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THERMAL AND ELECTRICAL MEASUREMENTS ON SOLIDS
AT LOW TEMPERATURES
by
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MASTER

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This report summarizes the work done on this project from February, 1974 to the present. The participating staff consisted of H. Weinstock (Professor of Physics), C. Chau (Assistant Professor of Physics), S. Colucci, J. B. Hartman and Y. Pak (Research Associates), the following graduate students: B. Vis and J. Ho; and undergraduate students: D. Dziekan, R. Osa and H. Schwerdlin.

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I. SUMMARY OF RESEARCH PROGRESS

A. Thermal Conductivity of Radiation Damaged and Deformed MgO

Low temperature (0.4 K to 80 K) thermal conductivity studies of fast neutron-irradiated¹ and additively colored² MgO have been made previously. The neutron-irradiated MgO exhibited two resonant dips in conductivity vs. temperature curves at about 1 K and 20 K. With increasing dosage up to 2×10^{19} n.cm⁻², the 20 K dip became more pronounced, while the 1 K dip decreased and eventually disappeared. Correlation with optical measurements³ indicated that the high temperature resonance was associated with F-type centers, and it was postulated that the low temperature resonance was caused by small aggregate centers.

An MgO crystal, additively colored in magnesium vapor, and subsequently found (by optical absorption) to have an F center concentration of 1.8×10^{17} cm⁻³, also exhibited a thermal conductivity resonance dip at about 20 K. After UV irradiation, resulting in partial $F \rightarrow F^+$ conversion, the resonance was found to be much weaker. This increase in conductivity was attributed to a relaxation of neighboring ions due to the different charge state of the defect. Heating the UV bleached crystal to 700 K produced $F^+ \rightarrow F$ reversion and correspondingly a return to the same thermal conductivity vs. temperature behavior observed before bleaching. The absence of a 1 K dip and optical absorption bands associated with cluster defects⁴, confirmed that such a dip in neutron-irradiated MgO was due to cluster centers.

Current thermal conductivity studies of electron-irradiated MgO

for two specimens receiving total doses of about $3 \times 10^{18} \text{ e} \cdot \text{cm}^{-2}$ exhibit the same resonance behavior as observed in the earlier measurements on neutron-irradiated and additively colored specimens. Again, as for the additively colored case, the absence of a resonance at 1 K supports the argument that cluster defects are the cause of such a resonance in neutron-irradiated MgO. UV exposure produces a small increase in the resonance scattering, in contrast to the pronounced decrease in scattering for the additively colored specimen.

Thermal conductivity measurements are in progress⁵ on an MgO specimen which has been deformed approximately 2% along a [100] direction. Over the high and low ends of the temperature range (0.4 K to 70 K), the conductivity approaches that of undeformed specimens, but is somewhat depressed at intermediate temperatures (akin to the behavior observed for the specimens mentioned above). Optical measurements on the sample showed an absorption band at 5.7 eV, due probably to higher order point defects, with concentrations in the range of 10^{17} to $10^{18}/\text{cm}^3$. F^+ concentration of $\sim 2 \times 10^{16} \text{ cm}^{-3}$ can also be expected.⁶ At this time, it is not certain whether it is the small concentration of anion vacancies or the larger concentration of higher order defects that is responsible for the dip in the deformed crystal.

B. Phonon Thermal Conductivity of Superconducting Lead-Thallium Alloys

Mrstik and Ginsberg⁷ have shown that the reduced electronic thermal conductivity of a strong coupling superconductor (such as lead) may be obtained from BRT theory for weak coupling superconductors⁸ by simply scaling the gap parameter by a constant factor. This method should be applicable to lead alloys as well. With scaling, the phonon thermal conductivity in the superconducting state may be separated from the measured thermal conductivity. This report presents the thermal conductivity of lead-thallium alloys with emphasis on phonon scattering by the thallium impurity and by conduction electrons.

The heat conduction of six lead-thallium alloy specimens has been investigated. The thallium concentration ranges from 1 to 20% by weight. In this concentration range, the alloys are in the phase of random solid solution.⁹ All specimens are annealed at 275°C for one week. The thermal conductivity measurements are performed before and after annealing. The lead-thallium alloys are superconducting at temperatures below 7.2 K, and below T_c are driven normal by means of a 7 kOe magnetic field. Since the product of the cyclotron frequency and the electron mean free time is much less than one, the effect of the magnetic field on thermal transport should be negligible.

The thermal conductivity for the 3.5% Tl specimen is shown in Fig. 1. The magnitude of the superconducting state data increases significantly after annealing. This is probably due to the enlargement of the alloy grain size which restricts the phonon mean free path at low temperatures. The normal state thermal conductivity is not

Figure 1. Thermal Conductivity of 3.5% Tl lead alloy versus temperature

Figure 2. Phonon thermal conductivity of 3.5% Tl lead alloy versus temperature

affected by the annealing process. Hence, the electron and phonon mean free paths are not limited by the grain size of the specimen in the normal state.

To separate the electronic and phonon contributions to the heat conduction, the electronic thermal resistivity in the normal state is written as

$$1/K_{en} = \rho_0/L_0 T + B_{ep} T^2 \quad (1)$$

The first term is due to the electron-impurity scattering, where ρ_0 and L_0 are the residual resistivity and the Lorenz number respectively. The second term is due to the electron-phonon interaction. In the 1% Tl specimen, the electrons dominate the heat conduction process, permitting the value of B_{ep} to be obtained from the slope of a $1/K_n$ vs. T^3 plot. Assuming the same value of B_{ep} for all other specimens, K_{en} for each specimen is calculated according to Eq. (1) after measuring the residual electrical resistivity of the specimen. Using the Kadanoff-Martin expression¹⁰ for the ratio K_{es}/K_{en} , K_{es} is obtained from the known K_{en} value. Finally, subtracting K_{en} and K_{es} from the measured thermal conductivities, one deduces the phonon thermal conductivities K_{gn} and K_{gs} , which are shown in Fig. 2 for the 3.5% Tl specimen.

To analyze the phonon thermal conductivity in the normal and superconducting states, the Debye thermal conductivity integral with a combined relaxation rate is employed.¹¹ The phonon scattering mechanisms include grain boundaries, impurity point defects, the three-phonon normal process and phonon-electron interaction. First, one determines the boundary scattering term from the T^3 region of K_{gs} . Then one fits K_{gs} as a function of temperature (up to 2 K) by adjusting

the impurity scattering and the three-phonon normal process terms. The same three-phonon term is used for all specimens. The phonon-electron interaction is neglected in this case because there are very few normal electrons present in this temperature region. To handle the K_{gn} curve, one must take account of the phonon-electron interaction, and do so by adjusting the phonon-electron relaxation rate to fit the K_{gn} curve. Finally, one uses the BRT expression for the phonon-electron interaction in the superconducting state and the Debye thermal conductivity integral to calculate K_{gs} up to the transition temperature. The calculated thermal conductivity of the normal and superconducting states for the 3.5% Tl specimen is shown by the solid lines in Fig. 2.

From the analyses, it is found: (1) There is no definite correlation between crystallite grain size and thallium concentration. [This is different from the conclusion drawn by Gupta and Wolf for Pb-In alloys.¹² They found that the grain size decreases as the indium concentration increases.] (2) The impurity point defect relaxation rate is roughly proportional to the thallium concentration. However, due to the small mass and size differences between lead and thallium, one attributes this effect to the impurity strain-field scattering of phonons. (3) The phonon-electron relaxation rate increases with increasing thallium concentration. Its magnitude is similar to that obtained for Pb-In and Pb-Bi alloys.¹³

Specimens for this study were kindly supplied by Dr. W. C. Overton, Jr., of the Los Alamos Scientific Laboratory.

C. Effects of Low Temperature Neutron Irradiation on Nb₃Sn Composite Wires in Fields up to 10 Tesla

Neutron irradiation effects on practical superconductors are relevant to CTR designs where a superconducting magnet will be exposed to fusion products during operation at ~ 4 K. To approach CTR conditions, samples of Nb₃Sn wire were irradiated in the cryogenic facility¹⁴ of the CP-5 reactor at Argonne National Laboratory. The superconducting wire, provided by M. Suenaga¹⁵, is a Cu-Sn matrix containing 19 Nb filaments with a Nb₃Sn layer. Several wires were maintained at ~ 6 K and received a relatively low fluence (4.8×10^{17} n/cm²) of fast neutrons ($E > 0.1$ MeV). In situ $J_c(H)$ measurements were made¹⁶ up to ~ 3 T, stronger fields being prohibited by space limitations. To extend the study to higher fields, a method was devised to mount the samples in a four-terminal probe in IIT's 12 T magnet, without ever exceeding 77 K. Preliminary studies appear to indicate that $J_c(H)$ of radiation damaged samples at 4.2 K is unaffected by annealing to this temperature.

$J_c(H)$ was determined by passing a current through a sample wire at a given H and displaying the amplified $V(I)$ characteristics on an x-y recorder. $I_c(H)$ was then determined by extrapolating that portion of $V(I)$ with slope 125 $\mu V/A$ back to the $V = 0$ axis. Transitions from small to large slope are rather sharp and occur (by design) well below the linear flux flow region. In Fig. 3, $J_c(H)$ is shown for three cases: (A) unirradiated, (B) irradiated, and (C) irradiated and annealed to 300 K.

At 3.2 T, $J_c(H)$ for the irradiated sample (B) is increased 18% above the already high unirradiated value of 1.32×10^6 A/cm² and

Fig. 3. $J_c(H)$ at 4.2 K for unirradiated Nb_3Sn composite sample (A), sample irradiated to $4.8 \times 10^{17} \text{ n/cm}^2$ (B), and sample irradiated and subsequently annealed to 300 K (C). Data measured with increasing field are indicated by flags.

agrees with comparable in situ measurements.¹⁶ With increasing field, $J_c(H)_B/J_c(H)_A$ becomes larger until, at 10 T, J_c is enhanced 50%. (This contrasts with studies on Nb_3Sn irradiated at "reactor-ambient" temperature ($\sim 60^\circ C$).¹⁷ There, J_c appeared relatively constant for large values of H , finally showing a sharp decrease at higher neutron fluences.) Annealing to 300 K results in $J_c(H)_C$ lying between $J_c(H)_A$ and $J_c(H)_B$. At 10 T, $J_c(H)$ for the annealed sample is still 20% above the unirradiated value.

The pinning force density, $F_p(H) = J_c(H) \cdot H$, for the unirradiated sample exhibited a maximum value of 4×10^9 dyn/cm³ at about 2.75 T. The irradiated sample showed an increase of 22% in $F_p(H)_{\max}$ with the peak shifted to 4.25 T. As might be anticipated, the annealed sample had $F_p(H)_{\max}$ about 10% greater than that of the unirradiated value and was located at 3.75 T.

If no new species of pinning center is introduced by the irradiation, the reduced quantities, $f_p(h) = F_p(h)/F_{p\max}$ and $h = H/H_{c2}$, are expected to obey a scaling law.¹⁸ From the reported data, this requires H_{c2} to increase by 50% with irradiation. Although precise values of H_{c2} are not available, this large increase is not supported by experiment. It is possible that low temperature, fast n damage in Nb_3Sn produces a new species of pinning center, e.g., defect loop, which partially persists even after room temperature annealing. In fact, if one were to naively subtract $F_p(H)_A$ from $F_p(H)_B$ and $F_p(H)_C$, the resulting two curves both have small peaks at about 5.5 T. (It is understood, of course, that pinning strengths need not add algebraically.) Liberally interpreting the model of Kramer¹⁸, one might conjecture that the new pinning centers are weak and relatively

widely spaced.

For the small fluence studied, the additional pinning centers serve to enhance $J_c(H)$. A planned extension of the present work is to expose the samples to greater fluences in an attempt to determine the onset of $J_c(H)$ degradation. Future plans also include annealing samples to $T > 300$ K.

This study has been carried out in collaboration with Dr. B. S. Brown and Dr. T. H. Blewitt of the Argonne National Laboratory.

D. Thermal Conductivity of Tantalum Wire in the Superconducting State

The thermal conductivity of 10 mil diameter tantalum wire¹ as a function of temperature has been measured from 1.6 K to 6 K - the superconducting transition temperature for these tantalum specimens is 4.48 K. In order to reduce the time to reach thermal equilibrium, four Ta wires in parallel are used in the measurements.

The experimental results are shown in Figs. 4 and 5. In Fig. 4, K vs. T is plotted on a log-log plot. Up to 4 K the data may be approximated by a straight line implying a power law dependence of thermal conductivity on temperature. Figure 5, which is a plot of K vs. T^2 , indicates that this dependence is $K \propto T^2$. Since the specimens are in the form of 10 mil diameter wire, it is likely that a high density of dislocations are present. If dislocations represent the dominant scattering mechanism for phonons, then at temperatures well below T_c , a $K \propto T^2$ dependence is to be expected.

Employing the Wiedemann-Franz ratio and the measured normal state residual electrical resistivity of 1.9×10^{-6} ohm-cm for these wires, it is found that in the temperature range of $T \sim 2$ K, phonon thermal conductivity should dominate. At temperatures close to $T_c \sim 4.5$ K, electrons should be the dominant thermal current carriers. This interpretation is consistent with the fact that the $K \propto T^2$ dependence is no longer evident at such temperatures, with the deviation from this dependence increasing as temperature increases.

While of practical importance and of interest in themselves, these measurements have been carried out to provide the necessary parameters for evaluation of both theory and experiment on the dynamics

Fig. 4 Thermal conductivity vs. temperature for 10 mil tantalum wires (manufactured by Kawecki Chemical Co.)

of superconducting-normal interfaces in current-carrying wires. The measurements have been applied to the theoretical solutions¹⁹ of the heat flow equations for Ta wires and to the actual interface velocity data²⁰ so that the Kapitza boundary resistance between the Ta wires and the surrounding medium (either liquid helium or Stycast 2850FT) may be evaluated.

Currently, plans are being made to extend these thermal conductivity measurements over a wider temperature range and to apply a magnetic field (~ 10 kOe) so that normal state data may be obtained.

E. Thermal Conductivity of Vanadium in the Superconducting and Normal States

A study has been undertaken on vanadium, one of the few elemental type II superconductors, with the aim of investigating the mixed state thermal conductivity as a function of the electron mean free path. The first part of the study involves a high purity vanadium sample which has a long electron mean free path. Subsequently, the electron mean free path will be shortened in stages by means of neutron irradiation. This avoids the need to add impurities in order to produce the same changes in mean free path. Reported below are thermal conductivity measurements on a high purity vanadium sample (V-1) as a function of temperature in the normal and superconducting states.

The V-1 sample has been generously provided by Dr. S. T. Sekula of the Oak Ridge National Laboratory. This particular sample resulted from an unsuccessful attempt to produce a single crystal rod with the rod axis oriented along the [110] direction. The sample has been annealed to 1650°C for one hour under a pressure of 10^{-9} torr and the resistivity ratio (between room and liquid helium temperatures) was found to be 630. The normal and superconducting state thermal conductivity data are shown in Fig. 6.

The normal state thermal conductivity shows a peak around 10 K with a magnitude of about 5.5 W/cmK. Below the peak, the conductivity has a linear temperature dependence which indicates electronic conduction. In the superconducting state, the thermal conductivity has a T^3 dependence near the transition temperature. As temperature decreases, the conductivity decreases more rapidly than T^3 . For

Fig. 6 Thermal conductivity of vanadium sample V-1 as a function of temperature in the normal and superconducting states

temperatures below about 1 K there is a decrease in slope such that the thermal conductivity has a less than T^3 temperature dependence.

In metals, heat is conducted by electrons and phonons. The electronic thermal resistivity can be written as

$$1/K_{en} = A/T + BT^2 ,$$

where A/T is the electron-impurity scattering term and BT^2 is that for electron-phonon scattering. Since normal state heat conduction in the sample is dominated by electrons, T/K is plotted versus T^3 for the normal state data. From this plot, one obtains the coefficients A and B .

The ratio of the thermal conductivity in the superconducting state to that in the normal state as a function of reduced temperature is shown in Fig. 7. To analyze this result, one uses the Kadanoff-Martin expression²¹

$$\frac{K_{es}}{K_{en}} = \frac{3}{2\pi^2} \int_0^\infty d(\beta\epsilon) (\beta\epsilon)^2 \text{sech}^2\left(\frac{\beta E}{2}\right) \left[1 + \alpha \left(\frac{T}{T_C}\right)^3\right] \left[\frac{\beta\epsilon}{\beta E} + \alpha \left(\frac{T}{T_C}\right)^3\right]^{-1} ,$$

where

$$\beta = \frac{1}{k_B T} ,$$

$$E = \sqrt{\epsilon^2 + \Delta^2(T)}$$

$$\alpha = BT_C^3/A$$

The only adjustable parameter in this expression is $\Delta(0)/k_B T_C$.

Vanadium has 4s and 3d electrons. The high density d electrons are

Fig. 7 The reduced thermal conductivity vs. reduced temperature for vanadium sample V-1

mainly responsible for its superconducting properties. For a high purity specimen, the energy gap due to the d electrons becomes anisotropic. Radebaugh and Keesom²² have measured the specific heat of high purity vanadium and they found that the energy gap is anisotropic with a maximum value of $3.52 kT_c$ (BCS) and a minimum value of $3.2 kT_c$. They used 75% of the maximum value and 25% of the minimum value to fit their specific heat data. In fitting the current data, the average energy gap is found to be $3.34 kT_c$. The deviation of the experimental data from the calculated curve for $(T/T_c) < 0.3$ indicates that phonon heat conduction is important in the superconducting state.

Low temperature phonon thermal conductivity in solids is often limited by specimen boundaries. Using Radebaugh and Keesom's phonon specific heat data and the average phonon velocity calculated from the elastic measurements of Alers²³, one obtains the Casimir boundary limit values for thermal conductivity shown by the T^3 straight line in Fig. 6. The conductivity data approach this T^3 line with a temperature dependence less than T^3 , and at $T \sim 0.5$ K, they appear to cross this limit. There seem to be two plausible explanations for this behavior. The first is that the T^3 phonon thermal conductivity is due to diffusive boundary scattering. For specular boundary scattering, the temperature dependence will be less than T^3 . The other explanation is that there is a second energy gap due to the s electrons in vanadium. In their specific heat measurements Radebaugh and Keesom found a linear temperature dependence term in their data down to 0.6 K. They attributed this term to normal s electrons, which contribute a linear temperature dependence to the thermal conductivity.

If one assumes that both s and d electrons have the same electron velocity and electron mean free path, then the estimated electronic thermal conductivity for the normal s electrons is about $4 \times 10^{-4} \text{ T(W/cm}\cdot\text{K}^2)$. This result is on the correct order of magnitude to account for the observed low temperature thermal conductivity in the superconducting state.

F. Miscellaneous

Progress has been made in the growth of two species of insulating crystals with antiferromagnetic transitions at liquid helium temperatures. Crystal specimens suitable for thermal conductivity measurement are now in hand. Although measurements on these specimens does not have high priority, these will be carried out at some future date.

Thermal and electrical conductivity measurements on Cu:Fe and Cu:Cr Kondo alloys at helium dilution refrigeration temperatures (between ~ 0.02 K and 1.0 K) and in high magnetic fields will be carried out shortly. Again, while this effort does not have the highest priority, an effort has been made to set these up during the past year, and it now appears that the performance of these measurements is imminent.

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II. SUMMARY OF PUBLICATIONS: February 1974 to the present

1. "Low Temperature Magneto-Thermal Conductivity of Pyrolytic Graphite";
C. K. Chau and S. Y. Lu
J. Low Temp. Phys. 15, 447 (1974); (COO-1629-43)
2. "Low Temperature Thermal Conductivity of Fast Neutron Irradiated Pyrolytic Graphite";
C. K. Chau
Bull. Am. Phys. Soc. 19, 255 (1974); (COO-1629-40)
3. "The Specific Heat of the Linear Chain Antiferromagnet TMMC Below 4 K";
B. Vis
Ph.D. Thesis, IIT (May 1974); (COO-1629-42)
4. "Specific Heat of the One-Dimensional Antiferromagnet TMMC Above 1 K";
R. E. Dietz, L. R. Walker, F. S. L. Hsu, W. H. Haemmerle, B. Vis, C. K. Chau and H. Weinstock
Solid State Comm. 15, 1185 (1974); (COO-1629-45)
5. "Specific Heat of TMMC in the Region of Low Temperature Magnetic Phase Transition";
B. Vis, C. K. Chau, H. Weinstock and R. E. Dietz
Solid State Comm. 15, 1765 (1974); (COO-1629-46)
6. "Heat Conduction in Superconducting Lead-Thallium Alloys";
J. L. Ho and C. K. Chau
Bull. Am. Phys. Soc. 20, 58 (1975); (COO-1629-44)
7. "Thermal Conductivity of Deformed MgO";
J. B. Hartmann and H. Weinstock
Bull. Am. Phys. Soc. 20, 469 (1975); (COO-1629-47)
8. "Low Temperature Thermal Conductivity of Vanadium Crystals";
C. K. Chau and Y. L. Pak
Bull. Am. Phys. Soc. 20, 615 (1975); (COO-1629-48)
9. "Thermal Conductivity of Radiation Damaged and Deformed MgO";
J. B. Hartmann, H. Weinstock and Y. Chen
MgO Workshop, Canton NC, May 26-28, 1975; (COO-1629-49)
10. "Effects of Low Temperature Neutron Irradiation on Nb₃Sn Composite Wires in Fields Up To 10 Tesla";
S. L. Colucci, H. Weinstock, B. S. Brown and T. H. Blewitt
Submitted to LT-14; (COO-1629-50)

11. "Phonon Thermal Conductivity of Superconducting Lead-Thallium Alloys";
J. L. Ho, C. K. Chau, H. Weinstock and W. C. Overton, Jr.
Submitted to LT-14; (COO-1629-51)
12. "Thermal Conductivity of Radiation Damaged, Additively Colored and Deformed MgO";
J. B. Hartmann, H. Weinstock and Y. Chen
Submitted to Phonon Scattering in Solids Conference (Nottingham);
(COO-1629-52)

III. GRADUATE STUDENT PROGRESS

Benjamin Vis was awarded the Ph.D. degree in May, 1974 and is currently employed by Euratom in Milan, Italy.

Jen-Lie Ho has completed all requirements for the Ph.D. and will be awarded said degree during the summer of 1975. His thesis research on the thermal conductivity of Pb-Tl superconducting alloys will be reported at the Fourteenth International Conference on Low Temperature Physics and in an article to be submitted to the Journal of Low Temperature Physics. He has just accepted employment with the Burroughs Corporation's Chicago office.

It is anticipated that new graduate students will enter into laboratory research during the 1975-1976 academic year.