Measurement of the pair potential in aligned Pb-Cd lamellar eutectic composites

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Thermal-conductivity measurements have been used to study the pair potential in multilayer superconductor–normal-metal lamellar composites. Lamellar Pb-Cd eutectic samples were prepared by a high-speed directional solidification technique which generates large single-grain samples with all the lamellas lined up in the same direction. For thin Cd layers where the pair potential Δ is expected to be uniform throughout the Cd, the pair potential is found to be ~40% of the value in bulk Pb. This gives direct experimental evidence that the boundary condition requiring continuity of Δ/NV applies in the clean regime. Surprisingly, the electron mean free path in the Cd is longer than the width of the lamellas implying that Andreev scattering is suppressed at the Pb-Cd interface.

I. INTRODUCTION

When a normal metal is placed in electrical contact with a superconductor the normal metal can exhibit superconducting properties.[1,2] This so-called proximity effect has previously been studied primarily with thin-film specimens, but the use of thin films has several disadvantages. The electron mean free paths tend to be short, restricting the samples to the dirty limit; the small mass of the sample makes some measurements (e.g., specific heat) difficult, and there exists the possibility of an oxide layer forming at the interface between the films. Some of these difficulties can be alleviated with the use of multilayer lamellar samples prepared by directional solidification of eutectic composite materials. It is possible for such materials to have long electron mean free paths, the samples can be made sufficiently massive to permit accurate specific-heat measurements, and, because the sample is formed directly from the melt, there is less of a problem with oxide layers. Early studies on eutectics showed qualitative agreement with theory[3] for the transition temperature and the jump in specific heat[4,5] but quantitative agreement can only be attained by using a number of adjustable parameters. The samples used in these experiments consisted of many small grains with the lamellas in each grain unaligned with respect to the lamellas in neighboring grains and so were not well suited for transport measurements. More recently Spencer *et al.*[6] have developed techniques for preparing large-grain samples with all the lamellas aligned in a given direction permitting electrical transport and flux-flow measurements to be studied either parallel or perpendicular to the lamellas. For Pb-Cd eutectic the flux-flow measurments gave pinning forces at the lamellar interface smaller than expected for an abrupt decrease in pair potential. In addition, with electrical current flowing perpendicular to the lamellas, no resistance due to the Cd layers was observed once the Pb was superconducting.

In light of these experiments it was the purpose of the present study to measure the pair potential Δ in the Cd region of the composite and compare its magnitude with theory. The more common techniques used to measure Δ of a superconductor such as electron tunneling are unfeasible for a eutectic due to the lack of a macroscopic homogeneous surface. Somewhat fortuitously, the thermal conductivity parallel to the lamellas is dominated by electronic conduction in the Cd making it possible to determine Δ from the thermal conductivity. The data show that Δ in the Cd is reduced from the value in the Pb in accordance with the de Gennes boundary condition[7] which requires that Δ/NV be continuous across the boundary where N is the density of states at the Fermi surface and V is the electron-electron interaction potential which gives rise to superconductivity. A description of the eutectic samples is given in Sec. II. The experimental techniques are discussed in Sec. III and the results are presented in Sec. IV. Andreev scattering and its effects on the measurements are discussed in Sec. V.

II. EUTECTIC ALLOYS

When metals combined in the eutectic composition are cooled from the liquid, two solid phases are formed simultaneously in one of a variety of microstructures, depending on the metals involved. For Pb-Cd eutectic, as with the much studied Pb-Sn eutectic, the microstructure is lamellar as shown schematically in Fig. 1. The periodicity of the eutectic $(=d_S+d_N)$ is determined by the rate at which the liquid-solid interface is moved through the sample. For samples in the present study this was easily controlled by a motor-

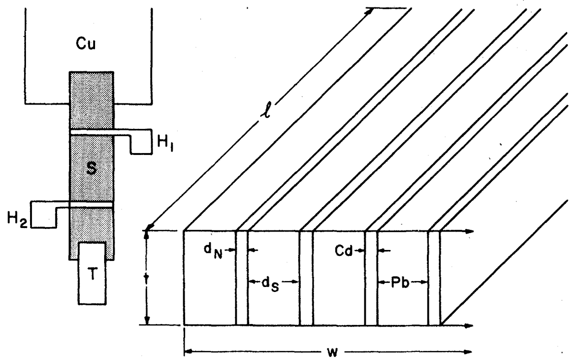

FIG. 1. Schematic drawing of a sample of lamellar eutectic composite. The sample is formed by high-speed directional solidification in a direction parallel to the length l. The widths d_N and d_S define the size of the Cd and Pb lamellas, respectively. Some of the dimensions are exaggerated for clarity. The method for measuring κ is shown on the left. The darkened region S is the sample, H_1 and H_2 are electrical heaters, and T is a germanium thermometer.

driven assembly which resulted in a uniform periodicity throughout a sample. Samples of Pb-Cd could readily be made with a periodicities of 0.5–8.0 μm. The periodicity was measured using optical and scanning electron micrographs of the samples. The ratio of Pb width d_S to Cd width d_N is of course fixed and determined by the eutectic composition which is 82.6-wt.% Pb and 17.3-wt.% Cd.[8] For this composition the ratio of thicknesses is $d_S/d_N \simeq 3.6$.

In early work on eutectics, samples were prepared in cylindrical tubes resulting in lamellas which had only limited extent and contained many grains. The lamellas in a given grain were parallel to each other but not necessarily parallel to the lamellas of neighboring grains. By means of high-speed directional solidification in a constrained geometry, it is possible to produce lamellas which extend over several centimeters with the grains all aligned perpendicular to the sample thickness. The process for making these samples has been described elsewhere.[6]

Because of the nonzero solubility of Pb in the Cd and of Cd in the Pb, the solid phases of the eutectic are not pure Pb and pure Cd but each contains some of the other metal. From the phase diagrams it is expected that the Pb contains 5.9-at.% Cd and the Cd contains 0.14-at.% Pb at the eutectic temperature of 248 °C. The corresponding figures at room temperature are approximately 1.4% Cd in the Pb and less than 0.06% Pb in the Cd.[8] These impurities are frozen in upon cooling to room temperature—at least in the Cd lamellas. In the Pb lamellas the Cd impurities appear to precipitate out of solution over a period of weeks. Evidence for this comes from scanning electron micrographs which show small Cd crystallites forming in the Pb after several days at room temperature. In addition the conductivity (either thermal or electrical) of the Pb phase increases by over a factor of 4 when the sample is allowed to remain at room temperature for 2–4 weeks. Allowing the sample to remain at room temperature for longer periods of several months increases the conductivity by a negligible amount of only a percent or two. No conductivity change is observed in the Cd phase within an accuracy of a few percent as is shown in the Appendix. Samples allowed to stay at room temperature for long periods are referred to as "annealed." Samples to be measured in the unannealed state are placed in liquid nitrogen immediately after solidification. Prior to being installed in the cryostat, the samples are removed from the liquid nitrogen for only ~8–10 h so that heaters and thermometers can be mounted.

The differing impurity contents of the two solid phases lead to different electrical conductivities making the eutectic very anisotropic. This in turn makes it easy to separate the contributions to the conductivities of the two phases. For thermal (or electrical) conductivity of the sample in the normal state in the direction perpendicular to the lamellas, the much cleaner Cd lamellas are essentially thermal (or electrical) shorts and all the resistance comes from the dirtier and thicker Pb lamellas. Similarly, for transport in the direction parallel to the lamellas most of the current flows in the Cd. From such measurements the individual conductivities of the two phases can be deduced. For the unannealed Pb phase a typical electron mean free path l_e is $l_{Pb} \sim 0.04$ μm while for the Cd it is $l_{Cd} \sim 2.5$ μm.[9] It is interesting to note that l_{Cd} is considerably larger than the width d_N of the Cd lamellas implying that electrons are specularly reflected at the Pb-Cd interface. This specular reflection seems reasonable in light of the smooth metal-metal interface expected in a eutectic. Mean free paths do depend somewhat on periodicity for samples with $d_S + d_N < 2$ μm but the effect is not large. For the sample with the smallest periodicity ($d_N = 1170$ Å) l_{Cd} is only slightly more than 1 μm. But this is still much greater than the mean free path expected from diffuse boundary scattering. Indeed, it is not clear whether this reduction in l_e is due to boundary scattering or to a higher Pb impurity content in the Cd because of the higher velocity of the liquid-solid interface during solidification. The Pb, at least, is dirtier in samples of smaller periodicity as deduced from the larger magnetic fields required to drive it normal.

Sample thickness t is typically 30–60 μm (Fig. 1). The sample width w is chosen to be 3–4 mm so that a sample contains many hundreds or thousands of lamellas. Sample lengths are usually 1–2 cm.

III. EXPERIMENTAL TECHNIQUES

Measurements of the thermal conductivity were made using a two-heater arrangement as shown in Fig.

1. This method was used instead of the more common two-thermometer method because the heaters were lighter than the thermometers and less likely to twist or strain the samples and also to reduce the number of thermometers required. Germanium thermometers were used to avoid the drift associated with carbon thermometers.[10] These thermometers were calibrated against a previously calibrated germanium thermometer which was checked against superconducting fixed points. Heaters were made of 0.005-cm-diam Manganin wire and both heaters and thermometers were attached to the sample with GE7031 varnish. The samples were enclosed in a vacuum can with one end of the sample mechanically and thermally anchored to a copper cold sink with GE7031. The cold sink was attached to a pumped ^{4}He bath through a weak thermal link so that the sample temperature could be controlled. Electrical leads were thermally anchored to the copper sample holder as well as at higher temperature points before being brought out of the cryostat. The sample was allowed to hang with the largest dimension in the vertical direction. A superconducting magnet outside the vacuum can provided the small magnetic fields (0–3 kG in the vertical direction) necessary to drive the sample normal. To obtain a datum, electrical power $\dot{Q}$ was applied to the first heater H_1, and the temperature T_1 read from thermometer T. The power was then removed from H_1 and the same power $\dot{Q}$ applied to heater H_2. Again thermometer T was read to obtain T_2 and the thermal conductivity κ calculated using

$$\kappa=(\dot{Q}/\Delta T)(l/A) \quad ,$$

where $\Delta T = T_2 - T_1$, A is the cross-sectional area of the sample ($=wt$), and l is the distance between heaters H_1 and H_2. To avoid trapped flux, the sample was warmed above the transition temperature before each datum taken in the superconducting state. Temperature measurement in the magnetic field was no problem inasmuch as the calibrated thermometer was situated well outside the solenoid and the sample thermometers were calibrated *in situ*. In addition the applied fields were sufficiently small that the change in calibration was never more than 0.1%.

IV. RESULTS AND DISCUSSION

A. Transition temperature

The electrical conductivity of a sample could not be measured simultaneously with the thermal conductivity for lack of leads in the cryostat as well as the conflicting requirements of the two measurements regarding thermal isolation. Nevertheless, a determination of the transition temperature T_c could be made from critical field measurements. A critical field $H_{1/2}$ was chosen as the field at which κ was halfway between its superconducting- and normal-state values. Plotting $H_{1/2}$ against T^2 gives a straight line, at least near T_c, from which T_c can be determined as in Fig. 2. Transition temperatures determined by this method are in good agreement with those determined by electrical conductivity measurements.[6] For all samples, T_c was decreased slightly from the transition temperature of pure Pb. The smaller periodicity samples had the greater reduction in T_c but this was always a small effect, never greater than a few tenths of a degree. A decrease in T_c of the eutectic from that of pure bulk Pb is expected from the proximity effect although the magnitude is difficult to predict, particularly for samples of small periodicity.[4] An additional small decrease is expected due to the Cd impurities.[5,11] It is interesting to note that after annealing T_c *decreases* still further. If the only effect of annealing came from the reduction of Cd impurities in the Pb then T_c would be expect to *increase*.[11] Presumably, then, the decrease is due to the proximity effect through the increase in coherence length with increasing electron mean free path. The simplest theory for the transition temperature of a eutectic, namely, a solution to the linearized Ginzburg-Landau equations as given in Ref. 4, does qualitatively predict the observed behavior (i.e., $dT_c/dl_{\rm Pb} < 0$) although it fails quantitatively. This observation of a change in T_c on annealing may prove useful in comparing theories of the proximity effect, for it allows the electron mfp dependence of a theory to be checked using a single sample.

B. Thermal conductivity

The thermal conductivity provides a measure of Δ because Δ controls the number of electrons which condense into the ground state and are thus unable to transport heat. This is observed as a reduction in the ratio of superconducting- to normal-state conductivity κ^S/κ^N. Before an estimate of $\Delta_{\rm Cd}$ can be deduced

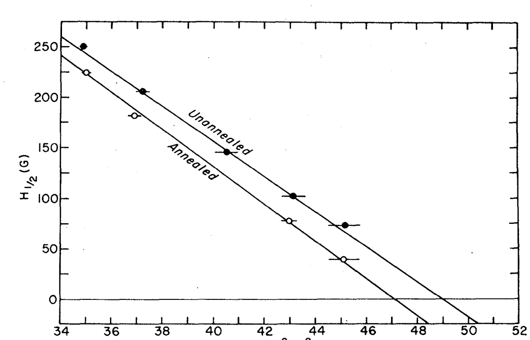

FIG. 2. Plot of $H_{1/2}$ as a function of the square of the temperature for a sample ($d_N = 1170$ Å) in both the annealed (open circles) and unannealed (solid circles) state. The field $H_{1/2}$ is defined so that $\kappa(H_{1/2}) = \frac{1}{2}(\kappa^S + \kappa^N)$. The transition temperature is determined from the intercept at $H_{1/2} = 0$.

from the experimental κ^S/κ^N measured parallel to the lamellas, two corrections must be applied to the raw data. The most important correction is the subtraction of the parallel conductance of the Pb which is known from measurements in the direction perpendicular to the lamellas. In the unannealed samples the Pb phase accounts for 10%–20% of the conductance. In addition, a small correction (always less than ~4% for these unannealed samples) for magnetoresistance is applied. With these corrections the ratio of the thermal conductivities of the Cd in the superconducting and normal states is shown in Fig. 3 as $\kappa_{Cd}^S/\kappa_{Cd}^N$. To demonstrate that these conductivities are due to electrons, upper bounds on the phonon conductivity are estimated. Most of the phonon conductance occurs in the Pb because of its lower Debye temperature and larger cross section. The normal-state electrical and thermal conductivities of the Pb are determined from measurements in the direction perpendicular to the lamellas. The ratio $\kappa/\sigma T$ gives the Lorenz number (2.45×10^{-8} W Ω/K^2) within the accuracy of the experiment, implying predominantly electronic conduction. Another estimate of the phonon conductivity can be obtained from measurements of the zero-field κ in the perpendicular direction at low temperatures when nearly all the electrons in the Pb have condensed into the ground state so that phonons transport most of the heat. The measured conductivity is $\lesssim 7\times10^{-4}$ W/K cm at 1.2 K which corresponds to a

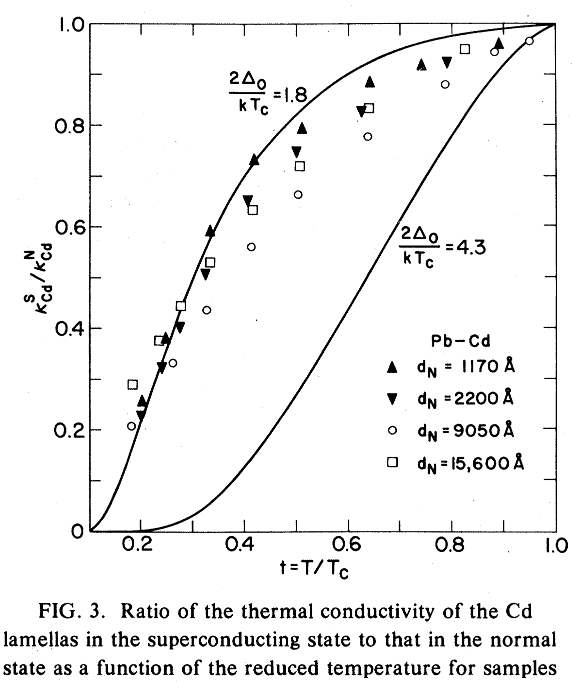

FIG. 3. Ratio of the thermal conductivity of the Cd lamellas in the superconducting state to that in the normal state as a function of the reduced temperature for samples with a wide range of periodicities. The solid symbols are for small periodicities, the open symbols are for larger periodicities. The curves are calculations using the Bardeen-Rickaysen-Tewordt theory for different values of Δ_0.

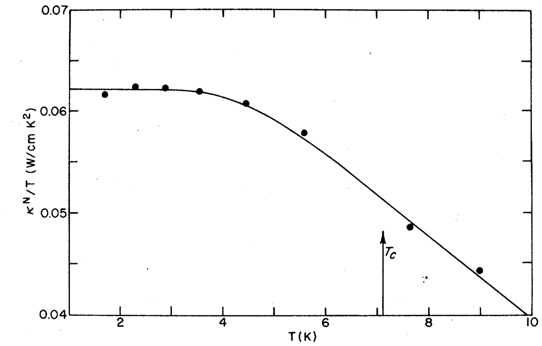

FIG. 4. Normal-state thermal conductivity measured parallel to the lamellas divided by temperature for a Pb-Cd sample ($d_N=2200$ Å, annealed). All samples, annealed and unannealed, showed a similar temperature dependence. Below 3–4 K, κ^N/T is temperature independent as expected for impurity scattering of electrons. Above this temperature phonon scattering of electrons becomes increasingly more important.

phonon mean free path of ~1 μm. This low conductivity is not unreasonable in view of the large strain expected due to the differential thermal contraction of the lamellas. From these measurements it is seen that for κ parallel to the lamellas, phonons cannot account for more than ~2% of the total conductivity when the sample is in the normal state and not more than ~5%–10% in the superconducting state, even at the lowest temperatures. This is consistent with the normal-state data (Fig. 4) showing a thermal conductivity nearly proportional to T as is expected for electrons scattered by impurities. Thus the data in Fig. 3 can be considered as the ratio of the thermal conductivities due to electrons.

For the case of electrons which are scattered by nonmagnetic impurities, the theory of Bardeen, Rickaysen, and Tewordt[12] (BRT) determines the ratio κ^S/κ^N as

$$\frac{\kappa^S}{\kappa^N}=\frac{12}{\pi^2}\int_{\Delta/2kT}^{\infty}x^2\,\mathrm{sech}^2x\,dx\ . \tag{1}$$

A curve calculated according to Eq. (1) for a value of Δ_0 appropriate to Pb ($2\Delta_0/kT_c=4.3$)[13] is shown in Fig. 3. All the data lie above this line indicating that Δ in the Cd is reduced from that in the Pb.

To make a quantitative estimate of Δ it is necessary to take into account the spatial variation of Δ in the composite. When a metal in the superconducting state is placed in good contact with a metal in the normal state, a nonzero pair potential Δ_N can be induced in the second metal.[1,2] If the temperature is sufficiently above the transition temperature of the second metal then Δ_N decays exponentially[1,14] away from the superconductor–normal-metal boundary with decay length K^{-1}. In an S-N-S $\cdots$

(superconducting-normal-superconducting- · · ·) structure, such as a eutectic, Δ_N takes the form of a hyperbolic cosine as shown in Fig. 5. For the case $K^{-1} >> d_N$ the pair potential is nearly uniform in the Cd whereas for $K^{-1} << d_N$ it resembles a well. For a clean metal (which the Cd is) K^{-1} is given by[1]

$$K^{-1} = \hbar v_F / 2\pi k T \quad , \tag{2}$$

where $\hbar$ is Planck's constant divided by 2π, v_F is the Fermi velocity,[15] and k is Boltzmann's constant. This gives $K^{-1} \simeq 1400$ Å at the transition temperature with K^{-1} increasing at lower temperatures. Thus samples can be made for studying either the regime $K^{-1} > d_N$ or $K^{-1} < d_N$. Unfortunately there is no full theory for the thermal conductivity in the proximity effect regime. Such a theory is needed to take account of both the spatial dependence of Δ and the density of quasiparticle states. Nevertheless, it is possible to draw some conclusions from the data for the two narrowest samples in Fig. 3 (the solid triangles). These samples are sufficiently thin (i.e., $d_N < K^{-1}$, at least at temperatures below 4 K) that Δ should be nearly independent of position.[16] This data is in reasonable agreement with the BRT theory [Eq. (1)] recalculated for a smaller ratio $2\Delta_0/kT_c = 1.8$. Thus the Cd lamellas in these samples behave as if they had a pair potential Δ_{Cd} given by

$$\frac{\Delta_{\mathrm{Cd}}}{\Delta_{\mathrm{Pb}}} = \frac{1.8\,T_c(\text{eutectic})}{4.3\,T_c(\mathrm{Pb})} = 0.41 \quad , \tag{3}$$

where Δ_{Pb} corresponds to the pair potential in the middle of the Pb lamellas. The width of the Pb is large enough so that this is essentially the bulk value. According to de Gennes the boundary conditions at a superconductor–normal-metal interface require that Δ/NV be continuous across the boundary.[7] So for

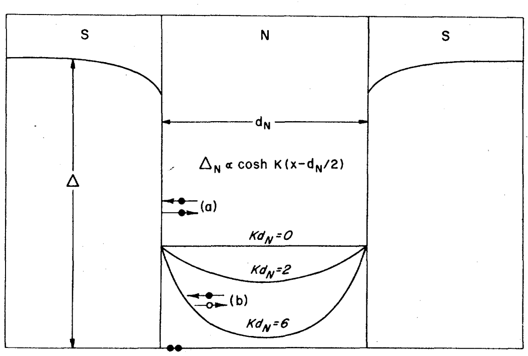

FIG. 5. Pair potential as a function of position for normal-metal (N) lamellas bounded by a superconductor (S). The pair potential is depressed slightly in S near the interface. The shape of the pair potential in N depends on the magnitude of Kd_N where d_N is the width of the N region. Process (a) represents an electron being specularly scattered at the Pb-Cd interface. Process (b) represents Andreev scattering occurring within the Cd.

these samples of nearly uniform Δ_{Cd}

$$\Delta_{\mathrm{Cd}} = \frac{(NV)_{\mathrm{Cd}}}{(NV)_{\mathrm{Pb}}} \Delta_{\mathrm{Pb}}(\text{interface}) = \frac{(NV)_{\mathrm{Cd}}}{(NV)_{\mathrm{Pb}}} \eta \Delta_{\mathrm{Pb}} \quad , \tag{4}$$

where η is defined as Δ_{Pb} (interface)/Δ_{Pb} which measures the reduction of the pair potential in the Pb near the interface. Equation (4), together with the measured value of $\Delta_{\mathrm{Cd}}/\Delta_{\mathrm{Pb}}$ and the known values of $(NV)_{\mathrm{Cd}}$ and $(NV)_{\mathrm{Pb}}$,[17] gives the experimental value $\eta_{\exp} = 0.9$. Theoretical estimates give values of η (Refs. 2 and 18) between 0.7 and 1.0, however, η must surely tend toward 1.0 as NV for the "normal" metal tends toward $(NV)_{\mathrm{Pb}}$. Since Cd has an NV a sizeable fraction of $(NV)_{\mathrm{Pb}}$, the experimental result is very reasonable.

Measurements have also been made on several samples with wide Cd lamellas (open circles and squares in Fig. 3) although these samples are more difficult to analyze because of the significant spatial variation of the pair potential. At temperatures below $t \sim 0.3$, where the pair potential is most nearly independent of position, the thermal conductivity data for the wide samples is similar to that of the narrow samples suggesting that at least at low temperatures, the pair potential in the wide Cd samples also satisfies the de Gennes boundary condition. At higher temperatures the analysis is more complicated but a qualitative explanation for the data is offered in Sec. V.

V. ANDREEV SCATTERING

Andreev[19] showed that quasiparticles undergo an unusual scattering process at a superconducting-normal (S-N) boundary in which the momentum and charge of the quasiparticles are reversed. This mechanism was used to explain the low thermal conductance of superconductors in the intermediate state; the quasiparticles had a high probability of reflection in flowing *across* the S-N boundaries. But even for heat flow *parallel* to the S-N boundaries it is possible for Andreev scattering to cause a reduction in κ.[20–22] Assuming the boundaries to be sufficiently close together (as in the case of a laminar intermediate state with a small spatial period) the highly nonspecular Andreev scattering will give rise to a reduced κ similar to boundary scattering.[23] For the geometry of a lamellar eutectic Andreev scattering is expected to reduce the electronic mfp and hence κ (assuming $l_e > d_N$) by a factor[20] of l_e/d_N. This is a reduction of over an order of magnitude for the two samples with the narrowest Cd lamellas in Fig. 3. Thus the ratio $\kappa^S_{\mathrm{Cd}}/\kappa^N_{\mathrm{Cd}}$ should be reduced to less than 0.1 slightly below the transition temperature ($t \sim 0.8$–0.9). Instead the measured $\kappa^S_{\mathrm{Cd}}/\kappa^N_{\mathrm{Cd}}$ is near unity, implying that specular reflection is still occurring at the Pb-Cd interface.

This might be explained by the long time over which Andreev scattering occurs. One expects the scattering to occur in time $\hbar/\Delta$ during which an electron travels on the order of a coherence length. On the other hand, the eutectic boundary is expected to be sharp, so that the specular reflection which occurs in the normal state[24] should scatter electrons quickly—before the electron travels more than a few lattice spacings. With the two scattering processes acting in parallel, Andreev scattering could be suppressed by two orders of magnitude. Another possible explanation is that if specular reflection were somehow to occur slightly ahead of the Pb-Cd boundary then electrons would never reach the *S-N* boundary to be Andreev scattered.

Measurements made on several samples with wider Cd lamellas (i.e., greater periodicity) displayed the interesting feature that the ratio $\kappa_{Cd}^S/\kappa_{Cd}^N$ is smaller than the ratio for the narrow Cd lamellas samples. This result is somewhat surprising since the spatial variation of the pair potential in the wide lamellas samples should result in a lower average Δ and an increase in $\kappa_{Cd}^S/\kappa_{Cd}^N$. This can be understood by noting that for electrons with energy less than Δ_{Cd} (interface), the *S-N* boundary occurs *within* the Cd lamellas. Without the competition of another scattering mechanism, Andreev scattering must occur leading to a reduction in κ_{Cd}^S. (The following paper[25] gives an example of just such a reduction in the thermal conductivity when a magnetic field is used to produce a spatial variation of Δ within the Cd.)

To summarize, measurements of the pair potential in the Cd lamellas are in agreement with the de Gennes boundary condition requiring continuity of Δ/NV. Samples with small Cd widths had electron mean free paths longer than d_N indicating that Andreev scattering was suppressed at the Pb-Cd interface.

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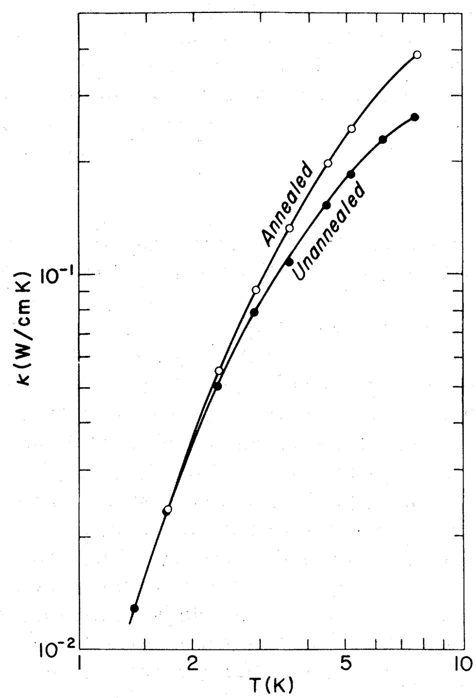

FIG. 6. Thermal conductivity measured parallel to the lamellas in zero magnetic field for a sample ($d_N = 1170$ Å) in both the annealed and unannealed state. The curves coincide below ~2 K indicating that the Cd lamellas are unaltered by annealing.

APPENDIX

The individual conductivities of the two solid phases of the eutectic can be determined from measurements parallel and perpendicular to the lamellas. Spencer *et al.*[6] have done this for Pb-Cd in the annealed and unannealed state and concluded that the Cd conductivity is unaffected on annealing. To check this conclusion note that well below the transition temperature the electronic contribution to the thermal conductance of the Pb will be vanishingly small. So the low temperature κ^S in the direction parallel to the lamellas should be a measure of only the Cd conductivity. As can be seen in Fig. 6, κ^S of the annealed and unannealed sample are indistinguishable at low temperatures confirming the lack of change in the Cd. At higher temperatures the conductivities differ because of the differing contributions of the Pb.

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