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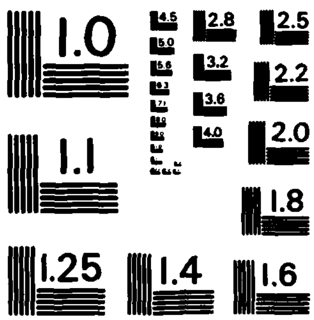

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Final Report
to the
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for research on
Structure–Property Relationships in Intercalated Graphite

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Contents

1.1 Abstract of Objectives

Experimental and theoretical studies have been carried out relevant to the structural, lattice, electronic, magnetic and superconducting properties of synthetic metals prepared by intercalating graphite. New synthesis methods have been developed for preparing magnetic transition metal chloride and potassium-hydrogen graphite intercalation compounds. The use of ion implantation to enhance intercalation has been explored and promising results have been obtained. Structural studies using high resolution x-ray scattering and transmission electron microscopy have been applied to study two-dimensional structural phase transitions such as the commensurate to incommensurate stripe phase transition in bromine intercalated graphite and the commensurate to glass phase transition in antimony pentachloride intercalated graphite. The construction of a Raman microprobe allows study of the spatial homogeneity (to 2 micron resolution) of the staging in specific intercalated graphite samples. Electrical and thermal transport studies have been carried out, providing new information on the dominant scattering mechanisms. The high field magnetoresistance anomaly in graphite identified with a charge density wave has been further explored with particular emphasis given to the role of impurities in pair breaking phenomena and pulsed electric fields in non-linear non-ohmic effects. Experimental and theoretical studies of two-dimensional magnetic phenomena have been successfully carried out in magnetic intercalation compounds. The theoretical model developed to explain the superconducting behavior in the first stage alkali metal compounds has guided studies on superconducting graphite intercalation compounds with higher transition temperatures with particular relevance to their superconducting behavior and their related Fermi surface and phonon mode properties.

1.2 Statement of Work

Statement of work in AFOSR contract #F49620-83-C-0011 on Structure-Property Relationships in Intercalated Graphite.

- Derive techniques for improved methods for the preparation and characterization of specific graphite intercalation compounds.
- Synthesize new intercalated systems and study their structure.
- Study in-plane structure and phase transitions in the intercalate layers with electron diffraction, lattice fringing, real space electron microscope imaging, and high resolution x-ray scattering.
- Deduce structural phase diagrams for specific graphite intercalation compounds.
- Investigate in detail commensurate-incommensurate phase transitions.
- Study lattice modes by infrared and Raman spectroscopy, and inelastic neutron scattering.
- Derive models for the phonon dispersion relations throughout the Brillouin zone, and apply these models to interpret lattice mode studies.
- Model the electronic dispersion relations and apply these models to interpret the experimental results relevant to the electronic properties.
- Measure and model thermal transport phenomena in intercalated graphite.
- Measure the temperature and field dependence of the magnetic susceptibility and heat capacity of magnetic graphite intercalation compounds and to construct magnetic phase diagrams for these systems.
- Study the superconductivity of specific graphite intercalation compounds.

2 Current Status of Research Effort

A summary of the current status of the research effort on the structure-property relationships in intercalated graphite is presented here following the listing given in the statement of work (section 1.2). In presenting the summary, we refer to the publications for the two year period which are listed in section 3.1 by number (#n).

2.1 Synthesis and Compositional Characterization of Graphite Intercalation Compounds

2.1.1 A New Method for Synthesis of Magnetic Graphite Intercalation Compounds

A new method has been successfully developed for the preparation of magnetic graphite intercalation compounds (GIC) with transition metal chlorides. This method does not require use of the $AlCl_3$ complexing halide, which was found to be retained in the GIC and to complicate the analysis of earlier magnetic susceptibility and heat capacity measurements. This new method requires several atmospheres of Cl_2 gas, which is introduced at room temperature, so that the gas pressure is approximately tripled at the growth temperature of 560°C. A one-month growth period has been used, and well-staged samples of $CoCl_2$-GICs with stages $n = 1, 2$, and 3 have been prepared under controlled conditions.

2.1.2 A New Method for Synthesis of Potassium-Hydrogen Graphite Intercalation Compounds

A new method has been developed for the synthesis of potassium-hydrogen graphite intercalation compounds, based on the intercalation of $K\bar{H}$ directly rather than the absorption of hydrogen by K–GICs. This method permits the preparation of KH_x–GICs in HOPG host materials (#63,#64). With this method we have successfully prepared stage 1 and stage 2 KH_x-GICs. This method also permits use of kish graphite as a host material.

2.1.3 Implantation-Enhanced Intercalation

We have discovered that if a graphite sample is first implanted, then the ability to intercalate certain species is greatly enhanced. Initial experiments were made with sodium, which does not readily intercalate graphite (#50). Here we have found that initial implantation of sodium into graphite enhances the subsequent intercalation of sodium. Since the implantation of carbon and argon into graphite also enhances the subsequent intercalation of sodium, we conclude that implantation-enhanced intercalation is due to a defect mechanism rather than to a catalytic process (#50,#55). The discovery of implantation-enhanced intercalation offers promise for the synthesis of new classes of graphite intercalation compounds.

2.1.4 Use of Rutherford Backscattering for Compositional Characterization of Graphite Intercalation Compounds

We have made further use of the Rutherford backscattering (RBS) technique to provide complementary information on the stoichiometry of GICs, particularly with respect to the depth profiling of the chemical constituents (#34). Our first measurements were on air stable compounds such as $SbCl_5$-GICs (#34). More recently we have extended the technique to highly reactive GICs such as KHg-GICs (#42) and K-GICs.(#61) Preliminary results from channeling spectra have been obtained for the K-GIC system showing that the graphite planes retain long range alignment (open channels). In some temperature ranges, weak channeling effects are also seen for the potassium.(#61) The Rutherford backscattering experiments were carried out at Bell Laboratories in collaboration with Drs. T. Venkatesan and J. Poate.

2.2 Structural Studies

2.2.1 In-Plane Structure Studies Using Electron Microscopy

High resolution Transmission Electron Microscopy (TEM) studies have been carried out on well staged KHg-GIC, $SbCl_5$-GIC samples and on partly desorbed Br_2-GIC samples.(#39) All the measurements have been made using both single crystal "kish" graphite and highly oriented pyrolytic graphite (HOPG) host materials. Early TEM measurements were made to study multiphase coexistence in alkali metal GICs.(#1)

For the KHg-GIC and $SbCl_5$-GIC systems, the staging perfection has been studied using c-axis lattice fringing techniques (#26,#39), and characteristic staging defects have been identified. Of particular interest is the long defect-free regions that are observed for both in-plane and c-axis fringes. For the case of the stage 1 KHg-GIC, three different commensurate phases have been identified from the lattice fringe spacings. These lattice fringe studies complement our previous observations of these phases by electron diffraction techniques. Of interest is the long range in-plane coherence of the $(2\times2)R0^\circ$ and $(\sqrt{3}\times\sqrt{3})R30^\circ$ phases (#39), in many cases extending beyond 1000Å.

Also of interest is the Br_2-GIC system where we have identified by the diffraction pattern a single domain of the $(\sqrt{3}\times7)R(30^\circ,0^\circ)$ structure, verifying previous observations using high resolution x-ray scattering.(#39) Commensurate in-plane structures have also been observed in the KH_x–GIC system, though domains of $(2\times2)R0^\circ$ and $(\sqrt{3}\times\sqrt{3})R30^\circ$ phases are found within the same samples. By variation of the temperature of intercalation, the relative importance of each of these phases can be varied.(#64)

2.2.2 In-Plane Structure Studies Using X-Ray Scattering Techniques

X-ray studies of intercalated graphite have been carried out to study both in-plane structure and phase transitions. A summary of the results obtained for the structural studies is given here, while results for phase transitions are summarized in Section 2.2.3.

The kinetics of KHg intercalation have been studied by x-ray scattering. Our results show that the intercalation process for this synthesis is sequential, with the potassium being intercalated initially to yield a well-staged compound of stage 1, after which the Hg intercalation proceeds. The x-ray results show that once the intercalant enters a layer plane, the growth of the in-plane structure is very rapid.(#33) These results are consistent with studies of the kinetics of the Br_2-GIC intercalation process (#31,#33), where the x-ray linewidth is found to be resolution-limited throughout the intercalation process. This small linewidth suggests the formation of large coherent in-plane domains early in the intercalation process.

In previous structural studies we have shown that the in-plane graphite lattice expands slightly for donors and contracts slightly for acceptors.(#3) A low temperature x-ray study showed that the lattice constant for the intercalate layer in an incommensurate intercalation compound has a very much larger coefficient of thermal expansion.(#6) This is a very important result and provides the driving mechanism for the commensurate-incommensurate phase transition discussed in Section 2.2.3.

2.2.3 Commensurate–Incommensurate Phase Transitions in the Br_2–GIC System

This work represents a significant contribution to our general understanding of two-dimensional phase transitions. The study was made in the Br_2–GIC system where a commensurate–incommensurate transition has been observed at 342K. (#13,#17,#25,#28,#31) The incommensurate phase which is obtained above 342K is a highly unusual phase whereby the intercalate unit cell in the $\sqrt{3}$ direction remains commensurate with the graphite while a more complicated structure (a stripe domain phase) characterizes the 7-fold direction. In the $\sqrt{3}$–fold direction the bromine structure is locally commensurate with the graphite structure within a domain, but in the domain wall region a phase slip of 2b/7 occurs where b is the length of an intercalate unit cell in the 7-fold direction.(#31) In this way, the size of the average bromine unit cell becomes larger than that in the commensurate phase below T_c = 342K. As the temperature increases above the critical temperature T_c the domain size continues to decrease, thereby resulting in an increase in the average intercalate unit cell size.

2.2.4 Commensurate-Glass Phase Transition in the $SbCl_5$-GIC System

Structural phase transitions in the $SbCl_5$-GIC system observed by transmission electron microscopy techniques show a transition from a commensurate intercalate phase at room temperature to a glassy phase below ~ 200K.(#22) This phase transition is very unusual insofar as the low temperature phase is the glassy phase. High resolution x-ray studies were carried out to identify this transition more fully, but no such transition could be observed with x-ray scattering. Further detailed high resolution TEM studies were then initiated. The TEM results show that the glassy phase is induced by electron beam irradiation effects.(#60) The characteristics of this novel phase transition are under further investigation.

2.2.5 Model for Staging in Intercalated Graphite

A model for staging has been developed (#56) based on an evaluation of the partition function for attractive in-plane and repulsive interplanar interactions. Mixed staging is found at high temperatures below the disordering temperature.

2.3 Lattice Mode Studies

2.3.1 Inelastic Neutron Scattering Studies in $SbCl_5$–GIC

Inelastic neutron scattering studies on the $SbCl_5$-GIC system were carried out at Oak Ridge National Laboratory in collaboration with the Oak Ridge and University of Kentucky groups.(#40) The neutron scattering data have been interpreted on the basis of a one-dimensional Born-von Karman model. The results for the $SbCl_5$-GIC system have been compared with inelastic neutron scattering results on other GICs to establish systematic trends in the magnitude of the interactions.

2.3.2 Raman Microprobe Studies

A Raman microprobe has been set up for particular application to the study of the Raman spectra in ordered and disordered graphites and graphite intercalation compounds.(#45) The availability of this new instrument has enabled us to use our knowledge of the phonon dispersion relations of GICs to characterize the spatial homogeneity of the staging in GIC samples (to $\sim 2\mu$m resolution).(#62) A particularly interesting application of this technique is the study of the spatial homogeneity of the staging in the KH_x-GIC system.(#63) One interesting new result is the observation of a depletion of intercalant $SbCl_5$ from within $\sim 10\mu$m of the sample edges, associated with a macroscopic contraction of the intercalate layer as the sample is cooled to room temperature from the temperature of intercalation.(#66)

2.3.3 Raman and Infrared Studies

A detailed examination of low frequency Raman-active modes in alkali metal-GICs has been completed with particular reference to the temperature dependence of these modes.(#10,#15) From these spectra, new information has been obtained about the changes in bonding and symmetry associated with the low temperature structural phase transitions previously identified by x-ray and electron diffraction.

Using infrared spectroscopy, the stage dependence of the lattice mode structure for the $CoCl_2$-GIC system has been studied. Analysis of the infrared spectra was used to obtain information on charge transfer in these compounds.(#21)

2.3.4 Models for Phonon Dispersion Relations

The models for phonon dispersion relations derived earlier (#2,#5,#9) have now been applied to understand the results of Raman scattering in some of the well–ordered materials (e.g., stage 1 and 2 KHg-GIC). For the first time, a large number of in-plane zone-folded peaks have been deduced by a model and the resulting modes have been correlated with the observed Raman spectra for stage 1 KHg-GIC samples.(#14) The general model for the phonon dispersion relations has also been applied successfully to interpret the observed mode shifts and symmetries in the Br_2-GIC system.(#19)

2.4 Electronic Structure

2.4.1 Models for Electron Dispersion Relations

The model for the electronic dispersion relations previously developed under the predecessor of this AFOSR contract has been applied to several projects, including the modeling of the Fermi surface of the KHg-GIC system of superconducting compounds.(#20,#43) An article on the basic electronic and lattice mode structure of graphite has been prepared and presented at a workshop on the electrochemistry of carbon.(#38) Attention has also been given to the controversial issue of whether the s-bands of the first stage heavy alkali metal-GICs are occupied.(#53)

2.4.2 Shubnikov-de Haas Effect in the KHg-GIC System

The KHg-GIC system is of particular interest because of its superconducting properties. Our discovery of intercalation techniques to prepare high stage compounds ($n \geq 3$) suggests the possibility of studying the transition from three-dimensional to two-dimensional superconductivity as the stage is increased.(#20,#43)

The importance of Fermi surface measurements for superconducting GICs relates to the Al–Jishi model for superconductivity in stage 1 potassium–intercalated graphite which indicates that superconductivity in GICs requires the presence of both intercalate s- and p-band electrons and graphitic π- electrons.

The commensurate nature of the stage 1 and 2 compounds results in some of the highest frequency periods yet measured in a graphite intercalation compound. Unfortunately the analysis does not conclusively identify any Shubnikov-de Haas frequencies with the intercalate for any of the stages that were investigated in detail ($n = 1, 2, 3$). For the higher stage compounds ($n \geq 2$) it is believed that the coherent intercalate domains are not sufficiently large to achieve the condition $\omega_c \tau >> 1$, thereby explaining the absence of intercalate Shubnikov-de Haas frequencies.

The magnetoconductivity experiments and Shubnikov-de Haas studies were all carried out at the Francis Bitter National Magnet Laboratory.

2.5 Transport Properties

2.5.1 Electrical Resistivity in Graphite Intercalation Compounds

A model has been developed for the temperature dependence of the electrical resistivity of graphite intercalation compounds, showing that the quadratic term in the temperature dependence is associated with out-of-plane phonon modes.(#54)

2.5.2 Thermal Transport Studies in Intercalated Graphite

The temperature dependence of the thermal conductivity of intercalated graphite has been measured for both donor and acceptor compounds. The significance of this work relates to the extremely high in-plane thermal conductivity of the graphite host material, as well as its very high thermal anisotropy. Of interest is the effect of intercalation on the magnitude and anisotropy of the thermal conductivity of the host material as a function of temperature.(#12) The electronic and lattice contributions to the thermal conductivity have been separated by measurements of the temperature dependence of the thermal conductivity in high magnetic fields.(#4) Measurement of the low temperature resistivity has confirmed the applicability of the Wiedemann-Franz relation.(#11) The anomalous stage dependence of the c-axis resistivity has also been studied.(#11) In contrast to the behavior of the thermal conductivity and the electrical resistivity, thermopower measurements on GICs have shown a very small stage dependence.(#11)

Anomalies in the low temperature thermal conductivity and thermopower of the $CoCl_2$-GIC system have been identified with a magnetic phase transition (#8) that is also observed in measurements of the magnetic susceptibility and heat capacity. Subsequent calculations have verified that the functional form of the observed temperature dependence is consistent with the identification of these anomalies with a magnetic phase transition.

2.5.3 High Field Magnetoresistance Anomaly in Graphite

Earlier work on the magnetoresistance anomaly in kish graphite at high magnetic fields was identified with a charge density wave instability. In connection with this anomaly, curious oscillatory behavior as a function of magnetic field has been observed in several kish graphite samples.(#36) The role of impurities and charge imbalance was recently studied and modeled (#46), showing that the detailed behavior at the magnetoresistance anomaly is highly sensitive to the concentration of charged impurities, which give rise to the breaking of pair states. These results also show that the observed magnetic field dependence differs in a subtle way (#49) from that predicted by Yoshioka and Fukuyama. By measuring the magnetoresistance with pulsed currents, it was shown that the magnitude of the anomaly exhibits a non-linear dependence on the electric field.(#65) In this work it was also established that the charge density wave occurs in the basal plane and not along the c-axis as had been calculated by Yoshioka and Fukuyama.

2.6 Magnetic Studies in Graphite Intercalation Compounds

The study of magnetic GICs has been actively pursued. High precision susceptibility measurements as a function of temperature and magnetic field have been instrumental in establishing the two-dimensional nature of the $CoCl_2$-GIC magnetic system.(#18,#24,#32,#48) These results have been correlated with measurements of the temperature dependence of the heat capacity at high magnetic fields.(#16,#30) Detailed attention to the materials characterization has been important in establishing the range of validity of the present work.(#51) Particular attention has been given to the stage variation of the temperature and magnetic field dependence of the magnetic susceptibility. The first stage samples show three-dimensional behavior for a wide range of temperatures and magnetic fields, whereas the higher stage compounds show more two-dimensional behavior.(#51,#52,#57)

Our theoretical modeling for this two-dimensional system is directed toward explaining the experimental results and extending currently available theoretical models.(#27,#52) A calculation of the effect of the finite size of the magnetic domains on the susceptibility has been made and has shown that finite size effects inhibit the divergence of the susceptibility at the Kosterlitz-Thouless transition.(#44,#47) A detailed study of the magnetic field dependence of the susceptibility and magnetization has provided important information on the magnetic phase diagram and has identified the spin flop transition for the stage 1 $CoCl_2$-GICs.(#58)

2.7 Superconductivity Studies in Graphite Intercalation Compounds

The Al-Jishi model for superconductivity in intercalated graphite requires both graphitic and intercalate electrons at the Fermi level.(#23) Therefore Fermi surface studies (see section 2.4.2) of superconducting materials were carried out (#20,#43) to test the validity of the Al-Jishi model. The large electron-phonon coupling, characteristic of superconducting materials, facilitated observation of zone-folding phenomena in the Raman spectra of stage 1 KHg-GIC samples (see section 2.3.4).

With regard to measurements of superconductivity in GICs, we have observed superconductivity in a stage-3 compound of KHg-GIC.(#20,#29) We have previously observed and more recently confirmed that different in-plane phases for stage 1 and 2 compounds are associated with different superconducting transition temperatures.(#20,#59)

2.8 Review Articles

A review article on intercalated graphite for *Physics Today* has been published and has had wide distribution.(#41) A short review article for the *Materials Research Encyclopedia* has been written.(#7) An educational module based on a tutorial lecture presented at the Materials Research Society has been published.(#35) In response to a request to summarize the highlights of the Third International Conference on Intercalated Graphite at Pont á Mousson (1983), a review article has been prepared.(#37)

3 Reports and Publications

3.1 Publications

1. "Scanning Transmission Electron Microscopy of Multiphases in Graphite–Alkali Metal Intercalation Compounds", H. Masurek, M.S. Dresselhaus and G. Dresselhaus, *Carbon*, 20, 297 (1982).

2. "Lattice Dynamical Model for Graphite", R. Al-Jishi and G. Dresselhaus, *Phys. Rev.* *B*26, 4514 (1982).

3. "The Effect of Intercalation on the Lattice Constants of Graphite", T. Krapchev, R. Olgivie and M.S. Dresselhaus, *Carbon*, 20, 331 (1982).

4. "High Magnetic Field Thermal Conductivity Measurements in Graphite Intercalation Compounds", J. Heremans, M. Shayegan, M.S. Dresselhaus and J-P. Issi, *Phys. Rev. B*26, 3338 (1982).

5. "Model for Raman Scattering from Incompletely Graphitized Carbons", P. Lespade, R. Al-Jishi and M.S. Dresselhaus, *Carbon*, 20, 427 (1982).

6. "**Low Temperature X-Ray Diffraction Study of Stage 1 Graphite – $FeCl_3$**", H. Masurek, G. Ghavamishahidi, G. Dresselhaus and M.S. Dresselhaus, *Carbon*, 20, 415 (1982).

7. "**Intercalation Compounds**", *Materials Research Encyclopedia*, Pergamon Press, M.S. Dresselhaus, accepted for publication.

8. "**Anomalies in the Thermal Conductivity and Thermopower in $CoCl_2$-Intercalated Graphite at the Magnetic Phase Transition**", F.J. Blatt, I. Zabala-Martinez, J. Heremans, J-P. Issi, M. Shayegan and M.S. Dresselhaus, *Phys. Rev.* *B*27, 2558 (1983).

9. "**A Lattice Dynamical Model for Alkali Metal Graphite Intercalation Compounds**, R. Al-Jishi and G. Dresselhaus, *Phys. Rev.* *B*26, 4523 (1982).

10. "**Raman Scattering from Low Frequency Phonons in Stage 2 Graphite-Rubidium**", J. Giergiel, P.C. Eklund, R. Al-Jishi and G. Dresselhaus, *Phys. Rev.* *B*26, 6881 (1982).

11. "**Temperature Dependence of C-Axis Electrical Resistivity and Thermopower of Graphite Intercalation Compounds**", J-P. Issi, B. Poulaert, J. Heremans and M.S. Dresselhaus, *Solid State Commun.* 44, 449 (1982).

12. "**Electronic and Lattice Contributions to the Thermal Conductivity of Graphite Intercalation Compounds**", J-P. Issi, J. Heremans and M.S. Dresselhaus, *Phys. Rev.* *B*27, 1333 (1983).

13. "**Commensurate-Incommensurate Transition in Bromine-Intercalated Graphite: A Model Stripe Domain System**", A.R. Kortan, A. Erbil, R.J. Birgeneau and M.S. Dresselhaus, *Phys. Rev. Lett.*, 49, 1427 (1982).

14. "**Observation of Superlattice-Induced Raman Modes in Graphite-Potassium Amalgam Compounds**", G. Timp, B.S. Elman, R. Al-Jishi and G. Dresselhaus, *Solid State Commun.* 44, 987 (1982).

15. "**A Study of the Temperature–Dependence of Low–Frequency Raman–Active Phonons in Stage–2 Graphite–K and Graphite–Rb Intercalation Compounds**", J. Giergiel, P.C. Eklund, R. Al-Jishi and G. Dresselhaus, *MRS Symposium on Intercalated Graphite*, edited by M.S. Dresselhaus, G. Dresselhaus, J.E. Fischer and M.J. Moran, (Elsevier, North Holland, New York, 1983), 20, p. 323.

16. "**Magnetic Heat Capacity of Stage 2 Graphite $CoCl_2$**", M. Shayegan, L. Salamanca-Riba, J. Heremans, G. Dresselhaus, and J.P. Issi, *MRS Symposium on Intercalated Graphite*, edited by M.S. Dresselhaus, G. Dresselhaus, J.E. Fischer and M.J. Moran, (Elsevier, North Holland, New York, 1983), 20, p. 213.

17. "**Commensurate-Incommensurate and Melting Transitions in Bromine- Intercalated Single Crystal Kish Graphite**", A. Erbil, A.R. Kortan J. Birgeneau and M. S.

Dresselhaus, *MRS Symposium on Intercalated Graphite*, edited by M.S. Dresselhaus, G. Dresselhaus, J.E. Fischer and M.J. Moran, (Elsevier, North Holland, New York, 1983), 20, p. 21.

18. "Susceptibility of Magnetic Graphite $CoCl_2$ Intercalation Compounds", M. Elahy and G. Dresselhaus, *MRS Symposium on Intercalated Graphite*, edited by M.S. Dresselhaus, G. Dresselhaus, J.E. Fischer and M.J. Moran, (Elsevier, North Holland, New York, 1983), 20, p. 207.

19. "Lattice Dynamical Model for Graphite-Bromine Intercalation Compounds", R. Al-Jishi and G. Dresselhaus, *MRS Symposium on Intercalated Graphite*, edited by M.S. Dresselhaus, G. Dresselhaus, J.E. Fischer and M.J. Moran, (Elsevier, North Holland, New York, 1983), 20, p. 301.

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21. "Electronic and Lattice Modes of Graphite-$CoCl_2$", C.W. Lowe, C. Nicolini, and G. Dresselhaus, *MRS Symposium on Intercalated Graphite*, edited by M.S. Dresselhaus, G. Dresselhaus, J.E. Fischer and M.J. Moran, (Elsevier, North Holland, New York, 1983), 20, p. 93.

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26. "High Resolution Diffraction Experiments on Graphite Potassium-Amalgam", G. Timp, A.R. Kortan, L. Salamanca-Riba, R.J. Birgeneau and M.S. Dresselhaus, *Bull. APS* 28, 347 (1983).

27. "Two-Dimensional Magnetic Model for Graphite – $CoCl_2$", K.Y. Szeto and G. Dresselhaus, *Bull. APS* 28, 265 (1983).

28. **"Two-Dimensional Phase Transitions in Bromine Intercalated Graphite"**, A. Erbil, R. Kortan, M.S. Dresselhaus and R.J. Birgeneau, *Bull. APS* 28, 264 (1983).

29. **"Two-Dimensional Superconductivity in Graphite-Potassium Amalgam"**, G. Dresselhaus, P.M. Tedrow, G. Timp and T. Sienko, *Bull. APS*, 28, 539 (1983).

30. **"Low Temperature Heat Capacity of Magnetic Graphite Intercalation Compounds"**, M. Shayegan, M.S. Dresselhaus, L. Salamanca-Riba, G. Dresselhaus, J. Heremans and J-P. Issi, *Phys. Rev.* B28, 4799 (1983).

31. **"Intercalant Structure, Melting and the Commensurate–Incommensurate Transition in Bromine–Intercalated Graphite"**, A. Erbil, A.R. Kortan, R.J. Birgeneau and M.S. Dresselhaus, *Phys. Rev.* B28, 6329 (1983).

32. **"Magnetic Properties of $CoCl_2$–Intercalated Graphite"**, M. Elahy, M. Shayegan, K.Y. Szeto and G. Dresselhaus, *Synthetic Metals* 8, 35 (1983).

33. **"Structure and Phase Transitions in Bromine and Potassium–mercury Graphite"**, A. Erbil, G. Timp, A.R. Kortan, R.J. Birgeneau, and M.S. Dresselhaus, *Synthetic Metals* 7, 273 (1983).

34. **"Stoichiometric Determination of $SbCl_5$–GIC using Rutherford Backscattering Spectrometry"**, B.S. Elman, L. Salamanca–Riba, M.S. Dresselhaus and T. Venkatesan, *J. Applied Phys.* 55, 894 (1984).

35. **"Structure–Property Relations in Intercalated Graphite"** M.S. Dresselhaus, *J. of Materials Education* 5, 553 (1983).

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38. **"Electronic and Lattice Mode Structure of Graphite"**, G. Dresselhaus, Proceedings on the Workshop on *Electrochemistry of Carbon*, edited by S. Sarangapani, J.R. Akridge and B. Schumm, August 1983, Vol. 84–5, p. 5, Case–Western Reserve University, Cleveland, Ohio.

39. **"Ultramicrostructure of Commensurate Graphite Intercalation Compounds"**, G. Timp and M.S. Dresselhaus, *J. Phys. C: Solid State Phys.* 17 (1984) 2641–2651.

40. **"Inelastic Neutron Scattering from Low Frequency (00q) Longitudinal Lattice Modes in Graphite–$SbCl_5$ Intercalation Compounds**, P.C. Eklund, V. Yeh, H.G. Smith, R. Nicklow, R. Al–Jishi, and G. Dresselhaus, *Phys. Rev.* B29, 2138 (1984).

41. **"Modification to the Properties of Materials by Intercalation"**, M.S. Dresselhaus, *Physics Today*, March 1984, p. 60.

42. **"Stoichiometric Determination of Graphite Intercalation Compounds Using Rutherford Backscattering Spectrometry"**, L. Salamanca-Riba, B.S. Elman, M.S. Dresselhaus and T. Venkatesan, *MRS Symposium on Ion Implantation and Ion Beam Processing of Materials*, edited by G.K. Hubler, O.W. Holland, C.R. Clayton, and C.W. White, Boston, Nov. 1983) (Elsevier, North Holland, New York, 1984) vol. 27, p. 481.

43. **"Shubnikov de Haas effect in KHg_x–graphite intercalation compounds"**, G. Timp, T.C. Chieu, P.D. Dresselhaus, and G. Dresselhaus, *Phys. Rev.* B29, 6940 (1984).

44. **"Kosterlitz-Thouless Phase Transitions in Finite Size Systems: Application to $CoCl_2$-Graphite Intercalation Compounds"**, K.Y. Szeto, M. Elahy, S.T. Chen and G. Dresselhaus, *Bull. APS* 29, 293 (1984).

45. **"Raman Microprobe Studies of the Structure of $SbCl_5$–Graphite Intercalation Compounds"**, L.E. McNeil, J. Steinbeck, L. Salamanca-Riba and G. Dresselhaus, *Bull. APS* 29, 253 (1984).

46. **"The Effect of Impurities on the Electronic Phase Transition in Graphite Under Strong Magnetic Field"**, Y. Iye, L.E. McNeil, and G. Dresselhaus, *Phys. Rev.* B30, (submitted).

47. **"Kosterlitz–Thouless Transition for Finite Size Systems"**, K.Y. Szeto and G. Dresselhaus, *Phys. Rev.* (submitted).

48. **"$CoCl_2$–Intercalated Graphite: A Quasi–Two–Dimensional Magnetic System"**, M. Elahy and G. Dresselhaus, *Phys. Rev.* (in press).

49. **"The Electronic Phase Transition in Graphite Under Strong Magnetic Field"**, Y. Iye, L.E. McNeil, G. Dresselhaus, G. Boebinger, and P.M. Berglund, *Proceedings of the 17th Conference on the Physics of Semiconductors,* Aug. 6–10, 1984, San Francisco (to be published).

50. **"Intercalation of Ion Implanted Graphite"**, H. Menjo, B.S. Elman, G. Braunstein, and M.S. Dresselhaus, presented at *Carbone 84 Conference*, July 2–6, 1984, Bordeaux, France (to be published).

51. **"Magnetic Phase Transitions in $CoCl_2$–Graphite Intercalation Compounds"**, S.T. Chen, K.Y. Szeto, M. Elahy, and G. Dresselhaus, presented at *Carbone 84 Conference,* July 2–6, 1984, Bordeaux, France (to be published).

52. **"2–D Magnetic Phase Transitions in Graphite Intercalation Compounds"**, K.Y. Szeto, S.T. Chen, G. Dresselhaus, and M.S. Dresselhaus, *Proceedings of the 17th Conference on Physics of Semiconductors*, Aug. 6–10, 1984, San Francisco (to be published).

53. "Occupation of the Intercalate Bands In Stage 1 Alkali Metal GICs", K. Sugihara and M.S. Dresselhaus, to be presented at the Materials Research Society Meeting, Boston 1984.

54. "Theory of Electrical Resistivity in Graphite Intercalation Compounds", K. Sugihara, to be presented at the Materials Research Society Meeting, Boston 1984.

55. "Enhanced Intercalation Induced by Ion Implantation", H. Menjo, G. Braunstein, B.S. Elman, L.E. McNeil and M.S. Dresselhaus, to be presented at the Materials Research Society Meeting, Boston 1984.

56. "Lattice Gas Model for Staging in Intercalated Graphite", J. C. Schön, D. Adler and G. Dresselhaus, to be presented at the Materials Research Society Meeting, Boston 1984.

57. "Magnetic Properties of Cobalt Chloride Intercalated Graphite", S.T. Chen, K.Y. Szeto and G. Dresselhaus, to be presented at the Materials Research Society Meeting, Boston 1984.

58. "Spin Flop Transition of $CoCl_2$ Intercalated Graphite from Field Dependence Measurements of Susceptibility", K.Y. Szeto, S.T. Chen and G. Dresselhaus, to be presented at the Materials Research Society Meeting, Boston 1984.

59. "Structural Order, Stoichiometry and Superconductivity in KHg_2-GIC", G. Roth, N.C. Yeh, A. Chaiken, G. Dresselhaus and P. Tedrow, to be presented at the Materials Research Society Meeting, Boston 1984.

60. "High Resolution Electron Microscopy and X-Ray Diffraction Studies on $SbCl_5$-GIC, G. Roth, L. Salamanca-Riba, A.R. Kortan, G. Dresselhaus, R. Birgeneau and J.M. Gibson, to be presented at the Materials Research Society Meeting, Boston 1984.

61. "Analysis of Structural Properties of Graphite Intercalation Compounds Using the Rutherford Backscattering–Channeling Technique", G. Braunstein, B. Elman, J. Steinbeck, M.S. Dresselhaus, T. Venkatesan and B. Wilkens, to be presented at the Materials Research Society Meeting, Boston 1984.

62. "Raman Microprobe Studies of Intercalated Graphite Fibers", L.E. McNeil, J. Steinbeck, L. Salamanca-Riba and G. Dresselhaus, to be presented at the Materials Research Society Meeting, Boston 1984.

63. "Raman Characteristics of Potassium-Hydrogen Intercalated Graphite", N.C. Yeh, T. Enoki, L.E. McNeil, G. Roth, L. Salamanca-Riba, M. Endo and G. Dresselhaus, to be presented at the Materials Research Society Meeting, Boston 1984.

64. "High Resolution Microscopy Studies of Potassium-Hydrogen Intercalated Graphite", L. Salamanca-Riba, N.C. Yeh, T. Enoki, M.S. Dresselhaus and M. Endo, to be presented at the Materials Research Society Meeting, Boston 1984.

65. **"Non-Ohmic Transport in the Magnetic-Field-Induced Charge Density Wave Phase of Graphite"**, Y. Iye and G. Dresselhaus, *submitted to Phys. Rev. Lett.*

66. **"Raman Microprobe Observation of Intercalate Contraction in Graphite Intercalation Compounds"**, L.E. McNeil, J. Steinbeck, L. Salamanca–Riba and G. Dresselhaus, *submitted to Phys. Rev.*

3.2 Advanced Degrees

- **"Lattice Dynamics of Graphite Intercalation Compounds"**,
  R. Al–Jishi, Ph.D., Physics, October 1982.

- **"Phase Transitions in the Intercalated Graphite–Bromine System"**,
  A. Erbil, Ph.D., Physics, October 1982.

- **"Optical Properties of Intercalated Graphite"**,
  C.W. Lowe, Ph.D., Physics, December 1982.

- **"High–Magnetic–Field Studies of Graphite Intercalation Compounds"**,
  M. Shayegan, Ph.D., Electrical Engineering and Computer Science, May 1983.

- **"Phase Transitions in Magnetic Graphite Intercalation Compounds"**,
  M. Elahy, Ph.D., Physics, September 1983.

- **"The Structural, Electronic and Lattice Dynamical Properties of Graphite Intercalation Compounds"**,
  G. Timp, Ph.D., Electrical Engineering and Computer Science, October 1983.

4 Personnel Involved with Research Program

- Mildred S. Dresselhaus – Principal Investigator
  Responsible for the research and the direction of all aspects of the program. The study of intercalated graphite is the major research activity in the research group.

- Gene Dresselhaus – Co–Principal Investigator
  Responsible together with the principal investigator for the research and the direction of all aspects of the program.

- Laurie McNeil – IBM Postdoctoral Fellow
  Responsible for measurements with the Raman Microprobe. (Left August 1984 to become Assistant Professor at the University of North Carolina, Chapel Hill).

- Gerhard Roth – Postdoctoral Fellow
  Responsible for high resolution x–ray measurements and for the synthesis and measurements of superconducting properties under pressure.

- **Ko Sugihara – Research Staff**
  Responsible for modeling transport properties of GICs.

- **Alison Chaiken – Research Assistant**
  Responsible for superconductivity studies in intercalated graphite.

- **S.T. Chen – Research Assistant and Graduate Fellowship Student**
  Responsible for the synthesis of magnetic intercalation compounds and for high precision measurements of magnetic susceptibility and heat capacity in external magnetic fields.

- **M. Elahy – Research Assistant**
  Was responsible for high precision measurement of the temperature dependence of the magnetic susceptibility for $CoCl_2$ intercalated graphite as a function of magnetic field. Also measured the microstructure of intercalate layer by transmission electron microscopy. Completed Ph.D. thesis in September 1983.

- **A. Erbil – Research Assistant**
  Was responsible for measurements of the temperature dependent Raman spectra in Br_2–GICs and for high resolution x–ray spectroscopy to study phase transitions in the graphite–bromine system (completed Ph.D. thesis February 1983). Also did modeling of the commensurate–incommensurate striped domain phase transition. The high resolution x–ray measurements were carried out in collaboration with Professor R.J. Birgeneau of the Physics Department, MIT.

- **C.W. Lowe – Research Assistant and Fellowship Student**
  Responsible for infrared spectroscopy measurements of intercalated graphite with emphasis on both the electronic and lattice mode structure. He also did modeling of optical electronic transitions and lattice modes in intercalated graphite based on his experimental results (completed his Ph.D. thesis in December 1982).

- **H. Menjo – Research Assistant and Fellowship Student**
  Responsible for synthesis and measurement of the Na-GICs using ion implantation to enhance the intercalation process.

- **L. Salamanca–Riba – Research Assistant**
  Responsible for structural studies of intercalated graphite using x–ray diffraction, real space imaging and lattice fringing, with particular emphasis on phase transitions.

- **M. Shayegan – Research Assistant**
  Was responsible for measuring high magnetic field dependence of the specific heat in magnetic graphite intercalation compounds and the thermal transport measurements on graphite intercalation compounds in high magnetic fields. Completed Ph.D. thesis in May 1983.

- **K.Y. Szeto – Research Assistant and Graduate Fellowship Student**
  Extension of two–dimensional xy model to calculate susceptibility for magnetic GIC including finite domain size effects, magnetic phase diagrams and spin flop transitions.

- **G. Timp – Research Assistant**
  Was responsible for structural studies of phase transitions using high resolution electron microscopy and high resolution x–ray scattering and Raman studies of the potassium–mercury–GIC system. Completed Ph.D. thesis in October 1983.

- **Nai–Chang Yeh – Research Assistant**
  Has been responsible for the synthesis and characterization of KHg and KH_x graphite intercalation compounds.

- **Maria Kudisch – Undergraduate Student**
  Has been assisting with synthesis of KHg-GICs and superconductivity measurements.

- **Tanya Sienko – Undergraduate Student**
  Assisted with synthesis of KHg–GICs and with superconductivity measurements. Completed B.S. thesis in June 1983.

4.1 MIT and Other Collaborators

- **A.N. Berker – Associate Professor, Physics, MIT**
  Provides expertise on phase transition theory.

- **R.J. Birgeneau – Professor, Physics, MIT**
  Provides expertise and equipment for carrying out high resolution x–ray scattering experiments.

- **P.C. Eklund – Associate Professor, Physics, University of Kentucky**
  Collaborates on lattice mode studies of intercalated graphite.

- **J. Murray Gibson – Research Staff, AT & T Bell Laboratories, Murray Hill, NJ**
  Collaborates on high resolution microscopy studies of intercalated graphite.

- **R. Guertin – Visiting Scientist, Francis Bitter National Magnet Laboratory**
  Provides expertise and equipment for carrying out superconductivity measurements under pressure.

- **L. Hobbs – Associate Professor, Materials Science and Engineering, MIT**
  Provides expertise in the electron microscopy measurements.

- **P.A. Lee – Professor, Physics, MIT**
  Provides expertise on theory of two–dimensional magnetism and high magnetic field anomaly in graphite.

- **R. Markiewicz – Associate Professor, Physics, Northeastern University**
  Collaborates on Fermi surface studies of intercalated graphite.

- **J.W. McClure – Professor, Physics, University of Oregon**
  Collaborates on electronic structure and transport studies of intercalated graphite.

- **R. Ogilvie – Professor, Materials Science and Engineering, MIT**
  Provides experimental expertise necessary to make precision (00ℓ) x-ray measurements.

- **H.G. Smith – Staff Member, Oak Ridge National Laboratory**
  Collaborates on neutron scattering experiments in intercalated graphite.

- **P. Tedrow – Staff Member, Francis Bitter National Magnet Laboratory**
  Provides expertise and equipment for carrying out measurements in the millikelvin range.

- **T. Venkatesan – Research Staff, Bell Communications Research, Murray Hill, NJ**
  Collaborates on Rutherford Backscattering studies of intercalated graphite.

- **B.J. Wuensch – Professor, Materials Science and Engineering, MIT**
  Provides expertise on the analysis of (00ℓ) x-ray intensity measurements.

- **G.O. Zimmerman – Professor, Physics, Boston University**
  Collaborates on susceptibility measurements in magnetic intercalation compounds.

4.2 Coupling Activities – Seminars and Conference Papers

The MIT group is strongly coupled to international activities on graphite intercalation compounds. We are frequently invited to write review articles (section 2.8), to present seminars at universities and in industry and to present papers at conferences. (The conference papers are listed under 3.0). Below are listed titles of seminars and symposia given over the two-year period relevant to the work supported under this contract.

- **October 13, 1982, Industrial Liaison Symposium, MIT, "Introduction and Overview of Synthetic Metals Field" (MSD).**

- **October 13, 1982, "Electrical and Thermal Transport in Intercalated Graphite" (MSD).**

- **November 2, 1982, Materials Research Society, Boston, "Structure–Property Relations in Intercalated Graphite", invited talk (MSD).**

- **November 11, 1982, Physics Colloquium, University of Texas, Austin, Texas, "The Physics of Intercalated Graphite" (MSD).**

- **November 19, 1982, Colloquium, Northeastern University, Boston, MA, "Two Dimensional Magnetic Phases in $CoCl_2$–Intercalated Graphite" (GD).**

- **December 7, 1982, Solid State Seminar, Bell Laboratories, Murray Hill, NJ, "The Modification of Graphite by Intercalation and Ion Implantation" (MSD).**

- **December 9, 1982, Solid State Seminar, Brown University, Providence, RI, "The Physics of Intercalated Graphite" (MSD).**

- **January 4, 1982, Company Seminar, Raychem Corporation, Menlo Park, CA, "The Physics of Intercalated Graphite and Graphite Fibers" (MSD).**

- **January 5, 1983, Colloquium, Department of Physics, University of California, Berkeley, CA, "The Physics of Intercalated Graphite" (MSD).**
- **January 10, 1983, Company Seminar, General Atomics, San Diego, CA, "Recent Advances in the Study of Intercalated Graphite" (MSD).**
- **February 18, 1983, NASA Lewis Research Center, Cleveland, OH, "Tutoral Lecture on Intercalated Graphite and Graphite Fibers" (MSD).**
- **March 9, 1983, Physics Colloquium, State University of New York at Stony Brook, "The Physics of Intercalated Graphite" (MSD).**
- **April 27, 1983, Solid State Seminar, IBM, Yorktown Heights, "Modification of Materials by Intercalation and Implantation" (MSD).**
- **May 16, 1983, Solid State Seminar, Oak Ridge National Laboratory, "Intercalation of Graphite and Graphite Fibers" (MSD).**
- **May 25, 1983, Third International Conference on Intercalated Graphite, Pont á Mousson, France, "Structure and Phase Transitions in Bromine and Potassium–Mercury Intercalated Graphite" (MSD).**
- **May 23, 1983, Third International Conference on Intercalated Graphite, Pont á Mousson, France, "Magnetic Properties of $CoCl_2$–Intercalated Graphite" (GD).**
- **May 27, 1983, Third International Conference on Intercalated Graphite, Pont á Mousson, France, "Conference Summary Paper" (MSD).**
- **July 18, 1983, 16th Biennial Carbon Conference, San Diego, CA, "The Physics of Intercalated Graphite", Plenary Lecture (MSD).**
- **July 18, 1983, 16th Biennial Carbon Conference, San Diego, CA, "Magnetic Properties of Graphite Intercalation Compounds", Conference Paper (GD).**
- **July 18, 1983, 16th Biennial Carbon Conference, San Diego, CA, "Structural Phase Transitions in Graphite Intercalation Compounds", Conference Paper (MSD).**
- **August 17, 1983, Plenary Lecture, Workshop on the Electrochemistry of Carbon, Case Western Reserve University, "Electronic and Lattice Properties of Graphite" (GD).**
- **August 24, 1983, Seminar, GA Technologies, San Diego, CA, "Structure–Property Relations of Boron in Graphite" (MSD).**
- **October 25, 1983, AIP Corporate Associates Meeting, XEROX Corporation, Palo Alto, CA, "The Physics of Intercalated Graphite" (MSD).**
- **November 28, 1983, Academy of Sciences, Shanghai, China, "The Physics of Intercalated Graphite" (MSD).**

- December 6, 1983, Academy of Sciences, Beijing, China, "The Physics of Intercalated Graphite" (MSD).
- December 10, 1983, Physics Colloquium, Science University of Tokyo, Tokyo, Japan, "Recent Developments in Intercalated Graphite" (MSD).
- December 12, 1983, Shinshu University, Nagano, Japan, "The Physics of Intercalated Graphite" (MSD).
- December 13, 1983, Showa Denko Company, Nagano Prefecture, Japan, "The Physics of Intercalated Graphite" (MSD).
- January 18, 1984, Physics Colloquium, University of North Carolina, Chapel Hill, NC, "The Physics of Graphite Intercalation Compounds" (MSD).
- February 3, 1984, RayChem Corporation, Menlo Park, CA, "Recent Developments and Applications of Intercalated Graphite" (MSD).
- February 18, 1984, General Colloquium, Bell Laboratories, Murray Hill, NJ, "Phase Transitions in Graphite Intercalation Compounds" (MSD).
- March 26, 1984, American Physical Society Meeting, Detroit, MI, "Shubnikov de Haas Effect in KHg_x-Graphite Intercalation Compounds", G. Timp, P.D. Dresselhaus and G. Dresselhaus.
- March 26, 1984, American Physical Society Meeting, Detroit, MI, "Kosterlitz–Thouless Phase Transitions in Finite Size Systems: Application to $CoCl_2$–Graphite Intercalation Compounds", K.Y. Szeto, M. Elahy, S.T. Chen and G. Dresselhaus.
- March 26, 1984, American Physical Society Meeting, Detroit, MI, "Raman Microprobe Studies of the Structure of $SbCl_5$-Graphite Intercalation Compounds", L.E. McNeil, J. Steinbeck, L. Salamanca-Riba and G. Dresselhaus.
- May 28, 1984, Physics Colloquium, University of Tennessee, Knoxville, TN, "The Physics of Intercalated Graphite" (MSD).
- June 18, 1984, Colloquium, Naval Research Laboratory, Washington, DC, "Phase Transitions in Intercalated Graphite" (MSD).
- July 5, 1984, Carbone 84 Conference, Bordeaux, France, "Intercalation of Ion Implanted Graphite", H. Menjo, B.S. Elman, G. Braunstein and M.S. Dresselhaus.
- July 5, 1984, Carbone 84 Conference, Bordeaux, France, "Magnetic Phase Transitions in $CoCl_2$-Graphite Intercalation Compounds", S.T. Chen, K.Y. Szeto, M. Elahy and G. Dresselhaus.
- August 6, 1984, 17th Conference on the Physics of Semiconductors, San Francisco, CA, "2-D Magnetic Phase Transitions in Graphite Intercalation Compounds", K.Y. Szeto, S.T. Chen, G. Dresselhaus and M.S. Dresselhaus.

- August 6, 1984, 17th Conference on the Physics of Semiconductors, San Francisco, CA, "The Electronic Phase Transition in Graphite Under Strong Magnetic Field", Y. Iye, L.E. McNeil, G. Dresselhaus, G. Boebinger and P.M. Berglund.

5 New Discoveries, Patents or Inventions

None.

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