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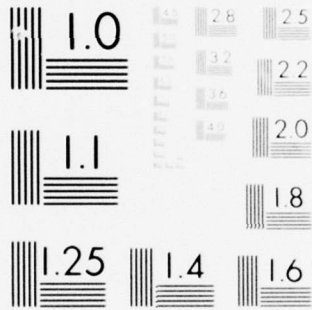
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6 EXPERIMENTAL STUDIES IN
SOLID STATE AND LOW TEMPERATURE PHYSICS

9 Progress Report for 1977

Submitted September 1977

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ABSTRACT

Experimental investigations are being carried out in a broad area of low-temperature and solid-state physics which includes superconductivity, magnetism in metals and liquid and solid helium. The pair-field susceptibility of superconductors is being studied. A propagating mode in the phase of the superconducting order parameter is under investigation. Superconducting fluctuations are being used to probe critical fluctuations in magnetic systems using the proximity effect. Heat capacities of superconducting films in the vicinity of T_c are also being investigated. The properties of thin film solid solutions of sodium in ammonia, sodium in argon, and mercury in xenon are being studied. Nuclear orientation studies of the dilute magnetic impurity problem in metals in the 1 mK temperature region are being carried out. Refrigeration requirements for this work are being met using enhanced hyperfine nuclear cooling.

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I. INTRODUCTION

The work described in this progress report consists of various experimental investigations in a broad area which may be called solid state and low temperature physics. The research is under the direction of Professors A. M. Goldman and W. V. Weyhmann, at the School of Physics and Astronomy in the Institute of Technology of the University of Minnesota and is supported by USERDA Contract E(11-1)-1569. Helium gas was supplied by the Office of Naval Research under Contract N00014-76-C-1086.

A brief discussion of the most important activities is presented in the following paragraphs. All of these contributions will be discussed in more detail later on in this progress report. The reader is cautioned that some of the results presented here are tentative and may be subject to modification prior to publication.

Experimental work on superconductivity is under the direction of Professor Goldman. An important aspect of this work is the study of the pair-field susceptibility of superconductors both above and below T_C . In particular, investigations of the effects of magnetic impurities on the susceptibility are being carried out. The pair-field susceptibility of films in current-carrying states is also being investigated. In such systems mode softening is believed to occur at the critical current. These softened modes are thought to be the nucleation fluctuations for the current-driven onset of phase-slip resistivity in weak links. In related work pair tunneling and the proximity effect are being used to study critical fluctuations in magnetic films with Curie temperatures just above the superconducting transition temperature.

Previous measurements of heat capacities of thin superconducting films revealed the existence of an anomaly near T_c . These studies are being repeated using ac calorimetry on films deposited on sapphire substrates.

The properties of thin solid films of sodium and ammonia have been studied. The metal-nonmetal transition in this system has been found to be anomalous. The same apparatus has been used to investigate the metal-nonmetal transition in the Na-Ar system. This transition does not appear to be anomalous, with the resistivity varying with composition in a manner consistent with percolation theory. Currently under study is the competition between superconductivity and the metal-nonmetal transition in films of Hg and Xe. This system should be a microscopic random dispersion of metal and insulator and as such is expected to have properties different from those of granular metals.

Professor Weyhmann has been conducting nuclear orientation studies of Mn in Cu at temperatures from 3 to 10 mK and fields from 5 to 40 kG. Excellent fits have been obtained to the theories of Luther and Emery and Götze and Schlottmann whereas the expression generated by Ishii does not fit the data. The fitted saturation value for the hyperfine field is in reasonable agreement with results obtained by specific heat measurements. Experiments on samples of Mn in Al and Mn in Ag have also been performed.

The apparatus used for ultra-low temperature studies of magnetism in metals and enhanced hyperfine nuclear cooling is currently being remodeled, with a new dilution refrigerator, dewar and magnet system being installed. With the new magnet system it will be possible to mount superconducting devices close to the sample being polarized thus allowing a variety of measurements other than nuclear orientation to be carried out.

II. DESCRIPTION OF RESEARCH

A. SUPERCONDUCTIVITY

1. The Pair-Field Susceptibility of Superconductors

Detailed information about the dynamics of the superconducting order parameter can be obtained from studying the wave-vector and frequency dependent pair-field susceptibility of superconductors.¹ This quantity is proportional to an excess current in the I-V characteristic of an asymmetric tunneling junction in which one electrode is the superconductor of interest and the other a higher T_c material. Work in this area has been directed at several goals. The first is the study of the dynamics of the order parameter above, below and in the immediate vicinity of T_c . There are two aspects to this investigation: the study of the effects of magnetic impurities on the dynamics of the order parameter and the study of mode softening which is predicted when the superconductor under study is in a current-carrying state near its critical current.² The latter effect is important because the slowed modes are believed to be the nucleation fluctuations for the current during the onset of phase-slip resistivity in weak links, a subject of theoretical and experimental interest. The second goal which has also developed into a well-defined experiment involves the study of the effect on superconducting fluctuations of a depairing parameter, with the aim being the characterization of the system responsible for the pair-breaking rather than the study of the fluctuations themselves. In particular, the critical behavior of the electron spin-flip scattering in systems undergoing magnetic phase transitions is being investigated.³

a. Superconducting Order Parameter Dynamics and Collective Modes
(F. Aspen and A. M. Goldman)

Work on magnetic impurity doped systems has emerged as a key to the possible definitive understanding of the time-dependent macroscopic equations for the order parameter and of the kinetic equations describing non-equilibrium superconductivity. In spite of an emerging theoretical consensus as to the nature of our previous results and the essential correctness of the theory of Schmid and Schön,⁴ a critical test of the theory has not been made. We have been attempting such a test by comparing our measurements of the pair-field magnetic susceptibility in the impurity system of Er in Al⁵ with the results of unpublished calculations of O. Entin-Wohlman and R. Orbach⁶ which are based on the work of Schmid and Schön. Experimentally we have been varying the Er concentration and studying the important limiting cases: the gapless superconductor, the superconductor with a gapless regime near T_c and a gap at lower temperatures, and the superconductor with a gap at all temperatures.

The behavior of two modes must be considered in describing the dynamics of the order parameter. The "longitudinal" mode corresponds to fluctuations of the amplitude of the order parameter and is always diffusive. The "transverse" mode couples to charge fluctuations and propagates when the energy gap is nonzero. The propagating mode is a branch imbalance wave⁷ with space charge fully compensated by the motion of the normal fluid. The propagating character of the mode is governed by the so-called "anomalous" term in the Eliashberg-Gor'kov equations.⁸ This term describes the coupling between the order parameter and the quasiparticle distribution function. When the magnetic impurity concentration is low and the superconductor has a gap, the "transverse" mode propagates. Increasing the magnetic impurity concentration gradually turns off the anomalous term and in both the gapless regime and in

resistivity in superconducting weak links. We have observed the softening of the "longitudinal" mode in some preliminary measurements which are at least qualitatively consistent with theory. The softening of the "transverse" mode should be observable in a limit where relaxation by inelastic electron-phonon scattering is augmented by spin-flip scattering by magnetic impurities. When we complete our attempts to observe this aspect of the softening of the "transverse" mode, we will submit this work for publication.

An interim report on all of the work described in this section was presented as an invited talk at the Gordon Conference on Quantum Fluids and Solids.

b. Magnetic Transitions and Superconducting Fluctuations
(F. Aspen and A. M. Goldman)

The second goal of our work on the pair-field susceptibility involves the use of the tunneling technique to study the fluctuations of a superconducting film in proximity with a metal film containing a sufficient concentration of magnetic impurities that it ferromagnetically orders with a Curie temperature just above the superconducting transition temperature. In this configuration the pair-field susceptibility of the superconducting half of the proximity sandwich is expected to have a quasi-Lorentzian shape. However, the apparent relaxation frequency of the superconducting order parameter is reduced by pair-breaking due to spin-flip scattering of the electrons in the magnetic film. This experiment would provide a new technique for the study of the onset of magnetic ordering in metals.³ It may allow determination of critical indices, which can be calculated by modern scaling theory but which are not easily measured in neutron scattering experiments.

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We performed experiments on Pd(Fe) films during calendar year 1976 which were necessary preliminaries to a full-fledged proximity effect tunneling measurement. A description of the results was given in the previous progress report.¹² We have not carried out additional studies on the Pd(Fe) system in the past eight months because apparatus and personnel have been engaged in continuing studies of the propagating mode which was deemed to have higher priority. Work on magnetic films will be resumed when the investigations described in the previous section are completed.

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2. Critical Behavior of Superconductors: The Heat Capacity of Thin Films
(N. Rao and A. M. Goldman)

We have realized the technique developed for measuring heat capacities

of films at Brookhaven National Laboratory by C. Varmazis and R. Viswanathan.¹ In this procedure films are deposited onto thin sapphire substrates which have a relatively small heat capacity and high thermal conductivity. A gold-iron and copper thermocouple² is used as a thermometer, with the thermocouple and all other electrical leads spot welded to gold contacts which are bonded to the sapphire. This bonding is accomplished by heating in an oven at 1450°C. As in the Brookhaven work, the film, heater, and thermometer are on separate pieces of sapphire which are greased together with a thermal contacting compound. In our work we have been using commercially ground sapphire substrates 0.001 inches thick which are obtained from Insaco Corp. Also, since in the particular experiment we are studying an excess heat capacity which is neither linear nor cubic, the effect at T_c stands out without carrying out measurements on a bare substrate. This makes these investigations somewhat easier than those being carried out at Brookhaven where an absolute heat capacity is required.

Thus far our results which have been confined to clean films ($T_c \lesssim 1.3$ K) show very sharp transitions with heat capacity jumps consistent with BCS. We are currently investigating dirtier films ($T_c > 1.8$ K) in search of the peaks in the temperature-dependent heat capacity, which we observed in such films in our earlier experiments,³ which appear to be in conflict with the work of other groups.⁴ The peaks which we observed previously were also larger than those predicted by theories of the critical behavior of short-coherence-length superconductors.^{5,6}

Because of the controversial nature of the situation, we have been proceeding with great care in developing these new measurements. As our earlier measurements were carried out with optical heating of the films, it

will be necessary to investigate possible effects of light on the heat capacity to take into account the nonequilibrium situation which might have resulted from the interaction of light with the film.⁷

Another feature of our earlier experiments which will have to be considered is the fact that the measurements were carried out using the resistive transition of the film as a thermometer. This was actually done by measuring the voltage drop across a film carrying a constant current. Consequently, at some temperature the measuring current is the critical current, and according to the recent calculations of Ambegaokar the transverse and longitudinal modes of the order parameter soften in such a situation.⁸ If the spectrum of the modes spans a broad enough range of phase space, a heat capacity anomaly could result. This possibility can be checked by measuring the heat capacity while the films are driven by a constant current source.

As this experiment is operational and working smoothly, the answer to these questions should be forthcoming in the near future.

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3. Other Superconductivity Experiments

- a. Electrical Conductivities of Solid Solutions of Na in NH₃, Na in Ar and Hg in Xe
(N. A. McNeal, K. Epstein and A. M. Goldman)

We have developed an apparatus for forming thin films at low temperatures using mixed molecular beams. The beams of gas mixtures are produced in a small oven suspended in a vacuum chamber surrounded by a tube cooled to liquid helium temperature.¹ The walls of the tube are gold-plated to reduce heat losses by radiation and also serve to cryopump the vacuum chamber that the beam traverses. The substrate is kept at liquid helium temperatures during film preparation. The composition of the beam is adjusted by varying the pressure of the gas in the oven and adjusting its temperature so as to obtain the desired vapor pressure of the metal in the oven.

In the case of films of sodium and ammonia the electrical resistance of the films was measured as a function of composition and a metal-nonmetal transition was observed.² The transition was found to be anomalous in that the resistivity was not a monotonic function of the sodium concentration. No evidence was found for the anomalously high electrical conductivities reported for bulk sodium-ammonia bulk solids which were quick-frozen from liquid solutions.³ At present there is no definitive explanation for the observed nonmonotonic character of the metal-nonmetal transition. One suggestion is that the resultant film is really a two component system: Na atoms and molecules which might be NaNH₂.⁴ The nonmonotonic resistance-composition curve then results from a percolation transition involving two constituents which have different electrical conductivities. Calculations indicate that as

the oven temperatures and gas pressures used in the experiments that this hypothesis is unlikely, as the NaNH_2 concentration from the usual reactions which form it is estimated to be less than one part in 10^3 .

This number is being checked experimentally by using a low temperature thickness monitor which we have built to search for H_2 which would be formed in a reaction in the oven producing NaNH_2 . The thickness monitor is a simple quartz crystal oscillator driven by a tunnel diode with all components at 4 K. The crystal will be used to collect a film and then will be warmed to 10-12 K, whereupon if H_2 is present there will be a shift in the frequency resulting from its evaporation. If H_2 is not evolved, then the proposed explanation may be incorrect, and another explanation for the anomalous character of the metal-nonmetal transition will be sought. The thickness monitor has been constructed and tested at 4 K and the results of this experiment should be available shortly.

The molecular beam evaporation apparatus has also been used to study the metal-nonmetal transition in sodium-argon films.⁵ The results are qualitatively consistent, with the metal-nonmetal transition being a percolation transition rather than with the sharp transition as reported by some workers.

The work on the Na- NH_3 and Na-Ar systems constituted the Ph.D. research of N. McNeal.¹ Upon completion of the thickness monitor studies, a detailed paper describing the two experiments will be submitted for publication. Additional investigations on the Na- NH_3 system are not planned until significant theoretical feedback is obtained on the present results.

The molecular beam apparatus is at present being used to study the competition between superconductivity and the metal-nonmetal transition in the Hg-Xe system. This is interesting because, in contrast with alkali-metal-

rare-gas systems, Hg-Xe is believed to exhibit a Mott-Anderson transition rather than a percolation transition.⁶ Preliminary results indicate that the superconducting transition temperature of Hg is depressed by the addition of Xe. It is not known at this time whether the present apparatus can be operated at a low enough temperature to follow the reduction of T_c with increasing Xe concentration as far as the metal-nonmetal transition.

b. Optical Superconductivity

We have proposed to study what might be called an "optical" superconductor - an array of small superconducting particles imbedded in a matrix which was a photoconductor. No experimental studies of this phenomenon were made in the past twelve months because apparatus and personnel were involved in other activities. As it has lower priority than other investigations described in the proposal, it will not actually be undertaken.

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B. EXPERIMENTS ON MAGNETISM IN METALS

1. Nuclear Orientation Studies

(D. Bakalyar, J. Babcock, J. Kiely, T. Manley, and W. Weyhmann)

The analysis of our work on manganese in copper has been completed and the work submitted by Mr. Bakalyar for his Ph.D. thesis. Very good fits have been obtained to the theories of Luther and Emery¹ and of Götze and Schlottmann.² The expression generated by Ishii³ clearly does not fit the data. Over the five to forty kilogauss range of magnetic fields used in our work, there is no distinction between the Götze-Schlottmann and Emery-Luther solutions. In the Götze-Schlottmann solution for high applied fields

$$\langle S_z \rangle = 1/2 \tanh [A(T)B^\epsilon] = H_{hf}/2 H_{sat}$$

we find the constants to be $H_{sat} = 97.1$ kG and $\epsilon = 0.23 \pm 0.06$. Götze and Schlottmann⁴ obtained a value of roughly 0.1 for ϵ from their analysis of low field susceptibility measurements.⁵ Those measurements, however, seem to indicate Brillouin function behavior for magnetization which is clearly at variance with our results. (It should be noted that the above expression is applicable only to high field results.) Our saturation value for the hyperfine field in the Luther-Emery fit is 302.7 kG. Both of these values for the saturation hyperfine field agree reasonably well with the results previously obtained by specific heat measurements.⁶ Our fit to the Luther-Emery theory gives values for longitudinal and transverse exchange constants that differ by a factor of 7. The theory, however, is based on an assumption of symmetric exchange. We do not yet know how serious this discrepancy is.

In this same system, we have studied the dependence of the hyperfine interaction on manganese concentration. With the addition of only 1/2 ppm of

manganese, the hyperfine field of 5 kG was lowered by about 4 percent, from 272 for the "carrier-free" ^{54}Mn sample to 261.5 for the 1/2 ppm sample. The addition of about ten ppm lowered the hyperfine field again by another several kilogauss. At the 40 kG applied field there is no measurable shift with concentration over these ranges. This implies that all data taken at low fields and concentrations of order 1 ppm are suspect, though this point deserves further investigation.

The data on manganese in silver shows a smaller variation in the hyperfine field over the same range of applied fields by roughly a factor of two. Thus at 4 mK Ag(Mn) seems to show a Kondo effect but with what would presumably be a lower Kondo temperature than for the Mn in Cu system.

In the laboratory the first half of 1977 has been spent dismantling the nuclear orientation apparatus and reconstructing it for use with our new dilution refrigerator, dewar and magnet systems. All components have been built and tested. The pumping system seems to work satisfactorily under gas load. We are presently mounting the new dewar and soldering in the dilution refrigerator stage. Hopefully, we will be running experiments again by early fall.

We have acquired an 80 kG magnet with a two-inch bore. This magnet has a counterwound compensation coil at one end such that four inches from the end of the magnet the field is less than 400 gauss and remains so from there out to infinity. With this magnet we hope to be able to mount superconducting devices fairly close to the sample being polarized, so that measurements other than nuclear orientation can be made on it. The 20 kilogauss demagnetizing magnet is also being replaced by a 40 kilogauss magnet we have built and reported previously.

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2. Enhanced Hyperfine Nuclear Cooling

(D. Bakalyar, J. Babcock, J. Kiely, T. Manley, and W. Weyhmann)

With our basic demagnetization apparatus being revised, we have not been able to make any new runs on materials for enhanced hyperfine cooling. We have, however, acquired a Centorr arc melting furnace for the preparation of materials without crucible. We previously reported our experience that materials remelted at Ames Laboratory by this technique were free of cracks; thus we hope that such remelting will provide materials with faster thermal equilibration times. This year we have constructed a sample pulling device for this furnace so that thin, cylindrical rods of the material being melted can be formed. By using several rods rather than one large ingot, the eddy current heating during demagnetization can be reduced. In addition, thin rods are preferable for measurements on the transport properties of these materials at low temperatures; and for this purpose fissure free materials are essential.

III. RECENT REPORTS

"Anomalous Conductivity of Quench Condensed Sodium-Ammonia Films,"
N. A. McNeal and A. M. Goldman, Phys. Rev. Lett. 38, 445 (1977).

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"Low Temperature Electrical Measurements of Quench-Condensed Sodium-Ammonia
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"Vapor Deposited Metal-Gas Alloys," N. A. McNeal and A. M. Goldman.

"Nuclear Orientation Studies of Manganese Copper Kondo System at Ultralow
Temperatures and High Applied Fields," D. Bakalyar, Ph.D. Thesis.

"Experimental Determination of the Order Parameter Response Function of
a Superconductor," A. M. Goldman

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