Effect of pressure on the parameters of the energy spectrum of graphite

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The dependence of the extremal sections and of the effective masses of the carriers of pyrolytic graphite were investigated at pressures up to 17 kbar and temperatures 2-4 K. A nonlinear variation of the extremal sections of the Fermi surface was observed at high pressures, and it is shown that this variation becomes linear when plotted in the coordinates $S$ and $V$, where $S$ is the specimen volume. The logarithmic derivatives with respect to pressure are determined. The logarithmic derivatives with respect to pressure are calculated for the parameters of the electron energy spectrum of graphite, and a comparison is made with the results of other experimental and theoretical studies.

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INTRODUCTION

Graphite and synthetic carbon materials on its base have a number of unique properties and have recently found extensive practical use. The distinct layered crystal structure of graphite causes all its properties to be highly anisotropic, since the interaction of the carbon atoms within each layer exceeds by one order of magnitude and interaction between atoms in neighboring layers.

A universally accepted model of the energy spectrum of graphite is at present the Slonczewski-Weiss model, in which the interaction between layers is taken into account by perturbation theory. The semimetallic properties are due to the presence of a weak interaction between atoms of different layers, an interaction described by the parameters $\gamma_i$ ($i = 1-6$), which are small compared with the parameter $\gamma_1$ that corresponds to the interaction of the atoms in the layer. The Fermi surface of graphite consists of electron and hole parts that are strongly elongated along the $\Gamma$-K-H edge of the Brillouin zone.

In the Slonczewski-Weiss model, the effective masses $m^*$ and the extremal sections of the Fermi surface in the basal plane perpendicular to the hexagonal c axis of the crystal lattice are respectively described by the following formulas:

$$m^*(\vec{K}) = \frac{4}{3} \frac{A}{h^2} \frac{c}{v_F} \cos \frac{\pi}{6} \left(1 + \frac{\delta_2}{\delta_1} \cos \frac{\pi}{6} \right),$$

$$m^*(\vec{V}) = \frac{4}{3} \frac{A}{h^2} \frac{c}{v_F} \cos \frac{\pi}{6} \left(1 - \frac{\delta_2}{\delta_1} \cos \frac{\pi}{6} \right),$$

$$S_v = 3\pi^2 \frac{h^2}{m^*} \frac{c}{v_F} \cos \frac{\pi}{6} \left(3 - \frac{2\delta_2}{\delta_1} \cos \frac{\pi}{6} - \frac{\delta_2}{\delta_1} \cos \frac{\pi}{2} \cos \frac{\pi}{6} \right),$$

$$S_s = 3\pi^2 \frac{h^2}{m^*} \frac{c}{v_F} \cos \frac{\pi}{6} \left(\frac{\delta_2}{\delta_1} \cos \frac{\pi}{2} \cos \frac{\pi}{6} \right),$$

where $\theta = \frac{k}{\sqrt{2}}$, $k$ is the wave number reckoned from the point $K$ of the edge of the Brillouin zone, $c_1$ and $c_2$ are the parameters of the graphite crystal lattice ($c_1 = 6.708 \text{ Å}, c_2 = 2.462 \text{ Å}$ for single-crystal graphite), $S_v$ is the maximum section of the electron part of the Fermi surface, $S_s$ is the minimum section of the hole part of the Fermi surface of graphite near the point $H$, $\delta_1$ is the Fermi energy, $\delta_2 = \cos \theta = (\sin \theta)^2$, and $\cos \theta = \sqrt{1 - (\sin \theta)^2}$. The accuracy of the cited expressions is $\pm 10%$. The
values of the parameters \( y_1 (i=0-6) \) and \( y_2 \) (in electron volts), determined from the data of oscillation, galvanomagnetic, and optical measurements, are given in Table I. The sets of the parameters are designated by the indices introduced in the paper of Dillou, Spain, and McClure: A—the simplified model of Anderson et al. \(^1\) where \( y_1 = y_2 = 0 \); B and C—the models of McClure et al. \(^1\), where the parameters \( y_1 \) and \( y_2 \) are fixed while the remaining parameters are chosen for a best fit to the experimental results under the assumption that the number of electrons \( N \) is equal to the number of holes \( P \) (the difference between cases of B and C is due to the fact that in the experiments we observed two frequencies of de Haas–Van Alphen oscillations pertaining to small sections of the Fermi surface of graphite: 

\[ E_n = -0.70 \times 10^{-6} \right \angle \left \langle n \right \rangle^2 \text{ ev} \]  

model B corresponds to the case of the larger section and model C (to the smaller one); J—the model of Johnson and Dresselhaus, \(^6\) where the parameters were obtained from optical measurements; N—the model of Nagayoshi et al. \(^3\).

Considerable interest attaches to an investigation of the influence of pressure on the energy spectrum of graphite. A distinguishing feature of graphite is the large anisotropy of the compressibility \( c_i \) (\( c_0 \) is the compressibility in the basal plane, \( c_2 \) is perpendicular to the layers). Therefore the hydrostatic pressure on the crystal is practically uniaxial along the \( c \) axis.

Galvanomagnetic measurements were made earlier \(^7\) at high temperatures \(-300-400 \text{ K}\) on graphite specimens at pressures up to 5 kbar, and the pressure dependences of the parameters \( y_1 \) and \( y_2 \) were calculated. Subject to a number of simplifying assumptions, the accuracies of \( 3 \text{ ln} N/3p \) and \( 3 \text{ ln} P/3p \) are estimated by the authors at \(-20 \) and \(-100\%\), respectively.

Anderson et al. \(^3\) and Balakirev and Fisher \(^1\) investigated quantum oscillation effects in graphite at helium temperatures and at pressures up to 4 and 8 kbar, respectively. The extremal cross sections obtained in these studies and their logarithmic derivatives with respect to pressure are listed in Table II. The pressure dependences of the effective masses were not measured in Refs. 3 and 11, where data by others \(^3\) were used to calculate the pressure dependences of the parameters of the Sonczewski–Weiss model. \(^1\) However, the parameters of the energy spectrum of graphite depend strongly on the temperature. For this reason the values of the parameters \( y_1 \) and \( y_2 \) and of their derivatives with respect to pressure, determined at high temperatures, can apparently not be used to calculate the pressure dependences of the remaining parameters from the data on the oscillation effects at low temperatures.

For a correct determination of the dependences of the parameters \( y_1 (i=1-6) \) and \( y_2 \) on the pressure it is necessary to use data obtained for the same specimen at identical temperatures. It is seen from expressions (1)-(5) that by measuring the pressure dependences of the extremal sections and of the effective carrier masses it is possible to determine the logarithmic derivatives of the parameters \( y_1 (i=1-6) \) and \( y_2 \) with respect to pressure (the parameter \( y_1 \), which characterizes the interaction of the atoms in the basal plane, can be assumed in first-order approximation, in view of the low compressibility of \( c_2 \) of graphite, as independent of pressure). To our knowledge, these measurements were performed by us here for the first time ever. In addition, it was of interest to carry out investigations in the pressure region above 10 kbar, where x-ray diffraction data \(^1\) indicate that the distance between the graphite layers has an essentially independent pressure dependence.

**MEASUREMENT TECHNIQUE, SPECIMENS**

We have investigated the quantum oscillations of the magnetic susceptibility and of the electric resistivity of samples of pyrolytic graphite at pressures up to 17.3 kbar in the temperature interval 2.1-4.2 K in fields up to 55 kOe, and also the behavior of the differential magnetic susceptibility \( \chi \) at \( p = 1 \) bar in fields up to 85 kOe.

The magnetic susceptibility was measured by a modulation technique.\(^2\) To determine the absolute value of \( \chi \) we used as references the values of the magnetic susceptibility of Bi. The measurement coils 2 x 1100 turns of PEV-0.02 wire on a teflon core) were placed directly in a high-pressure chamber channel of 4.5 mm diameter. The measurements were made in a fixed-pressure chamber similar to that described by Inkevich.\(^14\) The pressure-transmitting medium was a mix-

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**TABLE I.**

<table>
<thead>
<tr>
<th>Parameter</th>
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<th>C</th>
<th>D</th>
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**TABLE II.**

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<th>( \text{ln} P/3p )</th>
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<tr>
<td></td>
<td>2.36</td>
<td>0.044</td>
<td></td>
</tr>
</tbody>
</table>

References

1. Anderson et al.
2. Balakirev and Fisher
3. Nagayoshi et al.
4. Johnson and Dresselhaus
5. McClure et al.
6. Sonczewski and Weiss
7. Anderson et al.
8. Balakirev and Fisher
tire of 70% water-free kerosene and 30% transformer oil. The pressure in the chamber was determined from the shift of the superconducting transition temperature of a thin sensor placed in the immediate vicinity of the sample. The magnetic fields were produced by superconducting solenoids. We recorded simultaneously the oscillation curves at scans proportional to the direct (0) and reciprocal (1/H) magnetic fields. The harmonic analysis of the oscillation curves recorded in the reciprocal field were carried out with a computer by the procedure described by Moshchalkov. The effective masses of the carriers \( m^* \) were determined from the temperature dependences of the oscillation amplitudes (the determination of \( m^* \) is discussed in greater detail below).

Quasi-single-crystal samples of pyrolytic graphite were obtained by deposition of carbon from the gas phase at a temperature 2100°C followed by heat treatment under pressure at ~5000°C. The quasi-single-crystals prepared in this manner had a block structure (block size in the basal plane \( ~0.1 \) mm) and a lattice parameter \( c_0 = 6730 \) Å somewhat higher than that of single-crystal graphite (lattice parameter in the basal plane \( a_0 = 2463 \) Å). According to the data of galvanomagnetic measurements in strong magnetic field, the carrier decompensation in the investigated samples amounts to \( P - N \approx 3 \times 10^{12} \) cm\(^{-2} \), i.e., \(-9\%\) of the total carrier density.

To investigate the de Haas-van Alphen effect we used cylindrical samples \( 4 \) mm long in the c-axis direction and \( ~3 \) mm diameter. The samples used for the study of the galvanomagnetic effects were thin \( (~0.1 \) mm) plates with potential and Hall contacts to which copper electrodes were secured with silver paste. The accuracy of the calibration of the magnetic field \( B \) was \(-0.5\%\), that of the determination of the large Fermi-surface sections \( S \) was \(-1\%\), that of the pressure \( p \) was \(-0.1 \) kbar, of the absolute magnetic susceptibility \( \chi \) was \(-2\%\) relative to the calibration, and \( 3 \) Hz/ \( B p \) \(-2\%\).

MEASUREMENT RESULTS

Figure 1 shows plots of the oscillating part of the differential magnetic susceptibility \( \chi \) and of the magnetoresistance \( \rho \) against the magnetic field \( B \) (except for special cases, all the results that follow pertain to this magnetic-field direction). Figure 2 shows plots of the spectral density of the de Haas–van Alphen oscillations at various pressures.

The extremal sections and the effective masses at pressures \( p \) bar, as well as the logarithmic derivatives of these quantities with respect to pressure \( (p < 10 \) kbar) obtained by us and by others, are listed in Table II.

The magnetic-susceptibility oscillations in fields stronger than 4 kOe constitute a superposition of two frequencies corresponding to the electron and hole parts of the Fermi surface, whereas the oscillations of

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**Figure 1.** Oscillations of magnetic susceptibility at pressures 4.9 kbar (1) and 17 kbar (2) and of the magnetoresistance at \( p = 1 \) bar in pyrographite at \( T = 2.1 \) K. H//c.

The magnetoresistance are monochromatic (Fig. 1). The cross section determined from the Shubnikov–de Haas effect amounts to \((4.85 \pm 0.08) \times 10^{-2}\) cm\(^2\)/sec at \( p = 1 \) bar and pertains thus to the hole part of the Fermi surface. No oscillations of the magnetoresistance from the electron part of the Fermi surface were observed. The angular dependences of the large extremal sections of the electron and hole Fermi surface \( S_e \) and \( S_h \), obtained by measuring the phase shift of the de Haas–van Alphen oscillations in fields up to 55 kOe, can be traced all the way to angles \( \phi > 70\)° and are similar in character to the angular dependences obtained for natural single-crystal graphite. It appears that by virtue of the high symmetry of the Fermi surface of graphite the random orientation of the single-crystal blocks in the basal plane has practically no effect on the results of these measurements.

Figure 3 shows the plots of the extremal sections \( S_e \) and \( S_h \) against pressure, determined from the de Haas–van Alphen and Shubnikov–de Haas oscillation frequencies. It is seen that at pressures \( p \) higher than 10 kbar these plots differ greatly from straight lines.

Figure 4 shows the absolute susceptibilities measured at \( p = 1 \) bar in fields up to 85 kOe. The arrows indicate the fields, calculated by Ono and Sugihara, corresponding to the intersections of the Fermi level with the Landau levels numbered \( n = 1–5 \) for the electron part and \( n = 1–3 \) for the hole part of the Fermi surface. As seen from this figure, in strong magnetic fields some paramagnetic maxima of the oscillations are quite well resolved, so that the effective masses \( m^* \) of the carriers can be calculated from the temperature depen-
The Landau levels numbered
Shubnikov-de Haas-van Alphen oscillations, of the effective masses of pyrographite cannot be used by him are inapplicable in the present case. We chose for the calculation of \( m^* \) the oscillations at various temperature by the method proposed by Moshchalkov, since the approximations used by him are inapplicable in the present case. The Dingle temperature \( T_D \) was determined from the Shubnikov-de Haas effect, which amounts to \( 6.8 \pm 0.2 \) K at \( p = 1 \) bar and increases with increasing pressure at a rate \( 0.18 \pm 0.04 \) K/kbar. The values of \( T_D \) are independent of temperature, within the limits of the measurement accuracy, in the 2-4 K region, so that it is correct to determine the effective masses from the temperature dependences of the oscillation amplitudes. The values \( 3 \ln \alpha_0/(p) = 0.0464 \) kbar\(^{-1} \) and \( 3 \ln \alpha_0/(p) = 0.0464 \) kbar\(^{-1} \) determined from the dependences of the relative shift of the electron and hole maxima of the de Haas-van Alphen oscillations as functions of pressures at \( p = 10 \) kbar \( \Delta S(p) \) is the field in which the corresponding oscillation maximum is observed at the pressure \( p \) agrees within the limits of error with \( 3 \ln \alpha_0/(p) \) and \( 3 \ln \alpha_0/(p) \) obtained from Fig. 3 (see Table II). This confirms that the oscillation maxima have been correctly assigned to the electron and hole parts of the Fermi surface.

Figure 5 shows the pressure dependences, calculated by the procedure described above from the temperature dependences of the de Haas-van Alphen oscillations, of the effective masses of the majority electrons \( (m^*_e) \) and of the majority holes \( (m^*_h) \), as well as a plot of \( m^*_h \) against pressure, obtained from an investigation of the Shubnikov-de Haas effect, where oscillations from only the majority holes were observed. It is seen that within the limits of errors \( \pm 5\% \) the \( m^*_h(p) \) plots obtained by both methods agree. The smaller scatter of the points on the \( m^*_h(p) \) plot compared with \( m^*_e(p) \) is due to the better resolution of the oscillation maxima from the Landau levels of the electrons.

The extremal sections and the effective masses of the carriers were determined both with increasing and decreasing pressures in the chamber; their variation was reversible within the limits of errors, and the cross sections and masses returned to their initial values after the removal of the pressure.

Since there is no meeting of minds in the literature concerning the sizes of the small sections of the Fermi surface of graphite and concerning the positions of these groups of carriers in the Brillouin zones, we investigated the oscillations of the magnetic susceptibility in weak fields at \( p = 1 \) bar and their angular dependence (see Fig. 6 and Table III). As seen from Fig. 6(b), the observed cross section increases with increasing angle \( \theta \) between the directions of the magnetic field and of the c axis more rapidly than \( 1/\cos \theta \). This seems to indicate that the section \( S_e \) belongs to the neck of the hole part of the Fermi surface near the point H of the Brillouin zone.

The obtained values of the sections \( S_e \) and \( S_h \) of the investigated samples at \( p = 1 \) bar are somewhat lower than those of single-crystal graphite (see Table III), and the absolute value of the susceptibility in weak fields, \( |x_{12}| \geq 7.0 \times 10^4 \) cgs esu/cm\(^3 \) is somewhat high-

![FIG. 3. Plots of the extremal sections \( S_e \) (1) and \( S_h \) (2) of pyrographite vs pressure, obtained in investigations of the de Haas-van Alphen (\( \sim 4.2 \) K, \( 4-2.1 \) K) and of the shubnikov-de Haas (\( \sim 4.2 \) K) effects; \( H \perp c \).

![FIG. 4. Bulk differential susceptibility \( \chi_b \) of pyrographite vs magnetic field \( H \perp c \) at \( p = 1 \) bar and \( T = 4.2 \) K.

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FIG. 6. Oscillations of the magnetic susceptibility in pyro-
graphite in weak fields at H < c and p = 1 bar (a), and plot of the ra-
tio $S(H)/S(0)$ for the small external section vs the angle $\theta$ be-
tween the directions of the magnetic field and the c axis (curve 1); curve 2 is a plot of $1/cos \theta$.

er (according to the data of Refs. 17 and 23, $\chi|_{\theta=0} = 6.8 \times 10^3$ cgs emu/cm$^3$ for single crystal graphite).

This is apparently due to the higher value of the lattice parameter $a_0$ of the investigated pyrographite samples. We note that beyond the ultraquantum limit ($H > 72$ kOe) the susceptibility remains diamagnetic and does not differ in order of magnitude from $\chi|_{\theta=0}$ (see Fig. 4). This result agrees with the data of Chouteau and Cota, who investigated the field dependence of the magnetization of pyrographite up to 150 kOe, but disagrees with the data of Wallace and Spain, who took no account of the contribution of the filled bands to the susceptibility.

**DISCUSSION OF RESULTS**

To calculate the logarithmic derivatives of the parameters $\gamma_i (i = 1-6)$ and $c_0$ with respect to pressure it is necessary to use, in addition to the pressure dependence of the four characteristics of the electron energy spectrum of pyrographite (two extremal sections of the Fermi surface and two carrier effective masses) a number of additional assumption based on considerations of the symmetry of the graphite crystal lattice.

Dillon, Spain, and McClure have considered three possible approximations for the connection between the logarithmic derivatives of the parameters $\gamma_i (i = 1-6)$ with respect to pressure. These approximation are based on the fact that small parameters $\gamma_i (i = 1-6)$ are the result of the overlap of wave functions that decrease exponentially at large distances. In approximation I

$$\frac{\ln \gamma_1}{\partial p} = \frac{\ln \gamma_1}{\partial p},$$

in approximation II

$$\frac{\ln \gamma_1}{\partial p} = \frac{\ln \gamma_1}{\partial p},$$

and in approximation III

$$\frac{\ln \gamma_1}{\partial p} = \frac{\ln \gamma_1}{\partial p}.$$

From Eqs. (1) and (2) and from the $m^2(p)$ and $m^2(p)$ dependences obtained in the present paper it follows that $\frac{\ln \gamma_1}{\partial p}$, which does not agree with approximations II and III. We have therefore used approximation I in the calculations.

The minimum section $S_2$ determined experimentally in the present study agrees well with the calculated one for the set of parameters $C$ of the graphite spectrum (see Table II); the parameter sets $A$, $B$, $I$, and $N$ yield approximately double the value of $S_2$.

The set of parameters $C$ describes well the values of the extremal sections and of the effective masses of single-crystal graphite. In the investigated pyrographite samples, the distance between layers is ~0.32, larger, but under pressure it decreases and at a certain pressure $p_0$ it becomes close to the interlayer distance of single-crystal graphite. The variation of the interlayer distance in graphite at pressures up to 16 kbar was investigated by Kabalkina and Vershchagin and is described by the following empirical formula:

$$\Delta \alpha(p)/\alpha(0) = -25 \times 10^{-9} \times 9.26 \times p,$$

where $p$ is the pressure in bars, $c_0(0)$ is the lattice parameter at $p = 1$ bar, $c_0(p)$ is the value of this constant at the pressure $p$, and $\Delta \alpha(p) = c(p) - c(0)$. The value of $p_0$ obtained for our samples from (11) is ~1.2 kbar.

Table III lists the extremal sections and effective masses calculated at $p = 1$ for the set of parameters $C$, and our data recalculated for a pressure $p_0 = 1.2$ kbar. The cross sections are given in units of $10^{-6}$ g$^2$cm$^2$/sec$^3$.

**TABLE III.**

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<th>$S_1$</th>
<th>$S_2$</th>
<th>$S_{2c}$</th>
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**TABLE IV.**

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[Calculation of ... ] [Exp. work] [Brutt et al. 1980]
FIG. 7. Extremal sections $S_1(1)$ and $S_2(2)$ at $H \parallel c$ vs change $\Delta \rho_c(p)$ of the lattice parameter of pyrographite.

parameters $C$ in approximation I [Eqs. (6) and (7)] and the corresponding experimental values for $p < 10$ obtained by us here from Eqs. (1)–(5) in approximation I for the same set of parameter. To calculate the experimental values of the logarithmic derivatives of the spectrum parameters, we used the quantities

$$\delta \ln S/p, \delta \ln S/p, \delta \ln m_c/p, \delta \ln m_c/p, \delta \ln S_c/p = \langle S(p) - S(0) \rangle / \langle p - p_0 \rangle m(p),$$

The accuracy of the values $\delta \ln S/p (n=1-5)$ and $\delta \ln m_c/p$ obtained here is $-20\%$, and is limited mainly by the error in the determination of the effective carrier masses and by the accuracy of the initial equations (1)–(5). As seen from Tables III and IV, our results agree sufficiently well with the calculated data, obtained within the framework of the Slonczewski-Weiss model.

The deviation of the plots of $S$ and $S_c$ vs $p$ from straight lines in the pressure region above 10 kbar, which to our knowledge was observed here for the first time ever, is apparently due connected with the circumstance that the interlayer distance in graphite has a nonlinear pressure dependence. Figure 7 shows plots of $S$ and $S_c$ against the change $\Delta \rho_c(p)$ of the lattice parameter calculated from Eq. (11). It is seen from the figure that these plots are close to linear in a wide range of variation of the interlayer distance (up to $-5.5\%$). Owing to the low compressibility $\alpha$, this means that the sections of the Fermi surface depend linearly also on the volume $V$ of the sample.

Translated by J. G. Adashko