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Temperature Dependence of Shubnikov-de Haas Oscillations in Graphite Intercalation Compounds

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The Shubnikov-de Haas oscillations in the magnetoresistance are used to investigate the electronic properties of $FeCl_3$-graphite intercalated compounds. From the field and the temperature dependence of the amplitudes of the dominant frequencies of these oscillations the effective masses, the Dingle temperatures, the relaxation times, and the carrier mobilities are determined for stages 2 and 5. Details of the stage dependence of these oscillations at several different temperatures will be presented and discussed.

1. INTRODUCTION

The magnitude and angular dependence of the amplitude of de Haas-van Alphen (dHvA) and Shubnikov-de Haas oscillations (SdH) have been widely used to determine the shape of Fermi surfaces in solids. The temperature and magnetic field dependence of the amplitude of these oscillations determine the effective mass, scattering times, and the mobility of charge carriers. Therefore, in this work, we have used the experimental data of the magnetoresistivity SdH oscillations to investigate the electronic transport of several stages (stage 1 is one graphite layer sandwiched between two consecutive intercalant layers) of $FeCl_3$-graphite intercalated compounds (GIC's).

SdH and dHvA oscillation effects have been measured for several donor and acceptor GIC systems. Bender and Young [1] were the first to observe SdH oscillations in bromine-GIC. As more well characterized intercalated graphite samples became available, quantum oscillatory techniques were used extensively to determine the shape of the Fermi surfaces, transport coefficients, and the carrier density which leads to the charge transfer rate between the intercalant and the graphite [2-7]. Quantum oscillatory phenomena can also be observed in magnetothermal measurements [4,7]. One of the still unresolved problems in quantum ocillatory data is that while some GIC's exhibit stage dependent oscillations [6,8] others have shown stage independent oscillations [7].

2. EXPERIMENTAL

The $FeCl_3$-GIC samples were prepared using a standard two-zone furnace technique [9] where stage index was controlled by the temperature difference between the graphite host (HOPG) and the $FeCl_3$ powder. The samples were in the form of thin rectangular plates of dimensions 1.5x0.5x0.1 cm^3. Most of the samples measured were characterized by means of the Mössbauer effect, details are reported in references [10-11]. Samples, for resistivity measurements, of approximate dimensions 10μm by 5x5 mm^2 were peeled out from the intercalated samples using scotch tape. The leads were attached to the sample in the conventional four-prope configuration. Contacts were made on the sample using GC conductive silver print.

3. Results and Analysis

The data shown in Fig. (1) and Fig. (2) are the in-plane transverse magnetoresistivity ($\Delta\rho/\rho$) versus the applied magnetic field at room and liquid nitrogen temperatures, respectively, for stages 2, 3, 5, and 9. The numbers on the graph refer to the stage index. As shown in the figures, in all stages the magnetoresistances do not saturate up to the highest available field. The saturation field depends on $\omega\tau$ (ω is the cyclotron frequency and τ is the effective relaxation time) and one can qualitatively relate the lack of saturation to a long relaxation time in this GIC system. The quantity ($\Delta\rho/\rho$) for stages 2, 3, 5, and 9 at helium temperature is plotted versus the applied field in Fig. (3). The data also do not saturate at this temperature, as expected, however SdH oscillations are observed in all the stages. Stage 5 exhibits the most predominant oscillations among all other stages, which is related to a long relaxation time or low Dingle temperature ($T_D = \frac{2\hbar}{\pi k\tau}$) due to the two dimensional nature of this stage [14].

The temperature dependence of the SdH oscillations is shown for stages 2 and 5 in figures 4 and 5, respectively. The data shown in these figures are the transverse magnetoresistance versus the applied field at several different temperatures. As shown in the figures the oscillations persist at temperatures as high as 33 K. The effective mass, Dingle temperature, relaxation time, and the carrier mobility were determined by using the following equations:

$$m^* = \frac{\hbar eH}{2\pi^2 k_B}\frac{dln(A/T)}{dT} \tag{1}$$

$$T_D = \left(\frac{-e\hbar}{2\pi^2 m^* k_B}\right)\frac{d[ln(A/\sqrt{H})]}{d[(1/H)]} - T \tag{2}$$

$$\tau = \frac{\hbar}{\pi k T_D} \tag{3}$$

$$\mu = \frac{e\tau}{m^*} \tag{4}$$

H is the field value at which the amplitudes A are measured.

The data in table I are the dominant frequencies in stages 2 and 5, in stage 5 the amplitude of F_3 is relatively large in comparison with the other frequencies. In table II the calculated parameters, the effective masses, the Dingle temperatures, the relaxation times, and the carrier mobilities are listed for stages 2 and 5. In stage 5 we were not able to extract these parameters at the lower frequencies. At stage 5 we expect the dominance of one kind of carrier because the donoted electrons from HOPG are not enough to fill the intercalant vacancies. The data of stage 2 indicate that there are at least three distinguished Fermi surface extrema with different cross-sectional areas. In conclusion, at helium temperature all the stages exhibit SdH oscillations and the amplitude of theses oscillations are relatively large for stage 5 indicating longer relaxation time which is consistent with previously reported data [12].

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Table I
The observed dominant frequencies in stages 2 and 5 of $FeCl_3$.

	F_1	F_2	F_3	F_4
Stage 2	82	284	532	-
Stage 5	-	213	311	493

Table II
The calculated physical parameters for stages 2 and 5 $FeCl_3$.

	Parameter	F_1	F_2	F_3	F_4
Stage 2	m^*/m_o	0.077	0.192	0.221	-
	T_D(K)	6.53	29.8	37.3	-
	τ (10^{-13}S)	3.72	0.815	0.651	-
	μ (m^2 / V S)	0.850	0.075	0.052	-
Stage 5	m^*/m_o	-	-	-	0.158
	T_D(K)	-	-	-	23.7
	τ (10^{-13}S)	-	-	-	1.03
	μ	-	-	-	0.115

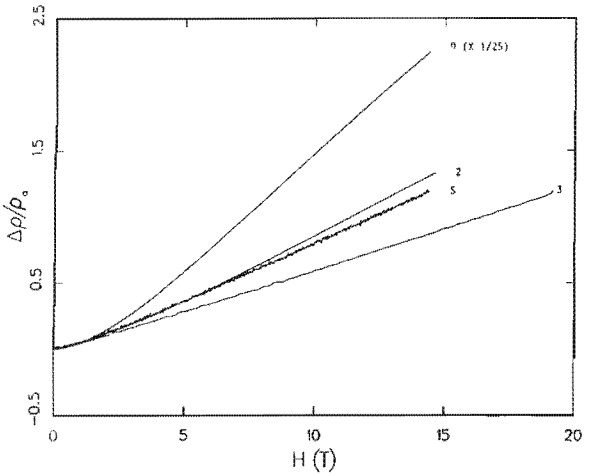

Fig. (1) Plot of ($\Delta\rho/\rho$) versus the field at 300 K for stages 2, 3, 5, and 9 of $FeCl_3$-GIC.

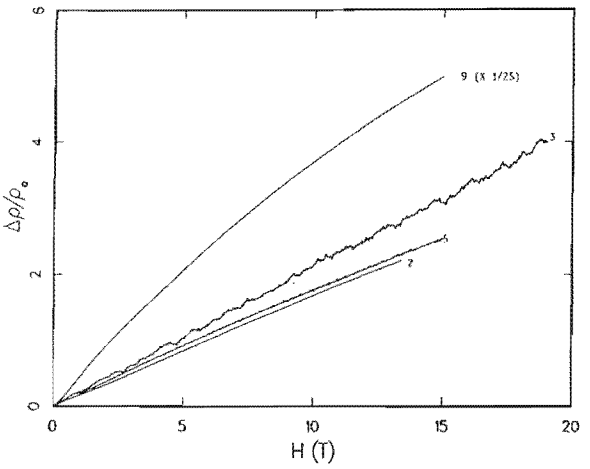

Fig. (2) The same plot as in figure (1) but the data here were taken at 77 K.

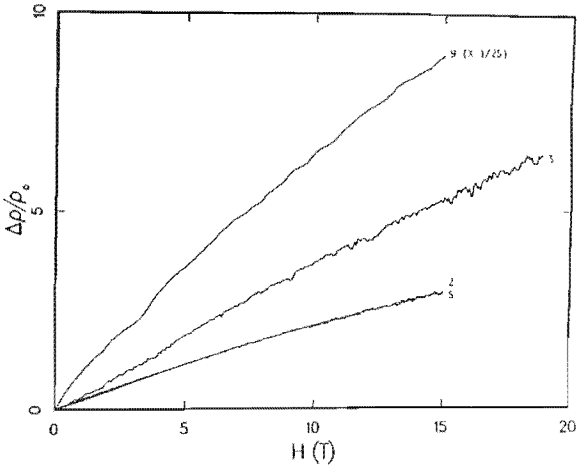

Fig. (3) The same plot as in figure (1) but the data here were taken at 4.2 K.

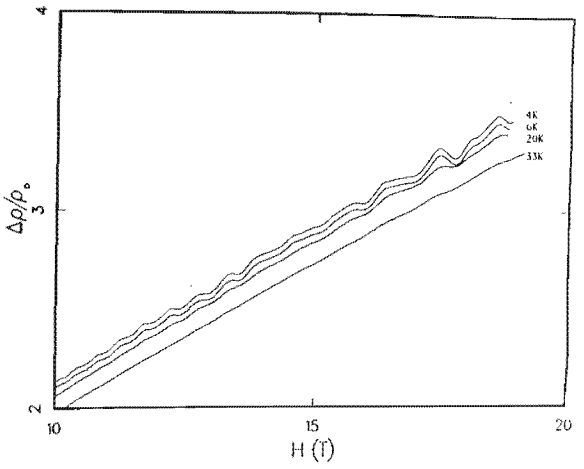

Fig. (4) The SdH oscillations are plotted versus the applied field at several different temperatures for stage 2 $FeCl_3$-GIC.

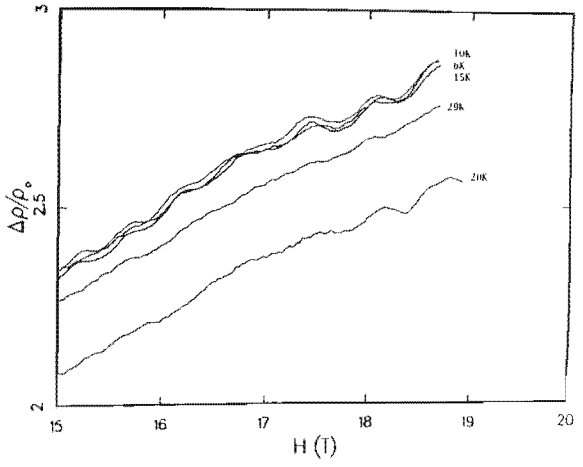

Fig. (5) The SdH oscillations are plotted versus the applied field at several different temperatures for stage 5 $FeCl_3$-GIC.