MASTER

ULTRASONIC ATTENUATION STUDIES OF THE ELECTRONIC
STRUCTURE OF METALS

Progress Report

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June 1, 1972 - May 31, 1973

Prepared for the U.S. Atomic Energy Commission under Contract No.

AT(11-1)-3331.

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Abstract:

The experimental measurements of ultrasonic attenuation and magnetization in the mixed state of niobium, in progress when last year's renewal proposal was submitted, have now been completed. Preliminary analysis of the data was presented at the Thirteenth International Conference on Low Temperature Physics in August 1972. A more thorough analysis is nearly ready for publication.

Measurements of beryllium's thermal conductivity as a function of temperature and magnetic field were begun during the current renewal period, and are still in progress. Unexpected experimental problems arose which are still not fully resolved, but good progress has been made towards obtaining the desired thermal conductivity information.

Previously suggested acoustic nuclear magnetic resonance studies in high-purity single crystal silver were discontinued after feasibility studies indicated that magnetic field homogeneities required for the experiment were not available.

Other measurements in progress, but in a relatively early stage, include velocity of sound variations at T_c in aluminum, and magnetoacoustic studies in beryllium.

PROGRESS REPORT:

a. niobium measurements:

The experimental measurements of ultrasonic attenuation and magnetization in the mixed state of niobium, in progress when last year's renewal proposal was submitted, have now been completed. These measurements were performed at

1.4K and 4.2K in two samples whose residual resistivity ratios (RRR) were 68 and 2600, as determined by Dr. Richard Reed of Oak Ridge National Laboratory at the conclusion of the experiments. The attenuation was measured for several configurations of magnetic field and ultrasonic propagation and polarization directions over as wide a range of ultrasonic frequency as was experimentally accessible. The extent of the measurements is presented in Table I, where the abbreviations L and T refer to longitudinal and transverse sound waves, respectively, || refers to magnetic field parallel to propagation direction, and $\perp$ refers to magnetic field perpendicular to both propagation and polarization directions. Magnetization was measured for the parallel geometry at both temperatures in both samples.

	Nb#3(RRR = 68)		Nb#4(RRR = 2600)	
	1.4K	4.2K	1.4K	4.2K
L	15-405	15-435	10-90	10-110
L $\perp$	15-405	15-405	10-90	10-110
T	10-210	10-250	10-50	10-50
T $\perp$	10-250	10-250	10-30	10-30

TABLE I

Frequency Ranges (in MHz) of Ultrasonic Attenuation Studies in Niobium

Preliminary analysis of the data was presented at the Thirteen International Conference on Low Temperature Physics in August 1972, in Boulder, Colorado. A preprint of the article to appear in the proceedings of this conference is included with this report as ATTACHED MANUSCRIPT A. A more thorough analysis of the data is nearly ready for publication. This includes a quantitative

detached

comparison with the theory of Houghton and Maki¹, as well as a more complete comparison with previous work. This analysis reveals that the agreement of the Nb#4 data with the theory of Houghton and Maki is even better than was originally appreciated.

Attempts to investigate spatial matching of ultrasonic wavelength and flux line lattice spacing using both pulse-echo and continuous wave techniques gave negative results. Also, the ultrasonic attenuation of transverse waves in the mixed state is not significantly different qualitatively or quantitatively from that of longitudinal waves, and it is our conclusion that collective excitation effects suggested by Caroli and Maki² do not significantly affect the ultrasonic attenuation.

Note: a Ph.D. thesis based mostly on these niobium measurements is currently in preparation by M. K. Purvis. A talk, based on these measurements, will be presented to the Department of Physics at Clark University (Worcester, Mass.) on March 6, 1973 by R. A. Johnson.

b. beryllium measurements (thermal conductivity):

As an outgrowth of studies on quantum oscillation waveforms in beryllium (see ATTACHED MANUSCRIPT B),³ it was discovered that measurements of beryllium's thermal conductivity as a function of temperature and magnetic field had not been performed in single crystal samples of high purity. Such measurements were undertaken during the current renewal period, and are still in progress. Unexpected experimental problems arose which are still not fully resolved, but good progress has been made towards obtaining the desired thermal conductivity information.

During summer 1972 a sample chamber was designed and constructed to allow simultaneous determination of thermal conductivity for two cylindrically-shaped beryllium samples of different resistivity ratio.⁴ The sample chamber was designed to be compatible with a glass dewar system associated with a conventional iron magnet as well as a superconducting magnet capable of supplying a maximum field of 65,000 Gauss.

The basic experimental arrangement is shown in Figure 1.⁵ Both samples are mounted in the vacuum can sample chamber on an isothermal platform whose temperature can be carefully controlled and measured. A heat input ($\dot{Q}$), of a few milliwatts nominally, is introduced via a heater at one end of each sample. Temperature is then measured at two points along each sample by previously calibrated carbon thermometers. Calibration is against a Cryocal model CR-1000 secondary standard.⁶ Resistances are measured via an ac Wheatstone bridge incorporating phase sensitive detection.

Initial zero-field, measurements were performed in the glass dewar system in late August. At first, the slopes of resistance versus temperature (R vs T) curves for the carbon thermometers were extracted by hand, and used to calculate the small temperature changes arising when $\dot{Q}$ was applied and a temperature gradient established. This proved tedious and unreliable, considering the small differences in temperatures we were attempting to measure (typically, several mK). As a result, some time was spent developing a computer program to fit the calibration data to Clement and Quinell's⁷ expression for the R - T behavior of low-temperature carbon thermometers. This procedure proved highly satisfactory; with the program, temperature gradients of a few mK in

the 1-10K region, and a few 10's of mK in the 10-20K region can be easily resolved. Overall accuracy of the thermal conductivity values is mainly limited by how accurately the sample dimensions, thermometer separation, and $\dot{Q}$ can be determined, and is estimated to be 4-5%. The relative accuracy of the results (e.g., the ratio of thermal conductivities at two different temperatures) is estimated to be of the order of 1%, based on calculations and experimentally observable scatter.

During the August experiments some smooth but abnormal behavior was observed for the calculated thermal conductivity values for both Be samples in the 13-18K region. Two well-defined "bumps" (see Fig. 2) were observed at the same temperatures in both samples, whereas above and below the 13-18K region, smooth behavior was observed. These bumps were seen in both the original hand and subsequent computer calculations. Because of the unexpected appearance of these anomalies, and the apparent low noise and excellent resolution of the rest of the measurements, considerable time and effort was put into testing the equipment and checking analysis procedures for possible errors. No apparent explanation could be found, and the entire external apparatus (ac bridge, current supplies, monitoring apparatus) was torn down, inspected, and rebuilt with careful attention to electrical shielding and grounding. Also, the experiment was shifted to the superconducting magnet, both to improve shielding (via the metal dewar system), and in preparation for field measurements.

When unusual behavior again appeared in the 13-17K region, it was decided to substitute a dummy copper sample as a control in place of one of the beryllium samples, but keeping the original thermometers. Since OFHC copper

conductivities are well known, this was a direct check on the apparatus and the analysis procedure. The thermal conductivity measured in copper was in excellent agreement with previously published values.⁸

With this reassurance, thermal conductivity for Be #1 has been remeasured in the 1.1 - 20K region. By allowing relatively long (≥ 20 minute) "settling-in" periods after each temperature change, data has been obtained which leads to the smooth thermal conductivity variation shown in Figure 3. Such long settling-in times are not associated with the natural time constant of the isothermal plane, as evidenced by the fact that they are not necessary when measurements are performed on the copper sample.

A further reliability check is routinely performed. This consists of measuring thermal conductivity for several values of $\dot{Q}$, with the sample temperature held constant. Within expected experimental error, the resultant thermal conductivity values remain constant for heat inputs that establish temperature differences from 7 mK to 300 mK along the sample.

With regard to the above-mentioned experimental difficulties associated with Be, for which we offer no explanation at this time, and which do not occur each and every time, we note that anomalous low-temperature results have been obtained in Be before. A 1966 article by Ahlers⁹ mentions that "The low-temperature heat capacity of beryllium was first measured by Cristescu and Simon¹⁰ on a sample of 99% purity at temperatures above 10°K. They observed an anomaly at 11°K which to them appeared similar to the anomalies which had been found in the heat capacities of superconductors."

However, a 1953 study by Hill and Smith¹¹ did not observe the anomaly...." Also, a 1971 article by Alekseevskii and Tsebro¹² contains an interesting reference to unusual behavior in beryllium: "Lazarev, Semenenko, and Sudovtsov⁸ discovered a superconducting modification of beryllium with a critical temperature of ~8K, which is formed in the condensation of the metal on a surface cooled to liquid-helium temperatures. When such films are warmed to temperatures above 30 K a transition from the superconducting to the ordinary modification is observed. This transition is also well manifested in the curves for the temperature dependence of the electrical resistance."

Finally, the initial measurements indicated higher thermal conductivity values for the less pure sample (Be#2) than for the purer one (Be#1).¹³ This effect will be carefully investigated in future measurements.

c. acoustic nuclear magnetic resonance studies:

Last year's renewal proposal contained a suggestion that the previously-developed cw ultrasonic spectrometer¹⁴ might be used to detect acoustic NMR in a bulk, single crystal, silver sample at high magnetic fields. Considerable effort was expended in considering the feasibility of such an experiment, and a collaborative effort was discussed with Professor George Seidel at Brown University. However, it was finally concluded that field homogeneities available at either facility were inadequate for the experiment, and further efforts were discontinued.

d. velocity of sound variations at T_c in type-I superconductors:

Work was begun using an available, although relatively dirty single crystal of aluminum, but is still in a relatively early stage. It was found

that the cryogenic system available for this experiment required modification if temperatures below the transition temperature of aluminum (1.19K) were to be attained. It was also appreciated that effort must be expended to cancel effects due to the earth's magnetic field. These two steps remain to be done.

e. beryllium measurements (magnetoacoustics):

Experiments were started to measure caliper dimensions of the Fermi surface of beryllium via magnetoacoustics. Considerable difficulty in bonding quartz transducers to the sample end faces has been experienced. After much trial and error, it was found that reasonable bonds could be obtained using pentene as the bonding fluid, and preparing the bonds at close to liquid nitrogen temperature in a specially designed bonding apparatus.¹⁵ These measurements have been temporarily delayed due to other research activities, but will be continued.

Personnel:

Two graduate students, one post-doctoral research associate, and the author of this progress report (principal investigator) have been involved in the research program during the current renewal period. One graduate student has completed the experimental portion of his Ph.D. work and is preparing his thesis, while the other is still engaged in the experimental portion of his thesis work. The author of this report devoted full time to the research effort during summer 1972, and is devoting 30% of his time during the 1972-73 academic year when he teaches 2 courses per semester.

References and Footnotes

1. A Houghton and K. Maki, Phys. Rev. B4, 843 (1971).
2. C. Caroli and K. Maki, Phys. Rev. 159, 316 (1967).
3. A. R. Hoffman, W. E. Nelson, P. Elterman, and K. D. Swartz, Phys. Letters 41A, 233 (1972).
4. a) Be #1: RRR $\approx$ 2000 (0.084" dia x 1" long; obtained from Oak Ridge National Laboratory
b) Be #2: RRR $\approx$ 800 (0.074" dia x 3/4" long; obtained from Franklin Institute.
5. sample chamber design is similar to that discussed by D. K. Wagner, J. C. Garland, and R. Bowers, Phys. Rev. B3, 3141 (1971).
6. absolute accuracies of Cryocal thermometer: 10 mK in the range 5-20K; 40 mK in the range 20-40 K.
7. J. R. Clement and E. H. Quinell, Rev. Sci. Instr. 23, 213 (1952).
8. see, e.g., Purdue University Thermophysical Properties Research Center Series, Vol. 1 (Thermal Conductivity of Metals), pp. 68-81.
9. G. Ahlers, Phys. Rev. 145, 419 (1966).
10. S. Cristescu and F. E. Simon, Z. Physik Chem. (Leipzig) B25, 273 (1934).
11. R. W. Hill and P. L. Smith, Phil. Mag. 44, 636 (1953).
12. N. E. Alekseevskii and V. I. Tsebro, J. Low Temp. Phys. 4, 679 (1971).
13. for Be#1, thermal conductivity values ranged from 0.05 w/cm-K^o at 2K to 0.8 w/cm-K^o and 6 w/cm-K^o.
14. A. R. Hoffman and M. K. Purvis, Rev. Sci. Instr. 43, 87 (1972).
15. we gratefully acknowledge the assistance provided by Dr. A. Hikata of Brown University.

SAMPLE CHAMBER FIG. 1

(NOT TO SCALE)

resistivity ratio 2000

Data taken August 1972

K&E 10 X 10 TO THE INCH 46 0780
7 X 10 INCHES
MADE IN U.S.A.
KRUHPEL & ESSER CO.

K (W/CM-°K)

T (°K)

BERYLLIUM #1 FIG 2A

The Δ represent a set of points taken on a single sweep when no abnormality other than the relatively high scatter occurred in the region. The dots (·) represent data sets in which the unusual dips are evident.

resistivity ratio 2000

Data taken August 1972

Resistivity-ratio 2000 slow sweep, Dec., 1972

Data taken in two sections: 6°K - 19°K , then 1.3°K - 8°K .