NEUTRON-SCATTERING STUDIES OF MAGNETIC SUPERCONDUCTORS

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Running Title: Neutron Scattering in Magnetic Superconductors

By

S. K. Sinha, G. W. Crabtree and D. G. Hinks
Argonne National Laboratory, * Argonne, Illinois 60439

and

H. A. Mook and O. A. Pringle
Oak Ridge National Laboratory, Oak Ridge, Tennessee 37830

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Argonne National Laboratory,* Argonne, Illinois 60439

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H. A. Mook and O. A. Pringle

Oak Ridge National Laboratory,† Oak Ridge, Tennessee 37830

Abstract

Results obtained in the last few years obtained by neutron diffraction on the nature of the magnetic ordering in magnetic superconductors are reviewed. Emphasis is given to studies of the complex intermediate phase in ferromagnetic superconductors where both superconductivity and ferromagnetism appear to coexist.

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In the last few years, our understanding of the details of the interaction between magnetism and superconductivity in ternary compounds has greatly increased due to much experimental and theoretical work. Neutron diffraction has proved to be an extremely useful tool in elucidating the behavior of the magnetic order parameter, particularly in the case of non-uniform states of magnetization, and has thus played a vital role in testing the applicability of the various theories. We will not dwell here on the historical development of the field, as there exists by now a vast body of literature on the subject, as well as several review articles [1]. Instead we shall in this paper simply review the information about magnetic superconductors that neutron diffraction has made available, and attempt to evaluate where present theories stand in relation to this data.

The earliest neutron diffraction investigations of superconductors containing magnetic ions appeared to confirm the belief that long-range magnetic order could not coexist with superconductivity. This belief was based on the notion that pair-breaking due to spin flip scattering of the conduction electrons by the magnetic ions would destroy superconductivity [2]. Thus, for superconductors which showed a tendency to magnetic ordering, such as $(\text{Ce}_{1-x}\text{RE}_x)\text{Ru}_2$ [3], neutron diffraction studies showed only short-range magnetic order. However, with the (almost simultaneous) emergence of the ternary superconductors of the form $(\text{RE})\text{Mo}_6\text{Se}_8$, and $(\text{RE})\text{Rh}_4\text{B}_4$, containing complete lattices of magnetic (RE) ions, true long range magnetic order was observed.

We consider first the antiferromagnetic superconductors [3]. In general, antiferromagnetic long-range order does not destroy superconductivity and the two persist down to the lowest temperatures, sometimes with more than one antiferromagnetic phase occurring. This is in accordance with the theoretical expectation that a uniform Meissner state is not precluded by an antiferromagnetic spin alignment. Of the Chevrel phase compounds, $\text{Gd}_{1.2}\text{Mo}_6\text{S}_8$, $\text{Er}_{1.2}\text{Mo}_6\text{S}_8$, $\text{Dy}_{1.2}\text{Mo}_6\text{S}_8$ and $\text{Tb}_{1.2}\text{Mo}_6\text{S}_8$ show anomalies in their upper critical field H_{c2} , resistivity and magnetic susceptibility [4] which suggest antiferromagnetic ordering in the superconducting state. (The extra 20% of rare earth ions are now believed to go into another phase where they combine with excess oxygen, rather than stay as interstitials in the Chevrel phase itself [5].) While the magnetic powder diffraction pattern of $\text{Er}_{1.2}\text{Mo}_6\text{S}_8$ shows a complex structure which has not been completely solved (owing perhaps to sample purity problems), neutron diffraction has yielded clear results for the magnetic structure of $\text{Dy}_{1.2}\text{Mo}_6\text{S}_8$ and $\text{Tb}_{1.2}\text{Mo}_6\text{S}_8$. The magnetic superlattice peaks which develop below $T_M = 1.05$ K are easily indexed with half-integer values, corresponding to an antiferromagnetic structure consisting of alternate (100) planes with opposite moments. Intensities of the various magnetic reflections were consistent with a moment (at $T = 0.07$ K) at the Tb sites of $8.28 \pm 0.2 \mu_B$ pointed along the unique rhombohedral axis (i.e., the [111] direction). Similar results were found for $\text{Dy}_{1.2}\text{Mo}_6\text{S}_8$. The widths of the magnetic peaks were not broadened beyond instrumental resolution implying a lower limit of ~ 300 Å for the magnetic correlation length, which requires long-

range magnetic order. The ordered moments are not equal to the free ion values, showing the importance of crystal field effects. Of the compounds of the form (RE) Rh_4B_4 , NdRh_4B_4 , SmRh_4B_4 and TmRh_4B_4 become antiferromagnetic while still superconducting at the lowest temperatures. TmRh_4B_4 was believed, from magnetization measurements [6] to behave like ErRh_4B_4 , i.e., to reenter, at lower temperatures, a normal ferromagnetic phase. This behavior has now been elucidated by neutron diffraction measurements [7] which show that the zero-field phase is indeed a sinusoidal antiferromagnetic structure of the Tm moments, propagating along the [101] axis. The Tm moments are aligned along the [010] direction. There is a certain similarity here with the modulated magnetic structure observed as the intermediate phase in ErRh_4B_4 , which will be discussed later. In NdRh_4B_4 , powder diffraction measurements [8] have shown two distinct antiferromagnetic phases below the superconducting transition temperature at $T_c = 5.4$ K, one at $T_{N1} = 1.5$ K and the other at $T_{N2} = 1$ K. While HoRh_4B_4 and ErRh_4B_4 order ferromagnetically (as discussed below), substituting Ir for Rh dramatically decreases the ferromagnetic transition temperatures. Hamaker et al. [9] have studied, by powder diffraction, the compound $\text{Ho}(\text{Rh}_{0.3}\text{Ir}_{0.7})_4\text{B}_4$, where a continuous antiferromagnetic phase transition occurs below $T_N = 2.7$ K from a normal paramagnetic phase. The structure corresponds to a set of stacked antiferromagnetic sheets of basal plane spins which alternate in orientation as one proceeds along the c-axis, with the Ho moment having a value equal to almost the full free-ion value and oriented along the unique c-axis. Superconductivity then sets in at a lower temperature of

$T_c = 1.34$ K and coexists with the magnetic order for lower temperatures. The onset of superconductivity has no discernable effect on the magnetic ordering, which is estimated to be long-range.

An interesting aspect of the antiferromagnetic superconductors is the behavior of the magnetic order under an applied magnetic field. For $Tb_{1.2}Mo_6S_8$ [10] long-range ferromagnetic order is seen to develop for $H > H_{c2} = 1.9$ KOe. However, for $Dy_{1.2}Mo_6S_8$ a relatively small field of 200 Oe $\ll H_{c2}$ produces long-range ferromagnetic order, which appears to coexist with antiferromagnetic order. Similar effects have been seen in $TmRh_4B_4$ [8]. This appears to be an example of a forced-ferromagnetic transition, and has been discussed theoretically by Sakai et al. [11].

To summarize the situation with regard to the antiferromagnetic compounds, we can say that neutron diffraction has clearly established the coexistence of long-range antiferromagnetic order and superconductivity, and yielded detailed information regarding the magnetic order parameter. This information has been useful for theorists who have modelled the behavior of quantities such as the temperature dependence of H_{c2} , etc., for which the periodicity and temperature dependence of the magnetic order parameters are needed as inputs. Theoretical explanations of the magnetic structures themselves await a deeper level of microscopic theory and may well involve the existence of superconductivity. It appears that the pair-breaking mechanism in these materials, i.e., the exchange interaction between the f-shells and the superconducting electrons must, in any case, be small.

The coexistence of long-range ferromagnetic order with superconductivity is a more subtle and complex issue. Anderson and Suhl showed many years ago [12] that superconductivity would inhibit the occurrence of ferromagnetism driven by the RKKY interaction because the conduction electron generalized susceptibility $\chi(q)$ is drastically reduced at $\vec{q} = 0$. They proposed an alternate form of magnetic order ("cryptoferromagnetism") based on a long wavelength spiralling structure. Their argument ignores spin-orbit interaction and also assumes that the same set of electrons which transmits the RKKY interaction become superconducting, so the conclusions may not apply to all metals. However, a more basic reason for the lack of coexistence is the effect of ferromagnetism on the superconducting order parameter. Assuming for the moment that the exchange effects are small, consider the electromagnetic interaction. A ferromagnetic long-range ordering would set up a macroscopic $\vec{B}$ field inside which a uniform Meissner state cannot be maintained (unless there are strong surface-current effects). Thus, ferromagnetic systems ultimately become normal at low temperatures, i.e., a reentrant behavior is observed, as in the classic case of ErRh_4B_4 [13,14] (see Fig. 1). Particularly interesting, however, is the possibility of a coexistence phase before normal ferromagnetism is established. This may arise because the system compromises in one of two ways: (1) by maintaining a uniform superconducting order parameter while developing a long-wavelength sinusoidal or spiral moment structure (so that no macroscopic $\vec{B}$ -field exists, yet neighboring spins are still almost parallel to each other), or (2) by allowing flux lines to penetrate in the

form of a vortex lattice. Such coexistence phases were in fact predicted independently by several workers [15-19]. Subsequent neutron small-angle scattering measurements on polycrystalline samples of ErRh_4B_4 by Moncton et al. [20] showed a peak at $Q = 0.6 \text{ \AA}^{-1}$ which appeared just above the re-entrant transition temperature T_{C2} and disappeared at T_{C2} . The intensity of the peak appeared to show hysteresis in temperature which was inverse to that of the ferromagnetic intensity. The conclusion was that there was indeed a modulated-moment coexistence phase, but that it appears simultaneously with a normal ferromagnetic phase. Later, similar behavior was seen above the reentrant temperature of the ferromagnetic superconductor HoMo_6S_8 [21,22].

Subsequently, two single crystals of ErRh_4B_4 were grown at Argonne National Laboratory, on which detailed neutron diffraction and bulk resistivity and magnetization studies could be done. The neutron diffraction work was carried out at the High Flux Isotope Reactor at Oak Ridge National Laboratory. The results [23] are summarized in Figs. 2 and 3. At temperatures below $\approx 1.2 \text{ K}$, ferromagnetic intensity appears at the reciprocal lattice points corresponding to the Bragg reflection, but the crystal remains a bulk superconductor. At the same, or slightly lower, temperature magnetic satellites are also observed at $\vec{r} \pm \xi \vec{a}^* \pm n \vec{c}^*$, where $\vec{r}$ is the position of the Bragg reflection, and $\xi = 0.042$, and $n = 0.055$, as shown in Fig. 2. These transitions appear to be continuous, but the magnetic order parameters of both the modulated and ferromagnetic components increase in a very unusual manner. A careful search for harmonics of the satellite at

$T = 0.81$ K (where the satellite intensity is largest) revealed no peaks at displacements of $(2\xi, 0, 2\eta)$, $(3\xi, 0, 3\eta)$, $(3\xi, 0, \eta)$ or $(\xi, 0, 3\eta)$ from the Bragg peaks, to within 2% of the main satellite intensity.

The length of the $\vec{q}$ vector corresponds to a periodicity in real space of 91.8 ± 2.7 Å and showed only a very slight temperature dependence over most of the observed range, although it increased somewhat at the highest temperatures to 103.7 ± 3 Å (at 0.97 K). The intensity pattern of the satellite was consistent with a linear transversely plane-polarized sinusoidal spin structure with the moments oriented along a [100] axis. At 0.7 K, the satellites abruptly vanish, while the ferromagnetic intensity suddenly increases and simultaneously bulk resistivity reappears. At lower temperatures, the behavior appears to be that of a normal ferromagnet. On warming, the transition to the superconducting phase (zero resistivity) and the reappearance of the satellite intensity occur at 0.085 K, with an accompanying decrease of the ferromagnetic component. The sharpness of the transition and the accompanying hysteresis of ~ 50 mK in both the satellite intensity and the bulk dc resistivity indicates that this transition is of first order.

More recently, further high sensitivity neutron diffraction scans have been performed on the crystal used in the earlier diffraction studies. This was accomplished by changing the two-axis diffractometer on the HFIR Reactor to a three-axis system by installing a nearly perfect Ge analyzer. The fact that the sample crystal was nearly perfect then permitted very high resolution to be achieved if scans were made in such a way that only the scattering angle of the sample was changed in the scan.

This was accomplished by scanning in the a^* direction from near the (002) reflection, and in the c^* direction from near the (100) reflection. Figure 4 shows such a high resolution scan through the satellite ($0.048 a^*$, $0, 0.055 c^*$) from the (002) reciprocal lattice point at 0.8 K, just above the temperature where the normal ferromagnetic phase sets in upon cooling. The narrow width observed ($0.001 a^*$) is equal to the instrumental resolution. If we assume that broadening of half that amount is detectable, we conclude that the magnetic correlation length for the sinusoidal ordering (or domain size) is larger than $2 \times 10^3 \times a = 10,000 \text{ \AA}$. This shows that if modulated moment (i.e., superconducting) domains exist, they are too large for the thin domain walls between ferromagnetic regions.

The intermediate or coexistence phase is sensitive to small magnetic fields. Figure 5 shows the effect of a magnetic field on the satellites and the (101) peak intensities. The magnetic field was applied along the (vertical) [010] axis. As the field increases, the satellite positions are fairly insensitive to field, although it appears that the satellite wave-vector increases just before they disappear. The coexistence phase seems to disappear for fields above 0.05 Tesla. Figure 6 shows that the hysteresis vs. temperature is not as large for an applied field of 0.0242 Tesla, which reduces but does not completely remove the satellite. A field of 0.0722 Tesla is strong enough to completely destroy the modulated phase and little if any hysteresis is observed in the order parameter vs. temperature curve (Fig. 7).

Because of the very narrow mosaic spread of the crystal, the nuclear and ferromagnetic reflections are subject to a high degree of extinction although this is less severe for reflections such as the (101) which have a relatively small nuclear structure factor. However, more recent measurements with Porter wavelengths have confirmed the value of $\sim 5.6 \mu_B$ for the spontaneous ordered moment/Er atom in the ferromagnetic phase at the lowest temperature. A distribution of ferromagnetic domains between the two easy axes ([010] and [100]) has also been found unlike the earlier measurements which indicated all the domains were polarized along [010]. (The irregular cylindrical shape of the crystal and the consequent demagnetizing factors in fact make these two axes inequivalent.) The considerable decrease from the full free ion Er moment of $\sim 8.0 \mu_B$ could be attributed to crystal field effects, but is inconsistent with Mössbauer hyperfine field measurements of a nearly full Er moment down to the lowest temperatures. This discrepancy remains a puzzle at the present time.

To summarize the conclusions of our measurements on ErRh_4B_4 we may say that a highly interesting intermediate or coexistence phase is established on the way from superconductivity to ferromagnetism. This phase does indeed show a modulated moment as was predicted theoretically, but its behavior is still more complex and puzzling than the early theoretical work indicated. The intermediate phase is characterized by the coexistence of bulk superconductivity, a ferromagnetic component, and a transversely polarized sinusoidally modulated moment structure. Both the ferromagnetic component and the sinusoidal moment increase with decreasing temperature,

but in a manner totally unlike a normal magnetic order parameter. Surprisingly, this coexistence phase persists up to the point where the spontaneous ferromagnetic moment reaches over 80% of its low temperature value; but very small magnetic fields are sufficient to convert the sample to a normal ferromagnetic phase. The temperature dependence of the periodicity of the sinusoidal structure is very weak, but its wavelength increases by $\sim 10\%$ at the highest temperatures. The magnetic field dependence is also very weak although at the highest fields at which it is observed there is a decrease in wavelength.

By general arguments mentioned earlier, we do not expect the superconducting order parameter to be uniform in the coexistence phase. (The only case where this would be possible is the pure spiral magnetic structure proposed earlier by Blount and Varma [15] and Matsumoto et al. [16], and this is ruled out by the present data.) The self-induced vortex structure [17] would show both ferromagnetic and modulated moment components. However, the lack of a strong field dependence of the periodicity, and the fact that the four satellites observed in the (010) plane around each Bragg reflection did not have equal intensities appears to weaken the case for such a structure in ErRh_4B_4 . More complex vortex-type structures have been proposed by Hu and Ham [24]. However, more detailed comparisons of the theory and the experiments have yet to be made for this case. Bulevskii et al. [25] have proposed a linear antiphase domain-type structure with a periodicity of the domains of about the value observed for the satellite. However, the higher (odd) harmonics of the satellites predicted by this

model do not seem to be observed. Recently, Machida [26] proposed the existence of a Ferrel-Fulde state for the intermediate phase in ErRh_4B_4 , characterized by both ferromagnetism and Cooper pairing of the conduction electrons with a non-zero momentum $\vec{q}$. This would lead to a conduction electron spin polarization of modulation vector $\vec{q}$, and via second-order f-conduction exchange, to a modulation of the f-moments with the same wave-vector. Such a state would revert to a normal ferromagnetic state on application of a sufficiently large field. This theory, however, predicts a continuous transition at lower temperature to a normal ferromagnetic phase, whereas the observed transition appears to be first order. Both Greenside, Blount and Varma [27] and Tachiki [28] have developed models for an intermediate phase in which the moments are linearly polarized and sinusoidally modulated, owing to magnetic anisotropy in the crystal. Tachiki's laminar vortex model [28] predicts a qualitative behavior for the magnetic intensities in the neutron diffraction which is very similar to that observed in the experiments. Recent developments of these models [29] involve inhomogeneous superconducting domains of such modulated phases coexisting with ferromagnetic domains in the intermediate phase. At the lower transition temperature T_{C2} , there is presumably a first order phase transition from this coexistence phase to a normal, uniform ferromagnetic phase. However, careful high resolution neutron diffraction scans have revealed that the size of these domains at least just above T_{C2} , must be $\geq 10^4 \text{\AA}$, and have not detected any macroscopic periodicity for this domain structure. This is consistent with evidence for such domain sizes obtained

from recent a.c. Josephson tunneling measurements on ErRh_4B_4 [30]. If one assumes that a magnetic field simply converts modulated-moment superconducting domains to ferromagnetic regions, the intensities with and without a field would indicate that, just above T_{C2} , about 20% of the sample is in the superconducting phase. However, the increase in the ferromagnetic intensity does not appear initially to be proportional to the decrease in the satellite intensity, so this interpretation may be open to doubt. The persistence of the strange upward-curving shape of the ferromagnetic intensity versus temperature even in the hysteresis-free finite applied field case, where the satellites no longer exist, is also mysterious. Clearly, a theory of the magnetic field dependence of the properties of the coexistence phase is needed.

An interesting intermediate phase with a long-wavelength moment modulation has also been observed in powder diffraction experiments from HoMo_6S_8 [21,22]. However, there are some important differences from ErRh_4B_4 , in that the modulation wave-vector decreases with increasing field and the intensity of the satellite increases. It is possible that the intermediate phase in this compound is different from that in ErRh_4B_4 .

Finally, we show in Fig. 8 the field dependence of the (101) intensity at 2 K. Both signatures of H_{C1} (up to which there is no induced ferromagnetic moment) and H_{C2} (at which there is a distinct change in slope) are apparent. Such measurements, combined with bulk magnetization moments [31] should in principle be used to separate the spin magnetization from that due to supercurrents as functions of field and temperature. However,

proper calibration of the internal field, taking into account the demagnetizing factor, must be made and this has not yet been done.

It may be seen that a wealth of new and detailed information has been provided by neutron diffraction from magnetic superconductors in the last few years. With the continuation of fruitful collaboration between experimentalists and theorists, and the advent of further new materials, it is expected that this field will remain interesting and exciting for some time to come.

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Figure Captions

- Fig. 1. DC resistance of single crystal ErRh_4B_4 measured by four point technique. The current direction in the crystal is not along any symmetry direction.
- Fig. 2. Schematic diagram of positions in reciprocal space where magnetic intensity was observed. The satellite positions relative to the Bragg peaks are shown on an enlarged scale for clarity. Large filled circles are ferromagnetic Bragg reflections, small filled circles are satellites due to the modulated moment. The open circles are weak reflections attributed to a set of four symmetrically equivalent satellites about each Bragg point related to the filled circles by a four-fold rotation about c^* .
- Fig. 3. Temperature dependence of the ferromagnetic intensity from the (101) Bragg peak, the satellite intensity, the DC resistance, and the ratio of the satellite to the ferromagnetic intensity for $T < 1.25$ K.
- Fig. 4. High resolution scan through the satellite. Steps are in units of a^* .

Fig. 5. Scans of the satellite and the (101) peak at various values of applied field.

Fig. 6. Temperature dependence of the magnetic order parameter in an applied field of 0.0242 T.

Fig. 7. Temperature dependence of the magnetic order parameter in an applied field of 0.0722 T.

Fig. 8. Intensity of the (101) reflection at $T = 2$ K as a function of the applied magnetic field.

NEUTRON COUNTS/50 MONITOR UNITS

$T = 0.780\text{K}$

NEUTRON COUNTS / 50 MONITOR UNITS

NEUTRON COUNTS / 10 MONITOR UNITS

$H = 0$

$H = 0.0106T$

$H = 0.0276T$

$H = 0.0446T$