tigation similar to ours, with Ti rather than Mo as the solvent. Their conclusions are similar to ours, except that they find an anomalously strong variation of κ_1' and κ_2 for all values of ξ/l, whereas in the present work the temperature dependence of κ_1' and κ_2 is compatible with the theory, within the experimental error, for the samples of shortest mean free path.

The theory of Neumann and Tewordt[13] accounts very well for the magnitude of H_{c1}/H_c in all samples. Their estimate of the temperature dependence of this quantity is in violent disagreement with our observations on pure Nb. In the case of the alloy samples, their calculation is at least in qualitative agreement with the data. There is some indication that the theory underestimates the rate of change with temperature, but the accuracy of measurement does not warrant definite conclusion.

One would, perhaps, expect to find systematic errors in H_{c1} due to surface effects.[26] The fact that theory[13] gives a good account of the observed magnitude of H_{c1}, and the almost complete absence of hysteresis in the alloy samples, suggests that this is not so. The reason why surface effects are absent is not clear; it is presumably related to some special surface condition in these samples.

ACKNOWLEDGMENTS

We are obliged to P. E. Madsen, C. H. Thomas, and G. Taylor for assistance in preparing the specimens, and to K. Mendelssohn for his critical interest. Financial support was provided by the Ministry of Technology.

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Ultrasonic Attenuation in Superconducting Mercury and Mercury-Cadmium Single Crystals*†

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This paper presents the results of low-temperature ultrasonic-attenuation studies in mercury single crystals, polycrystals, and mercury single crystals with varying concentrations of cadmium impurity. The frequency ranged from 10 to 130 MHz and the doping concentration from 0.01 to 0.10% by weight. The major emphasis is a systematic study of the deviations of α_s/α_n for longitudinal ultrasound from that predicted by the BCS theory. These deviations are a drop in α_s/α_n with decreasing temperature near T_c which is greater than that predicted by BCS. The rapidity of the drop increased with increasing frequency in all pure samples, was frequency-independent, and decreased with doping concentration in the doped samples. The deviations observed cannot be explained adequately by dislocation attenuation or by multiple anisotropic energy gaps.

I. INTRODUCTION

DURING the past several years there has been a growing interest in the significant deviations from theoretically predicted values displayed by the attenuation of longitudinal ultrasound in superconductors. Bardeen, Cooper, and Schrieffer (BCS),[1] Tsuneto,[2] and others calculate that the ratio of the electronic attenuation in the superconducting state to that in the normal state should be

$$\alpha_s/\alpha_n = 2\{1+\exp[\Delta(T)/kT]\}^{-1}, \quad (1)$$

where $\Delta(T)$ is the superconducting energy gap. Measurements of this ratio in pure superconductors seldom follow Eq. (1), the deviation usually appearing near T_c as a too rapid decrease of α_s/α_n with decreasing temperature. The most extreme example of this effect yet reported is by Deaton,[3] who worked with a very pure unstrained lead crystal, but other workers have observed the too rapid drop in other materials: polycrystalline mercury,[4] single-crystal mercury,[5] single-crystal tin,[6–8] single-crystal lead and lead doped with

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[1] J. Bardeen, L. N. Cooper, and J. R. Schrieffer, Phys. Rev. **108**, 1175 (1957).

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thallium,[9] and single-crystal thallium.[10] Most of the past work in ultrasonic attenuation of superconductors has been presented in a form which allows at best a very qualitative judgment as to whether the data actually do fit a BCS-shaped curve and no quantitative measure of the departure; but it seems safe to assert that non-BCS behavior has been observed in most pure superconductors.

The proposed explanations of the observed departures have generally involved one or more of the following:

(1) anisotropy of the superconducting energy gap[6] perhaps coupled with a multiplicity of gaps for the multiple sheets of the Fermi surface,

(2) temperature-dependent dislocation attenuation due to changes in the electron damping of the dislocation motion in the superconducting state,[11] and

(3) the fact that the first and most pronounced evidences of non-BCS behavior occurred in strong-coupling superconductors,[12] lead and mercury.

There has been recent emphasis both in the strong-coupling and dislocation explanations. A calculation has recently been published by Woo[12] which shows that in strong-coupling superconductors there should be a change in the electron mean free path when the sample goes superconducting with a corresponding change in the attenuation. Fate *et al.*[13] have applied this theory with good results to explain their experiments on lead. The recent measurements by Ferguson and Burgess[14] of the attenuation of hypersonic sound in mercury leads to attenuation ratios which give good fit to Eq. (1) for $2\Delta(0)=4.0\ kT_c$ and the BCS temperature dependence of $\Delta(T)/\Delta(0)$. They attribute the good fit to the fact that the dislocation attenuation is negligible compared to the electronic attenuation for such high frequencies.

Arguing from the results of the measurements reported herein, one can say the following:

(1) The anomalous behavior in mercury is not due to anisotropy of multiple gaps alone, although the attenuation is strongly anisotropic.

(2) Temperature-dependent dislocation attenuation seems to be ruled out also, but this case is not as strong as the one against anisotropy.

(3) No comparison with Woo's theory[12] can be made at present since the calculations necessary to do so for mercury have not been performed; however, any explanation of the anomalous behavior of α_s/α_n in pure superconductors which relies only on strong coupling cannot explain the presence of the rapid drop in weakly coupled superconductors.

It seems likely, therefore, that a combination of the proposed effects is necessary to explain all the observations, but in any event it is clear that more quantitative experimental studies of the fast drop are necessary.

We report here a study of the effects of propagation direction, sound frequency, and impurity concentration on the ultrasonic attenuation of pure single crystal, pure polycrystal, and cadmium-doped single crystals of mercury.

II. EXPERIMENTAL DETAILS

Sample Preparation

The samples were nearly cylindrical—about 1 cm in length and $\frac{1}{2}$ in. diam. The pure samples were made from mercury with a stated purity of 99.99999%, supplied by United Mineral and Chemical Co. The cadmium used was of 99.999% stated purity, supplied by Merck, Inc. The mercury in some of the cadmium-doped samples was 99.9995% pure, supplied by Wilt Corp., Latham, N.Y. The pure samples included single crystals in which the sound propagated along the twofold and the threefold directions and one polycrystal sample. The doped samples were all single crystals with propagation along the twofold direction. The nominal cadmium concentrations ranged from 0.01 to 0.1 wt% cadmium. The concentrations are not known precisely because of the oxidation of some unknown amount of the dissolved cadmium before sample growth and the effect of zone refining during sample growth.

The single-crystal samples were grown from oriented mercury seeds and were not ground or polished. Two transducers were grown onto the faces of the mercury crystals with no bonding material used. This method yielded a high percentage of good bonds for the twofold and polycrystal samples using optically polished or fine ground 10-MHz X-cut transducers, of $\frac{1}{4}$in. diam, but did not work well for the threefold samples. The faces were flat and parallel to within 0.0001 in. The method of growth is unusual and for that reason is described in some detail.

The mercury seeds were prepared by lowering a $\frac{3}{16}$-in.-diam thin-walled stainless-steel tube full of mercury slowly into liquid nitrogen. The tubes were about 8 cm long and were coated on the inside with graphite (Dag dispersion No. 154, Acheson Colloids). Besides protecting the sample purity the graphite suspension is also an invaluable mold release agent. Without it mercury will freeze very tenaciously to glass and to many clean metal surfaces. The seeds would freeze in about $\frac{1}{2}$ h with the lower end of the thin-walled tube just immersed in the nitrogen. This corresponds to a growth rate of about 2.6 mm/min.

The seeds were oriented by using the Laue back-reflection technique. While the x-ray pictures were being taken the seeds were kept below -70°C by being

[9] W. A. Fate and R. W. Shaw, Phys. Rev. Letters **19**, 230 (1967).
[10] H. J. Willard, R. W. Shaw, and G. L. Salinger (to be published).
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[12] J. W. F. Woo, Phys. Rev. **155**, 429 (1967).
[13] W. A. Fate, R. W. Shaw, and G. L. Salinger (to be published).
[14] R. B. Ferguson and J. H. Burgess, Phys. Rev. Letters **19**, 494 (1967).

clamped in the seed orienter and crystal grower shown in Fig. 1. The cooling was done by liquid nitrogen in a reservoir attached to the 1-in.-diam aluminum support rod, the upper part of the rod being in contact with the nitrogen. By rotating the seed holder and pivoting the arm, the crystallographic direction along which the sound was to propagate in the completed sample was made parallel to the aluminum support rod (vertical in Fig. 1). The block which held the seed holder was removed and rotated 90°. A platform which held the sample mold was attached to the support rod. The results of these operations are shown in Fig. 2. The sample mold is shown in Fig. 3. It was placed on the sliding platform with the sound propagation direction perpendicular to the plane of the page, as viewed in Fig. 2. Thus the propagation direction was along the desired

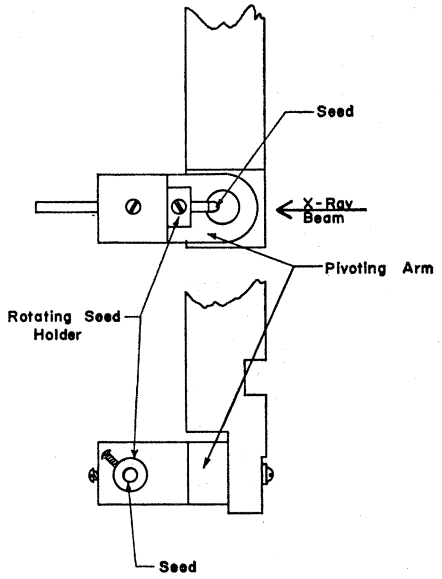

FIG. 1. Two views of a seed held in the seed orienter and crystal grower.

crystallographic direction. The propagation direction was within ±3° of either the two or threefold directions.

The mold was raised on the platform and the tip of the seed inserted into the melt. The heat of fusion was conducted up the seed to the support rod, gradually freezing the sample outward from the seed. Since molds were made of glass or Teflon, both having very low thermal conductivity, and the growing was done in a dry box to minimize the formation of frost on the still-liquid mercury, there was little tendency for stray nucleation to occur. The orientation was verified by taking an x-ray picture of the bottom of the sample. If this picture was identical to the pictures taken of the end of the seed before growth, this was taken as an indication that the growth proceeded correctly from top to bottom clear through the sample. On occasion a sample face was x-rayed, but the presence of the transducers

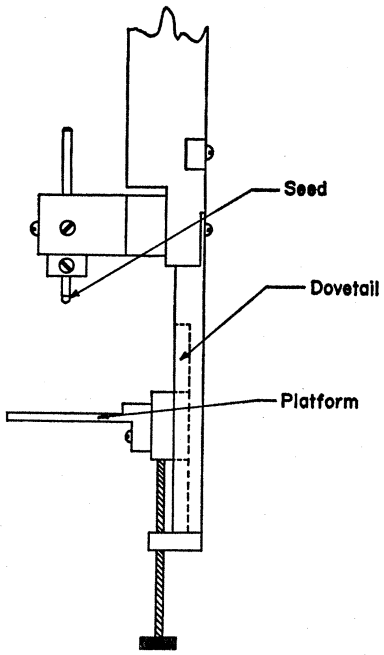

FIG. 2. Seed orienter and crystal grower with sliding platform attached and seed holder rotated 90°, ready for sample growth.

on the faces made this more difficult. The over-all accuracy of the crystal growth was ±3°—the major source of error being the play in the sliding dovetail necessary in order that it work smoothly at low temperatures. All orientations were performed with the help of stereographic projections of important directions and of the normals to important planes. These projections were made from tables of interplanar and interdirectional angles calculated especially for mercury, which has a rhombohedral crystal structure.

The polycrystal samples were grown in the same mold as the single crystals, but were not seeded. It is difficult to make useful polycrystal samples because a freezing rate which is slow enough to ensure samples free from fissures and possible gross internal imperfections will

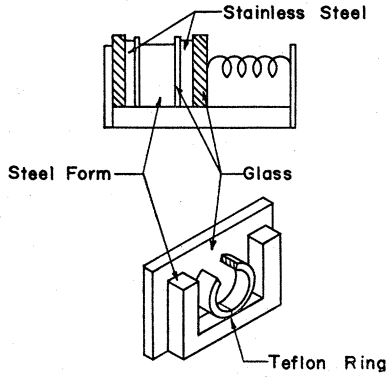

FIG. 3. Sample mold—the seed was inserted from above, through the gap in the Teflon ring.

result in a sample which contains only a few crystallites and is, therefore, not isotropic. For this reason data from only one polycrystal sample is given in this paper.

After growth, all samples used in this study were stored in liquid nitrogen and were kept below liquid-oxygen temperatures even when being installed in the sample holder for insertion into the low-temperature apparatus. These precautions were taken because it is reported[15] that mercury single crystals tend to recrystallize into polycrystals if they are kept at temperatures greater than about −100°C.

Instrumentation

The studies were conducted by the pulse-echo technique[5] at frequencies ranging from 10 to 130 MHz. There were calibrated variable attenuators matched to the lines both on the output of the pulsed oscillator and the inputs to the receiver. Generally two transducers were used, but on occasion one bond would fail as the sample was cooled to helium temperatures and the remaining good bond was used in a one transducer mode. The pulsed oscillator and receiver were part of a Matec ultrasonic generator. There were no nonlinear circuit elements between the sample and the calibrated attenuators. This obviously enhanced the accuracy of measurements made over a wide dynamic range but the 50-Ω attenuators had to be matched to the line. This was done by the double-stub tuner (Weinschell Engineering) and by preamplifiers which had tuned transformer coupled inputs with approximately 50-Ω input impedance. Several of these preamplifiers were constructed, each tuned for a different frequency. They consisted of two Nuvistor triodes, cascode connected, with tuned input and output. These simple cascode circuits gave about 20-dB signal gain with very little increase in noise—probably because the preamplifier bandwidth was less than the 4-MHz bandwidth of the receiver's i.f. section.

The use of two calibrated attenuators allowed an accurate check on the presence of amplitude-dependent attenuation. This is very important since the amplitude-dependent attenuation usually increases with decreasing temperature in the superconducting state and causes the rapid drop in electronic attenuation near T_c to appear even more rapid.[8]

The data were taken with the sample in liquid helium and the temperature was measured by a 56-Ω Allen-Bradley carbon resistor calibrated against the helium vapor pressure.

In order to measure the normal-state attenuation, superconductivity was quenched by a magnetic field of about 400 Oe applied parallel to the direction of sound propagation. For the pure samples, the attenuation in perpendicular field at low temperatures was too high for the signal to be usable. In an attempt to correct for the effect of the magnetic field on attenuation, the field was swept from 0 to 2 kOe at constant temperatures. Severe magnetoacoustic oscillations at low temperatures in the pure samples made the low-temperature normal-state data somewhat inaccurate; however, this error is judged to be small compared to that caused by uncertainty in the nonelectronic background attenuation which was subtracted from the raw data and which becomes increasingly important at low temperatures.

[15] D. M. Hill, Phys. Rev. **48**, 620 (1935).

III. RESULTS

Amplitude Independence

The amplitude dependence of the attenuation in the samples used in this work was less than $\frac{1}{2}$ dB over a total change of 40 dB in input amplitude at the lowest temperatures and at 50-MHz sound frequency. The method used to investigate the amplitude dependence involved inserting attenuation on the receiver attenuator and removing the same amount from the transmitter attenuator. If the attenuators are both correctly matched to the lines and there is no amplitude dependence, this should cause no change in the size of the received signal.

Pure Samples—Superconducting Behavior

The pure samples yielded results for α_s/α_n versus T/T_c as shown in Fig. 4. Note that the falloff near T_c becomes more rapid with increasing frequency, as observed by Deaton[3] in lead and Thomas *et al.*[5] in mercury. It is interesting to note that Fate, Shaw, and Salinger[13] in lead and Willard, Shaw, and Salinger[10] in thallium observe a decrease in the rapidity of the falloff with increasing frequency. A quantitative determination of the rapid drop cannot be made from a set of curves such as Fig. 4. In order to make quantitative measurements it is most convenient to invert Eq. (1), giving

$$2[\Delta(0)/kT_c]G(t)/t=2ln[2\alpha_n/\alpha_s-1], \qquad (2)$$

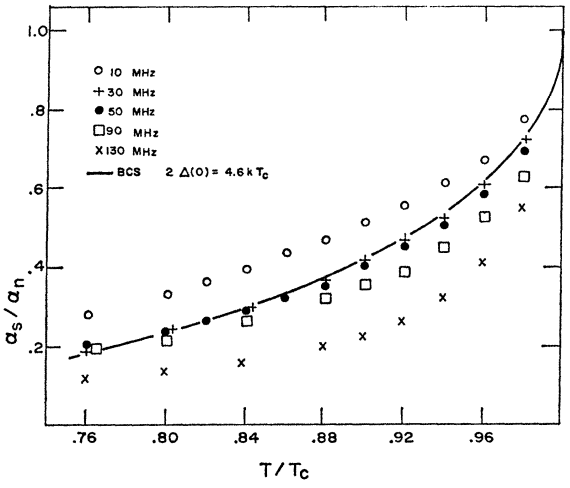

FIG. 4. Typical plots of α_s/α_n versus T/T_c for the pure twofold samples.

where $G(t)=\Delta(T)/\Delta(0)$ and $t=T/T_c$. According to BCS theory $G(t)$ will be the same for all superconductors. Mühlschlegel[16] has calculated $G(t)$ versus t and these values are used here.

Plotting $2\ln[2\alpha_n/\alpha_s-1]$ versus $G(t)/t$ should yield a straight line for a BCS superconductor and the slope of this line is $2\Delta(0)/kT_c$, which should be 3.52 for weakly coupled superconductors and about 4.5 for strongly coupled ones. Such a plot will be called a $G(t)/t$ plot hereafter.

In practice these plots are often nonlinear, typically displaying a greater slope near the origin (near T_c) than at low temperatures. This greater slope corresponds to the too rapid falloff of α_s/α_n near T_c with decreasing temperature. For such cases the identification of the slopes with a superconducting energy gap may not be justified but we will continue to do so for convenience. Figure 5 is a $G(t)/t$ plot of the extreme cases of Fig. 4.

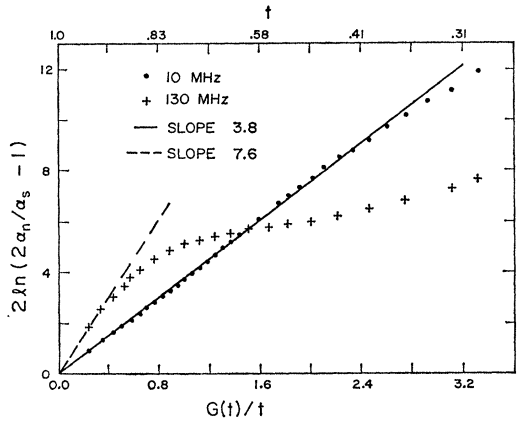

FIG. 5. Typical $G(t)/t$ plots using the pure twofold data of Fig. 4. According to BCS the slopes $=2\Delta(0)/kT_c$.

The 10-MHz data follows a BCS relation quite closely over the entire temperature range but with a slope of 3.8 instead of the 4.6 to be expected from tunneling measurements.[17] The 130-MHz data, however, display an extremely rapid drop near T_c, corresponding to a $2\Delta(0)/kT_c=7.6$, and a distinct flattening of the curve at low temperatures corresponding to $2\Delta(0)/kT_c$ of about 2.

Data were taken on several twofold and threefold samples and a polycrystal sample at several frequencies, and the slopes of the corresponding $G(t)/t$ plots display a systematic frequency dependence as is shown in Fig. 6. The falloff near T_c increases with frequency except for the threefold samples, where the data were not of as good quality because of poor transducer bonds. Also, the slopes of the $G(t)/t$ plots near T_c were less than those at low temperatures for the 10-MHz polycrystal

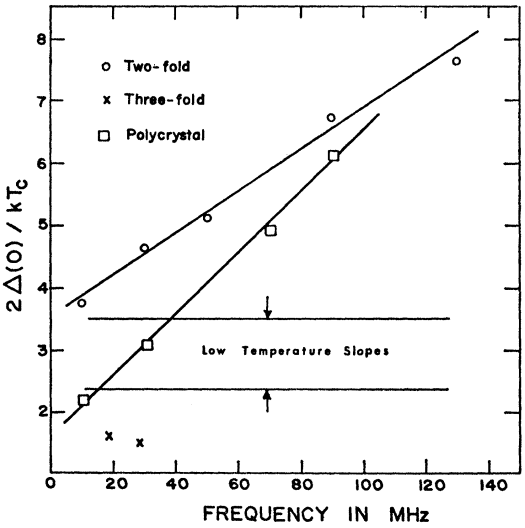

FIG. 6. Slopes of $G(t)/t$ plots near T_c for the pure samples with the range of slopes at low temperatures shown.

and the threefold samples. This fact is of significance and will be discussed in Sec. IV.

Doped Samples—Superconducting Behavior

The ratio α_s/α_n versus temperature for the twofold cadmium-doped samples was frequency-independent. There was, however, a marked and systematic variation with doping concentration. Figure 7 shows the $G(t)/t$ plot for 50 MHz, 0.01% cadmium-in-mercury sample. The solid curve represents the sort of behavior that might occur according to the multiple-gap picture of Morse[18] and Claiborne and Einspruch,[19] with two independent energy gaps—one for each sheet of the Fermi surface.[20]

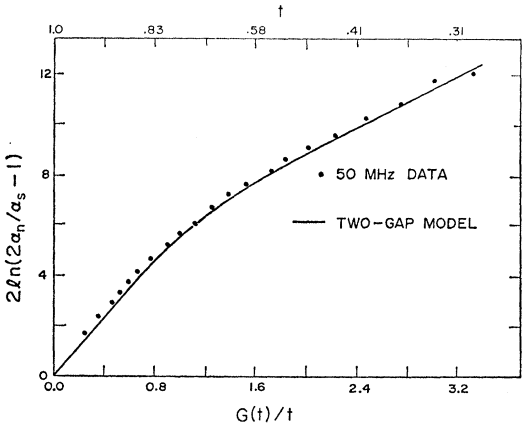

FIG. 7. Typical $G(t)/t$ plot for doped twofold samples—0.01% cadmium. The solid line is from the two-gap model for $\Delta_1(0)=6.8kT_c$, $\Delta_2(0)=2.6kT_c$, and $A=1.69$.

[16] B. Mühlschlegel, Z. Physik **155**, 313 (1959).
[17] D. M. Ginsberg and M. Tinkham, Phys. Rev. **118**, 990 (1960).
[18] R. W. Morse, IBM J. Res. Develop. **6**, 58 (1962).
[19] L. T. Claiborne and N. G. Einspruch, Phys. Rev. Letters **15**, 862 (1965).
[20] G. B. Brandt and J. A. Rayne, Phys. Rev. **148**, 644 (1966).

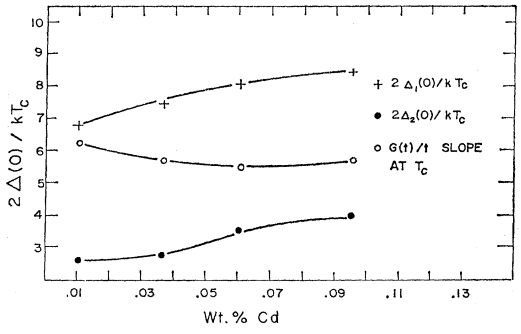

FIG. 8. Systematic changes of $\Delta_1(0)/kT_c$, $\Delta_2(0)/kT_c$, and $G(t)/t$ slope at T_c versus cadmium concentration.

The fact that doped samples display a simple two-gap behavior whereas the pure samples do not might be explained by arguing that the effect of modest dopant concentrations lowers the electron mean free path enough to eliminate anisotropy in the superconducting behavior but does not cause too much mixing of the two sheets of the Fermi surface. Thus the superconductor would be characterized by two nearly independent energy gaps.

According to the two-gap picture,

$$\alpha_s/\alpha_n = A[1+\exp(\Delta_1(t)/kT)]^{-1} + (2-A)[1+\exp(\Delta_2(t)/kT)]^{-1}, \quad (3)$$

where Δ_1 and Δ_2 are twice the superconducting energy gaps associated with the two sheets of the Fermi surface (Δ_1 is assumed to be the larger gap). The relative weighting of the two sheets is determined by A. Both $\Delta_1(t)$ and $\Delta_2(t)$ are assumed to be BCS shaped, i.e., $\Delta(t)=\Delta(0)G(t)$. One can show that at low temperatures the slope of the $G(t)/t$ plot will approach $2\Delta_1(0)/kT_c$ and that the slope of the $G(t)/t$ plot near T_c will be $A\Delta_1(0)+(2-A)\Delta_2(0)$.

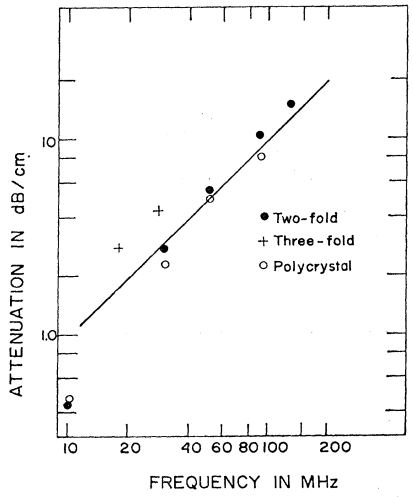

FIG. 9. Logα_n versus log$f$ for pure samples at $T=T_c$.

The good fit of the data which can be generated by the two-gap model should not be taken as a demonstration of its correctness since there are three adjustable parameters in the theory. It does offer some idea of the magnitudes of the differences needed to explain ultrasonic attenuation in this way, however, and serves as a convenient means of characterizing the data.

Figure 8 shows the systematic change of the various parameters of the two-gap model with cadmium concentration. With increasing cadmium concentrations the low- and high-temperature slopes each approach a value somewhere between four and five while the larger gap seems to increase with increasing concentrations.

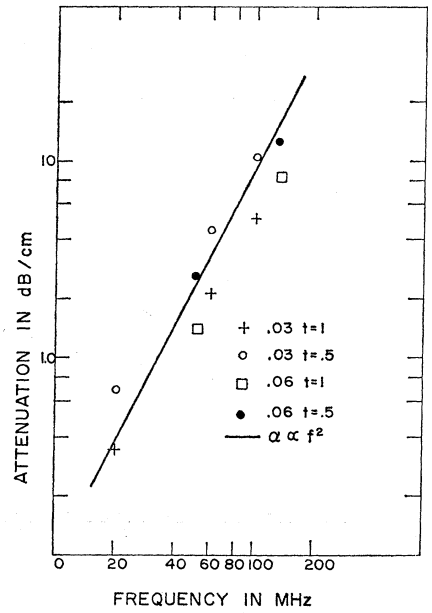

FIG. 10. Logα_n versus log$f$ for 0.03% cadmium and 0.06% cadmium in mercury samples at $t=1$ and 0.5.

Electron Mean Free Paths

The pure samples had a large electron mean free path l, as can be seen by Fig. 9. Even at T_c the plots of logα_n versus log$f$ are linear, with a slope of unity for frequencies >30 MHz. This implies that $ql \gg 1$, where q is the ultrasonic wave vector.[21] One can get quantitative estimates of ql by considering the ratios of Pippard's formula[21] for electronic attenuation versus ql at different frequencies and comparing these to the ratios of the measured attenuations.[12] Such a process yielded $ql \simeq 0.7$ at $t=1(T=T_c)$ and $ql>6$ at $t=0.3$ for $f=10$ MHz.

The doped samples had smaller ql's, as would be expected. Figure 10 shows logα_n versus log$f$ for the doped samples at various doping concentrations and temperatures. The slopes of these plots are nearly those which

[21] A. B. Pippard, Phil. Mag. 46, 1104 (1955).

would obtain for $ql \ll 1$. In this limit, $\alpha \propto f^2$. A more quantitative analysis as was done for the pure samples indicated that for the 0.03% cadmium sample $ql \simeq 0.5$ at $t=1$ and $ql \simeq 0.9$ at $t=0.5$ for $f=20$ MHz. This value is almost one-third that of the pure samples at T_c and less than one-tenth at lower temperatures.

Temperature-Dependent Backgrounds and Dislocation Attenuation

The deviations from BCS theory can be due either to anomalous electronic attenuation or to a temperature-dependent nonelectronic background attenuation. Since the BCS theory deals only with the attenuation due to electrons, it is necessary that the nonelectronic component be subtracted from the observed attenuation. All data presented in this study have been processed by subtracting the value obtained by extrapolating the attenuation in the superconducting state to 0°K from the measured superconducting and normal-state attenuation versus temperature. Thus the background was assumed temperature-independent.

A temperature-dependent background would not only effect the shape of α_s/α_n versus t but would also effect α_n-versus-frequency curves, such as Figs. 9 and 10. Because of the inability to obtain usable normal-state data in transverse magnetic fields it was not possible to make a quantitative assessment of how well the data fit Pippard's theory,[21] as did Fate and Shaw in lead.[9] Nevertheless, the qualitative behavior exhibited by Figs. 9 and 10 is quite close to the f and f^2 dependence expected, indicating that a temperature-dependent background is an unlikely explanation for the observed anomalous superconducting behavior.

Specifically, the above observations tend to rule out temperature-dependent dislocation attenuation[10,13] as a cause of the observed effects. This argument is supplemented by the presence of the rapid drop in the doped samples. For these samples, the background attenuation was reduced considerably (for the 0.06% cadmium in mercury at 130 MHz the background at $t=0.3$ was reduced from more than 6 dB/cm in the pure twofold sample to less than 1 dB/cm) showing that the dislocations were extensively pinned by the impurity atoms.

IV. CONCLUSIONS

To explain the results of these measurements with a model relying exclusively on multiple anisotropic energy gaps would require enormous differences between the average gaps of the two sheets (from $2.5kT_c$ for one sheet to $6.5kT_c$ for the other) and large anisotropies (at least 30% for both sheets). In addition, no simple picture of the shape of the energy-gap surface in k space seems capable of explaining all of the data.

As described in detail in Sec. III, there is considerable, but not airtight, evidence on the basis of these measurements that dislocation attenuation does not cause the observed anamolous behavior. In summary, these arguments involve the absence of a temperature-dependent background attenuation (unless the background varies linearly with frequency), the absence of significant amplitude-dependent attenutation, and the fact that the doped samples displayed the anomalous behavior even though the dislocations were pinned much more than in the pure samples.

As was mentioned in the Introduction, the necessary theoretical work has not been done to compare the results of this work with Woo's theory.[12] Fate *et al.*[13] have made very careful measurements in lead which show qualitative agreement with the theory.

It seems safe to say that the most striking examples of anomalous behavior reported in the literature are in lead and mercury—both strongly coupled. On the other hand, the presence of anomalous behavior in tin, thallium, and other weakly coupled superconductors shows that the strong-coupling theory does not explain the whole effect.

In summary, this work and the others cited seem to point toward state-dependent mean free paths and multiple anisotropic energy gaps as components in an explanation of the anomalous ultrasonic attenuation in superconductors, but more work clearly needs to be done. The systematic changes of the drop near T_c with frequency and with impurity concentration are thought provoking but as yet unexplained. Finally, the utility of presenting ultrasonic attenuation data in the form of $G(t)/t$ plots, or some other similar scheme, which allows quantitative systematic studies of the anomalous behavior, could hasten our understanding of the phenomenon.

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