Size effect on the lattice thermal conductivity of lead single crystals

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We have measured the temperature dependence of the thermal conductivity λ of lead single crystals with different sample dimensions in the superconducting state between 0.05 and 1.2 K. Experimental data reveal that apart from boundary scattering of phonons, an additional scattering mechanism, interpreted as scattering of phonons at dislocations, becomes increasingly important with decreasing temperature. By considering both scattering mechanisms, we demonstrate that the boundary scattering length is indeed limited by sample dimension. Additional λ measurements in the intermediate state indicate that an as-yet-unidentified heat transport across the boundaries between normal and superconducting regions may be the reason for the considerable discrepancy between experiment and theoretical calculations.

I. INTRODUCTION

In good conducting normal metals the low-temperature thermal conductivity λ is essentially due to thermal transport and scattering processes involving electrons. If the considered metal is a superconductor, however, the thermal conduction of electrons is increasingly suppressed at temperatures below the superconducting critical temperature T_c and other contributions to λ, e.g., lattice conductivity due to phonons, may become more important. According to various theories[1,2] this phonon-dominated regime is expected at temperatures $T/T_c < 0.15$, where, because of the energy gap in the electronic excitation spectrum of the superconductor, not only the electronic transport contribution has vanished, but also the scattering of phonons by electrons is negligible. In this temperature range, the superconducting metal can therefore be regarded as a thermal insulator and phonon-scattering processes other than phonon-electron scattering may be investigated.

Lead has a superconducting transition temperature of 7.2 K and hence, at temperatures below 1 K, its thermal conductivity is pure lattice conductivity. The first measurements of λ on a Pb single crystal below 1 K by Olsen and Renton[3] yielded an approximate T^3 dependence of the heat conduction in this temperature range. This temperature dependence is theoretically expected for boundary scattering of phonons.[4] However, the phonon mean free path as estimated from the experimental data of Ref. 3 disagreed considerably with theoretical calculations assuming boundary scattering only. This discrepancy could not be removed with subsequent investigations[5–7] and it was conjectured that the influence of subgrain structures and dislocation lines might be the reason for it. In Ref. 7, a quantitative comparison between dislocation line density and mean free path, based on an elastic string model due to Granato,[8] gave satisfactory agreement between theory and experiment. Finally in more recent work[9] on very high-quality single crystals, phonon mean free paths of the order of the sample diameter were reported. Nevertheless, a systematic study of size-limited thermal conduction in the phonon-dominated regime of a superconducting metal still does not exist and it is one of the aims of this work to investigate the size effect on the phonon thermal conductivity in metals, using Pb as an example.

Another size effect may be expected for λ in the intermediate state of a type-I superconductor. If a cylindrical sample of such a material is exposed to an external magnetic field $H > H_c/2$ (H_c is the critical magnetic field) oriented perpendicular to the cylinder axis, a splitting into alternate layers of normal and superconducting regions is observed. Early experimental work of Mendelssohn and Olsen[10,11] gave evidence that this splitting may cause a minimum in λ when H was varied between $H_c/2$ and H_c. Cornish and Olsen[12] argued that this reduction of λ was due to the fact that for a certain strength of the magnetic field, the size of the lamellar structure which depends on the sample diameter D and H (Ref. 13) is comparable to the phonon mean free path. This particular kind of size effect was demonstrated experimentally by Zavaritskii[6] and by Olsen and co-workers.[14] In these experiments, λ showed the same D dependence as theoretical predictions claim for the periodic length of the lamellar structure, but the observed values for λ were much higher than expected from a theoretical model developed by Laredo and Pippard.[15] With these results in mind, we also decided to investigate the thermal conduction in the intermediate state of a type-I superconductor such as Pb.

II. EXPERIMENTAL DETAILS

Lead single crystals were grown from a nominally 99.999% pure melt using Czochralski's method. From the initial crystals of about 6 cm in length and 5 mm in diameter, samples with appropriate dimensions were spark cut using low-power electroerosion. Special care was taken to prevent excess strain on the samples during mounting. They were kept in place with a stainless-steel tube as shown in Fig. 1. The glued connection between stainless-steel tube and sample was removed before closing the cryostat. For good thermal contact to the copper sample holder without clamping the crystal, the specimens were at one end carefully wrapped and glued into copper coil foil[16] over a length of about 10 mm. The thermometers consisted of sliced 100-Ω Matsushita carbon resistors wrapped

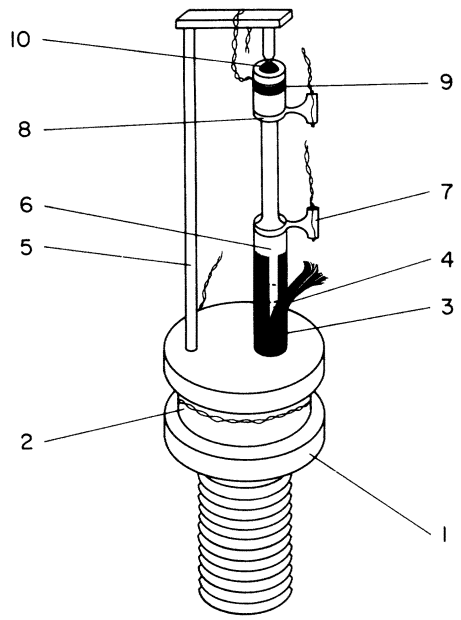

FIG. 1. Experimental arrangement of sample and sample holder: 1 is the Copper holder, 2 is the thermal anchoring of the wires, 3 is the coil foil, 4 is copper, 5 is the stainless-steel holder, 6 is the sample, 7 is the carbon thermometer, 8 is the silver foil, 9 is the heater, and 10 is the G.E. varnish (removed after mounting).

and glued into silver foil. Tails of this silver foil were then glued to the sample as shown again in Fig. 1. The geometric form factor l/F, where l is the distance between thermometers and F is the cross section of the sample, was determined photographically.

The thermal conductivity was determined from the temperature difference between the two thermometers when applying a steady heat current through the sample. Details of the experimental procedure have been published before.[17] After the first experiment with the initial diameter of the sample, its cross section was reduced by an *in situ* etching procedure using a mixture of 25 vol% H_2O_2 and 75 vol% acetic acid. After each etching, the sample was usually annealed for some days at room temperature.

The possible error of the absolute temperature is estimated to be of the order of $\pm 3\%$. Because of the finite areas of the thermometer contacts and some edge effects after etching, the form factor has an accuracy of only about $\pm 15\%$. The external magnetic field perpendicular to the cylinder axis of the samples was applied by means of a saddle-shaped coil which could be rotated around the sample axis. Table I shows the various dimensions of the different samples investigated, together with some parameters which will be discussed in the next section.

III. RESULTS AND DISCUSSION

A. Superconducting state

According to Casimir[4] and the later work of McCurdy and co-workers,[18] the lattice thermal conductivity λ_g in the boundary scattering limited regime can be written as

$$\lambda_g = c_v(\langle v^{-2}\rangle/\langle v^{-3}\rangle)\Lambda_c/3\,, \tag{1}$$

where c_v is the specific heat per unit volume, $\langle v^{-2}\rangle$ and $\langle v^{-3}\rangle$ denote average values of the inverse phonon phase velocities to the second and third power, respectively, and Λ_c is the Casimir length describing the effective phonon mean free path. For a cylinder with uniform cross section, Λ_c is equal to the diameter of the cylinder. In the temperature range considered here, the temperature dependence of λ_g is determined by the T dependence of the specific heat c_v, which in our case is proportional to T^3. Therefore, a plot of λ_g/T^3 vs T should yield a horizontal line. In Fig. 2 we display some of our data in this way. It may be seen, that a T^3 dependence of λ is observed between about 0.6 and 1.2 K. Below 0.6 K strong deviations from this temperature dependence are revealed, indicating that λ varies as T^n, where $n > 3$. From the results between 0.6 and 1.2 K we estimate the Casimir length Λ_c using a Debye temperature $\Theta_D = 105.4$ K (Ref. 19) and $\langle v^{-2}\rangle/\langle v^{-3}\rangle = 970$ m/sec taken from Ref. 20. Inserting these values into Eq. (1) we then obtain

$$\Lambda_c = 3.41\times 10^{-5}\lambda/T^3\,, \tag{2}$$

where λ is given in W/K m. The resulting values for Λ_c are given in Table I. It is quite clear that all values of Λ_c are always smaller than the respective sample diameter and no systematic trend for $\Lambda_c(d)$ is revealed.

The strong deviations from the T^3 dependence of λ below 0.6 K, however, indicate that additional scattering

TABLE I. Parameters used in the analysis of the thermal-conductivity data.

Sample number	Crystal orientation	Sample diameter (mm)	Λ_c (1 K) (mm)	N (m^{-2})	l_b (mm)	l_b^i (mm)	Remarks
13a	⟨100⟩	1.95	1.57	3.1×10^{11}	2.12	1.6×10^{-1}	
13b	⟨100⟩	1.6	0.56	1.45×10^{12}	1.55	1.28×10^{-1}	
13c	⟨100⟩	1.6	1.12	4.25×10^{11}	1.55	1.5×10^{-1}	Sample no. 13b annealed at ~80°C for 3 d
4	⟨100⟩	2.07	1.19	5.2×10^{11}	2.1	1.3×10^{-1}	
4a	⟨100⟩	1.61	0.60	1.35×10^{12}	1.6	1.28×10^{-1}	
4b	⟨100⟩	1.13	0.55	9.5×10^{11}	0.85	0.9×10^{-1}	
4c	⟨100⟩	0.79	0.95	2.8×10^{11}	0.95	0.74×10^{-1}	
5	⟨111⟩	3.29	1.75	3.35×10^{11}	2.85		

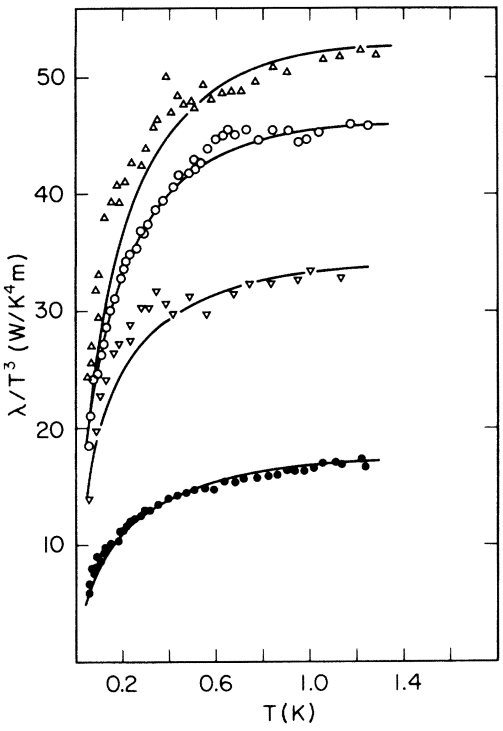

FIG. 2. Temperature dependence of λ/T^3 of Pb single crystals with different diameters in the superconducting state ($H=0$), below 1.2 K. The solid lines are fits considering phonon scattering by boundaries and dislocations.

processes are increasingly effective with decreasing temperature and their influence might also be non-negligible above 0.6 K. Similar temperature variations as shown in Fig. 2 for λ of superconducting Pb were reported for the same temperature range in earlier work of O'Hara and Anderson.[7] They attributed the additional scattering to interactions between phonons and vibrating dislocations, and they interpreted their results using the theoretical model of Granato.[8] Without any fit parameter they obtained more or less qualitative agreement. A more general theoretical treatment of the interaction between phonons and dislocations has been given by Ninomiya.[21] Later Suzuki and Suzuki[22] argued that for real cases, the rather complicated relaxation rate of Ninomiya can be used in a more convenient form considering that most of the scattering occurs far away from resonance of the dislocations. Since, in particular, O'Hara and Anderson did not find any evidence for resonant scattering, we shall discuss our data using the result of Ref. 22.

We first assume that λ may be calculated using the Debye model for the specific heat and a constant phonon velocity. The phonon thermal conductivity is then given by[23]

$$\lambda_g=\frac{k_B}{2\pi^2 v}\left[\frac{k_B}{\hbar}\right]^3 T^3\int_0^{\Theta_D/T}\tau(x)\frac{x^4e^x}{(e^x-1)^2}dx\ , \qquad (3)$$

where $x=\hbar\omega/k_BT$, Θ_D is the Debye temperature, $\tau(x)$ is the effective relaxation time for a phonon of energy $\hbar\omega$, v is the sound velocity, and k_B is the Boltzmann constant. Equation (3) may be generalized if various scattering mechanisms with different τ_i have to be considered. This is done by adding the scattering rates τ_i^{-1} to a total effective scattering rate $\tau_{\rm tot}^{-1}(x)$ (Ref. 14)

$$\tau_{\rm tot}^{-1}=\sum_i\tau_i^{-1}(x)\ . \qquad (4)$$

For the analysis of our results we considered the following scattering times: (i) boundary scattering with $\tau_b(x)\equiv\tau=l_b/v$; (ii) dynamic scattering from dislocations[22] with

$$\tau_d=\frac{xk_BT}{2\pi^2v^2Nh}\left\{\ln\left[\frac{2}{3}\left[\frac{xT}{\Theta_D}\right]^2\right]\right\}^2\ , \qquad (5)$$

where N is the dislocation density; and finally (iii) the static strain-field scattering[25] with

$$\tau_s=\left[N\gamma_G^2b^2\frac{xk_BT}{h}\right]^{-1}\ , \qquad (6)$$

where γ_G is the lattice Grüneisen parameter and b is the absolute value of Burgers vector of the dislocations. In our fitting procedure v was calculated from $\Theta_D=105.4$ K (see above) and b was assumed to be the lattice constant of Pb. We should mention that the scattering described in Eq. (6) is negligible in our case, and is merely mentioned for completion. The remaining parameters l_b and N enter the calculations as fit parameters.

The solid lines in Fig. 2 are fits to the appropriate experimental data and the resulting parameters l_b and N are again listed in Table I. As may be seen from Fig. 2, the temperature dependence of λ is rather well reproduced by considering the above-mentioned scattering mechanisms. The resulting dislocation densities N of about 4×10^{11} m^{-2} for our annealed samples are quite resonable if we recall that an etch-pit count of an annealed lead sample by O'Hara and Anderson gave $N=2\times10^{11}$ m^{-2} with a quoted uncertainty of 100%.[7] In our case, the effect of annealing is particularly evident when comparing the results for samples 13b and 13c (see Table I). λ of sample 13b was measured immediately after etching. After this experiment the sample was annealed *in situ* for about 3 d at about 80 °C and then cooled down again, now denoted as sample 13c. As expected, the fit to the data of sample 13c gives a considerably reduced value for N compared to the value of 13b, leaving, however, the mean free path l_b (due to boundary scattering) unchanged with respect to the value for sample 13b. In Fig. 3 we compare the mean free paths l_b with the respective sample diameters D. It clearly demonstrates the sought size effect on the phonon thermal conductivity in high-quality single crystals of Pb.

According to Winternheimer and McCurdy,[20] an anisotropy of the phonon thermal conductivity resulting from phonon focusing might be expected in oriented Pb single crystals. The calculations of Ref. 20 result in an effective mean free path l_b of the boundary scattering which should be 1.25 times the sample diameter if the temperature gradient is along the [100] direction of the crystal lattice and 0.93 times the sample diameter if $\delta T/\delta x$ is along [111]. These prefactors to D are calculated for cylindrical samples with the respective crystallographic directions along the cylinder axes and sample lengths and cross sections of 30 mm and 3 mm^2, respectively. Unfortunately, the presence of scattering mechanisms other than boundary

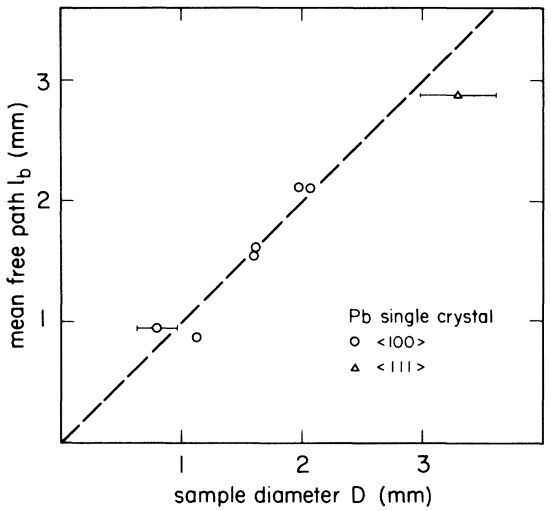

FIG. 3. Boundary scattering mean free path l_b as a function of sample diameter D, calculated from Eq. (3). ○ is the heat current $\vec{Q}$ parallel to the ⟨100⟩ axis, and △ is the $\vec{Q}$ parallel to the ⟨111⟩ axis.

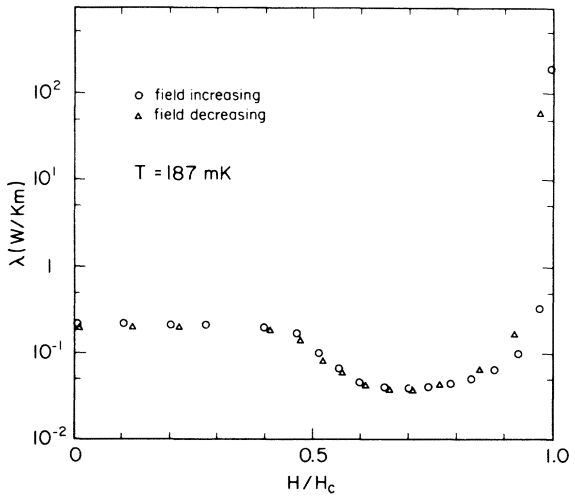

FIG. 4. Magnetic field dependence of λ of a single crystal of Pb at a fixed temperature $T=187$ mK. Note the reversibility of $\lambda(H)$ due to the rotating field method, as described in the text.

scattering reduce this anisotropy. Considering our uncertainty of the geometrical factor, an experimental confirmation of this phonon-focusing effect is therefore not possible from our measurements.

B. Intermediate state

As mentioned in the Introduction, a cylinder of a type-I superconductor exposed to a magnetic field perpendicular to its cylinder axis splits into alternating layers of normal and superconducting regions resulting in appreciable changes of the thermal conductivity. First attempts to measure these changes revealed that they depended on the magnetic history of the sample, i.e., whether the measurements were done in increasing or decreasing magnetic field, respectively. Walton[26] later showed that such effects could be avoided by rotating the magnetic field direction around the cylinder axis, and he argued that this procedure ensured that the normal and superconducting regions extend over the whole cross section of the cylindrical sample. Hence we adopted this procedure for our λ measurements in the intermediate state.

A set of thermal conductivity data in increasing and decreasing magnetic field at a fixed temperature is shown in Fig. 4. As may be seen, irreversibilities indeed can be avoided using the procedure mentioned above. The obviously small amount of frozen flux in decreasing field below $H/H_c=0.5$ can, of course, not be influenced by field rotation. Owing to slight deviations of our sample from being an ideal cylinder (see Fig. 1) the negative change of λ starts slightly below $H_c/2$.

Cornish and Olsen[12] and later also Laredo and Pippard[15] discussed the lattice thermal conductivity in the intermediate state. In the model of Cornish and Olsen, the imperfect thermal relaxation between electrons and phonons leads to a thermal-conductivity minimum in the intermediate state. In the case where the electronic thermal resistivity is negligible, the model of Laredo and Pippard seems to be appropriate. Within the model of Laredo and Pippard, the boundary scattering mean free path l_b has to be replaced by a new scattering length l_b^i of the intermediate state, namely

$$l_b^i=0.75a \tag{7}$$

where a is the periodicity of the normal and superconducting layer structure. This result is valid if $a_n \gg l_g^n$, where a_n is the width of the normal laminae and l_g^n is the phonon mean free path in the normal state. The electronic thermal conductivity in the superconducting state is neglected and that in the normal state is assumed to provide a thermal short circuit between superconducting layers. An estimate of l_g^n at 1 K yields a value of the order of 10^{-4} cm (Ref. 12) whereas the width a_n is at least 10 times larger for a sample of 1.5 mm diameter.[13] Therefore, we use Eq. (7) for the analysis of our experiments.

At constant magnetic field the periodic length a is also a constant in our temperature range. Thus again, the experimental thermal conductivity should be proportional to T^3 if the dominating relaxation rate is given by $\tau_b^{i\,-1}=v/l_b^i$. In Fig. 5 we show an example of this behavior for sample 4b with the special condition that $\lambda(T)=\lambda_{\min}(T)$, together with the corresponding data of λ in the superconducting state. We find indeed a T^3 dependence of λ in the intermediate state although at the lowest temperatures, a slight increase of λ/T^3 is observed. This enhancement of λ/T^3 is a feature of most of our samples.

We analyzed our data in the same way as for the purely superconducting state. The dislocation densities N were now taken from the fits as described in Sec. III A, and the boundary scattering length remained a fit parameter, now denoted as l_b^i. The solid line in Fig. 5 is an example of such a fit to intermediate-state data. The resulting parameters l_b^i for different samples are listed in Table I. We note that the influence of dynamic scattering of phonons [Eq. (5)] has only a tiny effect on the fit, but it enhances the disagreement between theory and experiment at the lowest temperatures (see Fig. 5).

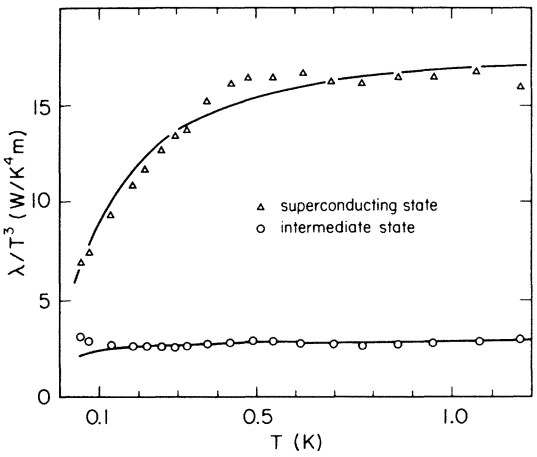

FIG. 5. Temperature dependence of λ/T^3 of a Pb single crystal below 1.2 K in the superconducting and intermediate state. The solid lines are fits including boundary and dislocation scatterings of phonons.

According to Eq. (7) l_b^i is proportional to the periodic length of the lamellar structure. The magnetic-field dependence of a has been discussed by Lifshitz and Sharvin,[13] and is given by

$$a=[\delta D/\phi(h)]^{1/2}\,, \tag{8}$$

where D is the sample diameter, δ denotes the surface-energy parameter related to the surface between superconducting and normal regions, and $\phi(h)$ is a dimensionless parameter tabulated by Lifshitz and Sharvin, and more extensively by Haenssler and Rinderer.[27] h denotes the reduced magnetic field H/H_c. The presently published values of the surface-energy parameter cover a wide range from 20×10^{-8} m to 120×10^{-8} m.[28,29] In Fig. 6 we show the values of l_b^i as they result from the experimental data

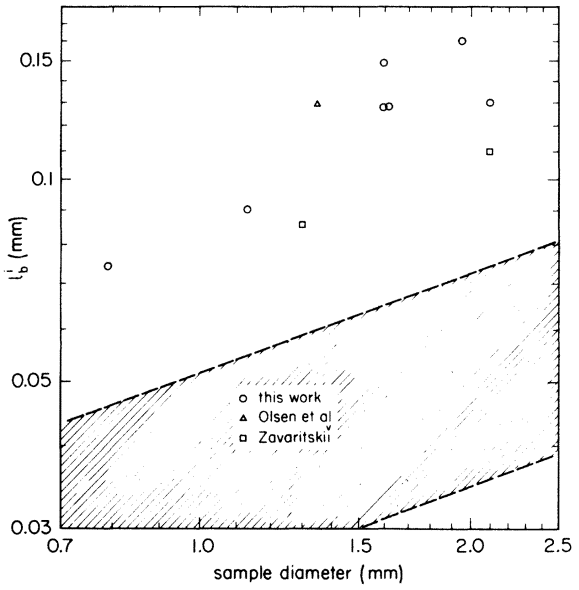

FIG. 6. Mean free path l_b^i in the intermediate-state minimum as calculated from Eq. (3). The dashed lines are the theoretical predictions from Ref. 24. For comparison, previously published data are also included.

together with calculated values using Eqs. (7) and (8). The dashed lines give the upper and lower limit of $l_b^i(D)$ according to the maximum and minimum values of δ. In the selection of experimental l_b^i's we also included some previously published data (see Refs. 6 and 14). The values of Ref. 12 were recalculated using the now established low-temperature Debye temperature $\Theta_D=105$ K, instead of 96 K. It is fairly evident that all experimental values of $l_b^i(D)$ follow the expected $D^{1/2}$ tendency; however, they all considerably exceed the theoretical maximum.

To explain the systematic discrepancy between experimental values from different sources and theoretical expectations, several reasons may be mentioned. The most unlikely possibility is that the lamellar structure includes regions with thermal short circuits. Both the T^3 dependence of λ and the absence of irreversibilities in the field dependence of λ indicate that this is not so. It is also not to be expected that similar deviations of that kind would be observed in three completely different sets of experiments. As mentioned above, a possible electronic contribution to the thermal conductivity across the superconducting regions was neglected. Demers and Griffin[30] calculated the tunneling probability of an electron through a superconducting layer between two normal regions. For an electron at the Fermi surface with an incident wave vector normal to the superconducting lamellae, the transmission probability due to tunneling is approximately given by[30,31]

$$W_{NN}\cong 4\exp(-2Ka_s)\,, \tag{9}$$

where a_s is the thickness of the superconducting layer, which at the intermediate-state minimum of λ is approximately given by $a_s\sim a/2$; Δ is the energy gap parameter of the superconductor and

$$K\cong\frac{\Delta}{v_F\hbar}\,, \tag{10}$$

where v_F is the Fermi velocity. With $v_F=1.82\times10^6$ m/sec,[32] $\Delta=13.7\times10^{-4}$ eV,[32] and $a_s=1\times10^{-5}$ m, we obtain $W_{NN}=4.7\times10^{-10}$, indicating that tunneling of electrons can be neglected. If we consider, however, that specific-heat measurements of van der Hoeven and Keesom[19] were interpreted by assuming an additional second smaller gap of $\Delta=3.4\times10^{-4}$ eV, which would lead to a tunneling probability of $W_{NN}=1.4\times10^{-2}$, then a substantial increase of the thermal conductivity could be imagined, however, it would have to retain a T^3 temperature dependence in order to agree with experiment.

Further evidence for the existence of an as-yet-unidentified contribution to thermal conduction in the intermediate state at temperatures below 1 K was also found by Bonnard and co-workers.[33] Their results of measurements of the thermal resistance of a normal to superconducting interphase boundary in high-purity Pb were interpreted by assuming Andreev scattering.[34] At higher temperatures ($T>2$ K) their experimental data agreed fairly well with calculated values but towards their lowest temperatures $T=1.5$ K an increasing discrepancy is observed, whereby the experimental thermal resistance is about an order of magnitude smaller than predicted by calculation.

IV. SUMMARY AND CONCLUSIONS

We have measured the thermal conductivity of Pb single crystals of various orientations, size, and quality in the superconducting and intermediate state at temperatures below 1 K. In the superconducting state we found clear evidence for the importance of dynamic phonon scattering at dislocations, which below about 0.6 K, dominates the traditional boundary scattering. The temperature dependence of the former term is well described by a theoretical scattering rate due to Ninomiya.[21] Inclusion of this contribution in the interpretation of our experimental data reveals a clear size effect for the phonon boundary scattering, to our knowledge for the first time borne out systematically by experiment in a superconducting metal. Unfortunately, this additional scattering of phonons at dislocations and the uncertainty in our geometrical factors inhibit, from our data, an experimental confirmation of phonon-focusing effects predicted by Winternheimer and McCurdy.[20]

In the intermediate state we find the theoretically predicted $D^{1/2}$ sample-diameter dependence of λ, however, the absolute values exceed theoretical predictions by a factor of about 2. In this case, further theoretical studies on thermal transport across boundaries between superconducting and normal regions might resolve this discrepancy.

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