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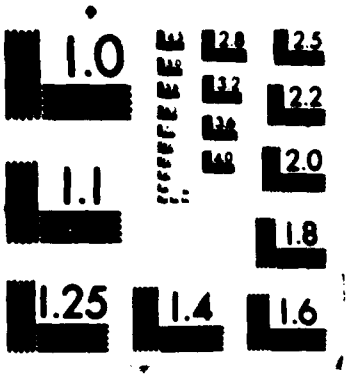
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SECURITY CLASSIFICATION OF THIS PAGE

REPORT DOCUMENTATION PAGE

1a. REPORT SECURITY CLASSIFICATION: Unclassified	1b. RESTRICTIVE MARKINGS
2a. SECURITY CLASSIFICATION AUTHORITY	3. DISTRIBUTION/AVAILABILITY OF REPORT: Unlimited
2b. DECLASSIFICATION/DOWNGRADING SCHEDULE	
4. PERFORMING ORGANIZATION REPORT NUMBER(S)	5. MONITORING ORGANIZATION REPORT NUMBER(S): AFOSR-TR- 87-0745
6a. NAME OF PERFORMING ORGANIZATION: Massachusetts Institute of Technology	6b. OFFICE SYMBOL (If applicable)
7a. NAME OF MONITORING ORGANIZATION: AFOSR/NE	
6c. ADDRESS (City, State and ZIP Code): Rm. 13-3005, Massachusetts Institute of Technology, 77 Massachusetts Avenue, Cambridge, MA 02139	7b. ADDRESS (City, State and ZIP Code): Building 410, Bolling AFB, DC 20332-6448
8a. NAME OF FUNDING/SPONSORING ORGANIZATION: AFOSR	8b. OFFICE SYMBOL (If applicable): NE
9. PROCUREMENT INSTRUMENT IDENTIFICATION NUMBER: F49620-83-C-0011	
8c. ADDRESS (City, State and ZIP Code): AFOSR/NP ATTN: H. Weinstock, Bolling AFB, Washington, DC 20332-6446	10. SOURCE OF FUNDING NOS.

PROGRAM ELEMENT NO.	PROJECT NO.	TASK NO.	WORK UNIT NO.
61102F	2306	C3	

11. TITLE (Include Security Classification): Synthetic Metals from Intercalated Graphite

12. PERSONAL AUTHOR(S): M.S. Dresselhaus and G. Dresselhaus

13a. TYPE OF REPORT	13b. TIME COVERED	14. DATE OF REPORT (Yr., Mo., Day)	15. PAGE COUNT
Semi Annual	FROM 10-1-86 TO 3-31-87	1987-5-7	10

16. SUPPLEMENTARY NOTATION

17. COSATI CODES			18. SUBJECT TERMS (Continue on reverse if necessary and identify by block number)
FIELD	GROUP	SUB. GR.	

19. ABSTRACT (Continue on reverse if necessary and identify by block number)

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20. DISTRIBUTION/AVAILABILITY OF ABSTRACT	21. ABSTRACT SECURITY CLASSIFICATION
UNCLASSIFIED/UNLIMITED ☒ SAME AS RPT. ☐ OTIC USERS ☐	Unclassified

22a. NAME OF RESPONSIBLE INDIVIDUAL	22b. TELEPHONE NUMBER (Include Area Code)	22c. OFFICE SYMBOL
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DD FORM 1473, 83 APR EDITION OF 1 JAN 73 IS OBSOLETE.

SECURITY CLASSIFICATION OF THIS PAGE

Fifth Semi–Annual Report
to the
Air Force Office of Scientific Research
for research on
Synthetic Metals from Intercalated Graphite

AFOSR Contract #F49620–83–C–0011
for the period
October 1, 1986 — March 31, 1987

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May 11, 1987

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1 Overview

This Semi-annual report contains a restatement of the proposed research under the contract and summarizes the research progress made during the fifth six month period of this contract, October 1, 1986 - March 31, 1987.

1.1 Abstract

During the six month period October 1, 1986—March 31, 1987, efforts on the Program "Synthetic Metals from Intercalated Graphite" were largely focused on studies of magnetic and superconducting graphite intercalation compounds. A summary of the accomplishments of the research program is presented.

1.2 Statement of Work

Statement of work in AFOSR contract #F49620–83–C–0011 on "Synthetic Metals from 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 "Synthetic Metals from Intercalated Graphite" is presented in terms of the progress made during the period October 1, 1986 to March 30, 1987. In presenting the summary, we refer by number (#[n]) to the publications for the six month period October 1, 1986 to March 30, 1987 following the publication numbers which are listed in section 3.1.

2.1 Magnetic Graphite Intercalation Compounds

Progress was made primarily in the areas of transport measurements of magnetic stage 1 and stage 2 $CoCl_2$–GICs, modeling of the transport properties of these magnetic $CoCl_2$–GICs, Monte Carlo simulations of spin arrangements and structural studies relevant to these magnetic intercalation compounds.

2.1.1 Transport Measurements of Magnetic $CoCl_2$–GICs

The magnetic intercalation compounds $CoCl_2$–GICs are well known as 2D–XY systems with a strong ferromagnetic in–plane nearest neighbor coupling and a relatively weak antiferromagnetic interplanar coupling. Although the magnetic properties of these compounds have been extensively studied, no detailed measurements of the transport properties had been carried out previously.

In the stage 1 compound, Nai–Chang Yeh in her thesis work has found an anomalous increase in the resistivity $\rho(T)$ as the temperature T is lowered below $T_{c2} = 9.7K$. while the stage–2 compound shows a sudden decrease in $\rho(T)$ below $T_{c\ell} = 9.2K$. These anomalies correspond to the temperatures of the susceptibility peaks for the stage–1 and stage–2 compounds and the observed anomalies are attributed to the interaction between carbon π–electrons and Co^{2+} spins. However, the spin fluctuation scattering due to the $\pi - d$ exchange interaction cannot account for the behavior of the stage–1 compound as the spin disorder scattering process is quenched by the onset of spin ordering. In this context, the behavior of the stage–2 compound is as expected. In the stage–1 compound, an antiferromagnetic interplanar exchange coupling J' (in the range $\sim 10^{-1} - 10^{-2}K$) induces an antiferromagnetic spin ordering below $T_{c\ell} = 9.7K$ of ferromagnetically arranged planes of spins.

Measurements of the transverse magnetoresistance with $\vec{j} \parallel$ a–axis and $\vec{H} \perp$ c–axis for the stage–2 $CoCl_2$–GICs below $T_{c\ell}(\sim 9.2K)$ show an approximately linear decrease of the resistivity in the low field range, and then saturation at higher H, typical for the resistivity derived from disorder–spin scattering. The total decrease of $\rho(H)$ from $H = 0$ to the saturation limit for $T < T_{c\ell}$ is only weakly temperature–dependent and has a magnitude of about -1%. For $T > T_{c\ell}$, a larger change in the resistance occurs as the field is increased, and the magnitude of $\Delta\rho$ in the field range of our experiments is about -1.2%. The negative magnetoresistance above $T_{c\ell}$ is not completely saturated up to the highest magnetic field in our measurements ($\sim 1.2kOe$). The observed temperature and field dependence of the resistivity is typical of spin–disorder scattering.

Magnetic ordering occurs below $T_{c\ell}$, where spins on a given plane align ferromagnetically and the interplanar antiferromagnetic alignment of the sequential planes of spins is established. The amount of spin–disorder scattering is smaller in this temperature range. The effect of the magnetic field for $T < T_{c\ell}$ is to provide additional magnetic ordering to the partially ordered spin system, thereby further reducing the spin–disorder scattering. Thus it is expected that the change in resistance with applied field will slowly decrease with decreasing temperature.

As T is increased above $T_{c\ell}$, the long–range ordering of spins breaks down, and the effect of an external magnetic field becomes more important. Therefore the magnitude of the negative magnetoresistance is expected to be larger in this temperature range, in agreement with our experimental results.

The magnetoresistance of the stage–1 $CoCl_2$–GIC in the same low field range ($0 < H < 1.2kOe$) shows a negative magnetoresistance for $T > T_{c\ell} \sim 9.8K$ which we attribute to spin–disorder scattering, since the long range magnetic ordering has already broken down. But for $T < T_{c\ell}$, two anomalous differences between stage–1 and stage–2 occur. For the stage–1 compound the resistivity remains nearly constant until $H = H_{c2} \sim 300Oe$, where a sharp decrease of $\rho(H,T)$ takes place, indicating a magnetic phase transition at $H = H_{c2}$. The resistivity then saturates for $H > 1kOe$. This type of plateau in resistivity is not apparent for the stage–2 compounds. Here H_{c2} is consistent with the upper critical field observed in the magnetic susceptibility measurements on stage–1 compounds where it has been attributed to the interplanar spin–flop/paramagnetic phase transition. We also find the order of magnitude of the negative magnetoresistance in stage–1 is about ten times larger than that in stage–2.

2.1.2 Modeling of Transport Properties in Magnetic $CoCl_2$–GICs

To explain the anomalous behavior of the stage 1 magnetic $CoCl_2$–GIC, the Fermi surface modification caused by the antiferromagnetic stacking of the ferromagnetic sheets has been considered, including the effect of this modification on the electron scattering and therefore on the resistivity. Our calculation can successfully account for an increase in the resistivity for the stage 1 compound below the Neél temperature. In addition, the Fermi surface modification is found to be more important than the spin disorder scattering mechanism which dominates the stage 2 behavior. Furthermore because the interplanar exchange interaction J' is much weaker for the stage 2 compound, the Fermi surface modification effects are much smaller than the spin–disorder scattering effect. The model also allows estimation of $J_{\pi d}$, the interaction energy between the carbon π–electrons and the spins on the magnetic Co^{2+} species. Evaluation of $J_{\pi d}$ for the stage 1 acceptor $CoCl_2$–GIC yields a value of $J_{\pi d}$ that is one order of magnitude smaller than the corresponding term for the donor GIC, C_6Eu.

2.1.3 Monte Carlo Simulations

In his thesis work, James Nicholls has been able to show the correspondence between the zero temperature mean field model of the antiferromagnetic 1D XY chain and the Monte Carlo simulations. Recent work with a finite temperature mean field model shows that the ratio of H_{c2}/H_{c1} is between 2 and 3 even when the angle between the applied magnetic field and the six–fold field is varied over the entire relevant range from 0 to $\pi/3$. This has enabled us to use the field dependent susceptibility (χ vs H) data to gain order of magnitude estimates of the interplanar exchange interaction J' and the six–fold symmetry–breaking field H_6 in the stage 1 $CoCl_2$–GIC. In stage 2 compounds we are not yet sure whether this model is appropriate, especially since there is some speculation that the stage 2 compound might be a spin glass. It is of interest that for the stage–1 $CoCl_2$–GIC, hysteresis has recently been observed in the in–plane χ vs H measurements, but only below $T_{c\ell}$.

2.1.4 Structural Studies

In his thesis work, James Speck has prepared $CuCl_2$–GICs in HOPG and kish graphite host material in stages 1 and 2. High resolution TEM studies on these samples at room temper-

ature show that the stage 1 $CuCl_2$–GIC has crystallographic correlations between different intercalate layers. Stage 2 samples do not display any interlayer correlations. Single crystals of stage 3 $CuCl_2$–GIC also lack any intercalate interlayer correlations as determined by single crystal x–ray precession photographs. $CoCl_2$–GICs with stage index 1,2, and 3 have been successfully prepared in several kinds of graphite host materials. High resolution TEM results and single crystal x–ray precession results indicate that the stage 1 $CoCl_2$–GIC samples have intercalate interlayer correlations. High stage $CoCl_2$ samples lack these correlations. Additionally, we have successfully prepared stage 2 $NiCl_2$–GICs and a mixed stage $PdCl_2$–GIC sample and the corresponding structural measurements will be carried out in the future on these samples. Finally, we have successfully prepared several mixed stage $InCl_3$–GIC samples. This material is of special interest because the intercalate layer in this system is commensurate with the graphite substrate. There is great hope that study of this system will eventually lead to a better understanding of the intercalate distribution on a layer plane.

2.2 Superconducting Graphite Intercalation Compounds

The temperature and angular dependence of the critical field have been measured by Alison Chaiken in her PhD thesis work in the stage 1 KHg–GICs and the experiments can be approximately fit by the Lawrence–Doniach (LD) model

$$\left(\frac{H_{c2}(\theta)\cos\theta}{H_{c2\|}}\right)^2+\left(\frac{H_{c2}(\theta)\sin\theta}{H_{c2\perp}}\right)^2=1$$

which was used previously by Iye et al. to fit data taken at about $T/T_c \equiv t = 0.55$. The parameters $H_{c2\|}$ and $H_{c2\perp}$ obtained from the fit to the data set taken at $t = 0.79$ are a perpendicular critical field $H_{c2\perp}$ of 117 gauss for $H \perp$ to the c–axis and a critical field anisotropy $(H_{c2\perp}/H_{c2\|})$ of 8. The magnitude of the disagreements between this higher temperature fit and the data are within the experimental uncertainty (about 15 gauss near $\theta = 90°$ and about 5 gauss near $\theta = 0°$). From a fit to the lower–temperature trace, the corresponding results are $H_{c2\perp} = 506$ gauss and $H_{c2\perp}/H_{c2\|} = 13$. Thus the fitted parameters at $t = 0.79$ and 0.30 sandwich the values previously reported by Iye at $t = 0.55$, as would be expected. Anomalous behavior is also observed for the temperature dependence of $H_{c2}(\theta, T)$ which appears to follow the $(1-T/T_c)$ functional form over a much wider temperature range than would be expected.

One would not expect the first stage C_4KHg_x to be two–dimensional because its c–axis coherence length $\xi_{\|}$ (as determined from the critical–field data) is on the order of 2300Å, far greater than the c–axis lattice constant of about 10.2Å, while these compounds may safely be assigned to the 3D limit. In the closely related alkali metal bismuth graphite intercalation compounds, the z–axis mean free path ℓ has been estimated to be less than I_c, the c–axis repeat distance. If similar transport properties are found in the alkali metal mercury GICs, then the superconductivity "dirtiness" parameter ξ_0/ℓ where ξ_0 is the BCS coherence length, should be much greater than one, and the compound should be in the dirty limit as far as interplanar transport is concerned. In this context it is of interest to note that the intercalated transition metal dichalcogenide superconductors are also in the dirty limit, where values of ξ_0/ℓ as high as 120 are obtained.

For these KHg–GICs which support superconductivity, high resolution structural studies are being carried out to understand the basic structure of the intercalant layer relative to the graphite, and the phase transitions that occur as a function of temperature. For the stage 2 compound, we have recently observed a structural phase transition near 140°C which we attribute to the intercalant melting. This will be studied in some detail in the Ph.D. thesis

work of Ali Kazeroonian. Some $(2 \times 2)R0°$ in–plane order persists above the transition temperature, suggesting a 2–dimensional phase transition. Moreover, a new incommensurate and orientationally locked structure was observed in this compound. For the stage 1 compound, we had previously observed interplanar correlation at room temperature, while the stage 2 sample showed no stacking correlation. Thus the stage 2 sample is expected to exhibit quasi 2–dimensional phase transitions. This work is being pursued in more detail in the coming months.

We have recently completed construction of a high purity hydrogenation system and are currently reacting some stage 1 KHg–GICs having low T_c values (i.e., $T_c < 1.5$K) with hydrogen. These samples will be studied with regard to their temperature and angular dependence of $H_{c2}(\theta, T)$ and the results will be compared to samples having T_c values $\simeq 1.5$K without hydrogen doping.

2.3 Other Projects

During the fifth reporting period of this contract, two new postdoctoral associates joined our group and initiated projects relevant to the overall goals of this program, branching out into new research areas as well.

2.3.1 "a-face" Studies on Graphite Intercalation Compounds

As a preliminary step toward a variety of possible "a–face" studies on intercalated graphite, effort was given to preparing a better "a–face" by oxygen–etching of pristine graphite (HOPG). The SEM pictures show a bright line parallel to the layers. The typical sizes of these lines are: 100μm parallel to the layers and 5μm perpendicular to the layers. Using an optical microscope it is clear that the reflectivity of light polarized parallel to the layers is greater than the reflectivity of light perpendicular to the layer. We estimate that in the visible $R_{\|}/R_{\perp} \sim 3$. This quality of "a-face" may be sufficient for many of the interesting optical measurements which need a high penetration depth.

2.3.2 Optical Transmission on Stage 2 AsF_5–GICs

Optical transmission has been done on very thin stage 2 AsF_5–GICs. We have measured with high accuracy the threshold for valence to conduction band transitions and the broadening associated with this transition (1.9eV and 0.026eV, respectively). We showed that the data cannot be fitted with the simple Blinowski–Rigaux model. The conclusion is that the electronic bands may not have a strictly linear $E(k)$ relation even in the $0 < k < k_F$ region.

3 Reports and Publications

3.1 Publications

1. **"Intercalation in Layered Materials"**, M.S. Dresselhaus, *Bulletin of the Materials Research Society*, xxx, (1987).

2. **"Transport Properties in $CoCl_2$ Graphite Intercalation Compounds"**, K. Sugihara, N.–C. Yeh, J.T. Nicholls, G. Dresselhaus and M.S. Dresselhaus, In *Extended Abstracts of the 18th Biennial Conference on carbon*, page xxx, (1987), July 19-24, 1987, Worcester Polytechnic Institute.

3. **"Low Temperature H_{c2} Measurements in C_4KHG_x"**, A. Chaiken and M.S. Dresselhaus, In *Extended Abstracts of the 18th Biennial Conference on carbon*, page xxx, (1987), July 19-24, 1987, Worcester Polytechnic Institute.

4. **"Studies on the Anomalous Transport Properties of Acceptor–Type Magnetic GICs"**, N.–C. Yeh, K. Sugihara, J.T. Nicholls, M.S. Dresselhaus and G. Dresselhaus, In *Extended Abstracts of the 18th Biennial Conference on Carbon*, page xxx, (1987), July 19-24, 1987, Worcester Polytechnic Institute.

5. **"Theory of Transport Properties of Magnetic Graphite Intercalation Compounds"**, K. Sugihara, N.–C. Yeh, J.T. Nicholls and G. Dresselhaus, *Bull. American Physical Society* 32, 895 (1987).

6. **"Transport Properties of Magnetic Graphite Intercalation Compounds"**, N.–C. Yeh, K. Sugihara, J.T. Nicholls, G. Dresselhaus and M.S. Dresselhaus, *Bull. American Physical Society* 32, 896 (1987).

7. **"Superconducting Properties of Hydrogenated KHg Graphite Intercalation Compounds"**, A. Chaiken, G.L. Doll, Y. Liu, M.S. Dresselhaus, and P.M. Tedrow, *Bull. American Physical Society* 32, 896 (1987).

8. **"Single Crystal X–ray and Calorimetry Studies of Graphite Intercalation Compounds"**, A. Kazeroonian, M.S. Dresselhaus and A.R. Kortan, *Bull. American Physical Society* 32, 926 (1987).

3.2 Advanced Degrees

None.

3.3 Honors

- M.S. Dresselhaus
  elected to Council, National Academy of Sciences, March 1987.

- M.S. Dresselhaus
  elected to Chairman, Engineering Section, National Academy of Sciences, February 1987.

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 has been 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.

- Ko Sugihara – Research Staff
  Responsible for modeling transport properties of GICs and of scattering processes in magnetic intercalation compounds.

- Gary Doll – Postdoctoral Fellow
  Responsible for superconductivity studies in hydrogenated $KHgH_x$–GICs, for optical and Raman studies on magnetic and superconducting GICs and for improved synthesis of KHg–GICs and transition metal chloride–GICs.

- Israel Ohana – Postdoctoral Fellow
  Responsible for developing improved a–faces for Raman spectroscopy studies of intercalant modes and for exploratory work on high temperature superconductors.

- Michel Laguës – Visiting Scientist
  In his 6 month visit to MIT was responsible for investigation of surface intercalant concentration relative to the corresponding bulk concentration in donor and acceptor GICs.

- Alison Chaiken – Research Assistant and Graduate Fellowship Student
  Responsible for superconductivity studies in graphite intercalation compounds.

- Ali Kazeroonian – Research Assistant
  Responsible for high resolution x–ray measurements of structure of graphite intercalation compounds. with special emphasis on the possible connection of structure to the stabilization of the superconducting transition in stage 1 KHg–GICs..

- James Nicholls – Research Assistant
  Responsible for the synthesis of magnetic graphite compounds. for susceptibility and magnetization measurements. and modeling of two–dimensional magnetic systems.

- James Speck – NSF Fellowship Student
  Responsible for investigation of the microstructure of magnetic graphite intercalation compounds using high resolution transmission electron microscopy techniques.

- Nai–Chang Yeh – Research Assistant
  Responsible for transport studies of magnetic graphite intercalation compounds from both an experimental and theoretical viewpoint.

4.1 Coupling Activities – Seminars and Invited Conference Papers

The MIT group is strongly coupled to international activities on graphite intercalation compounds. Below are listed titles of seminars, invited talks and symposia given over the six month October 1, 1986 to March 30, 1987 period relevant to the work supported under this contract. Significant coupling activities are also listed.

- September 19, 1986, Solid State Seminar, Purdue University, "Magnetic Phase Transitions in Graphite Intercalation Compounds", (GD).

- October 21, 1986, Meeting with Dr. L. Shepard and Dr. J. Perkins of Army Materials Research Center to discuss intercalation of specific transition metals into graphite. (MSD and GD).

- November 25, 1986, Materials Science Colloquium, Carnegie Mellon University, "Phase Transitions in Graphite Intercalation Compounds", (MSD).

- December 3-5, 1986, Organized Materials Research Society Symposium on "Graphite Intercalation Compounds", (MSD and GD).

- December 6-11, 1986, Visits for several days by several distinguished workers on intercalation compounds: Dr. M. Laguës, Professor M. Matsuura, Dr. H. Miyazaki, Dr. T. Takahashi, Dr. T. Enoki, Dr. M. Endo. (MSD and GD).
- December 10, 1986, Materials Science Distinguished Lecture Series, University of Connecticut (Storrs), "Phase Transitions in Graphite Intercalation Compounds", (MSD).
- December 22, 1986, Meeting with Dr. F. Huffman of Thermoelectron Corporation regarding possible applications of intercalation compounds, (MSD).
- January - mid March, 1987, Phi Beta Kappa Lectures on 8 campuses, including lectures on Frontiers of Solid State Physics, New Materials by Intercalation and Implantation, Phase Transitions in Intercalation Compounds, Superlattices, (MSD).
- January 23, 1986, Physics Colloquium, University of Wisconsin, "Phase Transitions in Graphite Intercalation Compounds", (MSD).
- January 29, 1986, Physics Colloquium, University of South Carolina, "Phase Transitions in Graphite Intercalation Compounds", (MSD).
- March 16-21, 1987, American Physical Society March Meeting, Several papers were presented and we interacted with many researchers, (MSD and GD).
- March 24, 1986, Physics Colloquium, SUNY Binghamton, "Phase Transitions in Intercalation Compounds", (MSD).

5 New Discoveries, Patents or Inventions

None.

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