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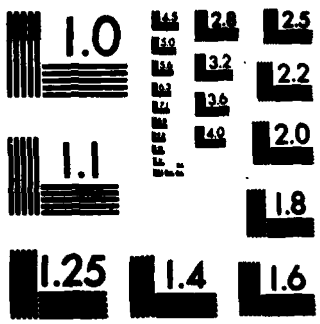

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Five major contributions to research in graphite intercalation compounds (GIC's) have been made. They are (i) the discovery of superconductivity in the mercurographitides ($KHgC_8$ and $RbHgC_8$) which was first seen in low temperature specific heat (C_p) studies, (ii) that low-energy phonon states appear to play a role in suppressing T_c for superconducting GIC's and may suppress superconductivity altogether, (iii) the re-awakening of interest in magnetic graphtie intercalation compounds arising in part from our specific heat studies

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which suggest the possibility of a magnetic spin-glass state in $FeCl_3$ and $NiCl_2$ compounds, and (iv) the confirmation that a low density of electronic states is common to a wide class of acceptor intercalation compounds. In addition (v) it permitted completion of research that showed for the first time the universality of "twin" phase transitions in donor alkali metal GIC's below stage 1. Of the above, (i), (iii) and (v) have led to a wealth of further research by other groups in recent years and have had lasting influence in this research area. Research performed under this grant was devoted primarily to the determination of the low temperature physical properties of a wide range of graphite intercalation compounds (GIC's) and the interpretation of these properties. In addition some new GIC's were synthesized and transport studies initiated elsewhere were completed.

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Attn: Dr. Max Swerdlow

FROM: David G. Onn, Department of Physics and Honors Program,
Principal Investigator

FINAL SCIENTIFIC REPORT ON GRANT AFOSR #77-3393

"Thermal and Physical Properties of Graphite Intercalation Compounds"

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I. INTRODUCTION AND SUMMARY OF RESEARCH:

Five major contributions to research in graphite intercalation compounds (GIC's) can be attributed to this grant, in whole or in part. They are, (i) the discovery of superconductivity in the mercurographitides ($KHgC_8$ and $RbHgC_8$) which was first seen in low temperature specific heat (C_p) studies, (ii) that low-energy phonon states appear to play a role in suppressing T_c for superconducting GIC's and may suppress superconductivity altogether, (iii) the re-awakening of interest in magnetic graphite intercalation compounds arising in part from our specific heat studies which suggest the possibility of a magnetic spin-glass state in $FeCl_3$ and $NiCl_2$ compounds, and (iv) the confirmation that a low density of electronic states is common to a wide class of acceptor intercalation compounds (not yet published). In addition (v) it permitted completion of research, initiated at University of Pennsylvania by Professor J. Fischer and the P.I., that showed for the first time the universality of "twin" phase transitions in donor alkali metal GIC's below stage 1.

Of the above, (i), (iii) and (v) have led to a wealth of further research by other groups in recent years and have had lasting influence in this research area.

Research performed under this grant was devoted primarily to the determination of the low temperature physical properties of a wide range of graphite intercalation compounds (GIC's) and the interpretation of these properties. In addition some new GIC's were synthesized and transport studies initiated elsewhere were completed. We first summarize the research classified by type of activity before discussing in more detail the most significant results obtained as outlined above.

A. Specific Heat (C_p) Studies: 0.5 K to 60 K

Methods for finding C_p of air sensitive and corrosive samples were specially developed for some of the compounds studied.

(i) Superconducting Compounds: $KHgC_8$, $RbHgC_8$ etc. These materials were first shown to be superconducting through our specific heat measurements which showed the entropy change arising from onset of the superconducting bulk state.[1,2] Extention of C_p studies to higher temperatures suggested the role of low energy phonon modes in the presence of superconductivity in GIC compounds in general.[3,14]

(ii) Magnetic Compounds: $FeCl_3$, $NiCl_2$ GIC's. The entropy associated with the onset of magnetic order was obtained and related to effective spin values. A spin-glass ordering state was suggested though no clear a.c. susceptibility signal was observed in $FeCl_3$ compounds.[4,5]

(iii) Other Donor Compounds: KC_8, CsC_8, RbC_8 were all synthesized in large blocks in highly oriented pyrolitic graphite (HOPG) and the linear (electronic density of states $N(E_F)$) and phonon contributions separated and compared with values for samples prepared in graphite powders, showing some influence of graphite base material on values obtained.(6,7)

(iv) Other Acceptor Compounds: GIC's of HNO_3, $AlCl_3$ and $SbCl_5$ were studied using C_p below 50 K. All showed very small linear terms, consistent with $N(E_F)$ values much lower than in donor compounds and all showed a dominant T^2 lattice contribution above 10 K compared with the T^3 contribution seen in donor compounds.(8,9,10)

B. A.C. Magnetic Susceptibility (χ)

This measurement system was developed originally for the study of spin-glasses under an NSF contract but was used extensively to search for superconductivity in GIC's and to determine the a.c. magnetic susceptibility of the $FeCl_3$ and $NiCl_2$ GIC's.(4,5)

C. Synthesis of Compounds:

Synthesis facilities were developed for a number of compounds, though many were obtained in collaborative efforts with other research groups (see III below). Synthesis at University of Delaware was limited by the absence of good X-ray facilities for characterizing materials after synthesis. Successful synthesis (verified elsewhere) of $KHgC_8$, $RbHgC_8$ was made as were KC_8, RbC_8 and CsC_8 themselves. HNO_3 intercalation was also performed since sample "stage" can be extimated from expansion and color combinations.

Facilities for hydrogenation of GIC's were also developed in cooperation with Dr. Daniel Guerard, University of Nancy, France during a visit to University of Delaware. New hydrogenated compounds were developed from this system.(11)

D. Thermo-electric Power (S):

A cryostat for the measurement of thermoelectric power S from 1.5 K up to 300 K was developed under this grant. It is capable of determining S vs. T for two air-sensitive compounds together in a fully automated system. Shortage of personnel, particularly well-qualified graduate students, has delayed completion of this cryostat until May 1983. It has been tested and calibrated using known materials and will be used in future studies of GICs and related compounds.

E. Electrical Resistivity (ρ), Magneto Resistance and Hall Effect:

Initial studies of electrical resistivity (ρ) of donor GIC's below 300 K were undertaken at the University of Pennsylvania in collaboration with Prof. J. Fischer. Magneto-resistance and Hall effect studies were also made. The first clear identification of the "twin" resistivity anomalies in all but Stage 1 alkali-metal GIC's was obtained.[12,13] This discovery has led to intensive research into structural facets of these phase transitions in other laboratories. Electrical resistivity measurements, again, fully automated, are now available at University of Delaware as a result of research support in a related area (amorphous metal alloys) and will be used for GIC's in the future.

F. Physisorbtion

In addition to hydrogen compound formation with GIC's some are known to physisorb hydrogen near 90 K. Preliminary studies were undertaken with Dr. Daniel Guerard, University of Nancy during a collaborative visit supported by this grant.

II. DETAILED DISCUSSION OF MOST SIGNIFICANT RESULTS FROM THIS RESEARCH:

A. Specific Heat and Superconductivity of Stage 1 and 2 Mercurographitides $KHgC_4$, $KHgC_8$, $RbHgC_4$, $RbHgC_8$:[1,2,3,14]

Specimens for this research were obtained by collaboration with Drs. Daniel Guerard and Phillipe Lagrange, University of Nancy, France. Later specimens were synthesized at University of Delaware. The air-sensitivity of these materials required that their specific heat be studied in the unique cryostat at U. of D. which can be reduced in size to permit sample loading within the confining space of an inert-atmosphere glove-box.

C_p for all four compounds was measured from about 0.7 K to 40 K using a thermal relaxation technique. Superconductivity in $KHgC_8$ and $RbHgC_8$ was first discovered in these compounds by observing the rise in C_p at the transition temperature T_c.[1] Analysis of the C_p into electronic and lattice contributions showed that the superconductivity was bulk in nature and could be described approximately in the BCS picture.[1,2] T_c for these two stage 2 compounds was below 2 K. We did not observe similar anomalies in C_p for stage 1 compounds ($KHgC_4$, $RbHgC_4$) which have since been shown to have T_c's lower than for the stage 1 compounds. This seemingly anomalous trend in T_c is still not clearly understood but may be related to the phonon properties as we describe next.

By extending our C_p studies of KC_8, CsC_8, RbC_8 and the above mercurographitides up to 50 K we have obtained a unique insight into the relationship between superconductivity and phonon properties in these layered GIC's. C_p values above about 10 K are dominated by the lattice contribution which may be compared with the "ideal" Debye model for phonon energy distribution. It appears that those materials with a phonon distribution closest to the Debye model show the highest T_c's, while materials with large anomalous contributions to C_p from low-lying energy levels show lower T_c's or do not superconduct. This research is in final analysis prior to publication[3,14]. It strongly suggests that the dominance of $N(E_F)$ as an indicator of superconductivity is not strong in the layered GIC's.

B. Studies of Magnetic Intercalation Compounds: $FeCl_3$, $NiCl_2$:[4,5]

Samples for these studies were obtained by collaboration with Dr. John Ritsko (Xerox) and Professor Serge Flandrois, University of Bordeaux, France. We studied both the low-field a.c. magnetic susceptibility (χ) and the specific heat C_p up to 60 K. The $NiCl_2$ (stage 2) compound showed a large magnetic anomaly in χ at about 23 K presumably associated with the onset of magnetic order. The C_p results were dominated at low temperatures by a large linear term which suggested that the magnetic state could be a spin-glass state.[4,5]

In contrast, the $FeCl_3$ compounds showed no signal in the a.c. susceptibility despite a very large anomaly in C_p at about 2 K for stage 2 and 3.5 K for stage 1. Again, C_p below the anomaly was almost linear and a spin-glass magnetic state was postulated though the near absence of a susceptibility signature remained a puzzle.[4,5] Later, further extensive studies of these and related magnetic GIC's have been undertaken by the MIT group (Dresselhaus, Dresselhaus et. al.) following our own initial research.

C. Stage 1 Donor Compounds: KC_8, CsC_8 and RbC_8:[6,7]

This study, of C_p alone, was the first ever undertaken on single piece bulk samples of these compounds synthesized in Highly Oriented Pyrolitic Graphite (HOPG) and directly compared with the parent graphite. Differences between our results and those of other authors who used graphite powder based samples suggests that both $N(E_F)$ and the phonon properties are mildly affected by the parent graphite. Apart from providing excellent values of $N(E_F)$ for HOPG based stage 1 compounds this study revealed important phonon effects. By extending C_p up to 80 K we observed for KC_8 that the behaviour is very "Debye-like" while for RbC_8 and CsC_8 remarkably strong deviations from Debye behaviour were observed that were attributable to low-lying energy levels possibly akin to those observed in disordered materials such as glasses. As noted above (II. A.) the absence of these low-lying levels seems to be correlated with a high superconducting transition temperature (T_c for C_8K has been reported as high as 0.55 K) while their presence appears to inhibit superconductivity (RbC_8, CsC_8 are not reliably known to superconduct or do so only near 0.1 K). A similar relationship between T_c and phonon behavior in the mercurographitides was also inferred as discussed in (II. A.) above.[3]

D. Acceptor Compounds: HNO_3, $AlCl_3$, $SbCl_5$.[8,9,10]

These compounds were obtained by collaboration with Professor J. Fischer (Univ. of Pennsylvania) (HNO_3), Professor Gil Hooley (University of British Columbia) ($AlCl_3$) and Professor Peter Eklund (University of Kentucky) ($SbCl_5$). In addition HNO_3 samples were synthesized at University of Delaware.

The results obtained for C_p studies of these compounds (which have not yet been published in full) show very similar results for all compounds and stages. In all samples studied the electronic contribution of C_p, which is directly proportional to the electronic density of states at the Fermi level $N(E_F)$ is very small, only two or three times that of the parent graphite. This contrasts with typical donor compounds where $N(E_F)$ is twenty to thirty times that of the parent graphite. The reported higher conductivity in these compounds is then presumably due to enhanced mobility of carriers rather than to enhanced $N(E_F)$. A further result, again shown by extending C_p studies to higher temperatures, is that for all of these compounds the phonon specific heat above about 5 K is dominated by a T^2 term rather than a T^3 term suggesting that the phonon spectrum is predominantly two-dimensional as is the case in graphite but not in donor compounds. These results in turn suggest that a different theoretical approach is necessary in discussing phonon properties of donor as opposed to acceptor GIC's.

E. Electrical Resistivity, Magneto Resistance and Hall Effects of Donor Compounds:[12,13]

This research component was undertaken in collaboration with Professor J. Fischer, University of Pennsylvania but was completed with support from this AFOSR Grant to University of Delaware.

An extensive survey of electrical resistivity of donor graphite intercalation compounds based upon alkali-metals and ranging from stage 1 to stage 4 was undertaken. Related magneto-resistance and Hall effect studies were also made. The report of the now well-known "twin" anomalies in ρ of the stage 2 to 4 compounds of K, Rb and Cs at temperatures below 300 K was the first evidence that these twin anomalies were common to all such compounds and to all stages except stage 1.[12] Since then a number of research groups have undertaken extensive structural studies of the "twin" phase transitions for which the anomalies in ρ first provided evidence. These later studies have verified the composition and stage dependence of the phase transition temperatures that we reported and have provided partial explanations for their occurrence.

In addition to the anomalies observed in ρ we also observed anomalous behaviour, in the same temperature regions, in the magneto-resistance and in the Hall effect.[13] In particular large changes in the magnitude and temperature dependence of the Hall effect occur at the lower transition temperature ($T_L \simeq 93$ K in KC_{24}), but only a small step in magnitude with no change in temperature dependence is observed at the upper transition ($T_L \simeq 123$ K in KC_{24}). While the Hall effect is often used as a measure of carrier concentration such a simple explanation cannot be made in this case since the donor GIC's are known to be two-carrier systems making the Hall effect and magneto-resistance complex functions of both carrier density and carrier mobility. As a result no quantitative explanation of the anomalies in the transport properties is yet possible, but more extensive studies should be undertaken in the future, especially since the characteristics of the Hall effect attributed to KC_{24} above are not universal in this class of material, and each compound seems to exhibit different behaviour.(13)

F. Hydrogen Interactions In GIC's: New Compounds and Physisorbtion:

As part of this research program Dr. Daniel Guerard, University of Nancy, visited on two occasions for periods of several months at the University of Delaware. In addition to collaborating in the synthesis of mercurographitide compounds (see II. A. above) and in the study of their superconductivity, collaborative studies on the interaction of hydrogen with GIC's was undertaken.

Hydrogen is known to form the compound $KC_8H_{2/3}$ and also to physisorb in KC_{24} and other stages of alkali metal compounds at temperatures near 90 K. Samples of $KC_8H_{2/3}$ and of $RbC_8H_{2/3}$ were synthesized, the latter being reported for the first time(11). Further collaborative studies of the physical properties of these compounds and their derivatives will be undertaken in the future.

In addition, preliminary studies of physisorbtion conditions for GIC's were undertaken and a higher rate of physisorbtion detected than had previously been reported. This research has not been published so far and will require further study for an effective and complete report.

III. National and International Collaborators in This Research:

A number of important collaborators in the U.S.A. and abroad have made this research possible and should be recognized in this report. They include:

Dr. A. Moore, Union Carbide Corporation (supply of HOPG)

Professor J. Fischer, University of Pennsylvania (transport study collaboration and supply of HNO_3 compound)

Dr. Daniel Guerard, University of Nancy, France (extensive collaborative visits and supply of samples, particularly superconducting mercurographitides)

Dr. Phillipe LaGrange, University of Nancy, France (supply of mercurographitide compounds)

Professor Serge Flandrois, University of Bordeaux, France (provision of $NiCl_2$ GIC)

Dr. John Ritsko, Xerox, Webster, N.Y. (supply of $FeCl_3$ GIC)

Professor Gil Hooley, University of British Columbia (supply of $AlCl_3$ GIC)

Professor Peter Eklund, University of Kentucky (supply of $SbCl_5$ GIC)

In addition, all of the above, and many others, contributed useful critique and discussions of ongoing research.

IV. Student and Post-Doctoral Participation in Research: Degrees Awarded

At the University of Delaware, Department of Physics the following personnel participated in this research project.

Professor David G. Onn, Principal Investigator: Promoted to Full Professor, Department of Physics and University Honors Program, September 1982. Several invited presentations on graphite intercalation compounds at other institutions. Member, International Advisory Committee, Third International Conference on Graphite Intercalation Compounds, Pont-a-Mousson, France, May 1983.

Mr. David Goshorn (M.S. candidate): Specific heat studies of several compound series including KC_8, RbC_8, CsC_8 and early studies of the superconducting mercurographitides. Mr. Goshorn moved to Exxon Research and Development Center, Linden, N.J.

Dr. M. Grayson Alexander. Ms. Alexander obtained her Ph.D. entirely with support from this research grant. In addition to her thesis research which related to the thermal properties of the superconducting mercurographitides she participated in studies of magnetic compounds and in early studies of the thermal properties of acceptor compounds. She also developed our synthetic capability in donor compounds. Her Ph.D. was completed in only 4½ years from her B.S. She is currently at Combustion Engineering, Windsor, Connecticut.

Dr. Yoshihisa Obi. Dr. Obi was a visiting Post-doctoral Fellow with primary support from the National Science Foundation to work on amorphous metal alloys. His spontaneous interest in GIC's led to his partial support on this grant for a limited time and participation in thermal and magnetic studies of various compounds. Dr. Obi has returned to the Institute for Research in Iron, Steel and Other Metals, Sendai, Japan.

Mr. Liu-Quan Wang: Mr. Wang is a mature graduate student visiting from Peking, PRC. He is primarily working in the area of amorphous metal alloys but has participated and collaborated in research on GIC's particularly in the area of thermal properties of acceptor GIC's. He will complete his Ph.D. in December 1983 after only three years in the U.S.A.

Dr. M. ElMakrini: Dr. ElMakrini was a graduate student of Dr. Daniel Guerard and Dr. Phillipe Lagrange, University of Nancy, France and participated in the synthesis of the superconducting mercurographitide samples.

Undergraduate research participants: A number of short-term undergraduate research participants have been involved in various aspects of this research especially in the areas of data reduction and computer programming.

V. Publications Resulting From This Research:

The following publications, past or pending, resulted from the research funded under AFOSR Grant #77-3393. They are listed by reference number related to the text of the report. Copies are attached when these are available.

PLEASE NOTE THAT ONLY REFEREED PUBLICATIONS ARE INCLUDED, AND THAT APS MEETING ABSTRACTS (Bull. Am. Phys. Soc.) ARE NOT: one or two such abstracts were published each year of the contract but have since, with a few exceptions pending, been updated to full publications as listed below:

1. "Superconductivity of the Graphite Intercalation Compounds $KHgC_8$ and $RbHgC_8$: Evidence from Specific Heat" M. G. Alexander, D. P. Goshorn, D. Guerard, M. ElMakrini and David G. Onn, Sol. State Comm. 38, 103 (1981)
2. "Synthesis and Low Temperature Specific Heat of the Graphite Intercalation Compounds $KHgC_4$ and $KHgC_8$" M. Grayson Alexander, D. P. Goshorn, D. Guerard, M. ElMakrini, P. Lagrange and David G. Onn, Synthetic Metals, 2, 203 (1980)
3. M. Grayson Alexander, Ph.D. Thesis, University of Delaware and to be published (with David G. Onn)
4. "Low Temperature Specific Heat and Low Field Magnetic Susceptability of Second State $NiCl_2$ and $FeCl_3$ Graphite Compounds" David G. Onn, M. Grayson Alexander, J. J. Ritsko and S. Flandrois, Proc. 16th Biennial Conf. on Carbon p. 44 (1981)
5. "Heat Capacity and Magnetic Studies of Graphite Intercalated With $FeCl_3$ and $NiCl_2$" David G. Onn, M. Grayson Alexander, J. J. Ritsko and S. Flandrois, J. Appl. Phys. 53, 2751 (1982)
6. "Specific Heat, 0.4 K to 90 D, of C_8K, C_8Cs, C_8Rb and Their Parent HOPG" M. G. Alexander, D. P. Goshorn, and David G. Onn, Proc. 14th Biennial Conf. on Carbon, p. 276 (1979).
7. "Low Temperature Specific Heat of the Graphite Intercalation Compounds KC_8, CsC_8 and RbC_8 and Their Parent Highly Oriented Pyrolitic Graphite" M. Grayson Alexander, David P. Goshorn, and David G. Onn, Phys. Rev. B22, 4535 (1980)

8. "Low Temperature Specific Heat of HNO_3 Graphite Intercalation Compounds" David G. Onn, David P. Goshorn and L. Q. Wang (in preparation)

9. "Low Temperature Specific Heat of $AlCl_3$ Graphite Intercalation Compounds" David G. Onn, G. Hooley, Y. Obi and L. Q. Wang (in preparation).

10. "Low Temperature Specific Heat of $SbCl_5$ Graphite Intercalation Compounds" David G. Onn, M. Grayson Alexander, P. Eklund and L. Q. Wang (in preparation)

11. "Ternary and Quaternary Compounds Derived From the First Stage Graphitide of Rubidium" P. LaGrange, D. Guerard, A. Herold and David G. Onn, Proc. 16th Biennial Conf. on Carbon. p.383 (1981)

12. "Resistivity Anomalies and Phase Transitions in Alkali-Metal Graphite Intercalation Compounds" David G. Onn, G. M. T. Foley and J. E. Fischer, Mater. Sci. and Eng. 31, 271 (1977)

13. "Electronic Properties, Resistive Anomalies and Phase Transitions in the Graphite Intercalation Compounds with K, Rb and Cs" David G. Onn, G. M. T. Foley and J. E. Fischer, Phys. Rev. B19, 6474 (1979)

14. "Superconductivity and Phonon Specific Heat of the Alkali Metal Mercurographitides" M. G. Alexander, D. Guerard, P. Lagrange, M. ElMakrini and D. G. Onn. Proc. 16th Biennial Conf. of Carbon p 56 (1981)