge.

High-resolution measurements of the field dependence of the thermal conductivity of quasi-2D HOPG samples have nowadays special relevance. In particular because evidence for a quantum Hall behavior in 2D samples in the QL has been recently reported.<sup>11</sup> The behavior of  $\kappa(T, B)$  in graphite is not only important to understand the nature of the QP in this material at the QL but also gives us the chance to study the thermal transport in the quantum Hall effect (QHE) regime. We note that the thermal transport in the QHE regime still remains an unclear problem since the discovery of this effect. In this work we have measured the longitudinal thermal conductivity  $\kappa$  of a well characterized HOPG sample as a function of magnetic field applied parallel to the  $c$  axis of the graphite structure. These measurements were accompanied by measurements of the longitudinal and Hall resistances. The results clearly show that the WF law is violated in the QL, whereas deviations are observed at lower fields.

## II. EXPERIMENTAL DETAILS

The HOPG sample from the Union Carbide company measured in this work was selected because of its clear quasi-2D properties observed in the electrical and Hall resistivity. This behavior is correlated with the small FWHM  $= 0.26^\circ$  of the rocking curve, which is a measure of the misorientation relative to the  $c$  axis of the crystallites in the sample. The measured FWHM is one of the smallest we have obtained for HOPG samples. We note that most of the graphite samples studied in the literature of the 1970's and 1980's had a much larger FWHM. Our experimental evidence indicates that in samples with FWHM values larger than  $\sim 0.5^\circ$  the transport properties show clear sign of 3D behavior and coherent transport in the  $c$  direction<sup>5,9</sup> and therefore they do not reflect true 2D behavior of ideal graphite. The dimension of our sample was length = 1.6 mm, width = 1.2 mm, and thickness  $\approx 60 \mu\text{m}$ . The room temperature out-of-plane/basal-plane resistivity ratio at  $B=0$  of the sample was  $\rho_c/\rho_b \sim 5 \times 10^4$ . Furthermore, this sample does not show any maximum in the  $c$  axis resistivity as a function of angle for fields parallel to the graphene planes.<sup>9</sup> This indicates incoherent electrical transport expected for weak-coupled conducting planes and for samples with a low density of defects.

The Hall resistance was measured using the van der Pauw configuration with a cyclic transposition of current and voltage leads<sup>19,20</sup> at fixed applied-field polarity as well as magnetic field reversal; no difference in  $R_H(H, T)$  obtained with these two methods was found. For the measurements, silver past electrodes were placed on the sample surface, while the resistivity values were obtained in a geometry with an uniform current distribution through the sample cross section. All resistance measurements were performed in the Ohmic

regime. The absolute value of the longitudinal (basal-plane) resistivity at zero field was  $\rho_b(6 \text{ K}, 0) \approx 2.4$  and  $2.6 \mu\Omega \text{ cm}$  at 10 K. The error in the absolute value is estimated to be  $\sim 30\%$  due to geometrical errors. At 10 K  $\rho_b$  reaches its low-temperature rest value within 10%. The resistivity increases by two orders of magnitude for an applied field of 1 T at  $T \leq 10 \text{ K}$ .

For the longitudinal thermal conductivity measurement the temperature gradient (of the order of 200 to 300 mK) was measured using a previously field- and temperature-calibrated type  $E$  thermocouples with a dc picovoltmeter.<sup>21</sup> The thermocouple ends were positioned one at the top and the other at the bottom of the main surface of the sample. A detailed calibration below 8 K was performed because in this temperature region the thermopower of our thermocouple is specially sensitive on the magnetic field with a nonmonotonous dependence.<sup>21</sup> The experimental arrangement was recently used to study the longitudinal and Hall thermal conductivities of high-temperature superconducting crystals.<sup>22,23</sup> We note that in general the measured thermal conductivity is  $\kappa = \kappa_i - TS^2\sigma_i$ , where  $\kappa_i$  is the "real" thermal conductivity of the sample,  $S$  the thermopower, and  $\sigma_i$  the electrical conductivity. In the case of our graphite sample the correction term to  $\kappa$  is four orders of magnitude smaller than  $\kappa_i$  at 10 K.

Our system enables us to measure  $\kappa(B)$  with a relative resolution better than 0.1% above 5 K. The thermal stability was better than 10 mK in the whole temperature range  $5 \text{ K} \leq T \leq 20 \text{ K}$  and magnetic field  $0 \text{ T} \leq B \leq 9 \text{ T}$ . The absolute error in the thermal conductivity was estimated to be  $\leq 30\%$ . The obtained absolute value of  $\kappa$  and its temperature dependence are similar to those from previous studies.<sup>2,17,18,24,25</sup> For example, at 10 K we obtain  $\kappa(0) \approx 130 \text{ W/mK}$  and  $\kappa(0) \approx 33 \text{ W/mK}$  at 5 K and zero fields. In all magnetic-field runs  $\kappa(B)$  showed reversible behavior. Irreversible behavior has been observed but we could prove that it was related to small temperature drifts since in the temperature range of the measurements  $\kappa$  depends strongly on temperature.

## III. RESULTS AND DISCUSSION

Figure 1(a) shows the reduced longitudinal thermal conductivity  $\kappa(T, B)/\kappa(T, 0)$  as a function of the field applied parallel to the  $c$  axis at different constant temperatures between 5 to 20 K. In Fig. 1(b) we show the Hall resistance of the same sample. The decrease of  $\kappa$  with field can be related to the decrease of the electronic contribution. The clear oscillations in  $\kappa(B)$  observed at  $B > 1 \text{ T}$  are apparently due to the quantization of the Landau levels and the crossing of the Fermi energy as the oscillations in the Hall effect indicate, see Fig. 1(b). Figure 2 shows the same data as Fig. 1 but in a linear field scale.

It is noticeable the appearance of the plateaulike features at  $B \sim 2$  and  $4 \text{ T}$  in the Hall resistivity that clearly suggests the occurrence of the quantum Hall effect (QHE) in graphite. In fact, the temperature dependence of the maximum slope  $(d|R_H|/dB)_{\text{max}}$  vs  $T^{-1}$  between two plateaus at 3.5 T, and measured to 70 mK shows a temperature dependence  $T^{-k}$  with an exponent  $k = 0.42$  similar to that found in QHE

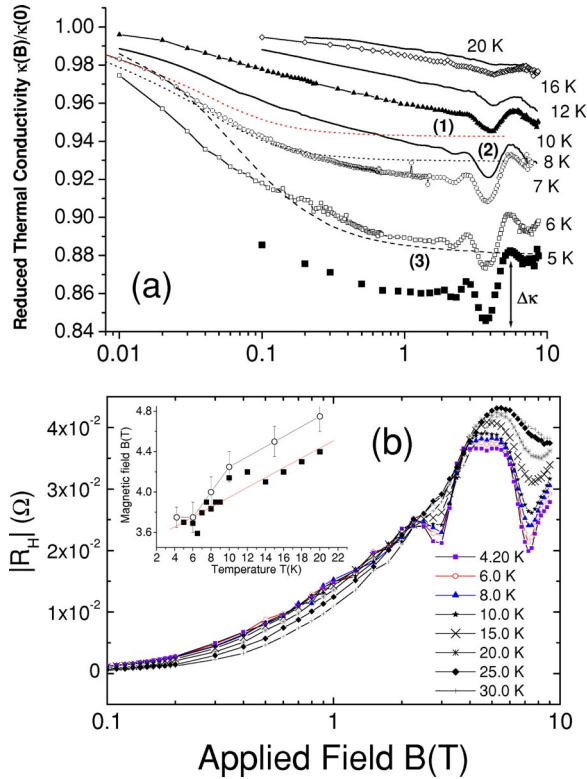


FIG. 1. (a) Reduced thermal conductivity  $\kappa(B)/\kappa(0)$  vs applied magnetic field at different constant temperatures. The lines (2) and (3) were calculated from the Wiedemann-Franz law, Eqs. (2) and (4), at 10 and 6 K using the measured field dependent electrical resistivity for the same sample. Curve (1) was obtained with the measured resistivity at 10 K but with a lower Lorenz number  $L = 2.0 \times 10^{-8} \text{ W } \Omega \text{ K}^{-2}$ . (b) Absolute value of the Hall resistance as a function of applied field. The inset shows the temperature dependence of the field at the onset of the plateau in  $R_H$  at  $\sim 3.7 \text{ T}$  ( $\circ$ ) and the position of the minimum in  $\kappa$  ( $\blacksquare$ ).

systems.<sup>11</sup> This result is not unexpected taking into account the quasi-2D structure of the sample. We note that the occurrence of the integral QHE in graphene has been predicted recently.<sup>26</sup> The reason why it was not found before is related to the sample quality which affects the 2D behavior of the transport properties. Our studies show that the dimensionality is strongly affected by internal lattice defects, some of them appear to short circuit the graphene planes. The fact that we want to stress is the good correspondence between the features measured in  $\kappa$  and  $R_H$  at the QL. To recognize this we show in the inset of Fig. 1(b) the temperature dependence of the field at the onset of the plateau in  $R_H$  at  $\sim 3.7 \text{ T}$  ( $\circ$ ) and the position of the minimum in  $\kappa$  ( $\blacksquare$ ).

Figure 3 shows in more detail the field dependence of the basal-plane and Hall resistances, taken at 4.2 K, as well as of the thermal conductivity at 5 K. The thermal conductivity data shown in this figure were taken increasing and decreasing field. As seen in the figure, no significant hysteresis is observed. This result is in contrast to the hysteresis observed by Ayache in his Ph.D. work.<sup>17</sup> We speculate that a small temperature drift might have been the reason for the observed hysteresis.

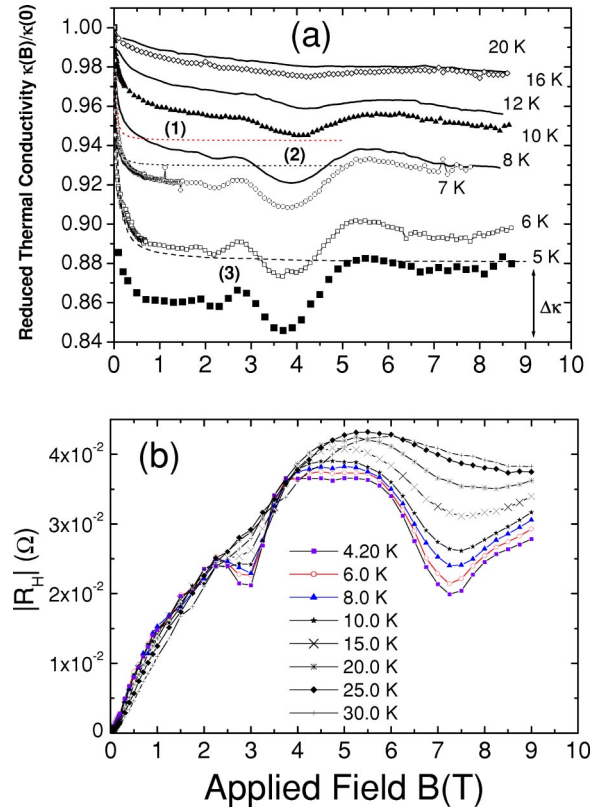


FIG. 2. The same data as in Fig. 1 but plotted in a linear field scale.

Can we understand the decrease with field and the oscillations observed in  $\kappa(B)$  within the Wiedemann-Franz relation? To answer this question we proceed as follows. We assume that the thermal transport of graphite is given by two contributions<sup>1</sup>

$$\kappa = \kappa_p(T) + \kappa_e(T, B), \quad (1)$$

where  $\kappa_p$  is due to the phonons, the contribution of the atomic lattice with the appropriate lattice anisotropy, and  $\kappa_e$

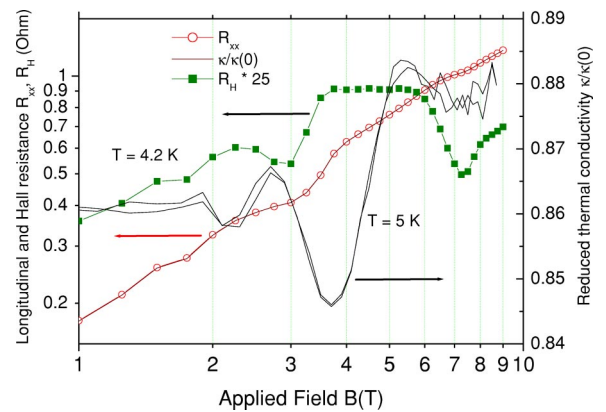


FIG. 3. Longitudinal and Hall resistances (left scale) and the reduced thermal conductivity (right scale) as a function of magnetic field applied parallel to the  $c$  axis. The two measured lines of the thermal conductivity data were taken increasing and decreasing field. Within the error there are no significant hysteresis.



due to conduction electrons. Usually one assumes that the field dependence of the thermal conductivity is given only by the electronic part  $\kappa_e(T, B)$ , which can be estimated with the WF relation. This universal relation relates the electrical resistivity  $\rho(T, B)$  with the thermal conductivity due to electrons by

$$\frac{\kappa_e \rho}{T} = L_0, \quad (2)$$

through the universal constant  $L_0 = 2.45 \times 10^{-8} \text{ W } \Omega \text{ K}^{-2}$  at low enough temperatures. One can recognize easily the difficulty to measure accurately the field dependence of the electronic contribution to the thermal transport above  $\sim 4$  K due to the small electronic contribution. From Eq. (2) we expect for well ordered HOPG samples at zero field a ratio between the electronic and total thermal conductivity  $\kappa_e / \kappa < 0.15$  at 5 K, and  $< 0.05$  at 10 K. Literature data are in general in good agreement with these estimates.<sup>17,24</sup>

The relation (2) holds strictly for elastic or quasielastic electron scattering and therefore the range of validity is usually set, either at low enough temperatures where the resistivity is temperature independent (impurity scattering dominates), or at high enough temperatures where the electron-phonon scattering is large.<sup>27</sup> For the sample measured in this work, the temperature dependence of the electrical resistivity indicates a saturation below 10 K (curves for a similar sample can be seen in Refs. 6,28) and therefore at  $T \leq 10$  K we expect to be roughly in the validity range of the WF law. From the measured field dependence of the electrical resistivity we can calculate the relative change of the total thermal conductivity at a fixed temperature as

$$\frac{\kappa(B)}{\kappa(0)} = \frac{\kappa_e(B) - \kappa_e(0)}{\kappa(0)} + 1, \quad (3)$$

assuming that the phonon conductivity does not depend on magnetic field. In Fig. 1(a) we show three curves calculated with Eqs. (3) and (2) using the measured  $\rho(B)$  at 10 K [curves (1) and (2)] and 6 K (3). Curve (1) was obtained with the same parameters as (2) but with  $L_0 = 2.0 \times 10^{-8} \text{ W } \Omega \text{ K}^{-2}$ , assuming a decrease of  $L_0$  due to the possible influence of the inelastic scattering.

From the comparison between the computed curves and the experimental ones one would tend to conclude that Eqs. (2) and (3) provide reasonably well the overall decrease of  $\kappa$  with field, within the geometrical errors in the measurement of both conductivities. Nevertheless we should note that the WF law and Eq. (3) do not reproduce accurately the measured field dependence, see Fig. 1(a). Due to the electrical resistivity increase (a factor  $\sim 100$  from zero field to 1 T at  $T \leq 10$  K) the electronic contribution, according to the WF law, becomes negligible. Therefore the electronic contribution to  $\kappa$  should be negligible, e.g., at  $B > 0.4$  T and  $T = 10$  K, and a saturation of  $\kappa$  at larger fields is expected. This is not observed experimentally. This means also that the relatively small oscillations observed in the longitudinal resistivity above 1 T that accompany the features in the Hall effect (see Fig. 2) should not affect  $\kappa$  according to Eq. (2) in contrast to the experimental results, see Fig. 1(a). These re-

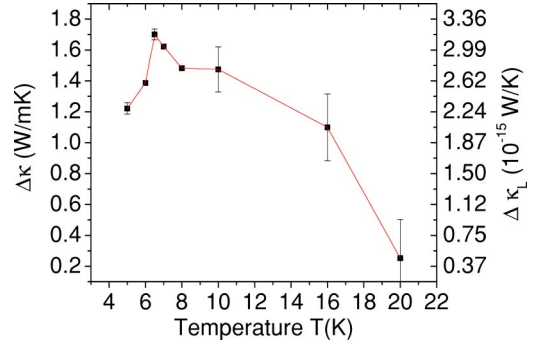


FIG. 4. The thermal conductivity difference between the minimum (at  $B \approx 3.7$  T) and maximum (at  $B \approx 5.5$  T)  $\Delta \kappa$  (see Fig. 1) as a function of temperature. The right scale provides the estimate values per graphene layer (within a geometrical error of  $\sim 30\%$ ).

sults clearly indicate that the WF law in its original form fails to explain the field dependence of the thermal conductivity in the QL of graphite and in the measured temperature range.

For any future comparison with theory it may be useful to provide the absolute value of the oscillation amplitude between minimum and maximum as defined in Figs. 1(a). Figure 4 shows the experimental  $\Delta \kappa$  obtained from Figs. 1(a) or 2(a). It shows a tendency to saturation around  $\Delta \kappa \sim 1.5 \text{ W/mK}$  for  $T < 10$  K. The oscillation amplitude per graphene layer can be estimated from  $\Delta \kappa_L \sim \Delta \kappa a / N$ , where  $a$  is the distance between layers and  $N$  the number of layers measured in parallel in our experiment within the  $60 \mu\text{m}$  thickness of the sample. Using this approximation we obtain  $\Delta \kappa_L \sim 3 \times 10^{-15} \text{ W/K}$  for  $T < 10$  K.

In the following we discuss our results taking into account relevant work. A recently published theoretical work calculated the thermal conductivity of a 2D electron gas at low temperatures and in a quantizing magnetic field.<sup>29</sup> Although some of the assumptions done there may not be valid for graphite, it is interesting to note that this work shows that the WF law is violated for small Landau level broadening and at low enough temperatures when the diffusion mechanism dominates. According to this work the deviations from the WF law are due to the energy derivatives of the longitudinal electrical conductivity. At low enough temperatures and small level broadening the numerical results show a “two-peak” behavior, i.e.,  $\kappa$  as a function of magnetic field shows two maxima in the field region between two neighboring plateaus in the Hall resistance. A similar result has been obtained in previous theoretical work.<sup>30,31</sup> In our case, however, we obtain the striking result that  $\kappa_e(B)$  decreases in this field region whereas increases with field in the plateau region and reaches a maximum at the end of the corresponding plateau, see Fig. 3. If we would have localized QP in the field region of the plateau we would naively expect a decrease of  $\kappa_e$ . On the other hand, the opposite behavior may be also possible, i.e., an increase of  $\kappa_e$  with field, if the density of interacting QP would decrease in this field region and the main scattering mechanism is given by QP-QP scattering, as in the case of high-temperature superconductors.<sup>32</sup> In this case the theoretical description of the thermal conductivity may become more complicated to handle.

According to recently published theory,<sup>7,33</sup> a magnetic field applied perpendicular to the graphene planes opens an insulating gap in the spectrum of Dirac fermions, associated with an electron-hole pairing, leading to the excitonic insulator state below a field dependent transition temperature. The experimental value of the critical field of the field-driven metal-insulator transition in graphite is  $\sim 50$  times smaller than the predicted in Refs. 7, 33. The discrepancy can be understood, however, assuming that the Coulomb coupling, given by the dimensionless parameter  $g = 2\pi e^2 / \epsilon_0 v$  ( $\epsilon_0$  is the dielectric constant),<sup>7,33</sup> drives the system very close to the excitonic instability. In this case, the threshold field  $B_c$  can be well below the estimated value of 2.5 T. The above analysis, together with the experimental evidence that only the perpendicular component of the applied field drives the MIT,<sup>9</sup> appear to support the theoretical expectations of a field-induced excitonic insulator state in graphite. In this case and according to Ref. 34 we would expect a monotonic decrease of  $\kappa_e(B)$  with field and a kink at  $B \sim B_c$  with a plateau region in the insulatinglike state of the QP at  $B > B_c$ . In the temperature range of our experiments the results do not show a clear kink at  $B \sim 0.1$  T nor a plateau at higher fields and  $T \geq 6$  K, see Fig. 1(a), although one may tend to recognize it at  $T = 6$  K and  $B \sim 0.5$  T when the data are plotted in a linear field scale, see Fig. 2(a). At the temperature limit of our system (5 K) the density of points at  $B < 1$  T is too low to assure the existence or nonexistence of a kink. Measurements at lower temperatures are needed to enhance the relative contribution of the QP to  $\kappa$  and check whether the predicted feature is observable. This issue will be studied in the future.

Can the oscillations in  $\kappa(B > 1$  T) be due to the lattice contribution  $\kappa_p(B)$  via electron-phonon interaction as, for

example, in antimony?<sup>35</sup> The temperature dependence of  $\kappa \propto T^{2.4}$  at 5 K  $< T < 30$  K speaks for phonon scattering by grain boundaries and not by electrons.<sup>1</sup> The inelasticity parameter  $\eta = v / \lambda \omega_c$  (here  $v$  is the sound velocity,  $\lambda$  the magnetic length and  $\omega_c$  the cyclotron frequency), that provides an estimate of the efficiency of the electron-phonon scattering, is  $\sim 0.01$  at 4 T for graphite. Therefore, unless there is an intersection of Landau levels that favors acoustic phonon transitions,<sup>36</sup> it does not seem that the phonon-electron scattering can be significantly enhanced at high fields in graphite. The overall correlation of  $\kappa(T, B)$  with the Hall resistivity indicates that the origin of the oscillations in  $\kappa(B)$  should be related to a pure QP phenomenon.

In summary, high-resolution measurements of the magnetic field dependence of the thermal conductivity in a quasi-2D sample of graphite show clear oscillations in the quantum limit. The Hall effect for the same sample shows quantum Hall effect features which are correlated to the features observed in  $\kappa$ . With the measured longitudinal electrical resistivity we show that the observed oscillations in  $\kappa$  cannot be explained with the original WF law. Lower temperature measurements as well as an appropriate theoretical framework for graphite are highly desirable.

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