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THERMAL AND ELECTRICAL MEASUREMENTS ON SOLIDS

AT LOW TEMPERATURES

by

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February, 1974

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on
Thermal and Electrical Measurements on
Solids at Low Temperatures

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Department of Physics
Illinois Institute of Technology

This report summarizes the work done on this project from February, 1973 to the present. The participating staff consisted of H. Weinstock (Associate Professor of Physics), C. Chau (Assistant Professor of Physics), D.S. Kupperman, J.B. Hartmann and J.W.M. Bakker (Research Associates), the following graduate students: B. Vis, S. Lu and J. Ho; and undergraduate students: M. Mueller, J. Parpia, S. Stopka, D. Dziekan, R. Osa and D. Fox.

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I. SUMMARY OF RESEARCH PROGRESS

A. Thermal Conductivity of Radiation Damaged and Additively Colored MgO

Earlier measurements of neutron irradiated MgO have been better understood as a result of a recently concluded study on additively colored MgO using a crystal specimen supplied by Dr. Y. Chen of Oak Ridge National Laboratory (ORNL). A report on the additively colored MgO measurements will appear shortly in the literature.

The additively colored MgO shows a "dip" in the thermal conductivity vs. temperature curve near 20 K. This dip is attributed to resonant phonon scattering associated with quasilocalized modes of the F center. After UV irradiation, resulting in partial $F \rightarrow F^+$ conversion, the dip was found to be much weaker. The increase in conductivity of the bleached sample is assumed due to a relaxation of neighboring ions associated with the different charge state of the defect. On comparison with the optical and thermal measurements on neutron irradiated MgO, there is good agreement provided that F centers constitute approximately 10% of the oxygen ion vacancies in the neutron irradiated crystals. This is indeed plausible.

Two specimens of electron irradiated MgO were also studied. Irradiation for one was carried out at the University of Illinois, Urbana; the other, at ORNL with the assistance of Dr. Chen. Both the crystals had received a radiation dose on the order of 10^{18} e/cm², for electron energy 1.5 MeV. The expected F center concentration for this dose is $\sim 10^{17}$ cm⁻³.

The thermal conductivity of the two samples has been measured and found to be similar to that of pure unirradiated crystals at temperatures < 2 K. Phonon scattering observed in neutron irradiated samples around

1 K has been attributed to aggregate formation and correlated with optical absorption bands at 352, 573, and 975 nm.³ Aggregate formation would be expected in electron irradiated MgO only for electrons with energies >29 MeV, much higher than used here. There was indeed no evidence for aggregates either in the low temperature thermal conductivity or in the optical absorption spectrum.

At higher temperatures, between 2 K and 55 K, preliminary measurements of the thermal conductivity of one electron irradiated sample indicate much less scattering than would be expected for the estimated F center concentration. However, this correlates with the optical absorption spectrum of the material which, from the F center band at 250 nm, gives an F defect concentration of only $3 \times 10^{16}/\text{cm}^3$.

The thermal conductivity of the other sample did have a resonant scattering of the magnitude expected from the estimated F center concentration. However, the optical absorption was anomalously low, possibly from damage during the chemical polishing of the surface.

Thermal conductivity and optical measurements of these samples are being continued to check the correspondence between F center concentration and phonon scattering.

B. Thermal Conductivity of Lead Films in the Intermediate State

When a type I superconductor is cooled below its transition temperature and subsequently exposed to a magnetic field perpendicular to its largest surface, an intermediate state results which is characterized by a partial penetration of magnetic flux into the superconductor. It has been observed that some long normal domains are randomly distributed over the superconducting matrix.⁴ As the external

field is increased, the width of the normal domains grows until the critical field is reached. However, if the thickness of the film is less than the coherence length of the superconductor, the orderly mixed-state results, which is similar to that in type II superconductors. The purpose of this investigation is to study the scattering of electrons and phonons at the normal-superconducting phase boundaries in lead films.

A 3μ (greater than the coherence length) pure lead film specimen was fabricated by Dr. R.P. Huebener at the Argonne National Laboratory, (ANL). The ratio of the specimen's resistivity between room temperature and liquid helium temperature is 550. The thermal conductivity of this lead film has been measured as a function of external magnetic fields between 1.5 K and 7.3 K. A typical thermal conductivity vs. magnetic field curve is shown in Fig. 1. From this curve, the critical field H_{c2} and the conductivity slope near the critical field $(dK/dH)_{H_{c2}}$ are obtained. A plot of H_{c2} vs. T is shown in Fig. 2. The $(dK/dH)_{H_{c2}}$ vs. T curve is shown in Fig. 3.

According to the Caroli-Cyrot theory⁵, the conductivity slope near H_{c2} may be written as

$$\left(\frac{dK}{dH}\right)_{H_{c2}} = - \frac{2\pi ck_B}{e} \frac{\left(\frac{\rho_c}{2}\right) [\psi^{(1)}\left(\frac{1}{2} + \frac{\rho_c}{2}\right) + \frac{\rho_c}{2} \psi^{(2)}\left(\frac{1}{2} + \frac{\rho_c}{2}\right)]}{\psi^{(1)}\left(\frac{1}{2} + \frac{\rho_c}{2}\right)} \left(\frac{dM}{dH}\right)_{H_{c2}} \quad (1)$$

$$\text{where } \rho_c = 3.63 \times 10^{-14} v_F l H_{c2}/T, \quad (2)$$

$$\left(\frac{dM}{dH}\right)_{H_{c2}} = \frac{1}{1.16(2\kappa_2^2 - 1)} \quad (3)$$

c is the speed of light, k_B is the Boltzmann constant, e is the electronic charge, v_F is the Fermi velocity of the electrons, l is the electron mean free path in Å, ψ is the digamma function and κ_2 is the

Ginzberg-Landau parameter. The transition temperature T_c is determined from the critical field curve. This curve is characterized by the equation⁶

$$\ln (T_c/T) = \psi \left(\frac{1}{2} + \frac{\rho_c}{2} \right) - \psi \left(\frac{1}{2} \right) \quad (4)$$

Since H_{c2} is proportional to $\rho_c T$, the correct choice of T_c will make $\rho_c T/H_{c2}$ independent of temperature. The results obtained from $\rho_c T/H_{c2}$ vs. T curves for three values of T_c are shown in Fig. 4. It is found that 7.4 K is the best value for T_c and $\rho_c T/H_{c2}$ is equal to $2.01 \times 10^{-3} \frac{\text{mK}}{\text{G}}$. Hence, ρ_c is known for all temperatures. With v_F equal to $2.28 \times 10^8 \text{ cm/sec}$ the electron mean free path ℓ is found to be about 240 Å, which is reasonable for the test specimen. With the known ρ_c , $(dK/dH)/(dM/dH)$ near the critical field is calculated according to Eq. 1, with the results plotted in Fig. 5. The experimental curve in Fig. 3 differs from that in Fig. 5 by a factor of $(dM/dH)_{H_{c2}}$. From the comparison of these two curves, it is shown clearly that κ_2 must decrease with increasing temperature. Currently, an attempt is being made to obtain κ_2 directly from magnetization measurements. Subsequent experiments will include the investigation of the field dependent thermal conductivity for a lead film with thickness less than the coherence length ($\sim 0.9 \mu$).

C. Thermal and Electrical Conductivity of Neutron Damaged Graphite

The thermal conductivity of highly oriented pyrolytic graphite has been measured from 0.45 to 20 K for a pure (PG-10) and three fast neutron irradiated specimens (PG-8, PG-5, PG-9). The irradiation was performed in the CP-5 reactor at ANL. The neutron dose for $E > 1 \text{ MeV}$ was 3.8×10^{16} , 6.6×10^{17} and $4.8 \times 10^{18} \text{ n/cm}^2$ for the specimens PG-8, PG-5 and PG-9, respectively. Since the fast neutron flux at the CP-5

has not been calibrated since 1965, the assumed neutron dose may be in error by up to a factor of 2.⁷ Furthermore, the temperature during irradiation is uncontrolled, so that some of the neutron produced defects may be annealed shortly after their production.

After subtraction of the electronic contributions, the lattice thermal conductivities of the four specimens are shown in Fig. 6. The data indicate that the thermal conductivity is strongly affected by the fast neutron produced defects in the irradiated specimens. At 4 K, the thermal conductivity of PG-5 is depressed by a factor of 40 from the thermal conductivity of the unirradiated sample (PG-10). Within the uncertainty of the neutron flux in the CP-5 reactor, the change of the thermal resistivity ($1/K - 1/K_{\text{pure}}$) is roughly proportional to the neutron dose.

At the lowest temperatures measured, the thermal conductivity of all four specimens have or are approaching a T^3 dependence. As temperature increases, each curve exhibits a temperature dependent upon its corresponding neutron dose. The results are summarized in Table I. Using Komatsu's semicontinuum lattice model⁸, one can explain the temperature dependence of the thermal conductivity in the unirradiated specimen. In this case phonons are predominantly scattered by the crystallite boundaries.

There are five possible types of defects in the fast neutron irradiated specimens: vacancies, vacancy loops, vacancy lines, interstitials and interstitial clusters. The phonon scattering by these defects is characterized by related relaxation rates. The forms of these relaxation rates have been described by several authors.⁹⁻¹¹ In the current study, the Debye theory with a combined relaxation rate for the defects is employed to analyze the thermal conductivity of the neutron irradiated specimens. As a result of this analysis, the

particular defect dominating the phonon scattering may be deduced by the frequency dependence of the relaxation rate: Vacancies and interstitials may give rise to a T^0 to T^{-1} dependence in the thermal conductivity; vacancy loops may yield a temperature independent thermal conductivity; vacancy lines can produce a T to T^2 dependence; and interstitial clusters may lead to a T^{-1} to T^{-2} dependence in the thermal conductivity. From the observed thermal conductivity measurements in the irradiated specimens, vacancy lines seem to dominate the phonon scattering process.

A report will appear shortly¹² on an earlier phase of this study on pyrolytic graphite.

D. Thermal and Electrical Conductivity of Kondo Alloys

In an attempt to improve the precision of measurements described in Progress Report No. 7, additional samples of CuFe (112 ppm, 380 ppm) and CuCr (32 ppm) have been obtained from Dr. W.A. Steyert of the Los Alamos Scientific Laboratory, (LASL). These are from the same cylinders of these alloys that were used to furnish the earlier samples, and upon which specific heat¹³⁻¹⁴, electrical resistivity¹⁵ and susceptibility¹⁴ measurements had already been made. The new specimens have an effective length three times that of the original ones. Currently they are being prepared for measurement in a helium dilution refrigerator and in magnetic fields up to 50 kG.

This study had been temporarily suspended upon the sudden departure of Ms. S.Y. Lu, who was undertaking it as partial fulfillment of the requirements for a Ph.D. degree. The study has recently been reactivated, with high priority, by Dr. J.W.M. Bakker.

E. Specific Heat of TMMC

The study reported in Progress Report No. 7 on the specific heat of the linear chain antiferromagnet $(\text{CH}_3)_4\text{NMnCl}_3$ - commonly referred to as TMMC - has been extended and for the most part been concluded. Two publications on this work, currently in rough draft form, will be submitted to either the Physical Review or Solid State Communications within the next month. The research reported is a collaborative effort between the IIT group and Dr. R.E. Dietz¹⁶ of Bell Telephone Laboratories.

Additional specific heat measurements have been carried out in the temperature range between 0.4 K and 6 K in zero magnetic field and (for the first time) in an applied field of 15 kG parallel to the c-axis, along which the linear chains are aligned. The zero field data show an anomaly at 0.83 K indicative of a phase transition, presumably to a form of three-dimensional (interchain) magnetic ordering. Application of the 15 kG magnetic field broadens the specific heat anomaly and shifts the maximum value to 1.0 K. One can understand this shift on purely thermodynamic grounds. Although the anomaly is broadened by the magnetic field, the total entropy associated with it is the same for both field values (to within experimental error).

Above 1.7 K, the data in zero field are found to fit an expression of the form

$$C(T) = aT + bT^2 \quad ,$$

and below 1.7 K

$$C(T) = aT + bT^2 + \Delta C(T) \quad ,$$

where $\Delta C(T)$ is the anomalous specific heat due to the phase transition, and has a different functional dependence above and below $T_c = 0.83$ K. In the entire temperature range of measurement, a and b are found to be identical: $a = 0.0858$ J/mole K^2 and $b = 0.0480$ J/mole K^3 . $\Delta C(T)$ is best approximated by a cusp-like singularity, with best fits of (for ΔC in J/mole K)

$$\Delta C(T) = 0.2995 (1 - \epsilon^{0.107}) \text{ for } T > T_c ,$$

$$\Delta C(T) = -0.0451 - 0.679 \ln \epsilon \text{ for } T < T_c ,$$

where $\epsilon \equiv |T - T_c| / T_c$.

F. Thermal Conductivity of PbTl Alloys

The thermal conductivity as a function of temperature has been measured from 1.2 K to 12 K for lead alloys with 1% and 20% Tl content, both in the normal and superconducting states. The normal state of the samples was obtained by the application of a magnetic field of about 10 kG. Experimental results are shown in Figs. 7 and 8.

Preliminary analysis shows that in the 20% sample, phonon conduction dominates in the superconducting state. Similar behavior had been reported by Gupta for PbIn alloys.¹⁷ Normal state data show that, in this 20% sample, the electron conduction dominates, with a relatively small phonon conduction component limited by electron scattering.

In the 1% sample, the situation is somewhat different. Normal state data show a predominance of electron conduction. The strong coupling character is reflected only by a weak phonon influence in scattering thermally conducting electrons.

In general, the thermal conductivity of a (normal) metal may be expressed as

$$K_n = K_{en} + K_{gn} ,$$

where K_{en} is the electron conduction component, and K_{gn} is the phonon conduction contribution. For a pure metal, it is generally true that $K_{en} \gg K_{gn}$. In this case, the electron-phonon interaction is the only important mechanism limiting the electron mean free path. For an impure sample, impurity scattering becomes more of a factor. Makinson¹⁸ showed that for the free electron model of a metal, the electronic thermal resistance for $T \ll \theta_0$

$$\frac{1}{K_{en}} = AT + BT^2$$

where $A = \frac{\rho_0}{L_0}$, $B = \frac{95.3 Na^{2/3}}{K_\infty \theta^2}$, Na is the

effective number of conduction electrons per atom, K_∞ is the limiting value of the electronic thermal conductivity at high temperatures, ρ_0 is the residual electrical resistivity and L_0 is the Lorentz number.

For the 1% sample, as shown in Fig. 9, $\rho_0 = 0.56 \mu\Omega/\text{cm K}$ in good agreement with the directly measured value of $0.57 \mu\Omega/\text{cm K}$. Increasing the impurity concentration reduces the electronic conduction component much more effectively and the phonon component of thermal conduction is no longer negligible and one must write

$$K_n = K_{en} + K_{gn} = aT + bT^2 ;$$

where $a = \frac{L_0}{\rho_0}$. The linear electronic conduction term represents an electronic mean free path limited by impurity scattering. The quadratic

phonon conduction is a result of phonon scattering by electrons. The 20% PbTl sample is found to be well represented by this relationship. Using it - see Fig. 10 - one finds $\rho_0 = 17.1 \mu\Omega/\text{cm K}$, which agrees well with $16.5 \mu\Omega/\text{cm K}$ obtained directly.

In analyzing the thermal data in the superconducting state, a computer program is being prepared to test the predictions of Kadanoff and Martin¹⁹ for the strong coupling PbTl alloys. The KM theory employs a Green's function technique and takes impurity and phonon scattering mechanisms into account. It has been found adequate to describe the thermal behavior in the superconducting state of several weak-coupling superconductors²⁰⁻²² and it is the purpose of the current effort to test it on the strong-coupling PbTl alloys.

G. Proximity Effect in Pb-In and Pb-Sn Thin Films Via Thermal Conductivity

This study was initiated by the Principal Investigator during a sabbatical year at the University of Nijmegen, The Netherlands. The motivation for this work has been discussed in the February 1973 Renewal Proposal. The thermal conductivity measurements have been performed on films with component thicknesses of from $10^{30}\text{\AA}$ to $4 \times 10^{30}\text{\AA}$ on glass substrates of 0.08 mm thickness. The change in conductivity is monitored (at a fixed base temperature) as a function of magnetic field applied parallel to the plane of the films.

Initial success in observing quantitative conductivity changes due to proximity occurred prior to the Principal Investigator's return to IIT. Currently, he is advising a graduate student (via written communication) at Nijmegen on the interpretation of the data and on various follow-up measurements. The effect of the proximity of superconducting

electron pairs in Pb on the thermal conductivity of Sn at a temperature above T_c for Sn is particularly striking, and it is expected that publication of this evidence will be made after the current series of measurements are completed and analyzed.

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TABLE I

T = 4 K				
	Fast neutron dose E > 1 MeV	Thermal Conductivity	Change of Thermal Resistivity	Temperature Dependence
PG-10	0	5.2×10^{-2} W/cm K	0	$T^3 \rightarrow T^{2.5}$
PG-8	3.8×10^{16} n/cm ²	3.3×10^{-2}	16 (cmK/W)	$T^3 \rightarrow T^2$
PG-5	6.6×10^{17}	1.3×10^{-3}	7.2×10^2	$T^3 \rightarrow T$
PG-9	4.8×10^{18}	5.7×10^{-4}	1.7×10^3	$T^3 \rightarrow T$

Fig. 1. Thermal Conductivity of 3μ Pb film as a function of magnetic field at 2.61 K.

Fig. 2. Upper critical field H_{c2} as a function of temperature.

Fig. 3. Measured slope $(dK/dH)_{H_{C2}}$ as a function of temperature.

Fig. 4. $\rho_c T / H_{c2}$ as a function of temperature for different values of T_c .

Fig. 5. Calculated $(dK/dH)/(dM/dH)_{H_{C2}}$ as a function of temperature.

Fig. 6. Thermal conductivity as a function of temperature for a pure and three fast neutron irradiated pyrolytic graphite samples.

Fig. 7. Thermal conductivity of $\text{Pb}_{80}\text{Tl}_{20}$ alloy as a function of temperature in normal and superconducting states.

Fig. 8. Thermal conductivity of $Pb_{99}Tl_1$ sample as a function of temperature in normal and superconducting states

Fig. 9. T/K_n as a function of T^3 for Pb₉₉Tl₁ sample.

Fig. 10. K_n/T as a function of temperature for Pb₈₀Tl₂₀ sample.

II. SUMMARY OF PUBLICATIONS: January 1973 to the present

"A Survey of Thermometric Characteristics of Recently Produced Allen-Bradley/Ohmite Resistors";

H. Weinstock and J. Parpia

Temperature, Vol. 4 (ISA, 1973) p. 785; (C00-1629-22)

"Thermal Conductivity of Neutron Irradiated MgO";

D.S. Kupperman, G. Kurz, and H. Weinstock

J. Low Temp. Phys. 10, 193 (1973); (C00-1629-32)

"Electrical Resistivity of Carbon Films";

D.S. Kupperman, C.K. Chau, and H. Weinstock

Carbon 11, 171 (1973); (C00-1629-33)

"Thermal Conductivity of Neutron and Electron Irradiated MgO";

D.S. Kupperman, G. Kurz, and H. Weinstock

Bull. Am. Phys. Soc. 18, 311 (1973); (C00-1629-35)

"Heat Capacity of Antiferromagnetic TMMC";

B. Vis, C.K. Chau and H. Weinstock

Bull. Am. Phys. Soc. 18, 450 (1973); (C00-1629-36)

"Thermal Conductivity of Additively Colored MgO";

D.S. Kupperman, H. Weinstock, and Y. Chen

J. Low Temp. Phys. 14, 277 (1974); (C00-1629-38)

"Low Temperature Magneto-Thermal Conductivity of Pyrolytic Graphite";

C.K. Chau and S.Y. Lu

J. Low Temp. Phys. (accepted for publication); (C00-1629-39)

"Low Temperature Thermal Conductivity of Fast Neutron Irradiated Pyrolytic Graphite";

C.K. Chau

Bull. Am. Phys. Soc. (March 1974); (C00-1629-40)

"Specific Heat of TMMC Above 2 K";

R.E. Dietz, B. Vis, C.K. Chau, and H. Weinstock

(to be submitted within one month to Phys. Rev. or Sol. St. Com.)

"Specific Heat of TMMC Below 2 K";

B. Vis, C.K. Chau H. Weinstock, and R.E. Dietz

(to be submitted within one month to Phys. Rev. or Sol. St. Com.)

III. GRADUATE STUDENT PROGRESS

Benjamin Vis has completed the rough draft of his Ph.D. thesis and will undergo his final oral dissertation examination during March 1974. His research on the specific heat of TMMC forms a major contribution to two papers, now also in rough draft form, which will be submitted for publication within one month.

Jen-Lie Ho has progressed well in his doctoral research on the thermal conductivity of Pb-Tl superconducting alloys - a summary of which appears elsewhere in this Report. It is expected that all requirements for the Ph.D. degree will have been completed by late 1974 or early 1974.

Shui-Ying Lu, while well along on her doctoral research on the thermal and electrical conductivities of Cu:Fe and Cu:Cr Kondo alloys, resigned for personal reasons in September 1974 and accepted a position at the Machlett Laboratories, Division of Raytheon, Stanford, Connecticut.