

SUPERCONDUCTIVITY IN TERNARY COMPOUNDS AT HIGH PRESSURE

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ABSTRACT

Present techniques allow routine measurements such as resistivity and susceptibility as a function of pressure with nearly the same ease as more traditional variables such as temperature and magnetic field. The combination of the ability to choose and tailor ternary materials with a variety of physical and electronic properties and the sensitivity of a simple measurable parameter such as the superconducting transition temperature (T_c) to various and subtle properties of materials, provides a powerful and sensitive laboratory. Indeed, the variety of materials and techniques available provides one of the major experimental problems: what to do first. It seems fitting then to summarize work done over the last decade in this area and to point out some directions for future work in the field.

INTRODUCTION

The effect of high pressure on T_c continues to play an important role in the understanding of superconductivity. The general decrease in T_c with pressure observed in the non-transition element superconductors has been generally attributed to the stiffening of the lattice with applied pressure¹ while the distribution of positive and negative pressure effects in the superconducting transition elements is generally attributed to electronic effects.² In those cases where non-linearities are observed in the elements (Re, U, La) various effects such as structural transformations, Fermi-surface topology and competitive phenomena, such as with spin density waves, have been invoked as possible explanations.² With few exceptions, however, the pressure effects on the elements and binary compounds and alloys are nearly linear, and small (usually $|dT_c/dp| \sim 10^{-5}$ K/bar).

The diversity of observed effects has grown as the complexity of the materials studied has grown. The advent of important ternary materials about ten years ago with the discovery of superconductivity in the Chevrel phase materials³ and the subsequent explosion in known ternary compounds⁴ combined with the now common-place hydrostatic high pressure techniques (to 25 kbar) has provided a tremendous laboratory for the study not only of superconductivity, but other fundamental physical properties of materials such as structural transformations and electronic instabilities. The basic crystal structure of many ternary systems can be retained even when one or more of the constituent elements is replaced, either partially or completely. For example, over a hundred ternary molybdenum chalcogenide⁵ (Chevrel phase) compounds can be formed with the stoichiometry $R_xMo_6X_8$. Within this class of materials examples of normal metals, superconductors, semiconductors, antiferromagnets and ferromagnets can be found with only minor variations in structure.

CHEVREL PHASE

The first ternary compounds for which extensive results under pressure were obtained were the Chevrel phase materials.^{6,7} The basic crystal

structure consists of clusters of Mo_6X_8 with $\text{X}=\text{S}, \text{Se}$ or Te , in a distorted cubic structure with the third element occupying sites between the clusters.⁵ The small cations such as Cu statistically occupy one of two six-fold sites giving such crystals an inherent disorder, at least at higher temperatures. The larger cations such as Pb or the rare-earth elements occupy unique sites between the Mo_6X_8 clusters. Superconductivity, when present in these materials, is attributed to the Mo clusters and the principle role of the third element is to "tune" the structure and electronic properties. A common feature in this structure is a distortion at lower temperatures, lowering the overall crystal symmetry from rhombohedral at high temperatures to triclinic at low temperatures. Even when this transformation is suppressed, however, the lattice remains soft, characterized by a low Debye temperature and pressure effects remain large throughout this class of materials.

Figure 1 shows T_C vs pressure for several ternary molybdenum sulfides. The pressure effects are large, exhibiting changes in T_C with pressure in excess of 1 K in many cases and T_C both rises and falls in different materials and even in the same material in several cases. The nonlinearities observed in Cu , Cd , Sn and Zn have been associated with structural instabilities induced either by pressure or composition. The data for Pb and Sn demonstrate the pressure effects may be strongly sample dependent. Recent studies indicate the presence of oxygen can strongly affect the superconducting properties of many Chevrel phase materials.⁸ This may explain the sample variability evident here.

Figure 2 shows T_C vs pressure for $\text{Cu}_x\text{Mo}_3\text{S}_4$ for various values of x (Ref. 9). Three distinct phases are evident in this figure: (I) a low x ($x < 1.45$), high T_C (10.5 K) phase; (II) an intermediate x ($1.45 < x < 1.60$), T_C (6.5 K) phase; and (III) a high x ($x > 1.60$), low T_C (4.5 K) phase.

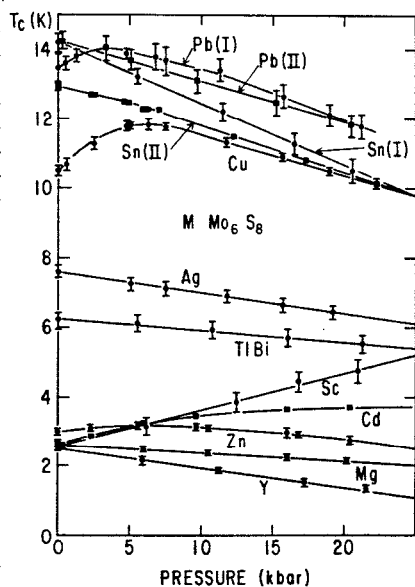


FIG. 1. T_C vs pressure for twelve ternary molybdenum sulfides.

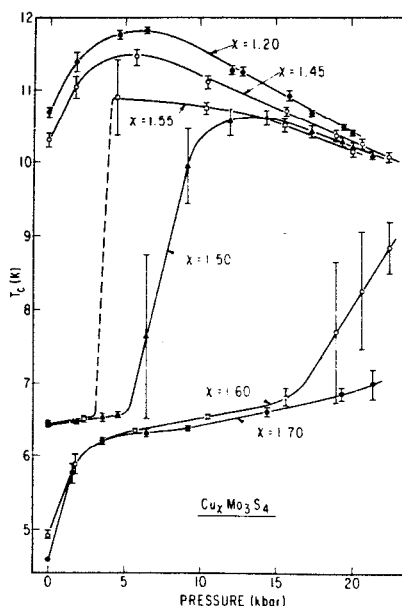


FIG. 2. T_C vs pressure for six $\text{Cu}_x\text{Mo}_3\text{S}_4$ compounds.

An increase in pressure or a decrease in x drives the system to a higher T_C phase. Independent structural data confirm this picture: application of pressure to low T_C phase III induces a structural transformation to phase II, which is superconducting near 6.5 K. If pressure is applied to a phase II sample, a transformation to the high T_C , phase I, material can be induced. Sufficient pressure, then, applied to a Cu-rich sample should induce the transformation sequence phase III-phase II-phase I, with the associated changes in the superconducting transition temperature. It was the peculiar behavior of T_C with pressure in this material which led to the elucidation of the structural transformations, which are rather subtle in this compound. Subsequently structural instabilities have been found in a number of the ternary molybdenum chalcogenides, including Ag, Zn, Cd, Eu, Ba, Sr, and possibly Pb.¹⁰

Related phenomena have been observed in the ternary molybdenum selenides⁶ as shown in Fig. 3. The spectacular enhancement of T_C in the Ag ternary shown in this figure is unusual, and very likely similar to the documented cases of $Cu_xMo_3S_4$ and $Cu_xMo_3Se_4$ (Ref. 11). The various pressure effects observed in the molybdenum chalcogenides are summarized in Fig. 4 where dT_C/dp is plotted vs T_C . It should be pointed out that the two samples of Sn and Pb each with different values of dT_C/dp may serve as an estimate of the variability possible with different sample preparation conditions. In those samples for which non-linear pressure dependences have been observed, only the slope at high pressure ($p > 10$ kbar) has been plotted. The overall scale is noteworthy, indicating pressure dependencies an order of magnitude greater than typically observed in the elements and binary compounds and the occurrence of both positive and negative values for dT_C/dp .

The solid line through several of the sulfides is drawn as a guide to the eye. While not rigorous, it is clear that for at least some compounds in this class dT_C/dp is nearly proportional to T_C . This trend is predicted by some theories which attempt to describe the effect of changes in the topology of the Fermi surface with pressure.¹² In this case the Fermi surface changes are presumably driven by the underlying structural transformation or instability. This should supply information about how the Fermi surface is changing which can presumably be checked by band structure calculations in the various symmetries involved. No compound falls far above this line in the dT_C/dp plane which suggests higher T_C 's could only be obtained in this class at the expense of even more strongly negative pressure effects.

The strong positive pressure dependence observed in $ScMo_6S_8$ can be expected to become smaller at higher pressures. It would be interesting to know if the slope at high pressure remains positive and falls near the solid line in Fig. 4 or becomes negative and falls near the cluster of rare-earth ternary molybdenum sulfides with dT_C/dp near -5×10^{-5} K/bar. The remarkable negative pressure dependence in $YbMo_6S_8$, with dT_C/dp almost twice as large as any other material in its class, is not well understood at present. One suspects mixed valence effects in this case, however a recent study suggests Yb is divalent in this compound.¹³

The question of superconductivity in the compound $EuMo_6S_8$ has been a source of considerable debate in recent years.^{14,15} Figure 5 provides a summary of the T_C vs pressure data available to date. The non-bulk nature of the superconductivity as evidenced by the incomplete Meissner effect is now well established. Sample preparation also plays a role as the behavior shown here is not observed in all samples. When observed, superconductivity has an onset near 7-10 kbar to a transition temperature of about 10 K. Various explanations have been put forth for this behavior, none of which has been completely satisfactory. A new possibility is superconductivity

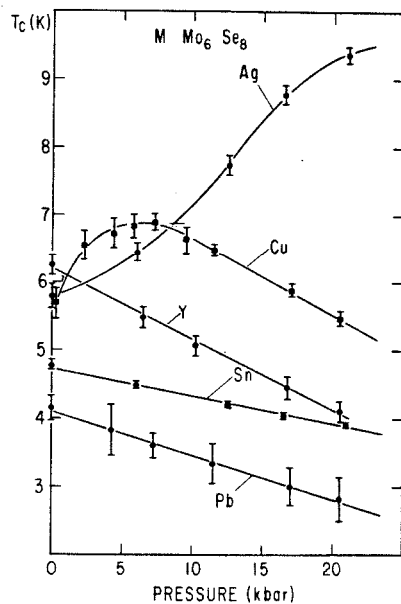


FIG. 3. T_C vs pressure for five ternary molybdenum selenides. Notice the spectacular behavior of $AgMo_6Se_8$.

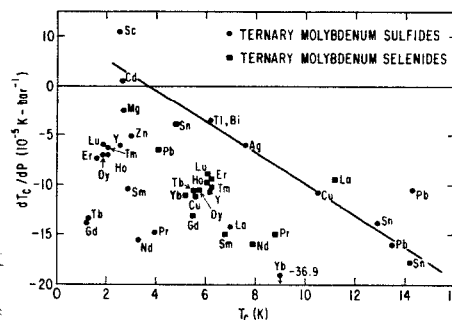


FIG. 4. dT_C/dp vs T_C for the known ternary molybdenum chalcogenides. The solid line, drawn as a guide for the eye, suggests a linear relationship between dT_C/dp and T_C (see text for details).

centered about trivalent Lanthanide impurities, common in Eu, which can percolate through the lattice when the host matrix undergoes a pressure induced triclinic to rhombohedral lattice transformation.¹⁶ This view is supported by the observation of a minute Meissner effect at 7 K and ambient pressure in two samples.¹⁵ Whatever the final resolution of this question, pressure will continue to play an essential role. Early results¹⁷ on $M Mo_6S_8$, with $M=Ba, Sr,$ and Ce , suggest similar possibilities in these materials.

TERNARY RARE-EARTH TRANSITION METAL BORIDES

There are now four distinct structures which are known to form with the stoichiometry MT_4B_4 ($T = Rh, Ru$ for example), the best known of which is the $CeCo_4B_4$ -type structure.⁴ Figure 6 indicates the pressure dependence of T_C for four members of this structure type, including $ErRh_4B_4$ the well-known reentrant superconductor.¹⁸ The cause of the nonlinearity in the pressure dependence of T_C in $LuRh_4B_4$ and YRh_4B_4 below 6 kbar is not known at this time; however, unlike the Chevrel phase compounds discussed above there are no known structural instabilities of any kind for this structure type. It may well be, then, that this anomaly is purely electronic in origin, resulting from the movement of some Rh d-like level through the Fermi level. Similar Fermi surface effects are believed responsible for non-linearities in the pressure dependence of T_C in Re.² The magnitude of this effect in $LuRh_4B_4$ and YRh_4B_4 should allow an estimate of the energy difference between the energy level involved and the Fermi level, providing a check on band structure calculations. Subtle features such as this are not easily discernable by other techniques.

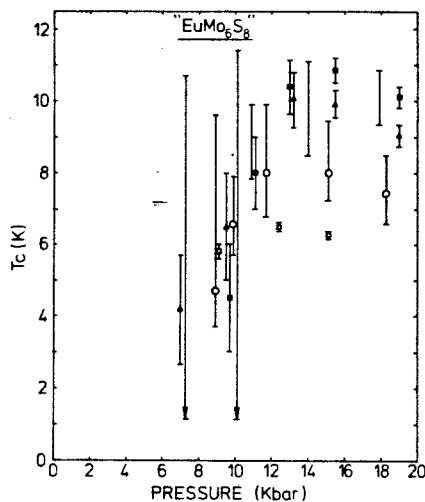


FIG. 5. T_c vs pressure for four samples of EuMo_6S_8 . Superconductivity is not observed in all samples studied.

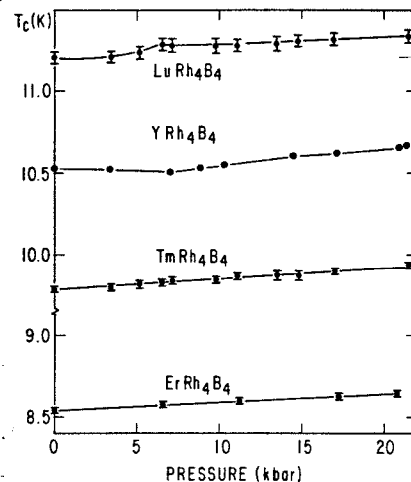


FIG. 6. T_c vs pressure for four ternary rare earth rhodium borides. All the ternary transition metal borides exhibit linear pressure dependencies except LuRh_4B_4 and YRh_4B_4 , shown here.

The Gd, Tb, Dy and Ho rhodium borides order ferromagnetically, but the ordering temperatures do not follow the usual de Gennes relation for magnetic ordering temperatures which should peak at Gd, instead of the observed peak at Dy (Fig. 7). Attempts to explain the magnetic ordering temperatures by including crystal field effects,¹⁹ while an important step forward, have not been entirely successful, predicting a maximum ordering temperature at Tb and not the observed Dy. The maximum in dT_M/dp at Tb distinctly violates the expected proportionality between dT_M/dp and T_M .²⁰ This implies the presently unknown contributions to the magnetic ordering temperature which do not originate with the RKKY indirect exchange interaction or crystal field effects are only weakly pressure dependent.

A second MT_4B_4 crystal structure, which is even more favorable to superconductivity in some ways than the CeCo_4B_4 -type discussed above, is the LuRu_4B_4 bct structure.²¹ The pressure derivatives are summarized in Fig. 8 for all the ternary borides where dT_c/dp is plotted against T_c . As in the CeCo_4B_4 -type compounds, the pressure dependencies of the LuRu_4B_4 -type compounds, including the $\text{X}(\text{Rh}_{0.85}\text{Ru}_{0.15})_4\text{B}_4$ compounds are of order 10^{-5} K/bar (Fig. 9) and exhibit both positive and negative values. The correlation between dT_c/dp and T_c is even more apparent here than in the Chevrel phase compounds where dT_c/dp is proportional to T_c for only a few of the compounds. In these two structure types, only one compound, $\text{Pr}(\text{Rh}_{0.85}\text{Ru}_{0.15})_4\text{B}_4$, is a major exception to the linear relation between dT_c/dp and T_c within a structure class.

The data displayed in Fig. 8 are remarkable in several respects. In Fig. 4, the variation of T_c for the various Chevrel phase materials can be understood in terms of the changes in electronic and structural properties from material to material. In Fig. 8, however, the major source of the variation of T_c is not structural or electronic in origin, but is due to the magnetic properties of the various rare-earths. The behavior of LuRu_4B_4

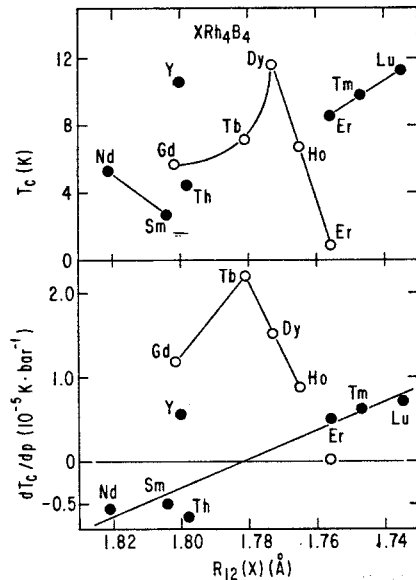


FIG. 7. T_C and dT_C/dp vs twelve-coordinate metallic radius of the rare-earth for the ternary rare-earth rhodium borides. Open symbols (○) refer to magnetic transitions and closed symbols (●) refer to superconducting transitions.

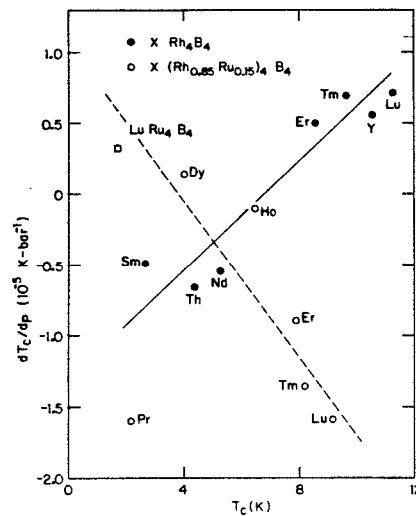


FIG. 8. dT_C/dp vs T_C for the ternary rare earth transition metal borides. The solid symbols (●) and solid line refer to the $CeCo_4B_4$ -type structure and the open symbols (○) and dashed line refer to the $LuRu_4B_4$ -type structure.

under pressure is consistent with the behavior of the other bct ternary borides, $X(Rh_{0.85}Ru_{0.15})_4B_4$, in spite of the difference in the valence of the transition metal. Finally, the linear relation between dT_C/dp and T_C for the two classes of materials in Fig. 8 is similar to that observed in Fig. 4 in spite of the order of magnitude difference in the pressure effects in the various classes of materials. Together these data suggest the linear relation between dT_C/dp and T_C is a rather universal first approximation and is independent of magnetic, electronic or structural contributions to T_C .

Only three other pseudoternary systems have been investigated to date: $(Er_{1-x}Gd_x)Rh_4B_4$ (Ref. 22), $(Y_{1-x}Th_x)Rh_4B_4$ (Ref. 23) and $(Er_{1-x}Ho_x)Rh_4B_4$ (Ref. 24). The second study indicated that the pressure effects are non-linear in YRh_4B_4 as well as the previously known non-linear pressure effects in $LuRh_4B_4$, the two highest T_C 's among the rhodium borides. In addition the non-linearities disappear in a complex fashion as Th is substituted for Y. In the third system superconductivity persists up to about $x=0.89$. At higher Ho concentrations only magnetism is observed. In this system both T_C and T_M are enhanced with pressure, T_M experiencing the greater enhancement such that at sufficiently high pressures magnetic order completely dominates (Fig. 9). By initially tuning the material through selecting a composition near the critical point in the phase diagram and then using pressure for the fine adjustments, various phase boundaries between superconducting, magnetic and coexistence regions can be delineated with reasonably high confidence, as shown in Fig. 10. The observation that dT_C/dp for $p < p_C$ and dT_M/dp for $p > p_C$ are similar

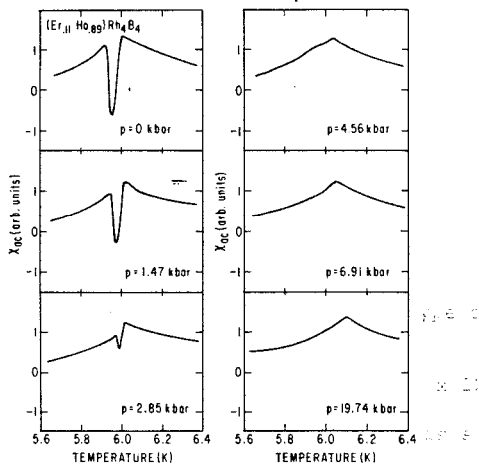


FIG. 9. X_{AC} vs temperature for six pressures for $(Er_{1-x}Ho_x)Rh_4B_4$ illustrating the quenching of the superconducting transition at high pressure.

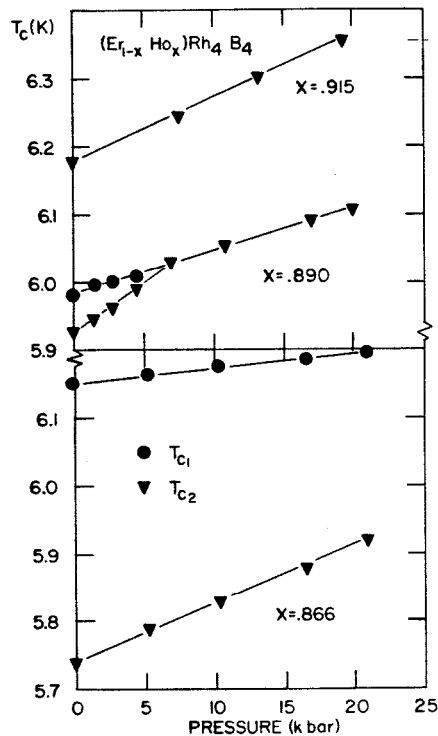


FIG. 10. Critical temperatures vs pressure for three compositions of $(Er_{1-x}Ho_x)Rh_4B_4$. Solid circles refer to superconducting transitions (\bullet) and solid triangles (\blacktriangledown) refer to magnetic transitions.

may simply reflect the influence of the density of states on both T_C and T_M . The reason for the change in dT_M/dp at p_C is not well understood, but this change is also evident in T_M vs composition studies. This is a largely untapped method for testing various theories dealing with magnetic superconductors. It should be pointed out that all of the high pressure studies to date, with the exception of some studies¹⁴ on $EuMo_6S_8$, have determined only T_C as a function of pressure. Studies of critical fields under pressure would be particularly useful at this point.

TERNARY IRON SILICIDES

The discovery of superconductivity in the $RE_2Fe_3Si_5$ (RE=Lu, Sc and Y) compounds²⁵ was surprising, principally because of the presence of the 3-d element, Fe, which is rarely found in superconductors. The dramatic effects of pressure and alloying on T_C in these materials²⁶ are shown in Fig. 11 and 12. Preliminary low temperature X-ray, neutron diffraction and resistivity studies do not suggest any structural instabilities,²⁷ as in several other superconductors with dramatic pressure effects. Recent heat capacity studies (Fig. 13) indicate an unusual linear term in the specific heat²⁷ below T_C , instead of the expected nearly exponential behavior. This has been attributed to a topologically complex Fermi surface in which a significant fraction of the entire surface (36%) does not participate in the superconductivity, but remains normal instead. In light of the previous

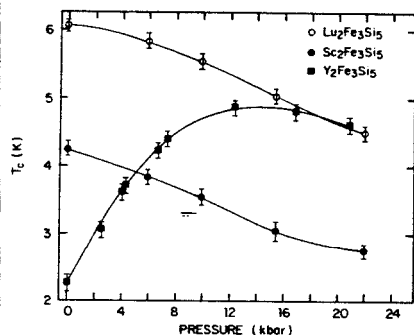


FIG. 11. T_c vs pressure for $\text{Lu}_2\text{Fe}_3\text{Si}_5$, $\text{Sc}_2\text{Fe}_3\text{Si}_5$ and $\text{Y}_2\text{Fe}_3\text{Si}_5$. (Ref. 26)

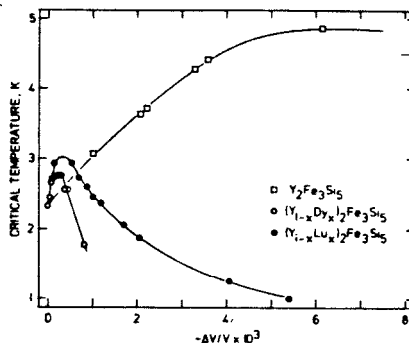


FIG. 12. T_c vs volume change. A bulk modulus of 2000 kbar was assumed to convert the pressure data on $\text{Y}_2\text{Fe}_3\text{Si}_5$ to the physically more relevant parameter, volume. (Ref. 26)

discussion this does not seem so unreasonable, as non-linearities can be generally associated with a rapidly changing Fermi surface, whether driven by structural changes or by the presence of bands near the Fermi level.

CeCu_2Si_2

Figure 14 shows T_c vs pressure for a single crystal²⁸ of CeCu_2Si_2 . This system has been reported to be a 'heavy fermion' superconductor, due to the apparent loss of resistivity and a linear term in the specific heat of about 1 J/mole.²⁹ Polycrystalline samples exhibit a T_c of about 0.5 K, while at ambient pressures single crystals are not superconducting. Recent experiments indicate antiferromagnetism in several closely related compounds and other investigations suggest antiferromagnetism may be responsible for the effects seen here also.³⁰ High pressure work is certain to play a major role in the understanding of the low temperature behavior in this compound, regardless of the nature of the phenomenon.

SUMMARY

Pressure work has led to a closer examination of structural instabilities in the Chevrel phase materials, and indeed provided the first indications that such instabilities existed. Features of the Fermi surface topology have been revealed in the superconducting ternary borides and silicides, which ultimately must be correlated with the band structure of these materials. The present understanding that large pressure effects and non-linearities, observed in each of the ternary systems investigated to date, can be attributed to changes in the electronic structure of the material is not entirely new but is confirmed in the ternary systems to a degree not seen in the superconducting elements and binary compounds. The complex electronic and crystallographic structure inherent in these compounds arises in a very natural way from the dominant role of the transition element, Mo, Rh, Ru, or Fe for example. Other ternary systems such as $\text{Sc}_5\text{Co}_4\text{Si}_{10}$ and $\text{LaFe}_4\text{P}_{12}$ with the dominant 3d transition elements might also be expected to have large pressure effects. If a ternary transition metal compound is superconducting, the Fermi surface is likely dominated by d-type bands. A complex Fermi surface can then be expected along with large and non-linear pressure effects. As a means of gaining a better understanding of the

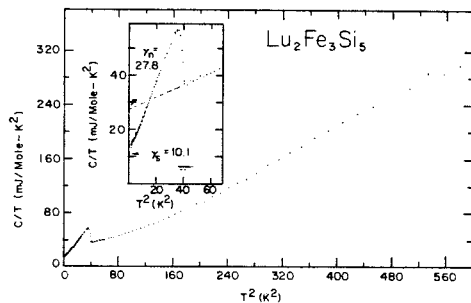


FIG. 13. The low temperature heat capacity of $\text{Lu}_2\text{Fe}_3\text{Si}_5$. The linear term below $T_C=6$ K indicates a large fraction (36%) of the conduction electrons do not form Cooper pairs.

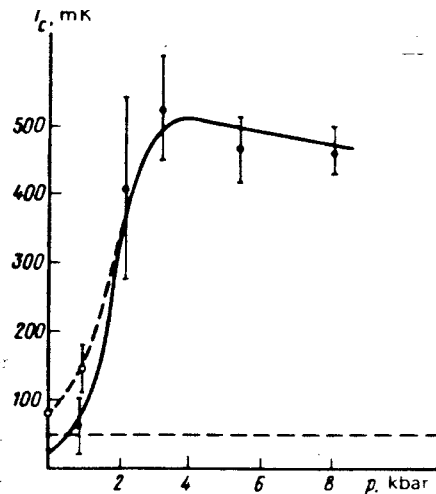


FIG. 14. T_C vs pressure for a single crystal of CeCu_2Si_2 . (Ref. 28)

competition between superconductivity and magnetism, the use of pressure has, unfortunately, remained almost completely undeveloped.

*Operated for the USDOE by Iowa State University of under contract No. W-7405-Eng-82. This work was supported by the Director for Energy Research, Office of Basic Energy Sciences.

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