

Combined Thermal-Conductivity and X-Ray Study of Hexagonal-Close-Packed Helium-4[†]

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Thermal-conductivity measurements made on hexagonal-close-packed helium-4 at 15.2-cm³ molar volume are reported for specimens whose quality and orientation were determined directly by an x-ray method. The highest thermal conductivity in the umklapp region occurred for a single crystal oriented in the basal plane. Specimens of inferior quality exhibited lower conductivities.

I. INTRODUCTION

In the quantum theory of heat conduction formulated by Peierls,^{1,2} the umklapp processes play a fundamental role in limiting the thermal conductivity of a perfect insulator. At temperatures much less than the Debye temperature Θ_0 , Peierls predicted that the conductivity κ of a perfect insulator would vary with absolute temperature T according to the relation

$$\kappa = A(T/\Theta_0)^n e^{\Theta_0/bT}, \quad (1)$$

where A , b , and n depend on details of the material ($b \approx 2$). When the thermal conductivity has the temperature dependence described by Eq. (1), it will be termed the "umklapp conductivity." In imperfect crystals, chemical and isotopic impurities and structural defects may severely limit the thermal conductivity and umklapp conductivity may not be observed.

Although it is not necessary to use a relaxation time approximation to obtain Eq. (1), such an approximation is useful in discussing experimental results.^{2,3} The simplest such approximation is given by the expression

$$\kappa = \frac{1}{3} C v l, \quad (2)$$

where C is the heat capacity per unit volume, v is some average sound velocity, and l is some effective mean free path. An example of the usefulness of Eq. (2) is the Casimir limit.^{2,4} This limit occurs in high-quality crystals at low temperatures when the mean free path becomes limited by scattering from the boundaries of the crystal. In that case, the mean free path becomes constant and the thermal conductivity is described by the equation

$$\kappa = \frac{1}{3} C v \alpha d, \quad (3)$$

where d is the diameter of the specimen and $\alpha \approx 1$ is a geometric factor which depends on the extent

of specular versus diffuse scattering at the surface of the specimen. Because of the temperature dependence of the heat capacity, the conductivity decreases as T^3 in the Casimir limit.

The first conclusive observations of umklapp conductivity in helium were made at Oxford⁵ some twenty years after Peierls's prediction. In experiments on solid helium at several densities, Wilkinson and Wilks⁶ found that the mean free path at different molar volumes, when computed by Eq. (2) using a Debye approximation for the heat capacity and sound velocity, could be described by an equation of the form

$$l = l_0 e^{\Theta_0/BT}, \quad (4)$$

with $l_0 = 6 \times 10^{-8}$ cm and $B = 2.3$. Equation (4) would describe exactly the theoretical temperature dependence if one has $n = 3$ in Eq. (1). However, the exponential factor increases so rapidly that the exact power of n cannot easily be determined from experiments.

At lower temperatures the mean free path observed in the Oxford experiments on helium increased more slowly; at the lowest temperatures the mean free path was an order of magnitude smaller than the specimen diameter. Thus the specimens were probably not single crystals. In other experiments, Crooks and Fairbank⁷ showed that the low-temperature conductivity of specimens solidified by the blocked capillary method used at Oxford could be increased a factor of 10 or more by annealing. This demonstrated that the small mean free paths previously observed at low temperatures were due to poor specimen quality rather than to some effect special to helium. Mezhev-Deglin⁸ was able to obtain a further improvement of specimen quality as deduced from conductivity measurements by using the constant pressure growth method of Shal'nikov.⁹ He was

able to observe Poiseuille flow in these high-quality specimens.¹⁰

The hexagonal-close-packed structure of solid helium allows anisotropy in the thermal conductivity; in such a case, the temperature gradient ∇T and the heat flux Q need not be parallel. In the usual experimental situation where the component of the temperature gradient along the direction of the heat flux is measured, the thermal resistivity is the natural experimental quantity.¹¹ The resistivity measured in this way is related to the two independent components of the resistivity tensor, R_1 and R_3 , by the equation

$$R(\theta) = R_1 \sin^2 \theta + R_3 \cos^2 \theta, \quad (5)$$

where θ is the angle between the heat flux and the c axis of the specimen. Under certain conditions a fine-grained polycrystalline sample will exhibit isotropic thermal resistivity given by the equation

$$R = \frac{1}{3}(2R_1 + R_3). \quad (6)$$

For Eq. (6) to be valid, (a) the thermal resistivity must be truly a bulk property (which is not true in the Casimir limit), (b) the boundaries between the crystalline grains must contribute no additional resistivity, and (c) there must be no preferred orientation of the grains.

Anisotropy of the thermal conductivity was suggested by the experiments of Mezhov-Deglin⁸ and of Bertman and co-workers.¹² The evidence was somewhat ambiguous because the mean free path in the Casimir limit was often small for specimens whose umklapp conductivity was small. Without direct evidence of specimen crystallinity and orientation, there seemed to be no more reason for the low umklapp conductivity to be due to orientation than to poor crystalline quality. Guyer and Hogan¹³ prepared a large number of specimens by constant pressure growth at 85 atm. The quality and monocrystallinity of their specimens may be inferred from the fact that they observed Poiseuille flow in many specimens and that the mean free path in the Casimir limit was comparable to the specimen diameter. They attributed the large variation they observed in umklapp conductivity at 1 K – a factor of 30 for specimens of comparable low-temperature mean free path – entirely to differences in orientation of their samples. They analyzed their data by assuming that Eq. (5) could be applied to the umklapp conductivity of samples grown by the constant pressure method of Mezhov-Deglin and that Eq. (6) could be applied for samples grown by the blocked capillary method. They concluded that the umklapp conductivity was greatest for heat currents in the basal plane and smallest along the c axis. In their experiments no direct determina-

tion of orientation was made to verify the results of the thermal-conductivity measurements.

In earlier experiments at this laboratory,¹⁴ we investigated the thermal conductivity of specimens grown by the constant pressure method of Mezhov-Deglin at higher densities than the work of Mezhov-Deglin and Guyer and Hogan. The mean free path of the highest conductivity samples could be described in the same form as the Oxford experiments [Eq. (2)] with $l_0 = 2.5 \times 10^{-8}$ cm and $B = 2.1$. The smaller value of B indicates that the conductivity increased faster as the temperature was lowered. This effect could be due to improved specimen quality or to anisotropy. Although variations in the umklapp conductivity were observed, they were much smaller than those reported by Guyer and Hogan at a lower density. In the present study, we have used an x-ray method to evaluate *directly* the crystalline quality and orientation of samples for which the thermal conductivity could also be measured. The results reported here are confined to samples grown at a constant pressure of 290 atm.

II. EXPERIMENTAL PROCEDURE

A metal cryostat with Mylar windows to pass x rays was used. The x-ray apparatus, located outside the cryostat, is described elsewhere.¹⁵ The sample tube assembly, described below, is attached to the exchange gas helium pot portion of the cryostat shown in Fig. 1. For solidification and annealing of samples ($T \approx 6.5$ K), the helium pot (8) is evacuated. A small amount of helium exchange gas is admitted through the exchange gas line (1) to the space confined by the metal bellows (7). This gas establishes thermal contact between the two copper exchange gas cylinders (5). The upper cylinder is hard-soldered to the brass bottom plate (4) of the helium reservoir; the lower cylinder is brazed to the helium pot (8). The temperature of the helium pot is determined from a calibrated germanium resistor attached at the copper post (9). The temperature control system used in our earlier experiments¹⁴ regulates the power to a heater on the helium pot to maintain the desired temperature. Before thermal-conductivity measurements the helium pot (40 cm³ volume) is filled by condensing a known amount of helium gas through the pot fill capillary (3). About 10^{-2} Torr exchange gas pressure is used during filling to minimize fluctuations in temperature at the pot. After filling, this exchange gas is removed and the liquid helium in the pot is evaporated through the support tube (6) and pump line (2). A small constriction (1 mm) at the pot enables the temperature to be maintained as low as

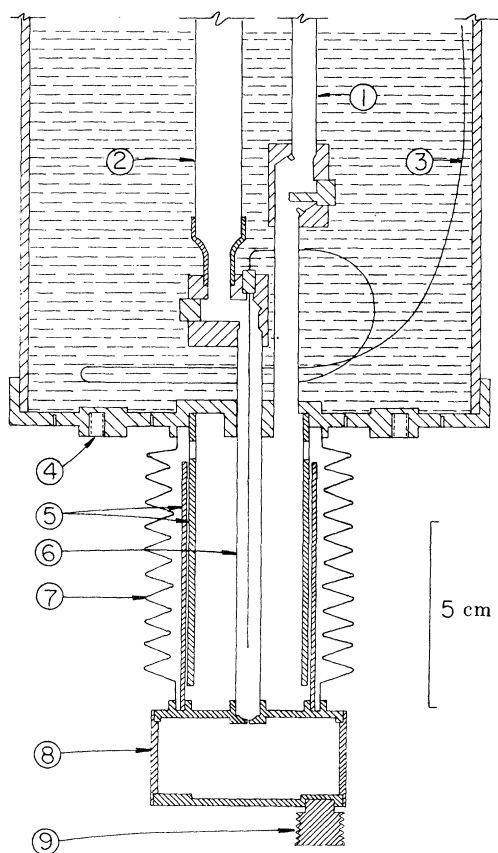


FIG. 1. Exchange gas helium pot portion of the metal cryostat. (1) Exchange gas line, (2) pot pump line, (3) pot fill line, (4) bolt holes for radiation shield, (5) exchange gas cylinders (copper), (6) pump line and support tube (stainless steel), (7) bellows to isolate exchange gas from high vacuum space (stainless steel), (8) helium pot (copper) with 1000- Ω heater, (9) threaded copper post for attaching sample tube assembly.

0.97 K with a mechanical vacuum pump. The amount of liquid remaining in the pot is determined by metering the gas pumped away.¹⁶ Thus sudden changes of temperature that may occur when the helium pot is emptied can be avoided.

The sample tube assembly is shown in Fig. 2. The sample tube (4) is machined from 1.2 cm o. d. extruded nylon rod.¹⁷ Nylon is used because of its low thermal conductivity and x-ray scattering and absorption. X-ray scattering and absorption are minimized by removing most of the nylon in the x-ray path. The x-ray beam passes through about 2 mm of nylon. The cylindrical specimen cavity is 2.2 mm i. d. and 23 mm long. Metal-to-nylon pressure seals are made by deformation of the nylon when the assembly is tightened at room temperature. The copper plugs [(3) and (7)] and the ends of the nylon tube have matching $\frac{1}{4}$ -28

threads; the brass screws [(5) and (6)] and the side holes in the nylon tube have matching 0-80 threads. A thin layer of indium is used on the sealing surface of the brass screws; no indium layer was necessary for the copper plugs. Sample gas is re-purified and pressurized by the same system used in the previous experiments¹⁴; it enters the specimen cavity through the capillary (10) soft-soldered into the lower copper plug (7). A heater (8) located on this plug is used during growth to prevent the capillary from freezing before the specimen cavity is filled with solid. This heater is also used to supply a known amount of power for thermal-conductivity measurements. Germanium resistance thermometers are mechanically mounted in two copper blocks [(2) and (9)]. Carbon resistance thermometers are attached to the brass screws [(5) and (6)] with low melting point solder. The rounded ends of these screws touch the solid sample but do not protrude into it. The portion of sample (10 mm long) between the screws is used for conductivity measurements. Heat is extracted during sample growth and thermal-conductivity measurements through the pointed copper plug (3) attached to the copper post of the helium pot with a brass nut (1).

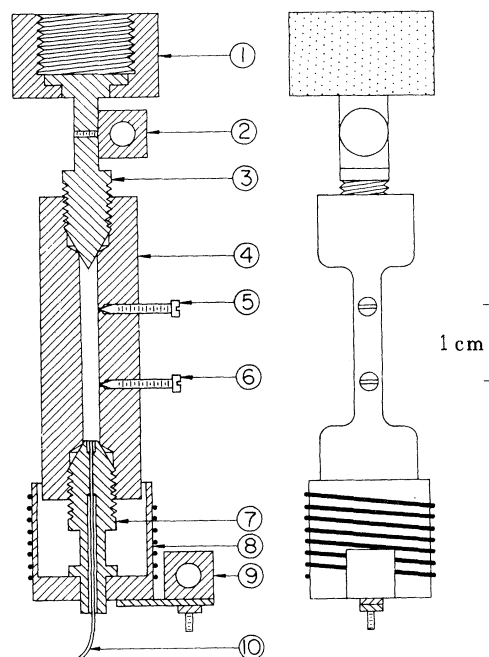


FIG. 2. Nylon sample tube assembly. (1) Knurled brass nut, (2) holder for upper germanium thermometer, (3) copper plug for upper end, (4) nylon sample tube, (5) and (6) brass 0-80 screws, (7) copper plug for lower end, (8) sample heater assembly, (9) holder for lower germanium thermometer, (10) inconel pressure capillary.

At the start of an experimental run the sample tube is flushed several times with helium gas at 100 atm and evacuated. After the sample tube assembly is cooled to 77 K, it is slowly pressurized to 290 atm with repurified helium gas. A helium mass-spectrometer leak detector is used to determine if the assembly is free of leaks. During transfer of liquid helium to the helium reservoir, the sample tube is maintained above 7 K. All the samples reported here were solidified by lowering the pot temperature at various rates determined by a motor-driven resistor in the temperature control circuit.¹⁴ The rate at which this temperature is lowered and the amount of power into the heater on the lower end of the sample tube determine the growth rate. Under certain conditions simple calculations can be used to predict the growth rate. The actual rate can usually be determined from chart records of the temperatures at the germanium and carbon resistors. After solidification is completed, the heater power is slowly reduced and the sample cooled to the temperature at which thermal-conductivity measurements are started. Temperatures are changed slowly during the measurements as a precaution against thermal shock. (Conductivity measurements were made by the same procedures as before.¹⁴)

For annealing of samples, the pot is usually warmed up rapidly from 4.2 K to a temperature near the melting point at the pressure of solidification. The lower end of the sample warms up slowly. Continuous monitoring of the temperature usually indicates when the pressurization capillary is free of solid. A slight change in pressure at the pressurization system can also be used to determine if the capillary is open. After x-ray pictures indicate that structural changes have stopped, the pot temperature is lowered slowly.

III. RESULTS

In Table I are presented the characteristics of six samples of hcp helium-4 at 15.2 cm³ molar volume. This table indicates the growth rates for four samples grown by unidirectional solidification from the fluid at a constant pressure of 290 atm. Sample 9-1 was grown from a seed crystal of about 1 mm of solid which formed immediately after nucleation; the seed was annealed for about 1 h. Sample 9-2 was grown from a seed crystal of about 3 mm of sample 9-1; the rest of sample 9-1 had been melted away. Samples 9-4 and 9-7 were grown without any pause after nucleation of the solid. Sample 9-5 was obtained by annealing sample 9-4. Sample 9-6 was obtained by annealing a specimen grown rapidly at 3 cm/h without any

pause. Considerable recrystallization occurred during these anneals. The same annealing procedure produced no change in x-ray photographs when carried out on the slowly grown sample 9-7 after conductivity measurements.

A short description of the x-ray evaluation of the 10-mm sample length used for thermal-conductivity measurements is also given in Table I. Because of the poor structural quality, it was not possible to determine the orientations of any grains in samples 9-1, 9-2, and 9-7. However, where only one large grain was present in the x-ray beam, its orientation could be determined. Where orientations are given, θ is the angle between the specimen tube and the c axis of the grain. The most important orientation determined is that of sample 9-6. In that case, practically all of the sample used for conductivity measurements was a single crystal. Figure 3 is a drawing made from a typical transmission Laue photograph of sample 9-6. The shape of the x-ray reflections indicates that the sample is not strained. Careful examination of the photographs revealed two subgrains separated by about 0.5°. A change in orientation occurred at the upper brass screw.

Figures 4 and 5 present the thermal conductivity of these samples as a function of temperature. Figure 5 includes for comparison the highest conductivity observed at 290 atm in our earlier study.¹⁴ Because the umklapp conductivity of sample 9-2 was the lowest observed, it is shown in both figures. The molar volume (15.2 cm³) was determined from the growth pressure using the results of Dugdale and Simon.¹⁸ The Debye temperature (58 K) was determined from the molar volume using the results of Franck¹⁹ and Ahlers.²⁰ The mean free path at 1.1 K was calculated from the experimental data using Eq. (2) and a Debye

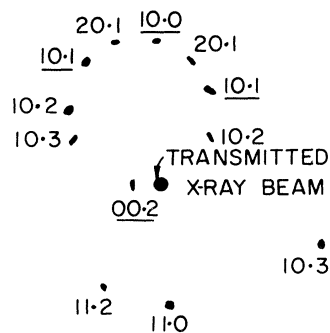


FIG. 3. Drawing made from typical transmission Laue photograph from portion of sample 9-6 used for conductivity measurements shown in Fig. 5. The underlined reflections were more intense than the others observed, as expected from the Debye-Waller factor. The spot shapes show no evidence of strain.

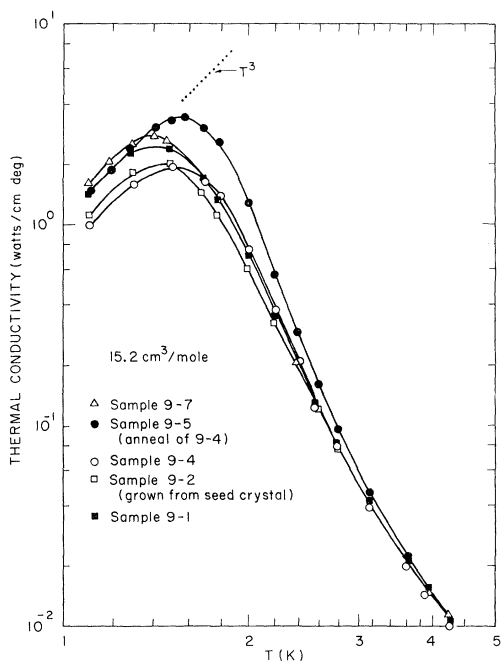


FIG. 4. Thermal-conductivity results for samples 9-1, 9-2, 9-4, 9-5, and 9-7.

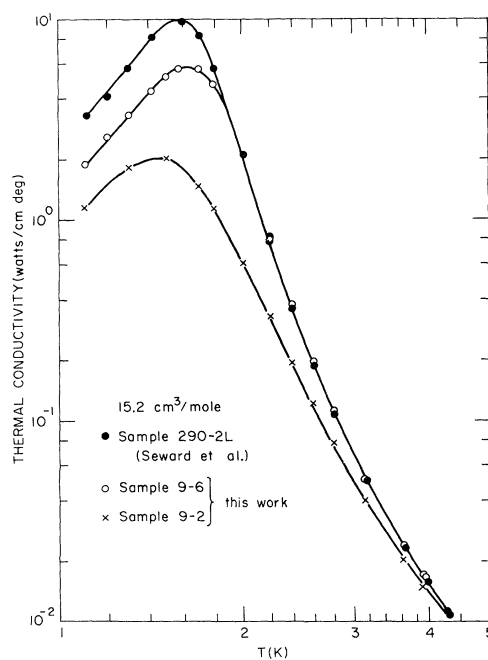


FIG. 5. Thermal-conductivity results from samples 9-2 and 9-6 compared with results of earlier study (Ref. 14) on samples of the same diameter (2.2 mm).

approximation for the heat capacity and sound velocity; the results are given in Table I. The absolute value of the mean free path has limited significance because of the uncertainty concerning the proper sound velocity to use in Eq. (2); no experimental sound velocity data are available at 15.2 cm³ molar volume. The thermal conductivity at 2.0 K is also given in Table I.

Because of the limited amount of data at this

molar volume, the thermal conductivity of the best specimen, sample 9-6, is compared in Fig. 6 with results at other densities. A plot of $\log \kappa$ versus Θ_0/T is used for comparison with Peierls's prediction [Eq. (1)]. One advantage of this type of plot is that no assumption is necessary regarding the variation of sound velocity with molar volume. The straight line shown for sample 9-6 is given by Eq. (1) with $n=0$, $A=1.1 \times 10^{-4}$ W cm⁻¹ K⁻¹, and

TABLE I. Six samples of hcp helium-4 at 15.2 cm³ molar volume.

Sample	Growth rate (cm/h) ^a	X-ray evaluation of 10 mm length used for conductivity measurements	κ at 2.0 K (W cm ⁻¹ K ⁻¹)	l at 1.1 K (mm)
9-1	0.6	At least three orientations; lineage structure (small angle grain boundaries)	0.69	0.85
9-2	1.2	At least two orientations; severe lineage poorest structural quality	0.61	0.69
9-4	6.0	Large grain about 4 mm long ($\theta=60^\circ$); otherwise poor quality	0.76	0.60
9-5	...	Three large grains ($\theta=?$, 60° , 90°)	1.3	0.88
9-6	...	Large single crystal 9.5 mm long; high quality (see Fig. 3), $\theta=88^\circ \pm 2^\circ$	2.1	1.14
9-7	0.3	At least three orientations; lineage	0.70	0.97

^aSee Sec. III of text.

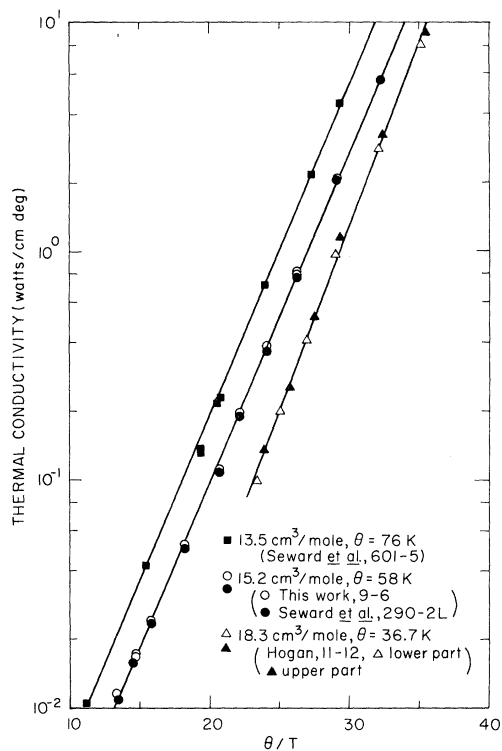


FIG. 6. Thermal-conductivity results for sample 9-6 compared with results of studies without x rays (Ref. 13 and 14).

$b = 3.0$. The conductivity data for the other samples in this work could be described equally well for $\theta_0/T < 30$ by Eq. (1) with $n=0$. The lowest observed umklapp conductivity (sample 9-2) is described in this manner by $A = 3.3 \times 10^{-4} \text{ W cm}^{-1} \text{ K}^{-1}$ and $b = 3.8$. The data at 13.5 cm^3 molar volume are for the best of seven samples grown from re-purified gas in our earlier work; the data at 18.3 cm^3 are for the best of 27 samples grown by Hogan. Neither of these prior studies provided an independent indicator of sample quality or orientation.

IV. DISCUSSION

The most important feature of the results is that the thermal conductivity of sample 9-6 is significantly higher than that of all other samples measured in the nylon tube. This sample was the best crystal obtained and was oriented essentially in the basal plane ($\theta = 88^\circ \pm 2^\circ$). The conductivities of sample 290-2L (see Fig. 5) and of sample 9-6 agree within experimental uncertainties at temperatures above 2 K. The difference below 2 K is probably due to the large-angle grain boundary present in sample 9-6 near the upper temperature probe. If the umklapp conductivity is dependent only on crystalline orientation, as Guyer and Hogan

have assumed, then the data indicate that a sample oriented in the basal plane was also obtained in the inconel sample tube (sample 290-2L). If there are no effects on the umklapp conductivity due to crystalline size or quality, then observation of umklapp conductivities lower than that of sample 9-6 (such as sample 9-2) is sufficient to show that the basal plane is the high conductivity direction. This is in accord with the conclusions of Guyer and Hogan. However, the present results are not conclusive in this regard, since crystals of poorer quality (e.g., sample 9-2) also show lower umklapp conductivity.

Because of the strong correlation between the specimen quality determined by x rays and the umklapp conductivity, more investigations of single crystals of known orientation would be useful. It is possible that the correlation is due to a preference for high-quality crystals to grow with the c axis perpendicular to the growth direction. The inability to obtain more than one single crystal of sufficient size for meaningful thermal-conductivity measurements was a definite handicap in the present investigation. The occurrence of lineage structure, bicrystals, and other defects in some specimens shows that a fitting process of the type used by Guyer and Hogan would determine only an "apparent" angle. In the case of a bicrystal, such an angle might have little relation to the orientations of the two parts.

Even if the assumption of Guyer and Hogan is correct that the two extreme observed resistivities at 18.3 cm^3 molar volume represent the tensor components R_1 and R_3 in Eq. (5), there is little reason to believe that Eq. (6) can be applied to samples grown by the blocked-capillary technique. Earlier x ray²¹ and neutron²² investigations have shown that this technique produces several large crystals rather than a fine-grained polycrystal with no preferred orientation. Thus the identification of the basal plane as the high conductivity direction should be considered tentative. Although the present identification does not require any assumption about samples produced by the blocked-capillary technique, it was necessary to assume that sample quality does not affect umklapp thermal conductivity.

The results of this experiment in the low-temperature region show that the mean free path at 1.1 K (see Table I) is lower for the samples grown rapidly from the fluid without annealing than for those grown slowly. However, it is hard to correlate the degree of lineage seen in the Laue photographs with this mean free path. Much more extensive measurements would be also useful in this regime.

The strong effect of annealing a rapidly grown

sample can be seen from Fig. 4 by comparing the thermal conductivity of samples 9-4 and 9-5. Because annealing seemed to give no improvement in slowly grown specimens, it appears that a small amount of lineage may be a thermally stable configuration. The screws in the side of the tube are believed to be the primary source of difficulty in obtaining large single crystals. Mezhov-Deglin has reported similar problems due to the insertion of platinum wires in glass sample tubes.²³

The comparison of conductivities at different molar volumes shown in Fig. 6 indicates that the observed dependence on reduced temperature is not the same at 13.5 and 15.2 cm³ as at 18.5 cm³. This difference could be due to different gas purity, sample orientation, or crystal quality. Since only the measurements on sample 9-6 were carried out on a sample of known orientation, it is difficult to compare the measurements quantitatively. Although the measurements of Webb and Wilks⁶ showed no change in dependence on reduced temperature in this molar volume range, their measurements were on specimens whose quality was poor. Therefore, their results are not conclusive.

Theoretical mechanisms are available, even in the absence of anisotropy, that will produce changes in the dependence of conductivity on reduced temperature. The exact values of A , n , and b in Eq. (1) depend very sensitively on the geome-

try of the frequency surfaces and the energy-conservation loci of the phonon spectrum.² Thus one would expect a volume dependence of n and b if the phonon spectrum does not scale exactly with the Debye temperature Θ_0 . Because of the possibility of a change in b with molar volume, more experiments would be useful on the volume dependence of thermal conductivity.

The absence of any theoretical calculations of thermal conductivity in the umklapp regime for an hcp material such as helium is unfortunate. It is clearly a formidable task to formulate an accurate theory of equilibrium properties from first principles.²⁴ A phenomenological theory which expresses the umklapp thermal conductivity for an hcp insulator in terms of the Debye temperature and Gruneisen parameters would be a vast improvement over the present situation.

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PHYSICAL REVIEW A

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Frequency Cusp, a Means for Discriminating between Convective and Nonconvective Instability

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The colliding-pole criterion for determining instability types from dispersion relations calls for a process of conformal mapping from the frequency plane into the wave-number plane. It is shown that this process can be inverted by mapping lines, parallel to the imaginary axis, from the wave-number plane into the frequency plane. Lines mapping into a cusp locate the frequencies at which the group velocity vanishes. These frequencies determine the instability character of the system.

I. INTRODUCTION

It was first recognized by Sturrock¹ that the stability character of wavelike perturbations $\propto e^{i\omega t - ikx}$ in linear media is determined by the topology of conformal mappings via the dispersion relation $D(\omega, k) = 0$. More precisely, it was found by the author² that nonconvective instabilities are due to a *selected* class of double roots, $k_n(\bar{\omega}) = k_m(\bar{\omega})$, with frequencies in a lower-half plane, $\text{Im}\bar{\omega} < 0$. The *selection rule* stated in the original article² requires that the roots $k_n(\omega)$ and $k_m(\omega)$ merge, in a process of analytic continuation toward real frequencies from below, across the real k axis. Subsequent works by Briggs,³ Hall and Heckrotte,⁴ and others differ in essence only in the methods of implementing the above selection rule. For example, Briggs³ determines the relevant saddle points $k_n(\bar{\omega}) = k_m(\bar{\omega})$ by mapping a sequence of lines parallel to the imaginary ω axis, from the ω plane [Fig. 1(a)] into the k plane [Fig. 1(b)]. A sequence of lines parallel to the real ω axis, i. e., Laplace integral paths, gives the same result.²

In an earlier attempt to implement the selection rule, the author² and, independently, Fainberg *et al.*⁵ mapped the real k axis into the frequency plane. We thereby found it necessary for nonconvective instability to occur, that the resulting contour loops around a branch point in the lower frequency plane, as shown in Fig. 2. This is because the branch point $\bar{\omega}$ then is accessible from the

lower frequency plane only by crossing the map of the real k axis, as required by the selection rule. The same result was later obtained by Baldwin and Rowlands,⁶ while Callen and McCune⁷ checked the presence of the branch point within the Fainberg contour by mapping the section A-C-B of the real ω axis [Fig. 2(b)] into the k plane [Fig. 1(b)]. Nonconvective instability arises only if the resulting contour is open, i. e., of the type γ in Fig. 1(b).

It has been pointed out by Briggs,³ that the Fainberg criterion for nonconvective instability, though necessary, is not sufficient in some cases. Rather more serious is the fact that all of the results discussed so far are strictly valid only when

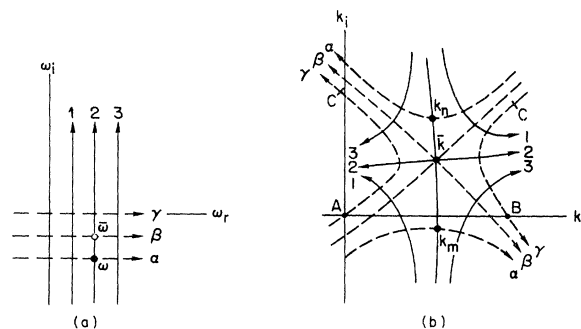


FIG. 1. Colliding pole criterion: broken line indicates Laplace integral paths mapped from ω plane; (a) into k plane, (b) after Derfler (see Ref. 2). Solid line indicates ordinate mapped from ω plane; (a) into k plane, (b) after Briggs (see Ref. 3).