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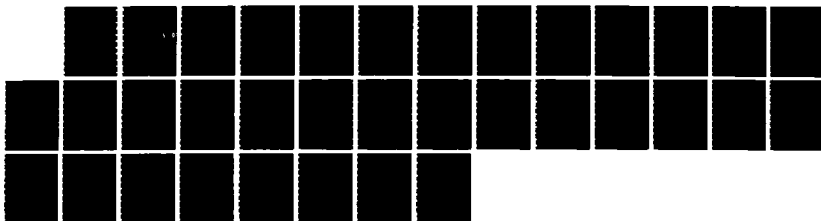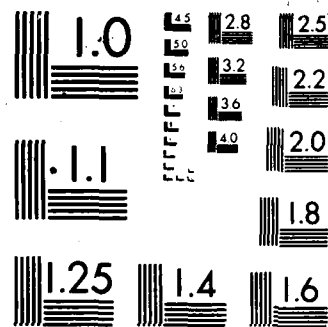
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SUPERLATTICE EFFECTS IN GRAPHITE INTERCALATION COMPOUNDS

R.S. Markiewicz
Northeastern University, Boston, Mass. 02115

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20. ABSTRACT *(Continue on reverse side if necessary and identify by block number)*

Our research was motivated by the observation of anomalous mixing frequencies in the deHaas-van Alphen spectra of graphite intercalation compounds. We have made a very detailed analysis concentrating on compounds containing Br_2, which has extremely large anomalies. Three sources can produce these anomalies:

- In many of our original samples, the very strong magnetic torques caused the samples to tilt in a magnetic field, and led to a <u>torque instability</u>, causing discontinuous jumps of sample position and hysteresis

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20. Abstract (continued)

→ - In rigidly mounted samples, _interband transfer_ causes significant lineshape distortion, which can be quantitatively accounted for by a simple theory. This theory confirms that the intercalation compound behaves as a true two-dimensional hole gas, with energy gaps between successive Landau levels.

- _Condon domain formation_, a phase transition within the electron gas, remains the most intriguing possibility. We have shown that in two-dimensions it is closely related to the quantum Hall effect. While this mechanism has not yet been observed, we can now predict which intercalation compounds it will appear in.

1.   Summary of Research Goals and Plans

i) To thoroughly examine the recently discovered Condon phase in Br_2 (and possibly other) intercalation compoun s, both to understand the soliton (domain wall) dynamics and to explore the predicted connections between this phase and the quantum Hall effect.

ii) To use magnetooscillations and x-ray diffraction as probes to study superlattice formation in graphite intercalation compounds, particularly in those situations in which magnetic breakdown or field-induced phase transitions suggest that the superlattice may be generated by Fermi surface instabilities.

iii) To search for pressure-induced phase transitions in intercalation compounds, especially via resistivity probes.

2.   Status of the Research Efforts

The basic premise of this research has been that graphite intercalation compounds, because of their extreme anisotropy, should display interesting two-dimensional behavior. To explore this we have concentrated primarily on stage 2 compounds with Br_2 as the intercalant, since this compound displays exceptionally intense magnetization oscillations. These oscillations were found to display a number of quite anomalous properties, including extremely sharp jumps, hysteresis, and a high-frequency "ringing" response which could be driven chaotic.[1] For a long time we interpreted these features as due to a "magnetic instability", which in a two-dimensional system can be considered as a Landau-level condensation. Based on more recent experimental evidence, we now believe that these are due instead to a different mechanism, a "torque instability", which shares many features in common with the magnetic instability. In this report, we will show how our new experiments led to this

reinterpretation, and that, when this instability is eliminated, the susceptibility data show clear evidence that the system is an ideal candidate for a two-dimensional hole gas. Indeed, it is the first two-dimensional gas-- electron, or hole--to display the energy gaps between successive Landau levels. The Landau level condensate remains an important theoretical idea. We will show that it provides interesting insights into the quantum Hall effect, and that it should be observable in other graphite intercalation compounds.

a) Torque Instability

Over this past summer, we carried out two new experiments on the Br_2-intercalation compounds, which caused us to question our earlier interpretation of Landau level condensation. In collaboration with J. Brooks (BU and National Magnet Lab), we made direct measurements of sample magnetization, M using a capacitance technique. These measurements of d.c. magnetization allowed us to observe even sharper structure than in the a.c. susceptibility. Indeed we observed discontinuous jumps in M(Fig. 1a). These were, however, of the opposite sign from what we might have expected. More direct evidence came from a second experiment -- optical studies. We found that in a magnetic field the luminescence and reflected light both show extremely large oscillations at the deHaas-van Alphen frequency in 1/B (Fig. 2). Subsequent study showed that the sample was physically moving in a magnetic field, thereby modulating the reflected light. This sample motion is caused by magnetic torque on the sample, which changes sign as the magnetization varies from para - to dia - magnetic. The torque effect is large enough to cause an instability, wherein the orientation of the sample jumps suddenly as the field is changed.

The torque instability[2,3] may be understood as follows. Our samples, sealed in glass ampoules, were held in position by pads of glass wool. The wool was not perfectly rigid but exerted a Hooke's law restoring force on the sample, which balanced the magnetic torque:

$$k\phi = -MB\sin\theta \qquad (1)$$

Here k is the effective spring constant, θ is the angle of tilt the sample makes in a nagnetic field, B, θ_O is the value of θ at B=0, and $\phi=\theta-\theta_O$. Since M is a function of $B\cos\theta$, Eq. 1 is an implicit function for ϕ. When B is lrge enough, ϕ can be multivalued. This is the torque instability. As B changes, ϕ must jump discontinuously between its allowed values, and there is a hysteresis in the jump position depending on the direction B is swept (Fig. 3). This is qualitatively very similar to the magnetic instability [3]. This jump will be reflected in all the properties of the sample. By directly measuring ϕ from the reflected-light image, we found that the changes were large enough that torque instability must exist, and indeed we could directly observe the sudden changes in ϕ. In a combined imaging and susceptibility (χ) experiment, we showed that the jumps coincided with sharp structure in χ. Finally we found that the torque instability provides a natural explanation for the sign at the jumps in M, and that the data could be reanalyzed in such a way as to eliminate the torque instability, and this essentially eliminated the jumps in M as well (Fig. 1b). Even the ringing we observed can be interpreted as an a.c. resonance of the sample motion.[4]

Landau gaps

We have been able to eliminate the torque instability by more rigidly clamping the sample into position. When this is done, all hysteresis effects are eliminated, and a well defined oscillatory pattern of χ is obtained which is reproducible from sample to sample (Fig. 4). The characteristic lineshape can be understood as due to the two whole bonds with strong interband transitions.[5] Fig. 4 shows that the fit to a simple theory is quite good. A key feature is the large negative spikes in the susceptibility. Ideally, these spikes would be infinitely sharp, but they are broadened out by density inhomogeneities ($\approx 0.4\%$ variation in density) in the sample. By using larger samples, we can reduce this broadening even further.

These spikes are the distinguishing features of a two-dimensional electron (or hole) gas--they occur because the Fermi level is forced to jump across an energy gap separating two Landau levels. The fit to the data shows that the Landau level bandwidth is 2.8meV, considerably less than the cyclotron energy, which separates successive Landau levels. We had earlier used the conductivity anisotropy to deduce an upper limit of 7meV for this band width[6]--the smaller value we have now found suggests that the c-axis conductivity is enhanced by interlayer hopping[7].

The observation of a Landau gap in a graphite intercalation compound is truly remarkable. It means that this is a genuine two-dimensional hole gas, despite the fact that the sample is a mm thick and consists of 10^6 coupled layers. Moreover, it is the only genuine two-dimensional hole or

electron gas known. Experiments on GaAs samples in which the quantum Hall effect has been observed have reached a consensus[8] that these materials do not have true Landau gaps. There is a considerable non-vanishing density-of-states throughout the gap. Fortunately, all that is necessary for the quantum Hall effect is the existence of a mobility gap--a range of energies for which all electronic states are localized.

Conversely, both theory [9] and experiment [10] have shown that a quantum Hall effect should persist in a thick, multilayer sample, as long as a Landau gap exists. This strongly suggests that the quantum Hall effect should be observable in these or other related graphite intercalation compounds. Unfortunately, because of the large carrier density and the many coupled layers, the Hall steps will be very small and closely spaced--almost impossible to distinguish in our inhomogeously broadened samples. While improvements in the samples will enhance our chances of seeing the steps, we have been looking at magnetransport for indirect evidence of the quantum Hall effect.

Longitudinal magnetoresistance is quite unusual in the presence of the quantum Hall effect. Due to the tensor nature of the conductivity, when the Fermi level is at a gap between Landau levels both the resistivity ρ_{xx} and the conductivity σ_{xx} vanish. Carriers are confined into perfect cyclotron orbits, yet current can be transported losslessly due to edge currents--a cyclotron orbit which bumps into the sample surface will skip along that surface carrying a net current. Hence we expected to find $\rho_{xx}=o$ at the gaps. Instead we found quite large values of ρ at the values of field corresponding to gaps (Fig. 5c,d). This was explained to us by von Klitzing, when he

visited the Magnet Lab. It is due to our anomalous probe arrangement. Since these samples are extremely air sensitive, we mount them under liquid N_2, using pressure-contact probes for a quick-connect. The probes are pressed against the face of the sample, away from its edges, in contrast to the usual arrangement in the quantum Hall effect, where probes attach directly to the sample edge. It is only in this latter case where edge currents are effective in transporting current between the two current leads. Our sample, with leads isolated from the edges, is topologically similar to a Corbino disc, and hence will measure σ_{xx}^{-1}. Indeed, theoretical values of σ_{xx}^{-1}, are in quite good agreement with our data (Fig. 5a,b). By looking at the out-of-phase components of a.c. susceptibility measurements, it is possible to derive an independent measure of σ_{xx}^{-1}, one that can be made on sealed samples. Fig 5c shows that this is consistent with our d.c. measurement.

The observation that graphite intercalation compounds can be prototypical two-dimensional hole gases open up considerable range for future work. There have been a number of predictions for properties of these lower dimensional samples, including heat capacity, thermopower, cyclotron resonance, etc., which have been difficult to verify for lack of sufficiently large sample volumes. The massive graphite intercalation compounds change that, and we can look forward to many years of exploring new and unusual properties of these two-dimensional materials.

c. Landau level condensation

One very striking new property is the Landau level condensation, which we mistakenly identified in the Br_2-compounds. It is

still a very fascinating theoretical system, and it would be quite important
to find an experimental verification. From the good fit of the susceptibility
of Br_2-graphite to theory, it is now possible to predict where this condensate
should be found. Fig. 6 shows that a number of graphite intercalation
compounds (but not Br_2) would be good candidates.

The importance of this state was first pointed out by Vagner, et
al.,[11] who predicted that it would share a number of features in common with
the quantum Hall effect, including the vanishing of ρ_{xx}, and yet is driven by
an apparently very different physical mechanism. The condensation is a phase
transition of a two-dimensional election gas into a phase with two types of
domains, with different values of net magnetic field. It is closely related
to the Condon domain states [3,12] in a three-dimensional metal, in that both
are driven by a magnetic instability. The magnetization is a function of the
total magnetic field in the solid, M=M(B), which is in turn influenced by the
magnetization of the other electrons, B=H+4πM. When a self-consistent
solution for M is found, it is seen that M can be multivalued if $4\pi\chi>1$ (where
$\chi=\partial M/\partial B$). This same sort of mechanism drives ferromagnetism, but in that case
the factor 4π is replaced by a much larger number, I, due to exchange
interaction. In an electron gas, the instability criterion can be satisfied
because M has strong oscillations in field (deHaas-van Alphen effect), so χ
can be large even though M is small. In a two-dimensional electron gas, this
domain phase has an important additional feature. Since χ is constant in a
partly-filled level, the condition $4\pi\chi>1$ means that partly-filled Landau
levels are unstable, and the domains consist entirely of filled Landau levels.
The domains differ in whether the highest, N_{th}, level is full or empty. This
pinning of the Fermi level in a filled Landau level over an extended field

range is the feature which connects the domain phase with the quantum Hall effect.

This was a very puzzling result. There appeared to be two independent mechanisms which could produce a quantum Hall effect--actually three, since the integer and fractional quantum Hall effects seem to have little in common. We have analyzed the problem, and find that all three effects are different aspects of a common phenomenon. The underlying feature is the existence of a field-induced energy gap--either due to Landau levels or electron interaction. This gap leads to a potential energy lowering of the electrons. The means by which this energy lowering is accomplished depends on whether charged or magnetic excitations are lower in energy. There is a transition between these two types of excitation as a function of sample thickness,[13] with (magnetic) domains being energetically favored in thick samples, and charged or "vortex" excitations in thin samples. This transition is very similar to the domain-to-vortex transition in a superconducting plate [1,14] and there is a very close analogy between superconductivity and Landau level condensation.[13] Alternatively, the three phases can be viewed as dominated by localization (integer quantum Hall effect), electron interaction (fractional quantum Hall effect), or magnetic interaction (Condon domain effect).[15]

Observation of the domain phase and its unusual properties would be important in confirming this theoretical picture, and would open the tantalizing possibility of studying the domain to vortex transition. It is unfortunate that the present research did not attain its ultimate goal of discovering this phase, but the physics we have unravelled will be valuable in

clearing the path to its discovery.

d.   Other experiments

In x-ray studies of $FeCl_3$-intercalated graphite, we observed a striking phase transition--the first low temperature (T<150K) phase transition we have seen in an acceptor graphite intercalation compound. $FeCl_3$ has an intense, incommensurate x-ray spectrum in graphite, which is essentially temperature independent. However at low T (T<50K), we observed a single new peak which appeared in the spectrum. This peak showed a very unusual history dependence. It first appeared at about 50K then on cooling disappeared below 20K and did not reappear on warming. It would reappear on cooling, always disappearing when the temperature is low enough--Fig. 7 shows several temperature cycles. After a few cycles, however, its intensity diminished and it vanished, and did not recur on subsequent cooldowns. Similar phenomena have been observed in other systems--cooling causes stress-induced dislocations which wash out fine structure. Unfortunately, this particular feature appears to be very structure sensitive, and we have not been able to reproduce this effect with fresher samples. Such sensitivity was to be expected--a scan through this peak along the c-axis suggested a repeat distance of 100A, requiring quite large correlation volumes.

We developed a probe to study the giant magnetothermal oscillations predicted [16] to occur in a two-dimensional electron gas. Unfortunately, the results we obtained were also affected by the torque instability (Fig. 8) and will have to be repeated.

In our optical studies, we observed a low temperature luminescence from the intercalated Br_2-molecules, comparable to the luminescence found in Br_2 condensed in Ar[17], but broadened and blue-shifted (Fig. 9). The spectrum is very different from solid Br_2 at that temperature, so we identify it with intercalated Br_2, rather than Br_2 on the graphite or ampoule surfaces. This discovery could provide an important tool for analyzing the intercalant and its chemical properties. It is the first luminescence reported from any intercalation compound. We also found evidence for similar luminescence in $FeCl_3$-graphite, but that was not analyzed in as much detail.

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FIGURE CAPTIONS

Fig. 1 Torque instability in magnetization. (a) Raw data, tilt angle (ϕ) vs. field B, showing jumps and hysteresis (solid line = increasing B; dashed line = decreasing B).(b) After analysis, resulting curves of M vs. B show greatly reduced hysteresis and jumps. Residual hystersis is presumably due to motion of sample inside ampoule, and has been eliminated in more rigidly mounted samples.

Fig. 2 Torque instability in magnetoreflection. The variations in reflected light intensity, I , Have the periodicity of the deHaas-van Alphen effect, and are due to physical motion of the sample.

Fig. 3 *Explanation of torque instability.* The variables y,x, and p are normalized forms of ϕ, B^{-1} and MB/k (from Eq. 1). For p=2, ϕ is a multivalued function of B, and the dashed lines show the discontinuous and hysteretic variation of ϕ with B that would be observed experimentally.

Fig. 4 Oscillations of susceptibility in a rigidly clamped sample of Br_2 graphite. (a)=experiment, (b)=theory.
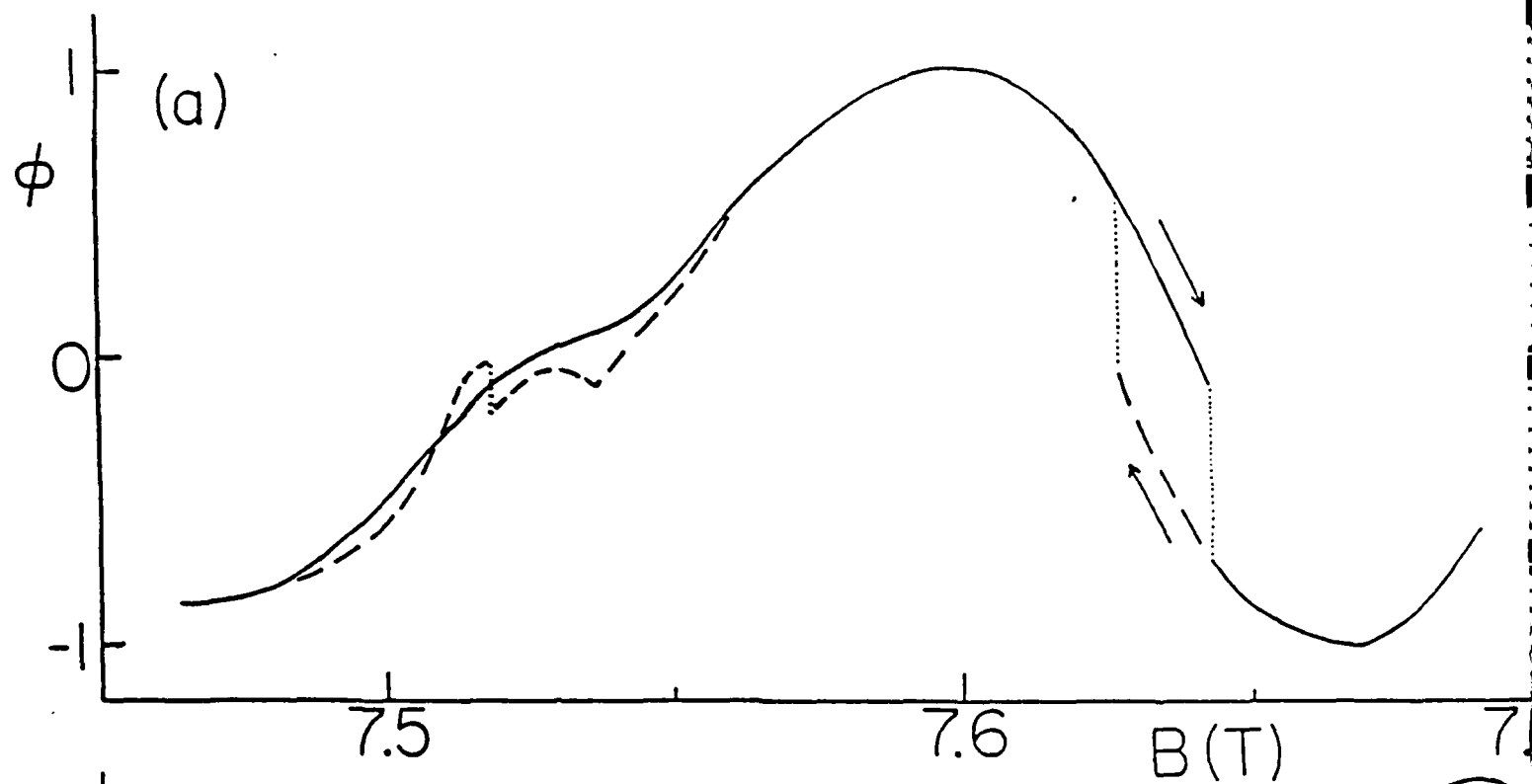
Fig. 5 Magnetoresistance oscillations in Br_2-graphite. (a)=data taken by four-probe measurement; (b)=theory (σ_{xx}^{-1}); (c)=data taken inductively, on a different sample; (d)=susceptibility oscillations, for comparison (plotted as $- 4\pi\chi$).

Fig. 6 "Phase diagram" of Condon domain formation, showing field (B) vs. deHaas-van Alphen frequency F. In stage 1 samples with a Landau level bandwith of 2.8meV, Condon domains could form at fields below the solid line, but would be two-dimensional only at fields above the dashed line. Note that several intercalation compounds have appropriate F values.

Fig. 7 Phase transition in $FeCl_3$-graphite. Curve shows x-ray intensity vs. T for extra peaks in spectrum. Traces are presented as a function of time, showing gradual loss of intensity of the extra peak.

Fig. 8 Magnetothermal oscillations of Br_2-graphite, for two different runs (arrows shows direction of field sweep). While the predicted large T-variations were observed (~0.2K at 4.2K), the sharp structure and hysteresis strongly suggest that the torque instability was present.

Fig. 9 Luminescence spectrum of Br_2 in graphite (a), compared to that of dilute Br_2 in an Ar matrix (b), from Ref. 16.
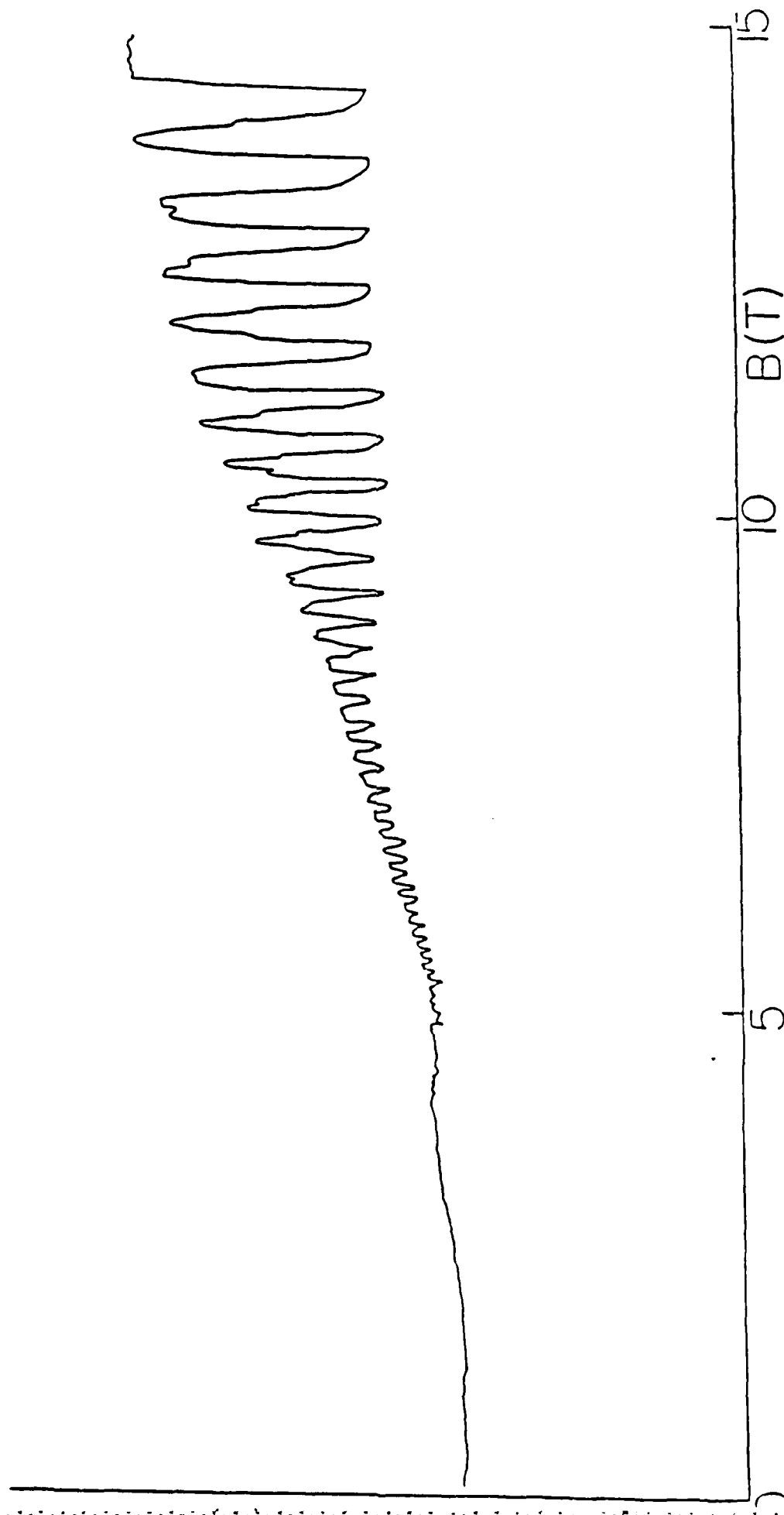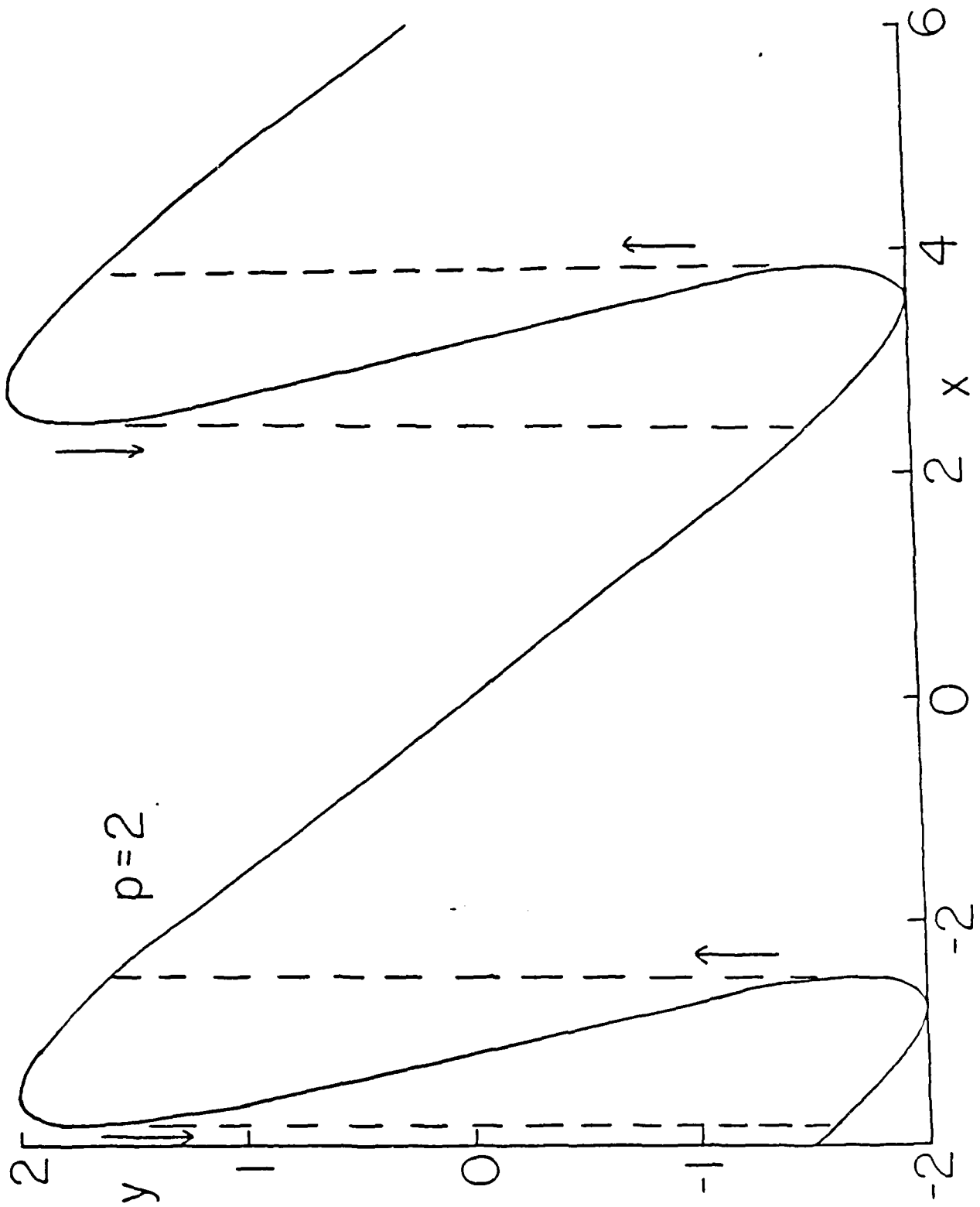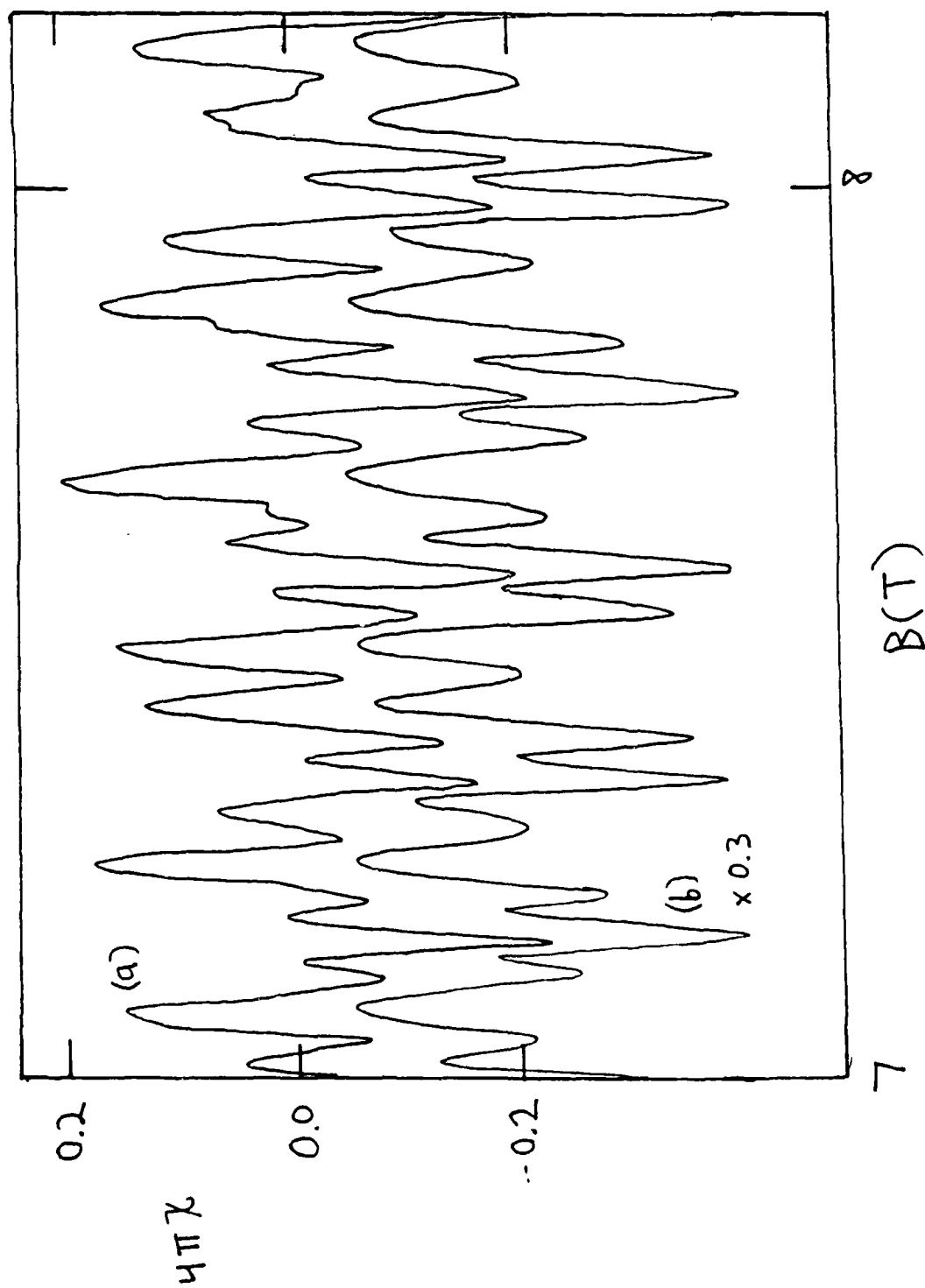
Fig 1

Fig. 2

Fig. 3

p=2
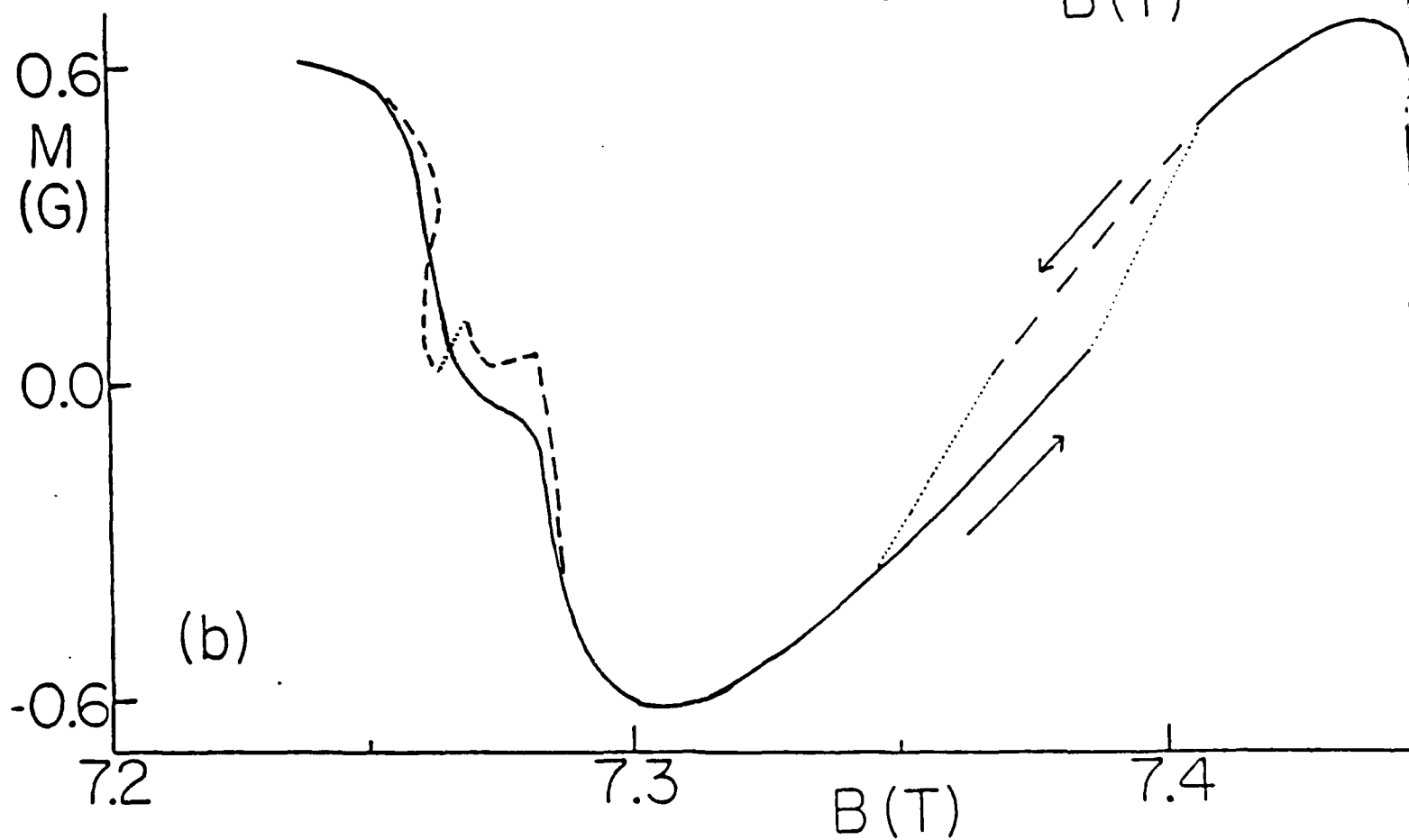
Fig. 4

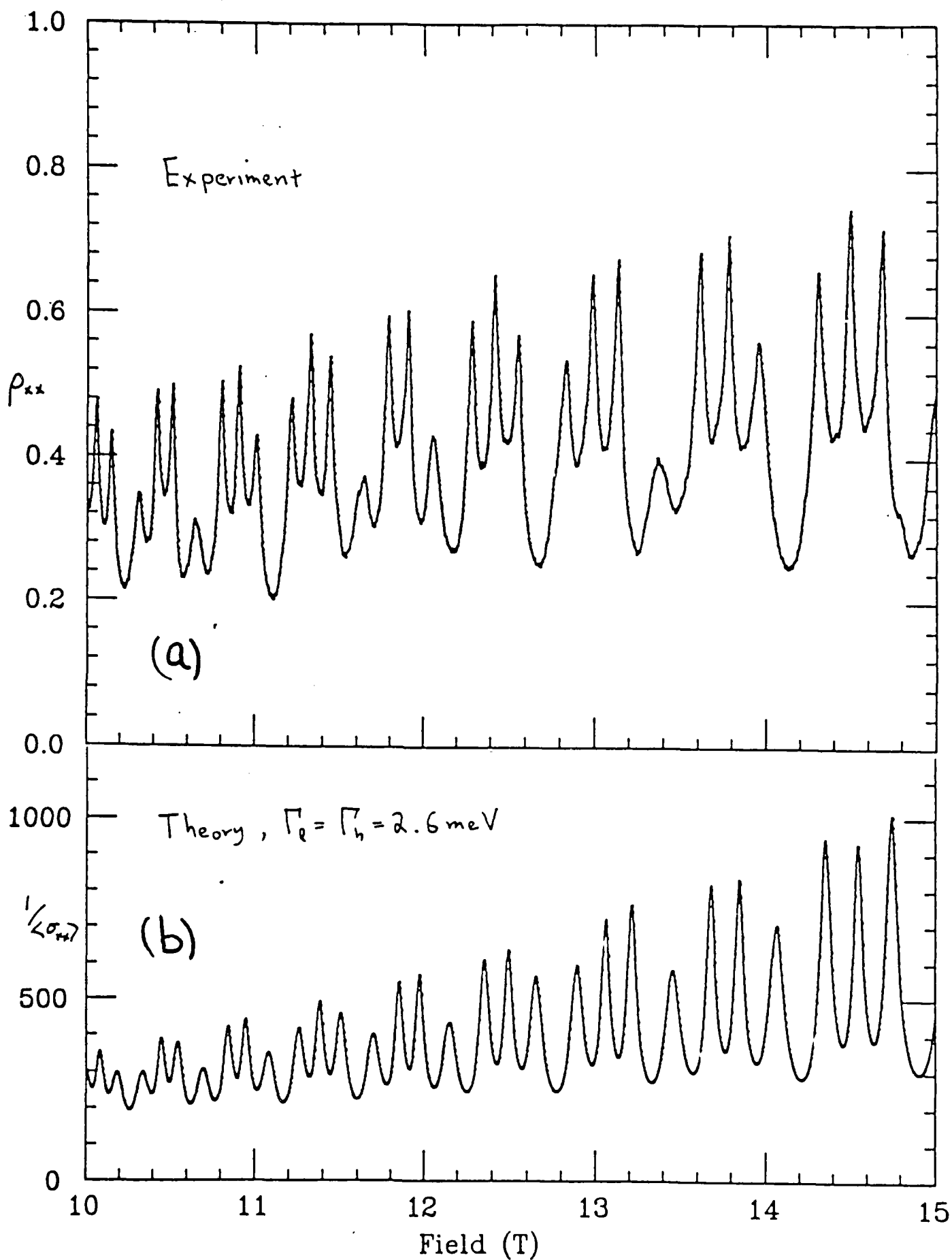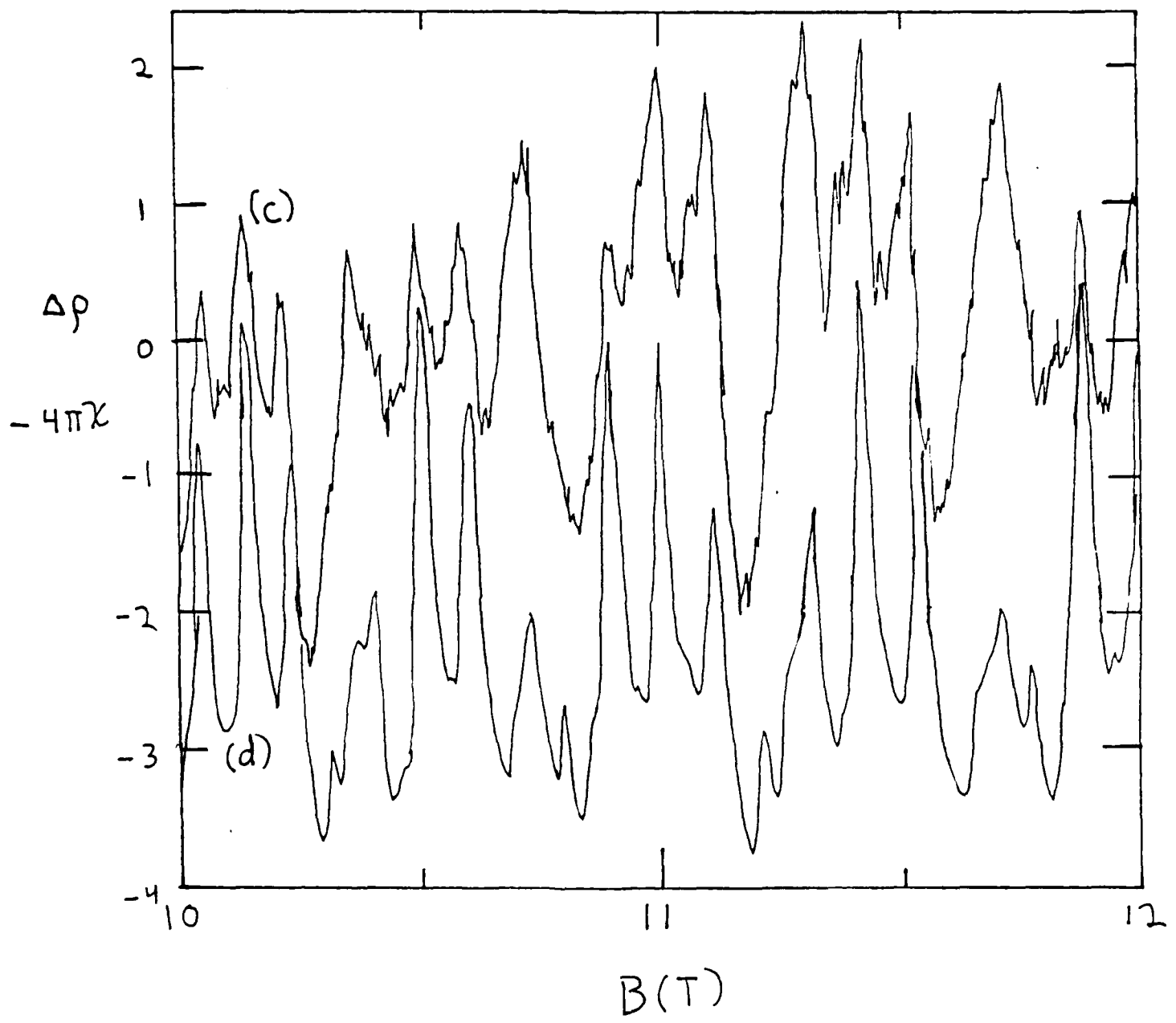Fig. 5, a,b

Fig. 5, c, d

Stage 1 Graphite Intercalation Compounds

2D Condon Domains

$F(T)$

$B(T)$

20

10

500   1000 $SbCl_5$ (H_2SO_4) 1500 AsF_5 2000

Fig. 6
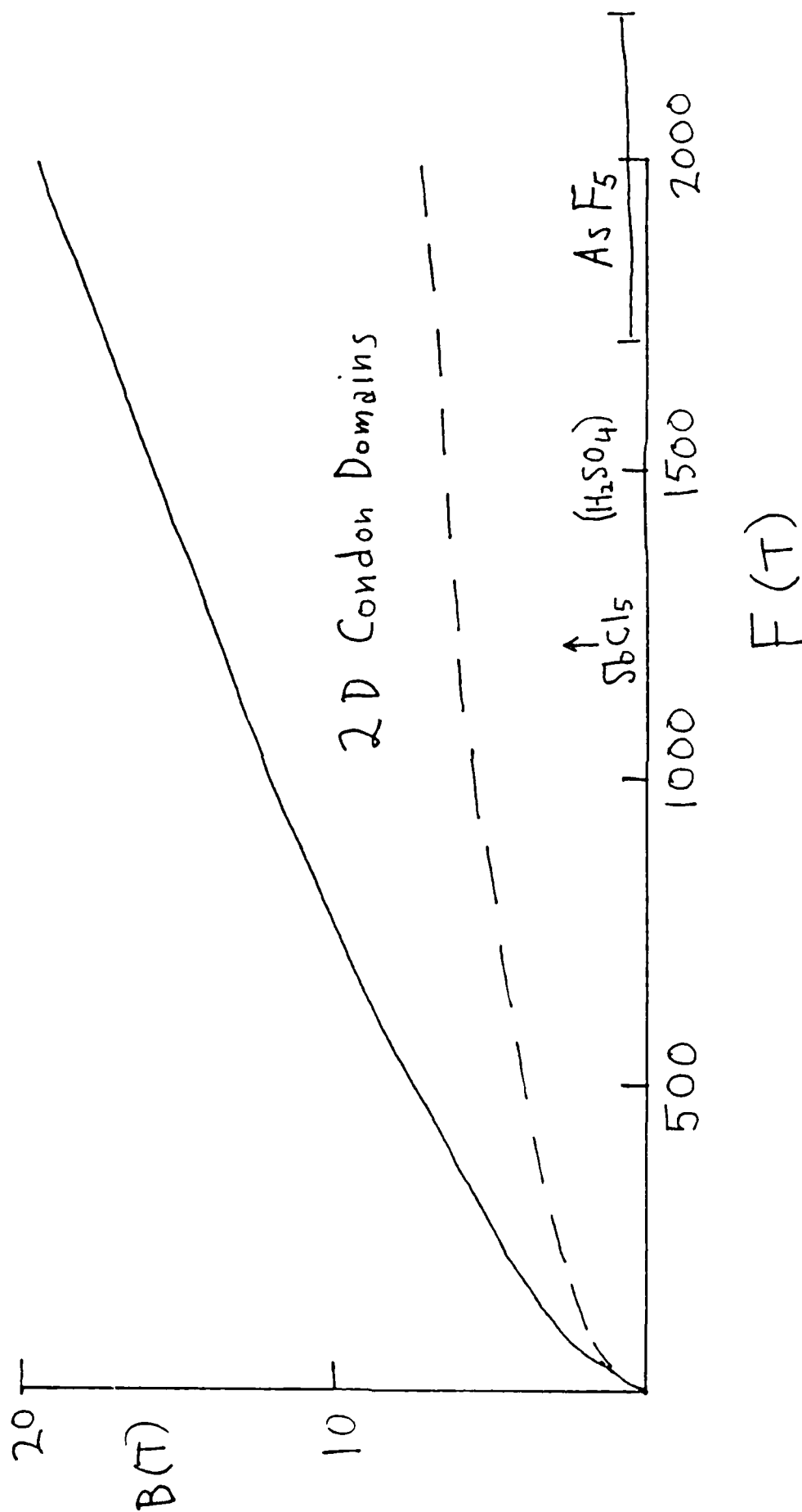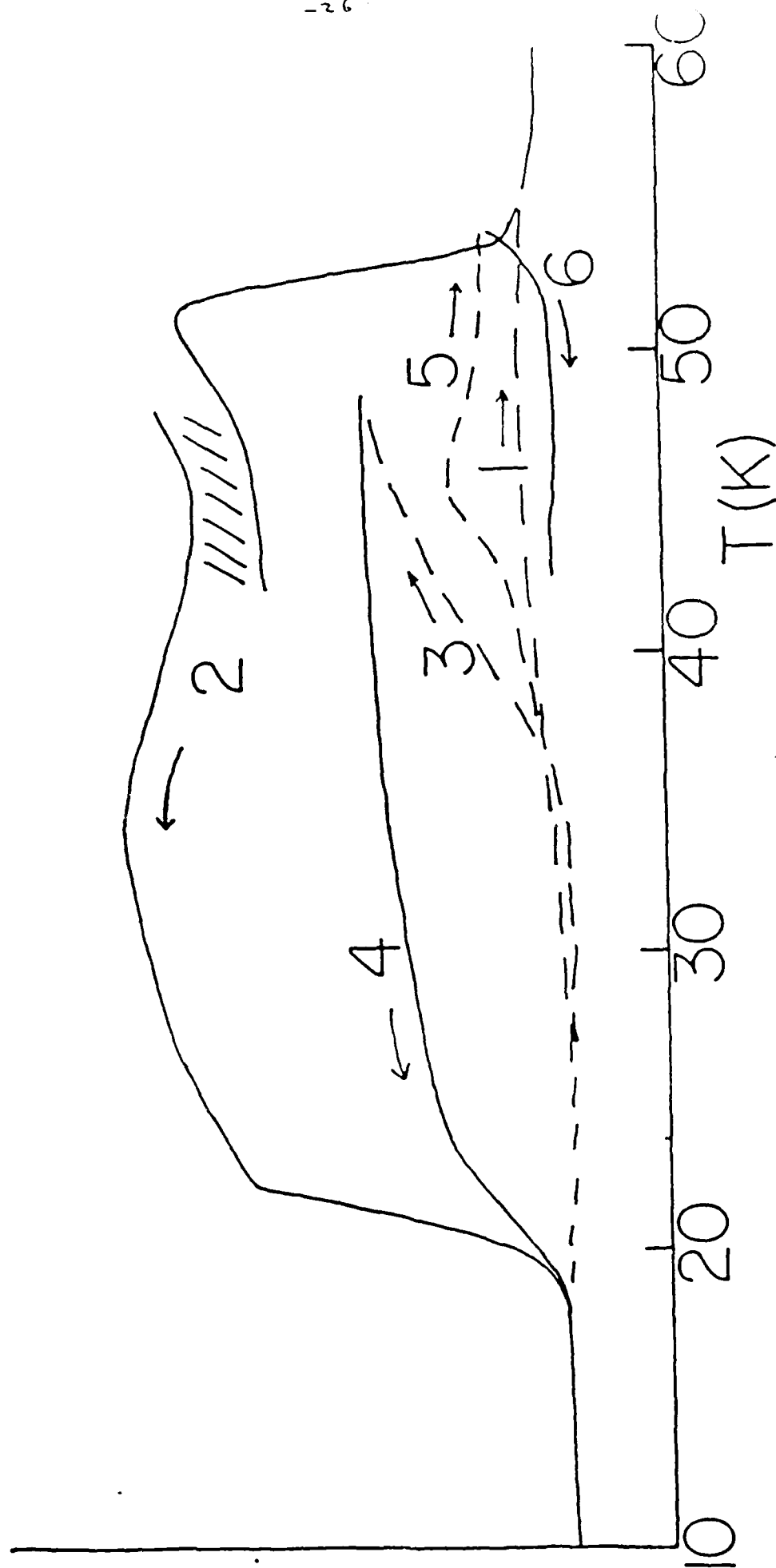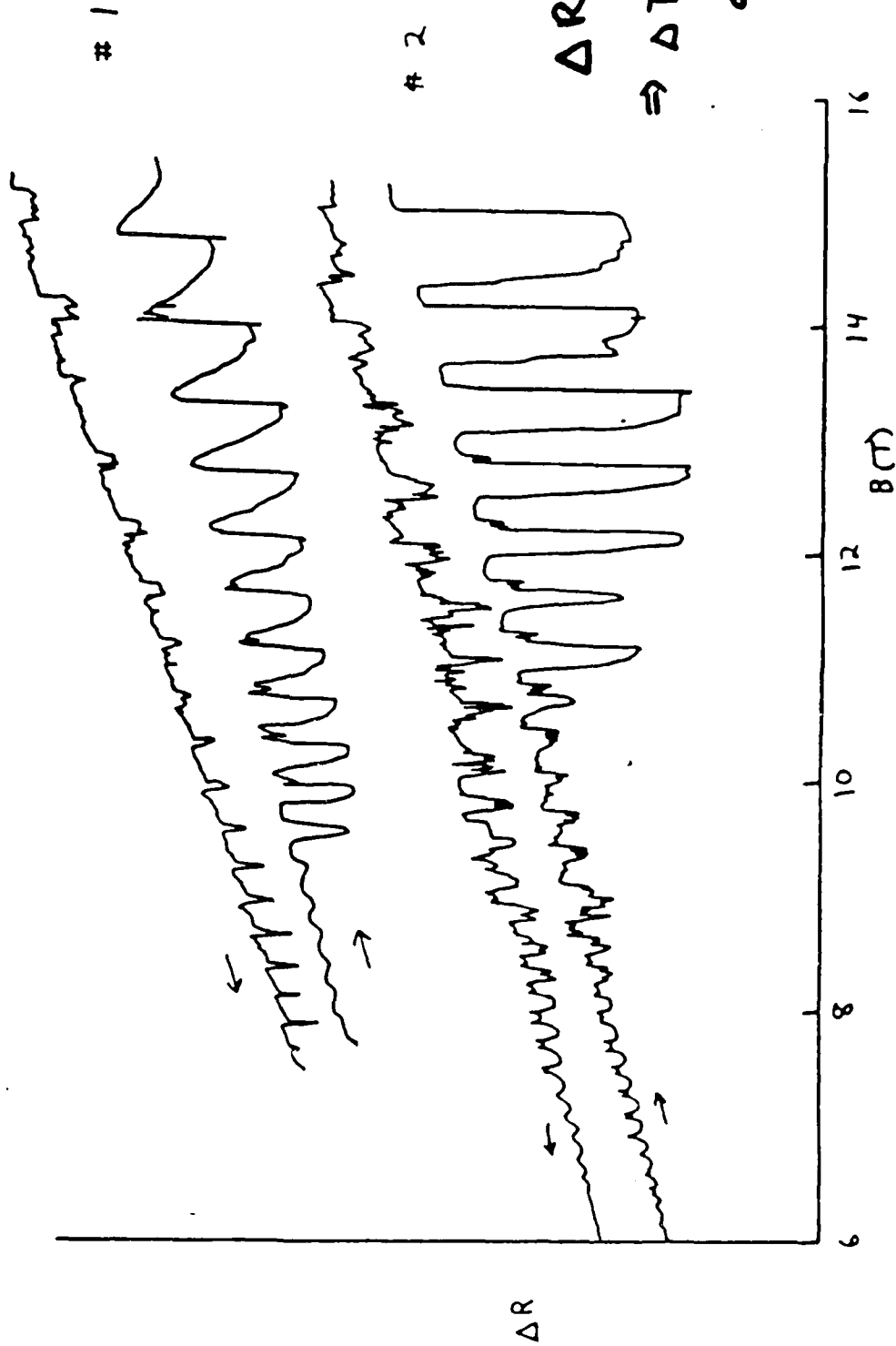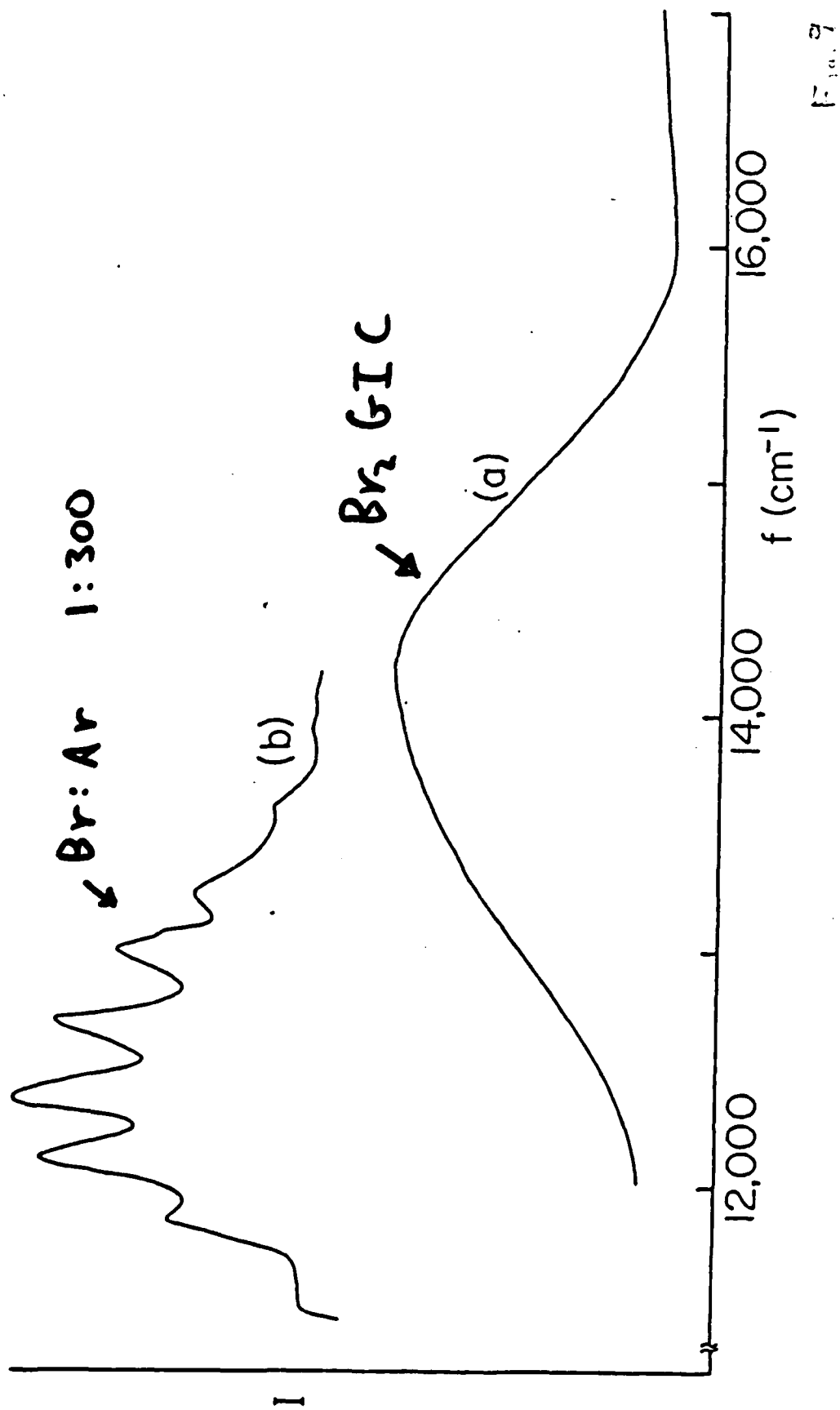
Fig 7

Fig. 7

3.   <u>Research Equipment Acquired</u>

HP 3561A Dynamic Signal Analyzer

PAR Model 113 Preamp

4. List of Publications

1. "Field-induced Phase Transition in AsF_5-graphite," R.S. Markiewicz, C. Zahopoulos, D. Chipman, J. Milliken, and J.E. Fischer in P.C. Eklund, M.S. Dresselhaus, and G. Dresselhaus, Eds. Graphite Intercalation Compounds: Extended Abstracts (Pittsburgh, Mat. Res. Soc., 1984), p. 42.

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16. R.S. Markiewicz, M. Meskoob, and B. Maheswaran, "Susceptibility of the Two-Dimensional Electron Gas: Diagmagntic Spikes and Gaps in the Density of States", submitted to Phy. Rev. B.

17. R.S. Markiewicz, "Effect of Torque Instability on deHass-van Alphen Oscillations," submitted to Phys. Rev. B.

18. R.S. Markiewicz, "Landau level Condensation in a Two-Dimensional Electron Gas: Stabilization by Electron-Electron Interaction or Localization," submitted to Phys. Rev.B.

5. Professional Personnel:

R.S. Markiewicz, Principal Investigator

C. Zahopoulos, Graduate Student[†]

M. Meskoob, Graduate Student

K. Chen, Graduate Student

B. Maheswaran, Graduate Student

L. Fotiadis, Graduate Student

X. Wu, Graduate Student (Reading Course)

M. El Rayess, Post Doc

[†]Received Ph.D. 3/85. Thesis "Fermiology of Acceptor Graphite Intercalation Compounds Using de Haas-van Alphen and Shubnikov-de Haas Measurements."

6. Interactions

a. Papers presented at scientific meetings:

(i) Refs. 1-3, presented at Materials Research Society Meeting, Boston, November 1984.

(ii) Ref. 5 presented at APS March Meeting, Baltimore, MD, March 1985.

(iii)   Ref. 6 presented at Int. Conf. on Graphite Intercalation Compounds, Tsukuba, Japan, May 27-30, 1985 (invited).

(iv) Ref. 11 presented at APS March Meeting, Las Vegas, Nevada, March 30-April 3, 1986.

(v) Refs. 12-15 (revised versions) presented at MRS Meeting, Boston, December 1986.

b. Seminars given or arranged:

(i) "Superlattices and Phase Transitions in Graphite Intercalation

(i) "Superlattices and Phase Transitions in Graphite Intercalation Compounds," IBM, Yorktown Heights, October 26, 1984.

(ii) "Giant Magnetic Interaction in a Two-dimensional System: Possible Connection to the Quantum Hall Effect," Northeastern Physics Dept. Colloquium, November 1984.

(iii) "Phase Transitions in Graphite Intercalation Compounds," Chemistry Dept., Northeastern, February 25, 1985.

(iv) "Giant Magnetic Interaction and Domain Dynamics in Two Dimensions," Boston University Physics Dept., May 1, 1985.

(v) Visit to J. Koplik and H. Levine, Slumberger, July 1985.

(vi) "Condon Domains and Chaos in Graphite Intercalation Compounds," Dresselhaus group meeting, MIT, September 19, 1985.

(vii) "Landau Level Condensation and Domain Wall Dynamics in Two Dimensions," Harvard Theory Lunch, January 30, 1986.

c. Collaborations

(i) Dr. David Chipman, A.M.M.R.C., Watertown Arsenal: Transmission x-ray studies.

(ii) Prof. J. Fischer, U. Penn., Philadelphia: magnetooscillations in mercurographites.

(iii) Dr. J. Milliken, NRL: magnetooscillations and x-ray studies of AsF_5-graphite with excess F.

(iv) Prof. R. Clarke, U. Mich., Ann Arbor: magnetooscillations and x-ray studies of single crystals of HNO_3-graphites.

(v) M.J. Brady, R. Webb and Dr. E. Pakulis, IBM, Yorktown Heights: formation of Bi microprobes to observe domains in Br_2-graphite.

(vi) Prof. J. Brooks, B.U., Boston, NMR and magnetization of Br_2 in Br_2-graphite.

(vii)   Prof. L. Falicov, U.C., Berkeley: calculation of magnetic

breakdown in 2-d.

(viii) Prof. G. Zimmerman and A. Ibrahim, B.U.: magnetic intercalation

compounds ($FeCl_3$).

(ix) H.A. Resing and M. Rubenstein, NRL: NMR in Br_2-graphite.

(x) P. Sagalyn, AMMRC: high-resolution C,F NMR in SbF_5- and $SbCl_5^-$

graphites.

(xi) C. Perry and L. Reinisch, NU: optical studies of Br_2-graphite.

7.   Patents

N.U. lawyers are doing patent search regarding possible patent on d.c.
transformer based on Condon domains.

END

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