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STUDIES OF ELECTRON TRANSITIONS USING SOLID He PRESSURE TECHNIQUES*

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ABSTRACT

Changes in the topology of the Fermi surfaces of metals occur with variation of temperature or stoichiometry in a number of metallic systems of current interest. Pressure generated by a variety of techniques has proven to be the most useful experimental variable in the study of these transitions, but in most cases only solid He techniques yield sufficiently hydrostatic conditions to permit direct measurements of the Fermi surface as a function of pressure.

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I. INTRODUCTION

Studies of the pressure dependence of the Fermi surface of metals have proven useful in unravelling the details of the electronic structure of a large number of metals and semimetals in the past few years. Efforts have centered in: i) testing of band theoretical models or descriptions of the metal and ii) which is the subject of this discussion, studies of electron transitions or changes in the topology of the Fermi surface (FS). Low-temperature pressure studies provide the best method for probing these topology changes because either temperature or alloying tends to smear out effects of these purely electronic transitions. There is a hierarchy of electron transitions of which the simplest situation is the electron or Lifshitz transition.¹ This is a change in FS topology with no change in the crystal structure and no change in the interatomic spacing. The next step in the hierarchy is a change in FS topology accompanied by a change in volume but no change in structure. This class includes the "mixed valence" and "electron promotion" situations found in an increasing number of narrow band systems which are receiving an enormous amount of attention currently. Included in this class are many of the metal-insulator and metal-semiconductor transitions. Finally, there is the third tier in the hierarchy where the topology change in the Fermi surface is accompanied by (or results from, depending on what is thought to be driving the transition) a change in both structure and volume. In this discussion, emphasis will be placed on the "simplest" situation, the Lifshitz transition, where it is possible in several cases to follow directly the changes in the Fermi surface

by use of deHaas-van Alphen or high field magnetoresistance techniques.

In Lifshitz,¹ original treatment of electron transitions he treated four situations: the creation or destruction of an entire sheet of the Fermi surface and the changes in connectivity involved in the creation or destruction of a neck or bridge between larger pieces of Fermi surface. Lifshitz dealt with a free electron density of states which caused half integer powers to appear in the free energy prompting him to dub these "transitions of order 2.5." The two in this 2.5 is meant to indicate that that transition is second order as well as isostructural. Anomalies then are predicted for essentially any properties depending upon the density of states.

Historically most of the experimental evidence² for electron transitions has come from observed nonlinearities in the pressure and concentration (in the case of alloys) dependence of the superconducting transition temperature T_c . This is because T_c depends exponentially upon the density of states, DOS, in the Bardeen-Cooper-Schrieffer³ framework and therefore extremely small changes in the DOS are reflected as easily measurable changes in T_c .

A vast amount of work has been published² based on such data in the case of Tl. A peak in T_c vs. pressure is observed near 2 kbar. A careful search using deHaas-van Alphen techniques under pressure failed to show any sign of a topology change⁴.

Holtham⁵ has hypothesized that the small sheet responsible has already been formed at normal volume but that corresponding peak in T_c occurs at higher pressure. While this is plausible it is perhaps more likely that some feature not simply related to the extremal cross sections measured to date is involved since the excursion in the DOS is only of the order of 0.1% of the total. Similar nonlinearities in T_c vs. pressure have been reported in Re⁶, in In and its alloys with Pb and Cd^{7,8}, in the intermetallic compound⁹ Bi_2K and in VRu alloys¹⁰ near the equiatomic composition. In all these materials, electron transitions have been suggested as the explanation for the anomalies but there has been no direct confirmation of Fermi surface changes.

There are a number of situations when direct evidence of Fermi surface topology changes have been reported. Itskevich and Voronovskii¹¹ presented convincing evidence from high field magnetoresistance studies that the arms of the 2nd zone hole sheet of the Fermi surface are formed in Cd by 15 kbar.

The electronic properties of the As structured semimetals have been studied extensively as a function of pressure. Itskevich and Fisher¹² reported a vanishing of the entire Fermi surface of $\text{Bi}_{.95}\text{Sb}_{.05}$ near 10 kbar. This information was obtained from the vanishing with pressure of the frequency of Schubnikov-de Haas oscillations in the magnetoresistance. Brandt and Ponomarev¹³ used similar techniques to study pressure-induced electron transitions in a series of Bi-based binary and ternary alloys. An electron transition involving changing the connectivity of the hole sheets of As by pinching off the " α " arms by 1.85 kbar was observed by Schirber and Van Dyke¹⁴. A volume-dependent band calculation based

on the Lin and Falicov pseudopotential for As was found to be consistent with this result. In Sb the cross-sectional area corresponding to the minimum area of the hole sheet is found to initially decrease with pressure, then increase passing through to the normal volume value near 5 kbar¹⁵. This behavior is probably due to a not as yet identified electron transition.

Low-pressure anomalies in the transport properties of Cs are probably related to an electron transition near 7 kbar. Beardsley and Schirber¹⁶ found that the dHvA signal corresponding to the "bellies" of the dimpled spherical Fermi surface disappear near this pressure for field directions where the orbit would be interrupted by the formation of [110] necks joining the spheres. Several band structure calculations indicate that these necks should be formed at modest pressures but the necks themselves were not detected.

Ribault has recently reported¹⁷ that a dHvA frequency disappears in fcc Yb at an extrapolated pressure of 12 kbar. This is very near the pressure at which the electrical properties indicate a change from semimetallic to semiconducting behavior.

In this paper we will discuss briefly what appears to be the best documented case¹⁸ of an electron transition -- AuGa_2 -- as an example of the types of anomalies that can be observed with various experimental probes. We will then describe recent studies on InBi and CeAs as similar analyses have been applied to account for various anomalies in the pressure and temperature dependence of electronic and magnetic properties.

In Section II the solid He technique¹⁹ is described which has proven invaluable in direct Fermi surface measurements. In Section III the AuGa_2 situation is reviewed and in Section IV we

summarize our results on InBi and CeAs. Section V summarizes the present situation in the study of electron transitions and presents some recommendations for further activity.

II. EXPERIMENTAL

As mentioned in the introduction, the emphasis in this discussion will be on direct determinations of Fermi surface topologies near electron transitions of the first kind -- those involving no changes in either volume or crystal structure. While a great deal of indirect information concerning an electron transition may be obtainable from temperature-dependent properties and from quasihydrostatic pressure studies of superconducting properties, extremely stringent conditions of hydrostaticity are required for direct measurement of the Fermi surface.

Direct measurements of the Fermi surface must be made at low temperatures because very long electron mean-free-paths are required. Above 25 bar, all pressure media are solid at 1 K. While a substantial amount of information has been obtained concerning the initial volume derivatives of cross sections using fluid He as the pressure medium, these techniques are inappropriate if one wishes to effect the transition with pressure. Therefore solid pressure transmitting media are required for these experiments. Itskevich²⁰ and coworkers and Brandt²¹ and coworkers have been able to use frozen fluid techniques in certain situations to obtain oscillatory magnetoresistance data. These situations usually involve small cross sections with small effective mass ratios. The amplitude factors for the oscillatory phenomena related to external cross sections of the Fermi surface involve products of the effective mass and the so called Dingle temperature which is proportional to the scattering of the electrons.

Thus for very small masses, observable amplitude oscillations may be observed. For more typical values of effective masses, the nonhydrostatic conditions obtained in these frozen water, kerosene and pentane mixtures usually preclude direct measurements of the Fermi surface. Brandt and coworkers²² have demonstrated that pulse heating of the sample so that the medium is liquified or softened can improve the amplitude of the signals in low-temperature high-pressure experiments using frozen iso-n-pentane mixtures, but no additional use of this technique has been reported to date.

On the other hand, it has been amply demonstrated that the solid He technique¹⁹ can give pressures to a maximum of 10-15 kbar which are experimentally indistinguishable from truly hydrostatic. This technique involves generation of pressure in fluid He as a temperature just above that at which the He will freeze. At 10 kbar this corresponds to about 60 K. This pressure is then maintained as the sample is cooled slowly from the bottom such that the fluid-solid line moves past the sample at constant pressure. The idea is to maintain a fixed position on the fusion curve until the sample is completely surrounded by solid He. When the fluid solid line reaches the capillary connecting the pressure vessel to the external system, it is considered blocked and subsequent cooling is assumed to be along an isochore. Within the validity of this assumption, the final pressure can be accurately determined from the known pressure-volume-temperature data for He. It is only fair to point out several shortcomings of the solid He technique. There is some danger in handling the high-pressure gas so volumes must be minimized. This difficulty in handling the gas limits the practical pressure limit of the technique to 10-15 kbar. A

thermal cycle is required to change the pressure which is fairly time-consuming and which precludes true constant temperature pressure derivatives.

A large amount of data have been accumulated on single crystal metals which indicate that even on the most fragile and anisotropic materials, hydrostatic conditions can be maintained with sufficient care. High-mass, high-frequency oscillations have been observed under solid He pressure conditions in many metals, usually without loss in amplitude. It is therefore possible with this technique to make detailed studies of the cross-sectional areas of the Fermi surface using the standard Fermi surface tools such as deHaas-van Alphen (dHvA) effect, the deHaas-Schubnikov oscillations and high field magnetoresistance. The field modulation technique for determining dHvA frequencies has in fact proven to be the most powerful technique because it gives cross-sectional areas, electron effective masses and information on scattering without the necessity of affixing leads to a specimen. The sensitivity with phase-sensitive detection techniques is such that the pickup and modulation can usually be external to the pressure vessel obviating the necessity of introducing leads into the high-pressure environment.

While the emphasis of this discussion is on direct Fermi surface studies and their relation to electron transitions, it is often difficult to obtain the necessary single crystal long mean-free-path samples necessary for such studies. The sensitivity of superconducting properties, particularly T_c , to these changes in the density of states, DOS, was mentioned in the introduction. Another DOS sensitive probe is the nuclear magnetic resonance

frequency shift or Knight shift which can be thought of as the product of a hyperfine coupling constant and the susceptibility. We have demonstrated that both pulsed and continuous wave resonance techniques can be employed in conjunction with solid He pressure generation. These techniques have been used to complement direct Fermi surface studies and in situations where the material is neither a superconductor nor can be obtained in single crystal form -- thus these measurements are the best available to probe the system in the region of an electron transition.

III. THE AuGa_2 STORY

As indicated in the introduction, there is fragmentary information concerning Lifshitz or electron transitions from many types of experiments on many different materials. However, AuGa_2 has probably the best understood electron transition from the standpoint of the diversity of experimental evidence and from the detail of the model description available. It seems appropriate then to briefly summarize the situation with regard to this in some sense ideal case.

AuGa_2 is a free-electron-like, valence 7 fcc metal which originally attracted attention because of anomalies in the magnetic properties which were called by Jaccarino, et al.²³ "the AuGa_2 dilemma." Specifically the Ga nmr showed a large continuous change from positive to negative near 100 K as the temperature is decreased. The Au nmr has no such anomaly nor do any of the nmr signatures in the isomorphous compounds AuAl_2 and AuIn_2 . dHvA measurements of Fermi surface cross sections in all the three compounds with pressure showed qualitative agreement with Switendick's band calculation²⁴ as a function of interatomic spacing²⁵. This band cal-

culation showed an anomalously flat band having predominantly Ga s character centered at the point Γ in the Brillouin zone and lying completely below E_F . This same band in the sister compounds AuIn_2 and AuAl_2 intersects the Fermi level and gives rise to the second band hole surface. This sheet is easily observable in dHvA work on AuIn_2 and AuAl_2 but is not seen in AuGa_2 . Subsequent measurement of the pressure dependence at various temperatures of the Au and Ga Knight shift and spin lattice relaxation time and of the pressure dependence of T_c are all consistent with the following band structure-electron transition picture¹⁸.

The flat Ga s band lies 0.012 eV below the Fermi level at 0 K and normal volumes. It can be pushed through the Fermi level with 7 kbar giving rise to the large abrupt changes in T_c and the Ga Knight shift and spin lattice relaxation. The Au nmr is not affected. With increasing temperature, the band actually drops with respect to E_F but is still sampled because of thermal smearing, thus giving rise to the anomalies in the temperature dependence of the Ga nmr properties and in the elastic and electrical properties. A schematic of this behavior is shown in Fig. 1.

This is a nearly ideal situation because the sheet of the Fermi surface involved has a high density of states associated with it. This DOS has a preponderance of Ga s character and Ga has a large moment so nmr is prominent. The large DOS change is reflected in very large excursion (doubling) of T_c . In addition, AuGa_2 is a relatively simple calcium fluoride structured material that can be prepared as good quality single crystals. Finally this high density of states peak is located very near the Fermi level and can be made to intersect E_F with an easily attainable pressure.

This combination of fortuitous circumstances combine to make AuGa_2 the showpiece electron transition material.

IV. RECENT RESULTS

A. InBi

InBi is a semimetal crystallizing in the tetragonal B10 structure instead of the usual cubic structures found in the typical III-V semiconductors. This material was the first compound metal in which direct Fermi surface data were obtained.²⁶ A number of anomalies in various properties of InBi with pressure and temperature variation have been reported. A change in sign of the In Knight shift and a disappearance of the electric field gradient at the In site were observed around at 230 K and 170 K respectively.²⁷ Anomalies in the resistivity were found near 15 kbar by Bridgman²⁸ and Rapoport, et al.²⁹ reported a jump in the resistance which they attributed to a first-order transition near 19 kbar. Gordon and Deaton³⁰ saw no volume discontinuity to 30 kbar. Broad anomalies in the pressure dependence of the acoustic mode velocities were observed by Fritz³¹ but no phase changes were indicated. Coupled with these conflicting data concerning anomalies, there is a certain amount of controversy about the Fermi surface of InBi because of the likelihood of spurious signals from oriented inclusions of In_2Bi and/or In_5Bi_3 . The most likely interpretation³² is that the largest sheet is an ellipsoid oriented along [001] associated with the γ dHvA oscillation. The β oscillations are associated with a sheet of one-half the volume of the γ sheet, also oriented along [001]. Two much smaller sheets (10^{-2} - 10^{-3} the volume of the γ) are observed. The α oscillation correspond to ellipsoids along [001] while the ζ sheet is probably an ellipsoid along [100]. We have measured the pressure derivatives for all of these cross sections using field-modulated dHvA tech-

niques in solid He as shown in Table I. We have also performed relativistic orthogonalized-plane-wave band calculations as a function of interatomic spacing.³³ The first cut band calculation did not agree with the accepted experimental Fermi surface. A modified picture in which bands were shifted small amounts within the calculational uncertainty accounts for the experimental Fermi surface with respect to compensation of holes and electrons and the general orientational features. The model also gives the pressure behavior of the four observed sheets as far as both sign and relative magnitude of the pressure derivatives are concerned. An electron transition which the experimental data indicate will occur near 12 kbar at 4 K as the α sheet disappears is postulated to be the cause of the anomalies in the elastic properties with pressure (see Fig. 2).

The model indicates that the electron pocket near R in the Brillouin zone has substantial In s character and changes in the proper direction to explain at least qualitatively the anomalies in the nmr properties. While some manipulation of the model was necessary to achieve a satisfactory description of the normal volume Fermi surface, this treatment does a good job of explaining a number of heretofore puzzling anomalies in pressure- and temperature-dependent properties of InBi.

B. CeP and CeAs

Anomalies in the temperature dependence of the resistivity, thermal expansion³⁴ and pnictide nmr³⁵ of CeP and CeAs have been reported. The explanations for these anomalies attempted include promotion of 4f electrons in the Ce, nonlinear hyperfine interactions and conduction electron effects. In these materials we have no possibility at present of obtaining direct Fermi surface

information of any kind. The materials are not superconducting so this probe of the DOS is not useful. Furthermore there is no detailed band theoretical picture presently available. We therefore have used measurements of the pnictide nuclear magnetic resonance frequency shift as a function of pressure to investigate these materials.

The results for the Knight shifts for the ^{31}P and ^{75}As in CeP and CeAs respectively at normal volume and at 4 kbar are shown in Fig. 3. The anomalous behavior is the flattening of the Knight shift below 50 K. Based on a large body of data in these types of rare earth compounds,³⁶ the Knight shifts were expected to show Curie-like behavior. The same data are shown in derivative form in Fig. 4.

Attempts to interpret these pressure derivatives by varying the crystal field parameters or invoking nonlinear hyperfine coupling give the wrong qualitative behavior with temperature. Instead of showing a decrease in the percentage change per kbar as the temperature increases, they show an increase. A model involving a sharp feature in the DOS which, upon fitting the normal volume and pressure dependence, turns out to be a 30 K wide peak 85 K below E_F moving towards E_F at a rate of 5 K/kbar. The solid line shown in Fig. 4 is the result of this model calculation and is seen to be in excellent agreement with experiment.

We have obviously leaned heavily on the much better documented cases of the AuGa_2 and InBi in evolving such a model. Recently Hirst³⁷ has discussed fluctuating valence descriptions for Ce which lead to narrow peaks in the DOS very near the Fermi level. Such models may ultimately legitimize our somewhat contrived

picture for these materials.

V. CONCLUSIONS

This brief review of past activities in the study of electron transitions and discussion of recent and ongoing work are perhaps indicative that progress is being made in the study of electron transitions. However much needs to be done before a clear picture of the detailed nature of these "simple" transitions will evolve. Other candidate materials that lend themselves to experimental and theoretical investigations as does AuGa_2 are sorely needed. A specific case that should be studied in depth is that of Cs as this is potentially the simplest situation we are likely to encounter -- the change from a simple spherical Fermi surface to a multiply connected array.

A second fundamental question is whether there is any connection between the various levels in the hierarchy of electron transitions. Do the simple electron or Lifshitz transitions serve in any fashion as precursors for more complicated transitions? The Russian investigators³⁸ have proposed that an electron transition occurs just before the valence change and associated isostructural lattice collapse in Ce. There is evidence as indicated above that an electron transition may precede the mixed valence state in CeP.³⁹ Again Cs is a good candidate for such considerations.

The intermetallic compounds will probably continue to provide the most opportunities for investigation of electron transitions. Because of the flexibility inherent in the possibility of forming closely related congeners, these materials are the best hope for providing the vehicles leading us to the understanding of electron transitions in particular and perhaps phase changes in general.

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Table I. Pressure derivatives $d\ln F/dP \equiv [F(P) - F(P = 0)]/F(P = 0)\Delta P$ for InBi. Orbit nomenclature is that of Ref. 31.

Field Direction	Frequency (MG)	Orbit	$d\ln F/dP$ (%/kbar)
[001]	0.137	α	-8 ± 1^a
			-8.3 ± 0.6^b
	3.19	β	$0.3 (\pm 0.1)^a$
			$0.3 (\pm 0.1)^b$
	7.78	γ	$0.03 (\pm 0.02)^a$
[100]	11.08	γ	$0.3 (\pm 0.1)^a$
	0.63	ζ	$9 (\pm 3)^a$
17 deg. from [110] in (001)	3.8	ϵ	$1 (\pm 0.5)^a$

^a Fluid He phase shift.

^b Solid He to 4 kbar.

FIGURE CAPTIONS

Figure 1. Schematic representation of relative position of Fermi energy (E_F) and the Ga s-band edge (E_0) in AuGa_2 under varying conditions of temperature and pressure. The bottom sketch indicates the energy range sampled in the Knight-shift measurements at 300 K.

Figure 2. Schematic energy band model of InBi. Bands not shown are assumed to not cross the Fermi energy. The effect of pressure is to increase the overlap of the bands at Λ and Z but with a net shift of the center of gravity and therefore E_F to higher energy. This decreases the overlap at Γ and increases it at R .

Figure 3. Temperature dependence of the Knight shift for ^{31}P and ^{75}As contained in CeP and CeAs at 0.3 ($\blacksquare$) and 4 kbar ($\bullet$) pressure.

Figure 4. Pressure derivatives of the ^{75}As Knight shift in CeAs as a function of temperature. The solid curve is derived with the model described in the text.

AuGa_2

$T = 4^\circ\text{K}$

$P = 0$

$T = 4^\circ\text{K}$

$P = 6.9 \text{ kbar}$

$T = 300^\circ\text{K}$

$P = 0$