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Thermal Boundary Resistance between Cerium Magnesium Nitrate and Liquid He^3

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The thermal boundary resistivity between cerium magnesium nitrate and liquid He^3 has been measured over the temperature range 0.07–0.7 K. The resistivity can be well described as $(55 \pm 10)/T^3$ $cm^2\,K\,W^{-1}$ which compares with $150/T^3$ $cm^2\,K\,W^{-1}$ as deduced from the Khalatnikov acoustic-mismatch theory of thermal boundary resistance. The theoretical values includes the surface-wave contribution. This agreement with acoustic-mismatch theory is in turn compared with that obtained for other materials, and is seen to be relatively good. Finally, the thermal-boundary-resistivity result has been used to reinterpret other experiments that measure the temperature decay following a change of magnetic field. It is shown that these experiments can be described by a relaxation process consistent, as far as size dependence, temperature dependence, and magnitude are concerned, with a phonon bottleneck.

I. INTRODUCTION

At low temperatures, the thermal relaxation time between cerium magnesium nitrate (CMN) and liquid He^3 was found by Abel *et al.* to be several orders of magnitude shorter than expected.[1] This effect was interpreted theoretically by Leggett and Vuorio,[2] and more recently by Guyer,[3] as a spin-spin interaction across the boundary between the spins of the cerium ions within the CMN and the He^3 nuclear spins in the liquid. Subsequent experiments by Black *et al.*[4] confirmed the existence of the effect, and showed that the enhanced coupling decreased when approximately one monolayer of He^4 was present on the crystal surface. In these experiments the energy interchange investigated was between the cerium spins, whose temperature was monitored with a magnetization measurement, and the liquid He^3. It seemed that a measurement of interest would be the thermal boundary resistance measured potentiometrically. The two methods are shown schematically in Fig. 1. Figure 1(a) shows the spin-temperature-decay method: A pulse of energy is transmitted either to the magnetic solid via a change in the ambient magnetic field, or to the liquid via an electrical heater. The solid can be a single crystal or a powder. The spin temperature of the solid is monitored with a magnetometer as the spin temperature and liquid temperature reach a common equilibrium. The thermal boundary resistivity R is given by

$$\tau = \frac{R}{A}\frac{C_1 C_2}{C_1 + C_2}, \tag{1}$$

where τ is the decay time constant, A is the contact area between solid and liquid, and C_1 and C_2 are the heat capacities of the solid and liquid. Internal time constants within solid or liquid are assumed negligible. Figure 1(b) illustrates the potentiometric method: a steady heat current flows across the boundary from solid to liquid, or vice versa. The temperature jump at the boundary is measured with thermometers close to the boundary. Corrections may have to be applied for gradients within the solid or liquid, for alternate heat paths and edge effects. The resistivity is given by

$$\dot{Q} = A \Delta T / R, \tag{2}$$

where $\dot{Q}$ is the heat current, A is again the contact area, and ΔT is the temperature jump at the boundary. In such an experiment for a dielectric it is expected that the heat current will be transported by lattice waves, and that the thermometers will register lattice temperatures. Such a measurement, for CMN, is described here. When this work was almost complete a value of the thermal boundary resistivity due to lattice waves was de-

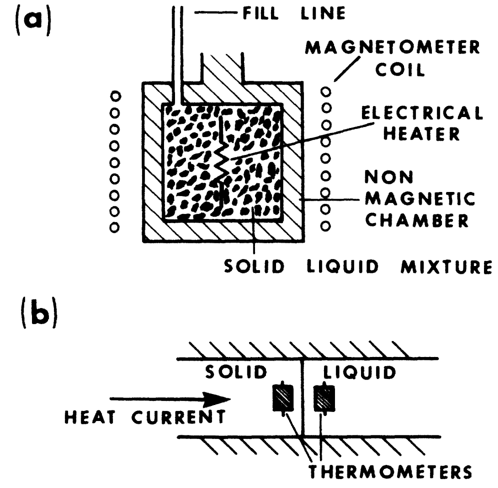

FIG. 1. Schematic illustration of the two types of boundary resistance measurements. (a) The spin-temperature-decay method, (b) the potentiometric method.

duced from a spin-temperature-decay experiment by Bishop *et al.*[5] The value quoted is far different from the result presented here. However it is found that the original data of Bishop *et al.* can be interpreted readily in terms of the present results and a phonon bottleneck.[6]

In the next three sections of the paper the present measurements are described. Then, an attempt is made to see the result in relation to measurements on other materials by a large number of experimenters. Finally, we resolve the discrepancy between the result presented here and that of Bishop *et al.*

II. SAMPLE PREPARATION

The CMN single-crystal preparation was the same as for the thermal-conductivity measurements previously described.[7] The sample was cut with a string saw from a clear section of the crystal and water polished to size. Thus no harsh treatment was accorded the sample, and so no surface damage should have resulted. Also, as the only contact with the surface was with tissue paper, no foreign matter should have deposited on the surface. It is known from the work of Bertinat *et al.*[8] that the surface of CMN is quite stable. The sample was 25-mm long with 4.45×5.28-mm rectangular cross section. The c axis of the crystal was in the cross section of the sample.

III. METHOD OF MEASUREMENT

Figures 2 and 3 show the sample and sample cell in vertical and horizontal cross section. The cell was of Epibond 100A with the mixing chamber of a dilution refrigerator at the top and a sample chamber at the bottom. The mixing chamber was sealed with a lid of Epibond 100A bonded with Epibond 121. The sample chamber was sealed with an Epibond 100A cap bonded to the chamber with a renewable indium seal. This cap contained electrical feedthroughs for the heater and thermometers within the sample chamber, and the He^3 fill line. Thermal contact between sample chamber and mixing chamber was made with 100 No. 32 copper wires. These wires, which extend from top to bottom of the cell, are embedded within the Epibond and lie just beneath the surface of both sample and mixing chambers.

The sample had two holes drilled in from the bottom. It was found possible to do this, without causing any visible damage to the sample. Into one hole was inserted a 1000-Ω nichrome heater with 0.075-mm constantan leads, and into the other was inserted a ground-down 100-Ω Speer carbon resistance thermometer. Both the heater and resistor were packed in with Apiezon N grease. There was a 0.4-mm gap between the sample and cell wall. Above and below the sample were Teflon end pieces, bonded to the sample with GE 7031 varnish. These were to prevent heat flow from the ends of the sample, and also they enabled the sample to be held centrally within the chamber. Teflon has an extremely small thermal conductivity at low temperatures.[9] A small hole was drilled up the chamber to permit a thermometer to be immersed in the He^3 close to the sample. In addition, a third thermometer was placed in a hole in the cell wall, drilled from outside. Again, thermal contact was made with N grease. The secondary thermometer for the experiments was a Scientific Instruments, Inc., Model 5 germanium thermometer, previously calibrated against the magnetic susceptibility of CMN. All thermometer resistances were measured with an SHE model 120 resistance bridge. As can be seen, the design was such that the thermometers measured temperature jumps at the boundaries between sample and liquid He^3, and liquid He^3 and cell wall provided either there were no temperature gradients in the plane of the cross section or any temperature gradients could be sufficiently accurately estimated. Since the thermal gradients within the sample were hard to estimate, it was important that the sample thermal conductivity be high. In fact, the purity and perfection of the samples was such that the mean free path of the phonons was equal to the crystal dimensions.[7] Thus, the temperature measured by the thermometer in the sample was appropriate for all points within the sample. The thermal conductivity of liquid He^3 has been measured and so the gradient within the liquid could be estimated. Since the copper wires were right at the surface of the chamber wall, and were of sufficient conductivity to carry

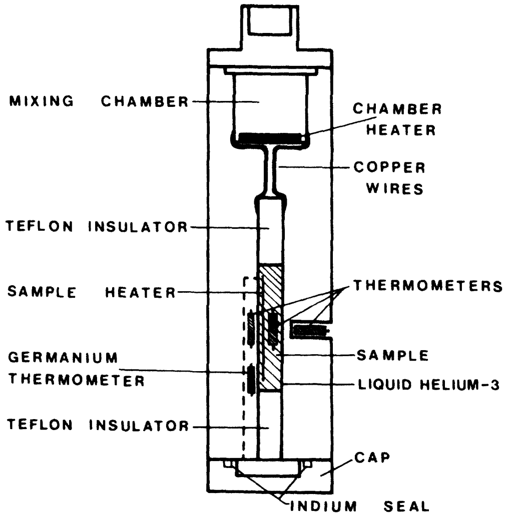

FIG. 2. Vertical cross section of the sample cell, showing the sample and thermometers.

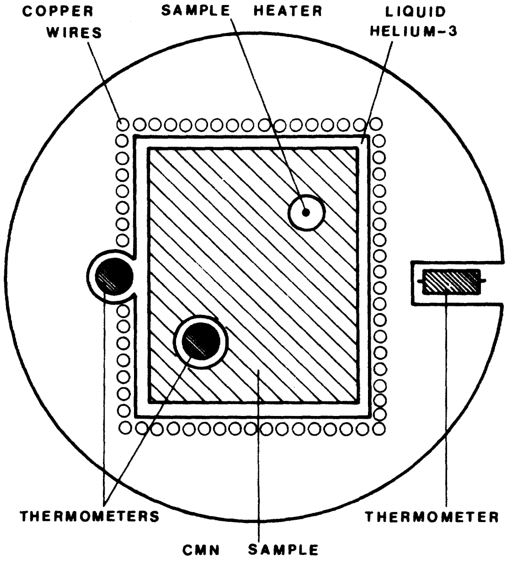

FIG. 3. Horizontal cross section of the sample cell at the center of the sample chamber.

all the heat to the mixing chamber, there were negligible thermal gradients within the cell wall. Thus, the thermometer in the wall should have accurately indicated the temperature of the wall at the boundary.

The experiment was performed as follows: After the refrigerator was started, the helium was admitted to the sample cell. This would warm the refrigerator to about 1 °K, but the recovery was quite quick, although the lowest temperatures were achieved only after a several-hour wait. This was almost certainly due to the large mass of He^3 which occupied the dead volume in the sample-chamber cap. Once the low temperature had been achieved, the sample heater was switched on, with current sufficient to give a temperature jump at the sample-liquid boundary of between 5 and 10% of the sample temperature. The three resistances were noted. Then the sample heater was switched off and the mixing-chamber heater switched on with current sufficient to bring the thermometer in the He^3 to its former value. The three thermometer resistances were again noted, as also was the germanium-thermometer resistance. These were the isothermal readings. The procedure was repeated for a series of temperatures by increasing the mixing-chamber heater current. At the higher temperatures the refrigerator became unstable and so the refrigeration cycle was stopped. The mixing chamber then became a large thermal mass. At no temperature was automatic temperature control required as the system was quite stable. From the isothermal readings, the calibration of the carbon thermometers was determined. From this calibration the temperature jumps across the sample-liquid and liquid-chamber-wall boundaries for the known heat current were determined, and hence the boundary resistances were calculated. Above about 0.5 °K, the thermal gradient in the liquid He^3 became significant. It was not obvious whether the thermometer in the He^3 would indicate the temperature of the He^3 close to the surface of the sample, close to the surface of the chamber wall, or in between the two. Thus, we will show the high-temperature data with error bars which reflect this uncertainty.

IV. RESULTS

Two experimental runs were made, separated by about six months; the first was with He^3 containing 0.5% He^4 and the second with He^3 containing 0.01% He^4. It is noted that the concentration of He^4 in the He^3 is not nearly as significant as whether the actual mass of He^4 present is sufficient to form a monolayer cover over the surface of the sample. Since in this experiment the He^3 was admitted through sintered-copper heat exchangers with a total surface area of 1 m^2, it is expected that during the first run, the sample surface would certainly have had a monolayer of He^4 formed on it, whereas for the second run, the crystal would at most have had 50% He^4 monolayer coverage, unless there were a good reason for the crystal to attract the He^4 away from the copper.

In Fig. 4, the thermal boundary resistivity between the He^3 and the Epibond cell wall is shown for the two runs. Above 0.15 K, the data can be well described by $R = 60/T^3$ and $35/T^3$ $cm^2 K W^{-1}$ for the two runs. The departure from the T^3 lines at low temperatures is attributed to the relatively high thermal conductivity of the He^3 which allows alternate heat paths into the cell wall. The decrease in resistivity between runs 1 and 2 is attributed to mechanical damage to the cell wall by thermal cycling. The magnitude of the results is in agreement with previous measurements as also is the variation with structure of the Epibond surface. Anderson, Salinger, Steyert, and Wheatley measured the boundary resistivity to be $R = 30/T^3$ $cm^2 K W^{-1}$[10] whereas Anderson, Connolly, and Wheatley[11] found the resistivity to be $R = 60/T^3$ $cm^2 K W^{-1}$, and on examination observed that the number of crevices was less for the latter Epibond sample.

The thermal boundary resistance between He^3 and the CMN sample is shown in Fig. 5. The data can be well described by $R = (55 \pm 10)/T^3$ $cm^2 K W^{-1}$. In Fig. 6 a plot of RT^3 as a function of temperature is presented. This shows that the exponent of T which best describes R is $-(3 \pm 0.3)$. The Khalatnikov acoustic-mismatch theory[12-14] gives an expected resistivity of $150/T^3$ $cm^2 K W^{-1}$ when

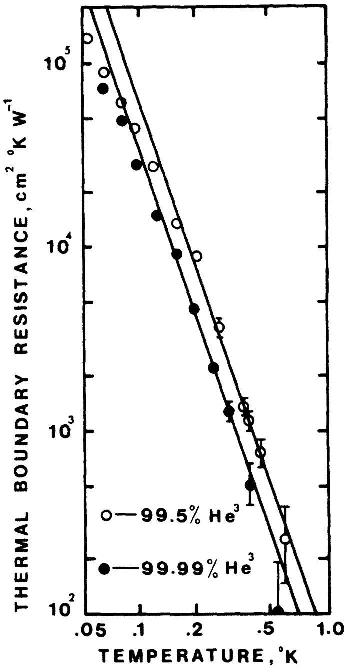

FIG. 4. Thermal boundary resistivity between Epibond 100A, with copper wires beneath the surface, and liquid He^3. Open circles: run 1 with 0.5% He^4 in the He^3. Closed circles: run 2 with 0.01% He^4 in the He^3. The straight lines shown a T^{-3} relationship.

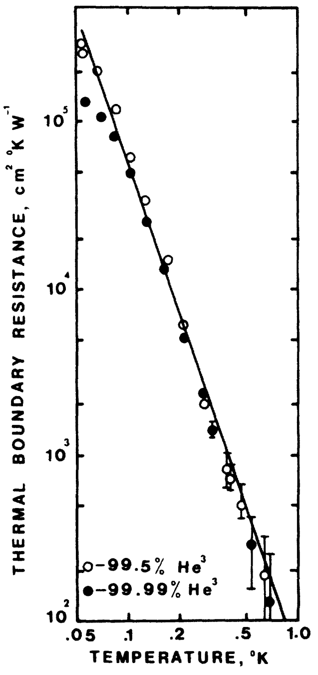

FIG. 5. Thermal boundary resistivity between CMN and liquid He^3. Open circles: run 1 with 0.5% He^4 in the He^3. Closed circles: run 2 with 0.01% He^4 in the He^3. The straight line is $R = 55/T^3$ cm² K W⁻¹.

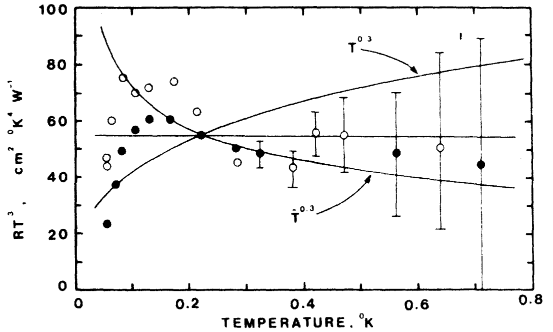

FIG. 6. RT^3 is plotted as a function of temperature. The solid lines indicate that $R \propto T^{-(3\pm0.3)}$.

a density of 2.0 g cm⁻³ and an average sound velocity of 2.12×10^5 cm sec⁻¹ are used. This expected resistivity was calculated on the basis that surface modes are contributing to the energy transfer across the boundary. Thus, experimental resistivity is 37% of the theoretical value, which in this field is quite close agreement. This comparison is seen in relation to that for other materials in Sec. V.

V. COMPARISON WITH OTHER RESULTS

The minimum possible boundary resistivity is that given by the phonon-radiation limit R_{PRL}. This limit, which has been described recently by Snyder[15] is the limit when all phonons impinging upon the surface of the solid from within the solid, are absorbed into the liquid. The limit is given by

$$R_{\mathrm{PRL}} = \frac{10}{\pi^2 k_B}\left(\frac{\hbar}{k_B T}\right)^3 v_s^2 ,$$

where v_s is an average velocity of sound in the solid. In terms of other parameters it is

$$R_{\mathrm{PRL}} = \frac{5}{2}\,\frac{\hbar\Theta_D^2}{\pi^4 k_B^2 T^3}\left(\frac{4\pi V}{3N}\right)^{2/3} ,$$

where Θ_D is the Debye temperature and N/V is the number density of atoms.

The maximum possible boundary resistivity is expected to be that given by the Khalatnikov acoustic-mismatch theory.[12–14] This is given by

$$R_{\mathrm{AML}} = F\frac{v_s\rho_s}{v_l\rho_l} R_{\mathrm{PRL}} ,$$

where F is a factor of order unity, v_l is the velocity of sound in the liquid, and ρ_s and ρ_l are the solid and liquid densities respectively. Thus

$$R_{\mathrm{AML}} = \frac{10F}{\pi^2 k_B \rho_l v_l}\left(\frac{\hbar}{k_B T}\right)^3 \rho_s v_s^3 ,$$

or in terms of other parameters,

$$R_{\mathrm{AML}} = \frac{5F}{3\pi^4 k_B \rho_l v_l}\,\frac{M\Theta_D^3}{T^3} ,$$

where M is the molecular weight of the solid.

The two boundary-resistivity limits differ by several orders of magnitude, and with one exception, all reported measurements lie at or within the limits. Since both limits do not depend simply upon one parameter of the solid, and since the ratio between the limits is not a function of any single solid parameter, the agreement between a measurement and the limits will be shown through the function

$$P=\frac{\ln R_{\text{meas.}}-\ln R_{\text{PRL}}}{\ln R_{\text{AML}}-\ln R_{\text{PRL}}} .$$

Thus, if a measurement is equal to the phonon-radiation limit then $P=0$. If a measurement is equal to the acoustic-mismatch limit, then $P=1$. Logarithms of R were chosen in the ratio P because of the several orders of magnitude difference between the limiting values. Since in few cases have measured boundary resistivities been found proportional to T^3, the ratio P is shown as a function of reduced temperature T/Θ_D. Figure 7 is such a plot for a variety of solids. First, such a plot makes evident the poor agreement of the great bulk of measurements, made above 1 K with the acoustic-mismatch theory. Next, the tendency for P to increase towards 1 at low temperatures is observed. In particular, the recent work on copper by Anderson and Johnson,[13] the very-low-temperature measurements of chrome potassium alum by Vilches and Wheatley[16] and the present results for cerium magnesium nitrate are cited. The anomalously large value of P for the CMN result of Bishop *et al.* is discussed further below. It is rather surprising to see the relatively good agreement with acoustic-mismatch theory for cerium magnesium nitrate extending to such large values of T/Θ_D. This would call into question any theories, used to explain the low values of P for other materials, based upon properties of the liquid. Such theories are the excitation-energy theory discussed by Anderson and Johnson,[13] the variable-acoustic-impedance theory of Challis, Dransfeld, and Wilks[22] and the shear-wave-dissipation theory of Saslow.[23] One fact concerning large-molecular solids should be mentioned. The actual value of Θ_D becomes an uncertain parameter since almost certainly the low-energy acoustic mode will not consist of entire CMN molecules vibrating as a unit. Going to the other extreme, by treating CMN as an atomic solid, with all atoms treated as of average mass connected by equal "forces," the Debye temperature is increased to 300 K. The dashed line on Fig. 6 is the present results plotted as a function of T/Θ_D with $\Theta_D=300$ K. The vertical displacement is for visual clarity. Still the P value does not show a decrease at T/Θ_D as high as 2.5×10^{-3}.

The anomalously large value of P for the CMN result of Bishop *et al.* is now considered. It is shown below that the raw data can be analyzed in terms of the present value for the thermal boundary resistivity and in addition this leads to a much simplified explanation for the experimental results. Bishop *et al.* made measurements of the decay of spin temperature to bath temperature following a magnetic pulse which either warmed or cooled the cerium spins with respect to the bath. The CMN was in two forms: First a single crystal $2.28\times2.28\times0.15$ mm, with volume $0.75\ \text{mm}^3$ and surface area $11.4\ \text{mm}^2$, and second a powder with mean diameter 0.055 mm, total volume $0.52\ \text{mm}^3$, and surface area $170\ \text{mm}^2$.[24]

Turning first to the single-crystal data, it is assumed that the measured relaxation time was due to an internal thermal resistance in series with the thermal boundary resistance given by $R=55/T^3$ $\text{cm}^2\,\text{K}\,\text{W}^{-1}$. It happens that the data are well described by

$$\tau/C_{\text{CMN}}=V(55/AT^3)+20\times10^4\ \text{cm}^3\,\text{K}\,\text{W}^{-1} , \quad (3)$$

where τ is the measured relaxation time, C_{CMN} is the specific heat of CMN (measured in $\text{J}\,\text{cm}^{-3}\,\text{K}^{-1}$), V and A are, respectively, the volume and surface area of the CMN. This is Eq. (1) under the condition that the heat capacity of the liquid is much larger than that of the CMN, as was the case for this experiment. The second term is interpreted in terms of the same phonon bottleneck as observed by Anderson and Robichaux and others.[6] In a similar vein, the measured relaxation time for the powder can be well represented by

$$\tau/C_{\text{CMN}}=V(3/AT^3)+6\times10^4\ \text{cm}^3\,\text{K}\,\text{W}^{-1} . \quad (4)$$

Again, we identify the first term with thermal-boundary resistance. The decrease in resistivity for the powder from $R=55/T^3$ to $R=3/T^3$ $\text{cm}^2\,\text{K}\,\text{W}^{-1}$ is not unexpected due to the harsh treatment given to the surface in powdering. Similar behavior has been observed recently by Anderson and Johnson[13] for sandblasted copper. The second term is again interpreted as a phonon-bottleneck term.

Looking now to the phonon-bottleneck terms, these are compared with theoretical estimates, in order to validate the interpretation. The relation between the phonon-bottlenecked spin relaxation time and phonon lifetime is[25]

$$\tau=\frac{n\pi^2 v_s^3\hbar^3}{6(\Delta\delta)k_B^2T^2}\,\tau_{\text{ph}} ,$$

where n is the concentration of spins, v_s is the velocity of sound, τ_{ph} is the phonon lifetime, and $\Delta\delta$ is the bandwidth of phonons in interaction with the spins. It is often assumed that $\Delta\delta$ is the same as $(\Delta\delta)_s$ the observed paramagnetic-resonance linewidth. However as pointed out by Giordmaine and Nash,[26] for homogeneously broadened lines,

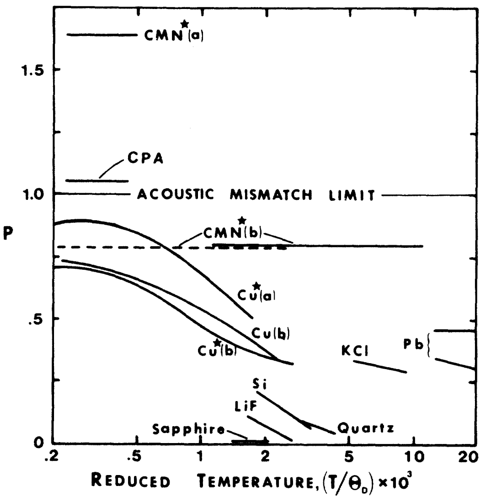

FIG. 7. A plot of $P = (\ln R_{\mathrm{meas}} - \ln R_{\mathrm{PRL}})/(\ln R_{\mathrm{AML}} - \ln R_{\mathrm{PRL}})$ as a function of reduced temperature (T/Θ_D) for a variety of solids. *A star indicates liquid He^3. Otherwise the liquid was He^4. The data sources are as follows: CMN* (a) Ref. 5; CMN* (b) this work; CPA, chrome potassium alum, Ref. 16; Cu* (a) Ref. 13; Cu (b) and Cu* (b) Ref. 11; sapphire, Ref. 17; LiF, Si, KCl, Ref. 18; quartz, Ref. 19; two values of Pb are from Ref. 20 (annealed) and Ref. 21 (ion bombarded) and are to represent metallic solids. The dashed line is discussed in the text.

the lattice spectral width can be appreciably larger than the spin-resonance width. Thus we will write

$$\Delta\delta = \xi(\Delta\delta)_s .$$

Now, the spin-resonance linewidth has been measured by Hudson, Kaeser, and Radford,[27] as 120–130 G, and by Al'tshuler, Valishev, and Khasanov[28] as 390 MHz (or 150 G). An average value of 140 G will be used. Also, the sound velocity in CMN can be taken as 2.12×10^5 cm sec^{-1}.[7] If τ_{ph} is written as $\lambda_{\mathrm{ph}}/v_s$ where λ_{ph} is the mean free path, then

$$\tau = \left(\frac{n\pi^2 v_s^2 \hbar^3}{6(\Delta\delta)_s k_B^2 T^2}\right) \frac{\lambda_{\mathrm{ph}}}{\xi} .$$

From the second terms in Eqs. (3) and (4) values of $\lambda_{\mathrm{ph}}/\xi$ are deduced. These are shown in Fig. 8 plotted as a function of smallest sample dimension. Also shown is the measurement made by Anderson and Robichaux[6] for comparison. First, it should be pointed out that no size effect was observed by Anderson and Robichaux over the thickness range 1–4 mm. However, as can be seen there does seem to be a linear relation between $\lambda_{\mathrm{ph}}/\xi$ and thickness when the value for the least-thick sample measured by Anderson and Robichaux and the two points deduced from the work Bishop *et al.* are compared. There is some doubt as to an acceptable value of λ_{ph}. From thermal-conductivity measurements on a sandblasted single-crystal sample of CMN, a phonon mean free path for elastic or inelastic scattering was found to be equal to the smallest sample dimension.[7] On the other hand, from the interrupted-phonon-avalanche experiment of Mims and Taylor[29] on a sample of CMN cut with a string saw from a single crystal, the mean free path for inelastic scattering was found to be about 10 times the smallest dimension. For the present, since Fig. 8 strongly suggests a size effect, and since the samples surfaces may have been sufficiently damaged for the surfaces to cause inelastic scattering, it will be assumed that the mean free path for the phonons created uniformly throughout the sample is equal to half the smallest dimension. Then, a value of $\xi = 1.0$ is deduced. It is interesting that this value of ξ means that $\Delta\delta$, the half-width of the hot-phonon spectrum is almost identical to the (400 ± 50)-MHz half-width of the hot-longitudinal-phonon spectrum measured by Valishev and Khasanov[30] by a Mandel'shtam-Brillouin scattering technique. Certainly the result that $\xi = 1.0$, the correct temperature dependence and the size effect seem to confirm the fact that the magnetic-pulse-decay data can be interpreted as a phonon-bottleneck resistance in series with the usual thermal-boundary resistance.

However, what of the cerium-spin–He^3-spin coupling? It had been found by Black *et al.*[4] that 100 ppm of He^4 in He^3 was the limiting purity for observing the effect, and the experiments of Bishop *et al.* were performed with 10 ppm He^4 in He^3. However, the important parameter as mentioned before is the mass of He^4. The cell design of Bishop *et al.* was such that considering the cell as a whole, because of the large volume of thermom-

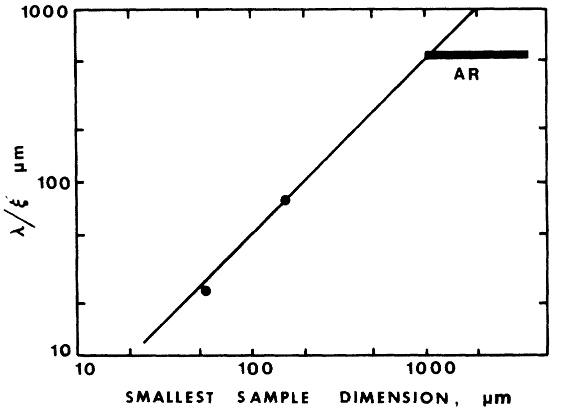

FIG. 8. A plot of reduced phonon mean free path λ/ξ, derived from the phonon-bottleneck equation as a function of minimum sample dimension. Data derived from the work of Bishop *et al.* and Anderson and Robichaux (AR) on relaxation times in CMN. The straight line is the equation $\lambda/\xi = l$, where l is half the minimum sample dimension.

eter powdered CMN, there would have been insufficient He^4 for monolayer coverage (by about a factor 5). However the method of filling was such that the sample and sample cell, with about 2 cm^2 of surface area was exposed to 0.3 cm^3 of He^3 before the He^3 came into contact with the large volume of thermometer powder. A volume of 0.3 cm^3 of He^3 containing 10-ppm He^4 has 50 times the He^4 required for monolayer coverage. Thus, it is doubtful that the experiment of Bishop *et al.* could have shown spin-spin contact.

VI. CONCLUSION

The thermal boundary resistivity between CMN and liquid He^3 has been measured as $55/T^3$ $cm^2 K W^{-1}$ compared to the acoustic mismatch theoretical value of $150/T^3$. It has been shown that this agreement compares favorably with that found for other solids and liquid helium. The agreement extends to relatively high temperatures indicating that the lack of agreement in other cases may be due to properties of the solid rather than the liquid. The difference between the present value for boundary resistivity and that from other work is resolved by reanalyzing the other data in the light of the present value. The present data do not extend to a low-enough temperature to observe the anomalous spin-spin coupling across the boundary.

ACKNOWLEDGMENTS

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