ANNUAL SCIENTIFIC PROGRESS REPORT

National Nuclear Security Administration Stockpile Stewardship Academic Alliance Research Grant #DE-FG52-06NA26205

The focus of this grant, entitled "Experimental investigations of magnetic, superconducting, and other phase transitions in novel f-electron materials at ultra-high pressures using designer diamond anvils," is to explore the novel properties of f-electron compounds under pressure, with a particular emphasis on the physics of superconductivity, magnetism, and their interactions. This report is a synopsis of the research that was undertaken from 6/2007 – 6/2008.

I. High temperature superconductivity in oxypnictide compounds

a. The arsenides: $LaFeAsO_{1-x}F_x$ and $CeFeAsO_{1-x}F_x$

A new class of superconductors consisting of layered materials with the chemical formula LnTPnO, where Ln is a lanthanide element, T is a transition metal, and Pn is either P, As, or Bi, has recently emerged. The phosphorus-based versions of these compounds, LaFePO and LaNiPO have rather low superconducting critical temperatures, T_c of 3 [1] and 5 K [2], respectively. Much higher T_c values were achieved by fluorine-doping the corresponding arsenic-based compound to produce $LaFeAsO_{1-x}F_x$, where doping to $x \sim 0.11$ produces $T_c \sim 26$ K [3]. The T_c appears to pass through a maximum as a function of fluorine doping. Subsequently, it was found that under a modest pressure of 40 kbar, the T_c of $LaFeAsO_{1-x}F_x$ increases to 43 K [4], becoming the first non-cuprate superconductor with a T_c higher than that of MgB_2. Replacing lanthanum with heavier rare-earth elements also leads to high T_c values, as in $CeFeAsO_{1-x}F_x$ with T_c up to 41 K [5]. As of this writing, the highest T_c reported for this class of materials is about 55 K, which was achieved in the compound $SmFeAsO_{1-x}F_x$ [6]. The T_c of optimally doped $SmFeAsO_{1-x}F_x$ initially decreases with pressure [7].

We have performed several high-pressure resistivity experiments on the recently discovered superconductors $LaFeAsO_{1-x}F_x$ and $CeFeAsO_{1-x}F_x$. For the lower portion of the pressure phase diagram, we performed measurements of T_c vs. pressure using hydrostatic clamp and Bridgman anvil cell techniques for both of the samples, respectively; the highest pressures for both samples were achieved utilizing the diamond anvil cell technique. At ambient pressure, these materials have superconducting onset temperatures T_c of 28 K and 44 K, respectively. While the T_c of $LaFeAsO_{1-x}F_x$ passes through a maximum between 10-68 kbar, the T_c of $CeFeAsO_{1-x}F_x$ decreases monotonically over the measured pressure range. At 265 kbar, the T_c of the cerium-based compound is suppressed below 1.1 K.

The strong dependence of T_c on pressure in these materials is rather remarkable. The bulk modulus of LaFeAsO is only 98 GPa [8], significantly smaller than that found for the cuprate superconductors. It is likely that the strong dependence of T_c on pressure for $LaFeAsO_{1-x}F_x$ and $CeFeAsO_{1-x}F_x$ is related to their high compressibility. Experiments to determine structural parameters under pressure would help to clarify the effect of structural properties on T_c. For the oxypnictides, it is likely that increasing pressure leads to an increase in carrier concentration, as in the cuprates. The initial increase in T_c with pressure for $LaFeAsO_{1-x}F_x$ may thus be due to the sample being underdoped. Indeed, it was reported [9] that increased doping achieved through high-pressure synthesis raises T_c to 41 K in $LaFeAsO_{1-x}F_x$. In the high-T_c cuprate superconductors, it is found that T_c generally increases with pressure in optimally doped samples, highlighting the fact that the effect of pressure is more complicated than simply changing the carrier concentration. The negative pressure dependence of T_c that we find for apparently optimally doped $CeFeAsO_{1-x}F_x$ combined with that previously reported for $SmFeAsO_{1-x}F_x$ points to a possible difference between the oxypnictide and cuprate superconductors. A systematic study of the effect of pressure on T_c across a wide range of dopings is clearly needed in order to obtain a better understanding of the optimal conditions for high-T_c values in the oxypnictide superconductors.

b. The phosphides: LaFePO

Superconductivity in this series of materials was discovered in LaFePO in 2006 [10], for which values of T_c that range from 3 K to 7 K have been reported. However, in a recent study of polycrystalline materials, it was concluded that this compound is metallic but non superconducting at temperatures as low as 0.35 K [11]. We synthesized single crystals of LaFePO, with superconducting transitions at 6.6 K and 6.0 K, according to electrical resistivity and magnetic susceptibility measurements, respectively. However, there is no specific heat jump at T_c, suggesting that only a small fraction of the sample is superconducting. The superconductivity appears to be a property of the single crystals, since the resistively measured upper critical field is quite anisotropic. It is possible that the superconductivity is associated with oxygen vacancies that dope a small fraction of the compound with charge carriers. Electrical resistivity measurements were made under high pressures of 54, 106, 158, and 204 kbar using a diamond anvil cell. A rather moderate pressure of 106 kbar was sufficient to nearly double the onset of superconductivity from 7K to 14 K, above which pressure acts to suppress T_c.

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II. Charge density waves, magnetic order, and superconductivity in rare-earth tritelluride compounds

Charge density waves (CDWs) are electronic instabilities found in low-dimensional materials with highly anisotropic electronic structures [1]. Since the CDW is predominantly driven by Fermi-surface (FS) nesting, it is especially sensitive to pressure-induced changes in the electronic structure. A well known example is $NbSe_2$, for which the CDW can be completely suppressed by an applied pressure of 35 kbar, favoring the competing superconducting phase. Chemical pressure (the incorporation of larger or smaller ions to expand or contract the crystal lattice) can be used to mimic the effect of external pressure, providing a valuable tuning parameter for such materials. In this regard, rare-earth containing compounds are particularly valuable because the lattice parameter can be varied over a wide range in an almost continuous fashion while keeping the band filling essentially unchanged. The rare-earth tritelluride $R\mathrm{Te}_3$ compounds form for almost the entire rare-earth series, with R = La – Nd, Sm, and Gd – Tm, and provide a unique opportunity to follow the effect of chemical pressure on FS nesting and CDW formation, and its competition with other ground states. For most of the rare earths, the material has an incommensurate lattice modulation at room temperature, with a single in-plane wave vector of approximately $(2/7)c^*$ ($c^*=2\pi/c$).

In order to further understand the electronic effects produced by changing the lattice parameters, we performed pressure experiments (in collaboration with Professor Ian Fisher's group from Stanford University) on two members of the rare-earth tritellurides, $CeTe_3$ and $TbTe_3$. Ce and Tb were strategically chosen for being located at the beginning and near the middle of the lanthanide series, respectively, allowing us to map a vast range of lattice parameters with the pressure techniques available in our laboratory. We found that the two CDW ordering temperatures present in $TbTe_3$ collapse into a single one and disappear at a pressure value of 23 kbar. The CDW of $CeTe_3$ happens well above room temperature, so we were expecting to lower its ordering temperature with pressure. We found that the system orders antiferromagnetically at low temperatures, and we followed the evolution of this ground state up to 150 kbar. The results obtained on these rare-earth tritelluride experiments are being analyzed and manuscripts reporting the results are in preparation.

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III. Superconductivity, hidden order, ferromagnetism, and quantum criticality in $URu_{2-x}Re_xSi_2$

a. URu_2Si_2 (x = 0)

The moderately heavy fermion compound URu_2Si_2 was discovered over 20 years ago, but still remains a perplexing and interesting compound owing mostly to its transition into an ordered state near T_0 = 17.5 K. The order parameter of this state has yet to be identified, even after 20 years of research, and, as such, this ordered state has come to be known as the “hidden order” (HO) state. In addition to this HO state, URu_2Si_2 exhibits another phase transition into a superconducting state at T_c = 1.5 K. It has been inferred previously from neutron diffraction data that these two ordered phases at ambient pressure coexist, prompting concerns over the nature of the HO state and its relation to superconductivity. To that end, we have prepared high-quality, single crystal specimens of URu_2Si_2 and prepared them for high-pressure, electrical resistivity measurements, utilizing a hydrostatic piston-cylinder clamp device in our facility, to explore the HO and superconducting states up to approximately 25 kbar.

The HO transition manifests itself in the resistivity as a trough-and-peak structure reminiscent of the spin density wave (SDW) transition of elemental chromium. We found that the qualitative shape of this transition persists up to the highest pressures measured. The HO transition temperature, T_0, was found to exhibit a distinct kink in its pressure dependence at P_c = 15 kbar: below P_c, $T_0(P)$ is linear with a slope near 0.1 K/kbar; and, above P_c, $T_0(P)$ is linear with a slope near 0.23 K/kbar. This kink at P_c corresponds to a dramatic change in the magnitude of the ordered moment as determined through neutron diffraction, where the magnetic moment above P_c is consistent with bulk antiferromagnetism (AFM). With increasing pressure, the superconducting critical temperature T_c was smoothly and monotonically suppressed towards zero temperature near 15 kbar, or P_c. The coincidence of the disappearance of superconductivity and the kink in T_0, possibly indicative of a crossover from a HO state to an AFM state, suggested that the HO and superconducting states were in competition. Previous specific heat measurements suggested the HO state partially gapped a portion of the Fermi surface (FS). From this, we analyzed the transition temperatures of the ordered states of URu_2Si_2 in the context of a competition for FS fraction. Through this analysis, we found that the increase in T_0 corresponds to an increase in the portion of FS gapped by the HO transition. The increase in this gapped portion of the FS leaves fewer electrons to undergo pairing into the superconducting state. Our analysis agrees extremely well with previous specific heat studies under pressure, and indicates that the HO transition fully gaps its portion of the FS near P_c.

By fitting the electrical resistivity to a form including scattering from gapped spin excitations, the magnitude of a gap in the spin-excitation spectrum could be quantified. We found that the magnitude of this gap changes near P_c. Furthermore, the height of the resistive anomaly associated with the HO state decreases with increasing pressure up to P_c, after which it remains roughly constant. This behavior is consistent with the gapping of the FS, indicated by our previous analysis. As the HO state gaps more of the FS, there are fewer states into which quasiparticles can scatter, thus reducing the magnitude of the resistivity. Above P_c, when the HO state has fully gapped its portion of the FS, the number of states into which quasiparticles can scatter is pressure-independent, yielding a pressure-independent value for the magnitude of the resistive anomaly.

The presence of a gap in the spin-excitation spectrum and a gap at the FS strongly suggests that a SDW-like instability occurs at the HO transition temperature T_0. The onset of bulk AFM above P_c is intriguing and could be explained in a SDW scenario where a SDW instability induces local ordering.

b. $URu_{2-x}Re_xSi_2$

The Re-substituted URu_2Si_2 system, $URu_{2-x}Re_xSi_2$, provides a unique opportunity to examine the pressure dependence of a HO state whose ambient pressure transition temperature and associated correlations are suppressed with increasing x. Using high-quality single crystals, we measured several compositions of $URu_{2-x}Re_xSi_2$ under pressure. The qualitative trough-and-peak structure of the HO transition in the pure compound persisted with increasing Re-content, although the absolute value of the resistivity changed due to impurity effects. With applied pressure, the HO transition temperature T_0 exhibited a kink in its pressure dependence at P_c = 15 kbar, identical to the pure compound, for all values of x. This persistence of the value of P_c with Re-content suggests that Re-substitution does little to affect the crossover from the HO state to the bulk AFM state.

c. Testing the hydrostaticity of the pressure medium

During our hydrostatic pressure measurements on URu_2Si_2, it became apparent that the nature of the hydrostatic pressure medium had dramatic effects on the reliability of the study. Because they freeze under fairly low pressure, we were forced to abandon the popular Fluorinert liquids in favor of a mixture of isoamyl alcohol and n-pentane, which remains hydrostatic to 30 kbar. We have completed a comparison of electrical transport measurements of the URu_2Si_2 HO transition performed in different pressure media and are planning a complementary study of the superconducting transition. These experiments may shed light on the disagreement in values of P_c reported recently.

IV. The single elements: pushing to higher pressures

a. Thorium and the new MP35N gaskets

Elemental thorium is a conventional superconductor with an ambient pressure critical temperature T_c = 1.4 K. Previous pressure-dependent measurements revealed a dramatic decrease in T_c with applied pressure; however, above approximately 100 kbar, the value of T_c remained roughly constant, showing a flat pressure dependence up to nearly 160 kbar. Using high-quality, single crystals, we have investigated the superconducting state of thorium up to high pressures. This work was performed using a beryllium-copper diamond anvil cell

(DAC) from our facility. We used a designer diamond anvil equipped with microprobes for electrical resistivity measurements, obtained through our collaborations with Dr. S. T. Weir of Lawrence Livermore National Laboratory and Dr. Y. K. Vohra of the University of Alabama, Birmingham. The thorium sample was mounted within a beryllium-copper gasket along with a ruby manometer. Ultra-high pressure measurements at very low temperature were performed in the Kelvinox MX-100 ^{3}He – ^{4}He dilution refrigerator within our facility.

The critical temperature was tracked up to pressures near 400 kbar, approaching the limit of the beryllium-copper gasket. The pressure-dependent evolution of the superconducting state was similar to previously reported results: T_c decreased with increasing pressure—although not as steeply as the previous results, possibly due to pressure gradients arising from a lack of a pressure-transmitting media; above approximately 100 kbar, T_c (P) flattened and exhibited little pressure dependence out to nearly 400 kbar. In addition, measurements of the critical field curves were attempted and showed little change at high pressures, consistent with the weak pressure dependence of T_c. Attempts to measure the superconducting upper critical field of Th at low temperatures yielded unreasonably large values, presumably due to magnetic shielding of the small sample by the large metallic gasket. Magnetization studies of spring steel, which we originally intended to use as a gasket to achieve higher pressures, indicated that the material is magnetic at low temperatures. We have opted instead to use the nonferrous alloy MP35N, which will allow us to extend our pressure range to 1 Mbar, and may allow for the accurate measurement of the upper critical field to observe whether it changes even while the transition temperature stays constant.

V. Other ongoing collaborations

Ongoing projects include: the study of Yb-based heavy fermion compounds using DACs; studies of Bi-based high temperature superconductors, utilizing Bridgman and DAC pressure cells, in collaboration with Professor Zhi-Xun Shen (Stanford University) and Dr. Tanja Cuk (Currently a Miller Fellow, UC Berkeley and Lawrence Berkeley National Laboratory); studies of filled skutterudite compounds in collaboration with Professor Zygmunt Henkie (Institute of Low Temperature and Structure Research, Polish Academy of Science, Wroclaw, Poland).

PROJECT PARTICIPANTS

Faculty

Name: M. Brian Maple

Percent Contribution: 10%

Contribution to Project: Research group leader and Principal Investigator.

Graduate Students

Name: Nicholas P. Butch

Percent Contribution: 100%

Contribution to Project: Prepares intermetallic samples and performs measurements of magnetic and transport properties of f-electron materials including high pressure measurements.

Name: Diego A. Zocco

Percent Contribution: 100%

Contribution to Project: Performs high pressure electrical resistivity and susceptibility measurements and prepares intermetallic samples.

Undergraduate Lab Assistants

Name: Colin McElroy

Percent Contribution: 25%

Contribution to Project: Prepares intermetallic f-electron samples and assists in electrical resistivity, magnetization, and specific heat measurements to characterize materials with potentially interesting high pressure properties.

PUBLICATIONS

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- D. A. Zocco, J. J. Hamlin, T. A. Sayles and M. B. Maple, “High pressure studies on $CeTe_3$” (in preparation)

Ph.D. THESES COMPLETED WITH SUPPORT PROVIDED BY THIS GRANT

- J. R. Jeffries, “Correlated Electronic States Under Extreme Conditions,” (2007). ***Current position: postdoctoral research physicist at Lawrence Livermore National Laboratory.**

- N. P. Butch, “The Search for Quantum Criticality near the Convergence of Hidden Order and Ferromagnetism,” (2008).

- T. A. Sayles, “Magnetism and Superconductivity in Pr-based Filled Skutterudite Arsenides,” (2008).

ABSTRACTS

- S. Francoual, N. Harrison, M. Jaime, S. Baily, A. Lacerda, N. P. Butch, and M. B. Maple, “Effects of Rhenium Doping on the High Magnetic Field versus Temperature Phase Diagram of URu_2Si_2,” *Bull. Am. Phys. Soc.* **51**, 574 (2007).

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INVITED PRESENTATIONS

- M. B. Maple, “Novel types of superconductivity in f-electron materials,” Conferment of the Honorary Professorship of the W. Trzebiatowski Institute for Low Temperature and Structure Research, Polish Academy of Sciences, Wroclaw, Poland, September 6, 2006.

- M. B. Maple, “Strongly correlated electron phenomena in filled skutterudite lanthanide osmium antimonides,” 6th International Conference on f-elements, Wroclaw, Poland, September 8, 2006.

- M. B. Maple, “Tuning of hidden order and superconductivity in URu_2Si_2 by applied pressure and Re doping,” Fall MRS’06 Actinides III Symposium, Boston, Massachusetts, November 27, 2006

- M. B. Maple, “Experimental investigation of magnetic, superconducting, and other phase transitions in novel f-electron materials at ultrahigh pressures,” National Nuclear Security Administration Stewardship Science Academic Alliance Symposium, Washington, D.C., February 5-7, 2007.

- J. R. Jeffries, “Competing Ordered Phases in URu_2Si_2,” Arete Associates, Thousand Oaks, CA, March 16, 2007.

- N. P. Butch, “Probing the Unusual Properties of URu_2Si_2 via Applied Pressure and Re Substitution,” Los Alamos National Laboratory, Los Alamos, NM, March 23, 2007.

- J. R. Jeffries, “Competing Ordered Phases in URu_2Si_2: Pressure and Substitution,” Lawrence Livermore National Laboratory, Livermore, CA, March 27, 2007.

- J. R. Jeffries, “Competing Ordered Phases in URu_2Si_2: Pressure and Substitution,” Sandia National Laboratory, Livermore, CA, May 9, 2007.

- J. R. Jeffries, “Competing Ordered Phases in URu_2Si_2: Pressure and Substitution,” Stanford University, Stanford, CA, July 11, 2007.

- N. P. Butch, “Experimental Investigation of Magnetic, Superconducting, and Other Phase Transitions in Novel f-electron Materials at Ultrahigh Pressures,” National Nuclear Security Administration Stewardship Science Academic Alliance Symposium, Washington, D.C., February 26-28, 2008.

POSTER SESSIONS

- J. R. Jeffries, N. P. Butch, D. D. Jackson, S. T. Weir, Y. K. Vohra, and M. B. Maple, “Evolution of Ordered States under Pressure in *f*- and *d*-electron Systems,” Poster Session: National Nuclear Security Administration Stewardship Science Academic Alliances Symposium, Washington, D. C., February 5-7, 2007.

- D. A. Zocco, N. P. Butch, J. R. Jeffries, J. J. Hamlin, and M. B. Maple, “Pressure dependence of electronic ground states in *f*-electron materials,” Poster Session: National Nuclear Security Administration Stewardship Science Academic Alliances Symposium, Washington, D. C., February 26-28, 2008.