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SUPERCONDUCTIVITY IN PRESSURE QUENCHED CADMIUM SULFIDE AT 77 K

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Observations of magnetic field induced collapse of Meissner sized diamagnetism with a coincident decrease in electrical conductivity in pressure quenched CdS material at 77 K are reported. These results are consistent with the existence of superconducting regions in the specimens at this temperature.

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INTRODUCTION

Recently we have reported large diamagnetism of Meissner proportions in pressure quenched CdS at 77 K.[1] At present only the superconducting state is known to exhibit such large diamagnetism, so it was natural to suggest that a superconducting state was also responsible for the large diamagnetism observed in this material. Zero resistance, the other well known characteristic of superconductivity, has not been observed in these specimens. Metallographic observations show that the specimens are inhomogeneous with gross lenticular platelets parallel to the plane of the disk,[2,3] and the finite resistance we believe results simply from incomplete interconnectivity between the superconducting regions; that is a zero resistance percolation pathway from one side of the specimen to the other does not exist. On this structural basis the disappearance of large diamagnetism with a simultaneous decrease in conductivity in an active specimen of pressure quenched CdS would be strong evidence for the existence of superconducting regions in the specimen, involving itinerant electrons, rather than, for example, the superdiamagnetic state.

In this report we describe the observation of such superconducting behavior in several specimens of pressure quenched CdS, namely the simultaneous disappearance of large diamagnetism with a coincident decrease in the

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[1]E. Brown, C. G. Homan, and R. K. MacCrone, Phys. Rev. Lett. 45, 478 (1980).
[2]C. G. Homan and D. P. Kendall, Bull. Am. Phys. Soc. 24, 316 (1979). Details are presented in ARRADCOM Technical Report No. ARLCB-TR-79004, available from NTIS, Springfield, VA, AD No. A069-609.
[3]C. G. Homan, D. P. Kendall, and R. K. MacCrone, Solid State Comm. 32, 521 (1979).

electrical conductivity. We believe that these observations provide very strong evidence for the existence of some kind of superconducting state at temperatures far in excess of those encountered heretofore. Several new aspects of the electrical and magnetic behaviors in this material are also presented.

EXPERIMENTAL CONSIDERATIONS

The diamagnetic properties of interest are known to depend critically on the purity of the starting material, the pressurizing history, the thermal history and the magnetic history. In addition, the specimens are metastable at room temperature and below,[2,3] so that it is not possible to perform magnetization and electrical measurements sequentially and make meaningful comparisons; the condition of the specimen will simply not be the same. To obtain incontrovertible results, it is necessary to observe the collapse of flux exclusion and at the same time a concomitant decrease in conductivity in one and the same specimen.

A special specimen holder for a vibrating specimen magnetometer was constructed so that dc and ac resistance measurements could be made while the magnetic moment was being measured. Essentially the device consisted of a silica tube carrying the lower copper electrode, through which a second concentric silica tube passed and which carried the upper copper electrode.

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[2] C. G. Homan and D. P. Kendall, Bull. Am. Phys. Soc. 24, 316 (1979). Details are presented in ARRADCOM Technical Report No. ARLCB-TR-79004, available from NTIS, Springfield, VA, AD No. A069-609.

[3] C. G. Homan, D. P. Kendall, and R. K. MacCrone, Solid State Comm. 32, 521 (1979).

In this way, the electrode faces were maintained in alignment and the miniature co-axial shielded cable brought to the outside world through a series of judiciously placed holes and slots. The specimen is simply slipped between the electrodes and maintained under light pressure from small springs far removed from the pick up coils. A cryostat provides controllable temperatures from 20 to 300 K (Air Products Helitran).

The relatively complex holder with wires and electrodes is not as magnetically inert as that used in the previous work.[1] The empty specimen holder shows a small temperature and field dependent magnetization. The specimen holder has been repeatedly run at a variety of temperatures and magnetic fields. At any temperature, the response of the specimen holder over one field sweep may differ slightly from another run on another day, due to uncontrollable variables encountered in mounting and demounting the rod, for example. On the other hand, the reproducibility during a single run is very good, with excellent temporal stability. Relative changes in the magnetization of a single specimen can thus be determined with higher accuracy. We have analyzed the overall statistical error and find a conservative estimate of the standard deviation in the magnetic moment to be 1.5×10^{-2} gauss below 1 kOe and twice that at higher fields. Our thrust is to establish not only diamagnetism but also to observe the disappearance of diamagnetism accompanied by a decrease in conductivity.

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[1]E. Brown, C. G. Homan, and R. K. MacCrone, Phys. Rev. Lett. 45, 478 (1980).

dc Resistance measurements were made using a current amplifier in a simple series circuit, with one to six volts supplied from a battery. ac Capacitance and conductive (or loss) measurements were made at 1 kHz using a G.R. 1615 capacitance bridge. The output of the magnetometer and the dc current or the capacitance and conductance off balance (which may be achieved by using two lock-in amplifiers) were simultaneously displayed on X-Y recorders, the abscissa of each being driven by the same time-base generator. In this way it is possible to obtain a complete one-to-one correspondence between any changes taking place amongst these quantities. The balance of the capacitance bridge was adjusted as required during the experiment, and similarly the current amplifier gain and/or offset.

For electrical contacts we relied solely on the mechanical pressure of the copper disks previously described. The metastability of the samples precludes the painting or evaporization of electrodes. Gentle tapping and the rotation of the upper and lower contacts revealed that the contacts were stable. To test for surface effects, numerous I versus V curves at 77 K have been taken which show ohmic behavior, reversibility, and no polarization effects. During capacitance measurements, dc bias up to ~ 200 V/cms was applied from time to time which resulted in a (reversible) capacitance change of only ~ 1 percent. At room temperature, where this sample showed no anomalous flux exclusion, the relative dielectric constant ε of CdS was determined from the capacitance measurements to be 5.3 ± 1.0, which compares favorably with literature value of 5.2.[4] This agreement is taken as evidence

[4] J. C. Phillips, Phys. Rev. Lett. 20, 550 (1968).

that any contribution from the surface capacitance of a Shottky barrier is essentially absent. In this two terminal measurement, the stray components of capacitance are not magnetic field dependent.

The sample material used in this study, Optronic grade CdS powders from Alpha Inorganic stock no. 20130, was from the same lot used in the previous studies.[1-3] Preliminary chemical analysis of the starting powder yielded total metallic impurities in the 20 ppm range.[1] An x-ray fluorescence spectroscopic analysis was performed by NBS to give a qualitative chemical analysis of all impurities ($Z > 11$). In addition both the starting materials and the pressure quenched samples have been characterized by x-ray measurements, differential scanning calorimetry, optical microscopy and metallography, results which have been reported elsewhere.[2,5] Electron and ion microprobe analyses have been performed on both starting materials and pressure quenched, magnetically active samples.[6] These techniques have provided a quantitative chemical analysis of all elements $Z > 1$ in our material and indicate that the starting materials used in these studies were heavily contaminated or doped with Cl and Si to levels in the 1 wt % range.

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[1] E. Brown, C. G. Homan, and R. K. MacCrone, Phys. Rev. Lett. 45, 478 (1980).
[2] C. G. Homan and D. P. Kendall, Bull. Am. Phys. Soc. 24, 316 (1979). Details are presented in ARRADCOM Technical Report No. ARLCB-TR-79004, available from NTIS, Springfield, VA, AD No. A069-609.
[3] C. G. Homan, D. P. Kendall, and R. K. MacCrone, Solid State Comm. 32, 521 (1979).
[5] P. J. Cote, G. P. Capsimalis, and C. G. Homan, Private Communication, to be published in Applied Physics Letters, May 1981.
[6] Private Communication - U.S. National Bureau of Standards, Report No. 553-33-Y-81, 1980 (unpublished).

The data presented in this report were obtained from samples pressure quenched at a lower rate than the preceding paper.[1] The pressure quenching was accomplished at a nominal rate of 10^6 bars/sec, but a conservative estimate based on the measured unloading rate suggests that the quench rate for these samples was greater than 3×10^5 bars/sec. The same technique applied in the previous work yielded a minimum quench rate of 1×10^6 bars/sec.[1]

A systematic study of the role and interrelationship of the impurity and quench rate variables is still under active investigation. This work represents an effort of considerable magnitude and will be subsequently submitted.

EXPERIMENTAL RESULTS

Diamagnetism and ac Conductivity as a Function of H

A pressure quenched CdS specimen was cooled to 77 K and the magnetization and ac conductivity measured as a function of magnetic field. In this case the dissipation, D, was measured rather than the conductance, G, which in this specimen was small. The results are shown in Figure 1.

At low fields the specimen exhibits a diamagnetic susceptibility amounting to six percent of $-1/4\pi$, the Meissner value. At about 600 Oe the specimen shows the onset of some instability accompanied by a capacitance and loss decrease. The flux exclusion is four percent at this point. Between 825 and 1500 Oe, the diamagnetism decreases to zero, while the capacitance and

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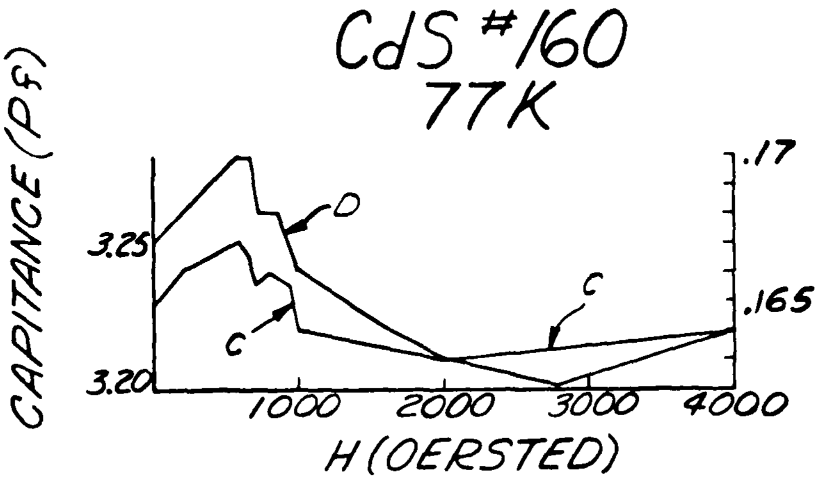
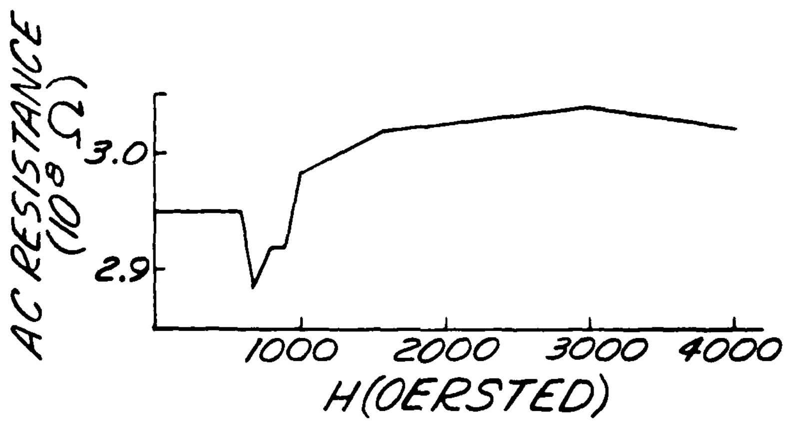
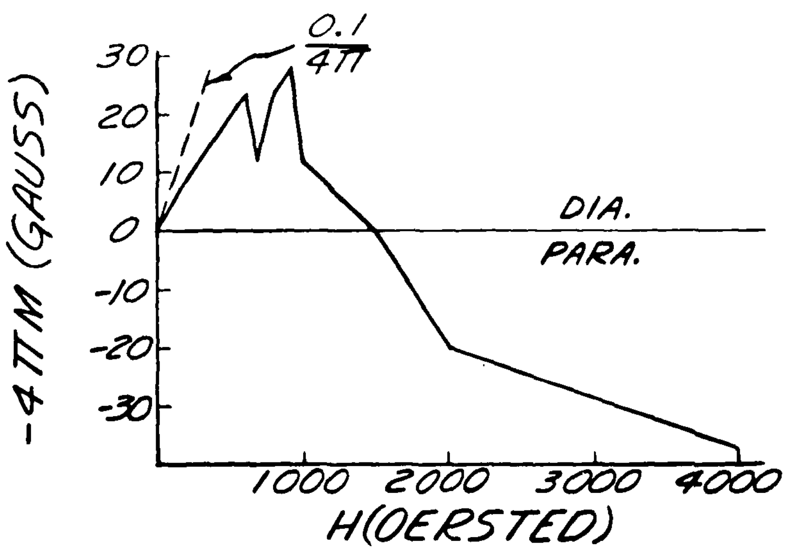
[1]E. Brown, C. G. Homan, and R. K. MacCrone, Phys. Rev. Lett. 45, 478 (1980).



Dielectric response of pressure quenched CdS as a function of applied magnetic field.



ac Resistance of pressure quenched CdS as a function of applied magnetic field.



Magnetic moment of pressure quenched CdS as a function of applied magnetic field.

FIGURE 1

loss show a sharper drop which is almost complete at 1000 Oe. Above 1.5 kOe the specimen is in the positive magnetic state[3] in which the capacitance and loss show only a smooth behavior with no sign of phase transitions.

Diamagnetism and dc Conductivity as a Function of H

A pressure quenched CdS specimen was cooled to 77 K and the magnetization and dc conductivity measured. The results are shown in Figure 2.

As the magnetic field is increased beyond 100 Oe, the dc conductivity decreases as the diamagnetism drops and flux enters the sample. (This specimen, showing constant diamagnetism up to 60 Oe, had been previously subjected to several exposures of fields of the order of 10 kOe while ac measurements were being made.) The ac results for the earlier runs for the sample were essentially the same as shown in Figure 1.

DISCUSSION

The observed relation between the magnetic behavior and the electrical transport behavior of these specimens is in general agreement with that expected of an inhomogeneous material containing superconducting regions below some percolation limit. The fact that a resistance increase is observed on an already large resistance implies that the normal state is highly resistive rather than metallic of low resistance. This is consistent with the observed decrease in the capacitance with the disappearance of the diamagnetism.

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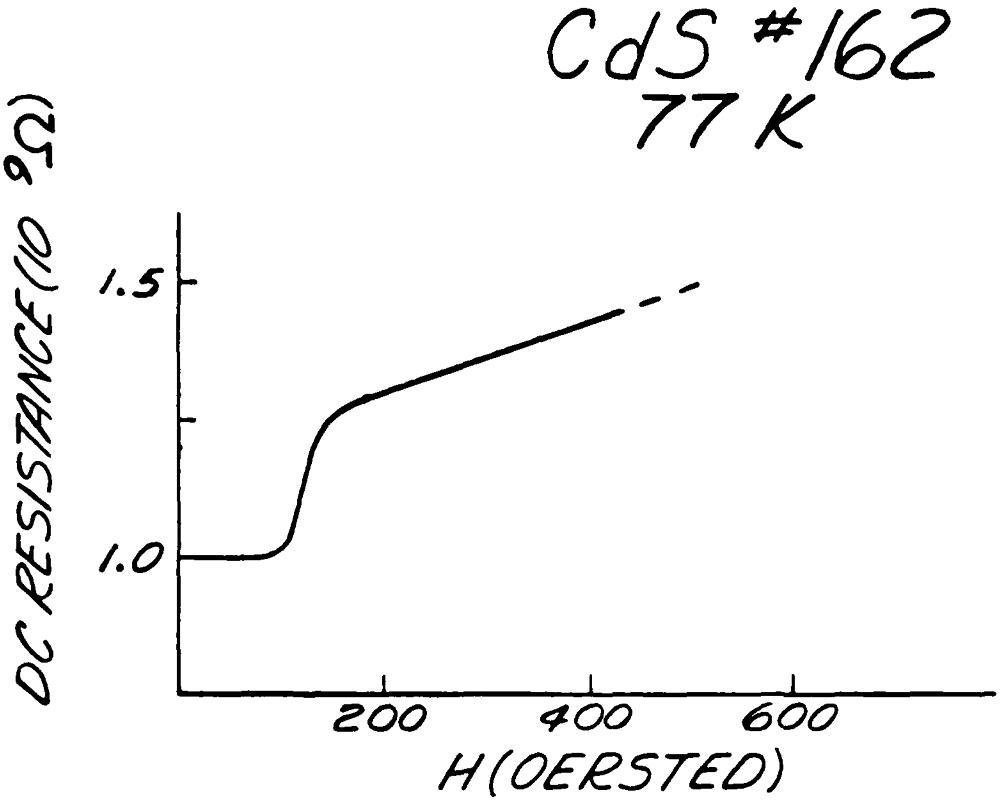
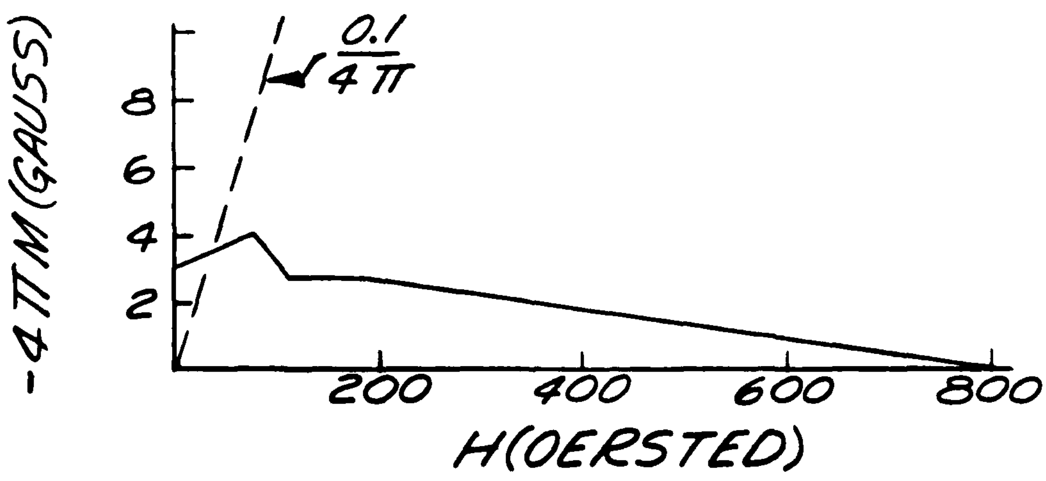
[3] C. G. Homan, D. P. Kendall, and R. K. MacCrone, Solid State Comm. 32, 521 (1979).



dc Resistance observed during the magnetic moment.



Magnetic moment of pressure quenched CdS as a function of applied magnetic field.

FIGURE 2

Rigorous analysis of the behavior expected of inhomogeneous material containing superconducting elements which become normal confirms the intuitive expectations. Computer calculations of general random resistive networks always show a drop in conductivity as the value of a resistive element is increased, corresponding to a superconducting region becoming normal.[7] As pointed out by Landauer, the correspondence of J and E to D implies a corresponding decrease in capacitance as a capacitance element is added (again corresponding to a region becoming normal).

Thus the electrical behavior observed at the collapse of the diamagnetism in Figures 1 and 2 is fully consistent with the electrical behavior expected of an inhomogeneous semiconductor containing superconducting inclusions. This conclusion is very general and is not dependent on ad hoc assumptions or specific morphology.

However, some simple analysis is of interest. Consider, as a very crude model of the system, that the superconducting regions consist of thin sheets lying parallel to the plane of the disk. Then if d_s is the total thickness of superconductor, d the total thickness of specimen, we find:

$$\frac{\Delta\sigma}{\sigma} = \frac{\Delta c}{c} \simeq \frac{d_s}{d}$$

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[7] R. Landauer, "Electrical Conductivity in Inhomogeneous Media," in A.I.P. Conference Proceedings, No. 4, Eds. J. C. Garland and D. B. Tanner, 1978, "Electrical Transport and Optical Properties of Inhomogeneous Media," Ohio State University, 1977, p. 2.

The electrical results of Figure 1 imply, for these ac measurements, that

$$\frac{d_s}{d} \simeq 0.02$$

with values ranging up to 0.09 in other specimens. The data of Figure 2 imply a ratio of

$$\frac{d_s}{d} \sim 0.16 \quad .$$

These differences are within the uncertainties of this crude model, particularly as far as the ac response is concerned which does not address the frequency dependence. The order of magnitude agreement we consider satisfactory. Furthermore, the correlation between the estimate of this volume fraction of superconductor based on electrical behavior above and the volume fraction of platelets based on direct metallographic observation, ~ 15%[2] typical of this material, is remarkable. Occurrence of superconductivity in the platelets produces a natural explanation for both the less than Meissner flux exclusion and the lack of zero resistance in these specimens. We note that the magnetic flux exclusion decays as the electrical conductivity.

CONCLUSIONS

The disappearance of large diamagnetism with a simultaneous decrease in both ac and dc conductivity has been shown to occur in pressure quenched CdS at 77 K. Usually such measurements would be taken as evidence for the

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[2] C. G. Homan and D. P. Kendall, Bull. Am. Phys. Soc. 24, 316 (1979). Details are presented in ARRADCOM Technical Report No. ARLCB-TR-79004, available from NTIS, Springfield, VA, AD No. A069-609.

presence of superconductivity. Our data is striking because of the relatively high temperature involved. We have not observed lossless electrical transport in our samples, which can be qualitatively understood by the unusual morphology of the pressurized samples. Several additional tests of superconductivity are presently being investigated. However, a possibility still exists that some new high temperature collective quantrum state might be involved, as pointed out in our earlier work, and a re-interpretation of the data may eventually take place.

We are forced to conclude at this time that the conductivity and diamagnet'c behavior of our pressure quenched CdS materials must be attributed to superconductivity at 77 K.

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2. C. G. Homan and D. P. Kendall, Bull. Am. Phys. Soc. 24, 316 (1979). Details are presented in ARRADCOM Technical Report No. ARLCB-TR-79004, available from NTIS, Springfield, VA, AD No. A069-609.
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4. J. C. Phillips, Phys. Rev. Lett. 20, 550 (1968).
5. P. J. Cote, G. P. Capsimalis, and C. G. Homan, Private Communication, to be published in Applied Physics Letters, May 1981.
6. Private Communication - U.S. National Bureau of Standards, Report No. 553-33-Y-81, 1980 (unpublished).
7. R. Landauer, "Electrical Conductivity in Inhomogeneous Media," in A.I.P. Conference Proceedings, No. 40, Eds. J. C. Garland and D. B. Tanner, 1978, "Electrical Transport and Optical Properties of Inhomogeneous Media," Ohio State University, 1977, p. 2.

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