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Magnetism and Superconductivity in $(Er_{0.16}Ho_{0.84})Rh_4B_4$

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The superconducting and ferromagnetic phase boundaries in the $(Er_{1-x}Ho_x)Rh_4B_4$ mixed ternary alloy system meet in a multicritical point at $x_{cr} \approx 0.9$. For $x < x_{cr}$, the compounds first become superconducting as the temperature is lowered, and then lose superconductivity in a transition to ferromagnetism. The coexistence of superconductivity and ferromagnetism for alloys near the erbium-rich end of the phase diagram is well established. It has also been suggested that ferromagnetism and superconductivity coexist in alloys with x just below x_{cr}. We have carried out neutron-diffraction, ac magnetic susceptibility, and heat-capacity measurements on a sample of $(Er_{0.16}Ho_{0.84})Rh_4B_4$ to investigate the possibility of coexistence of ferromagnetism and superconductivity for $x \approx x_{cr}$. We find that there are minor discrepancies in the superconducting and magnetic transition temperatures reported for different samples of $(Er_{0.16}Ho_{0.84})Rh_4B_4$, but that ferromagnetism and superconductivity do occur simultaneously over a narrow temperature range in this sample. We suggest that an inhomogeneous state occurs, consisting of separate ferromagnetic and superconducting regions, rather than microscopic coexistence.

I. INTRODUCTION

The $(Er_{1-x}Ho_x)Rh_4B_4$ mixed ternary alloy system has been studied extensively in recent years to investigate the interaction between superconductivity and long-range magnetic order. The $(Er_{1-x}Ho_x)Rh_4B_4$ low-temperature phase diagram[1] is shown in Fig. 1. The phase boundaries have been determined from ac magnetic susceptibility[2,3] and neutron-diffraction[4,5] measurements. The phase diagram exhibits separate regions of superconductivity and ferromagnetism, as well as a region where superconductivity and ferromagnetism coexist over a small temperature interval. The superconducting and magnetic phase boundaries for this alloy system meet at a multicritical point at $x_{cr} \approx 0.9$. For $x < x_{cr}$, the alloy first becomes superconducting at T_{c1}, and then loses superconductivity in a transition to ferromagnetism at T_{c2}. For $x < 0.3$, there is a region above T_{c2} where superconductivity and a sinusoidally modulated magnetic phase coexist with normal ferromagnetism.[6–8] The Er^{3+} and Ho^{3+} magnetic moments exhibit different magnetic anisotropies and order independently.[4]

Lynn *et al.* have reported a region of coexistence of ferromagnetism and superconductivity for x near 0.9.[9,10] For a sample of $(Er_{0.16}Ho_{0.84})Rh_4B_4$, their ac magnetic susceptibility measurements showed onset of superconductivity at $T_{c1} = 5.95$ K and loss of superconductivity at $T_{c2} = 4.95$ K. Neutron-diffraction measurements on the same sample showed a transition to ferromagnetism beginning at $T_M = 5.30$ K. A possible interpretation of these results is that an internal magnetization of the Ho^{3+} moments develops in the superconducting phase and eventually quenches the superconductivity.[10]

The values of T_{c1} and T_{c2} obtained by Lynn *et al.* are significantly lower than those given by the $(Er_{1-x}Ho_x)Rh_4B_4$ low-temperature phase diagram of Johnston *et al.*[3] For $x = 0.84$, $T_{c1} = 6.33$ K, and $T_{c2} = 5.57$ K can be obtained from this phase diagram. The disagreement between the two sets of T_c's is well outside the experimental uncertainties in transition width, and raises the question whether the coexistence of superconductivity and ferromagnetism near $x = x_{cr}$ is a fundamen-

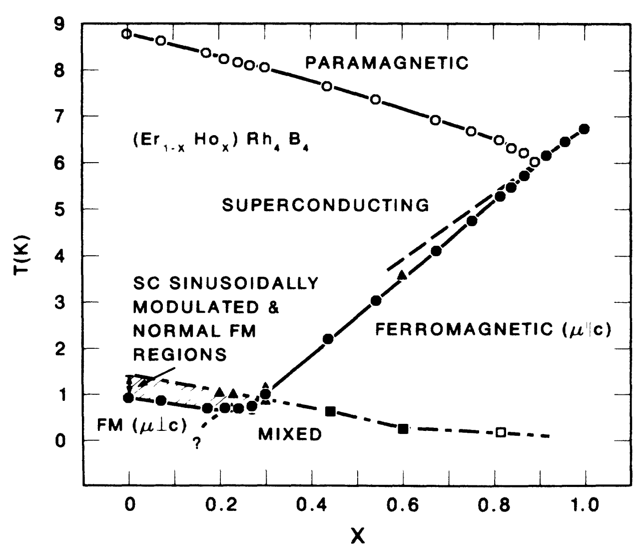

FIG. 1. Low-temperature phase diagram of transition temperature vs composition x for the $(Er_{1-x}Ho_x)Rh_4B_4$ system (Ref. 1).

tal property of the $(Er_{1-x}Ho_x)Rh_4B_4$ alloy system or is a sample-dependent effect. To further investigate the interaction between magnetism and superconductivity in the $(Er_{1-x}Ho_x)Rh_4B_4$ alloy system for x near x_{cr}, we have made neutron-diffraction and ac magnetic susceptibility measurements on a sample of $(Er_{0.16}Ho_{0.84})Rh_4B_4$, using the same cryostat, sample container, and thermometer for both measurements. We have also made heat-capacity measurements on both the neutron-diffraction sample and another sample spark cut from the same ingot. Our magnetic-susceptibility measurements give essentially the same values of T_{c1} and T_{c2} as obtained from the data of Johnston *et al.*,[3] while our neutron-diffraction measurements suggest the coexistence of superconductivity and ferromagnetism, in agreement with the conclusion of Lynn *et al.*[9,10]

Another interesting feature of the $(Er_{1-x}Ho_x)Rh_4B_4$ system is the mean-field-like behavior of the alloys for x near 1. Mean-field theory provides an excellent description of the properties of $HoRh_4B_4$ (Ref. 11) and has been used to help understand the transition to ferromagnetism in $(Er_{0.4}Ho_{0.6})Rh_4B_4$.[4] Mean-field-like behavior has also been observed in neutron-diffraction and heat-capacity measurements on a number of other holmium-rich $(Er_{1-x}Ho_x)Rh_4B_4$ samples.[10,12] Our neutron-diffraction and specific-heat data show that such a description is appropriate for $(Er_{0.16}Ho_{0.84})Rh_4B_4$, although detailed agreement is lacking, perhaps due to inhomogeneities in our sample.

II. EXPERIMENTAL METHODS

The $(Er_{0.16}Ho_{0.84})Rh_4B_4$ sample was synthesized by arc melting the constituent elements on a water-cooled copper hearth in a Zr-gettered atmosphere, followed by annealing in a manner previously described.[4] Boron enriched in ^{11}B was used to reduce neutron absorption. A powder sample produced by crushing an ingot was enclosed between two thin aluminum plates, placed inside a sample container filled with one atmosphere standard temperature and pressure (STP) of helium, and then mounted in a pumped helium cryostat. The neutron-diffraction measurements were made at the High Flux Isotope Reactor at Oak Ridge National Laboratory (ORNL) using a standard triple-axis spectrometer set for elastic scattering. The neutron wavelength was 2.351 Å. Neutron counting times were made versus a monitor counter placed in the reactor beam to account for any small variations in reactor power. Zero-field low-frequency (100 Hz) ac magnetic-susceptibility measurements were carried out at ORNL using the same sample, cryostat, sample container, and thermometer as the neutron-diffraction measurements. Magnetic susceptibility and specific-heat measurements were also made at the University of California, San Diego (UCSD) on the same sample as studied at ORNL, using apparatus described previously.[13] The specific heat of the crushed sample was determined by mixing the sample with Crycon grease and loading it into a copper holder.[13] These measurements permitted a comparison of the thermometry at the two laboratories.

III. RESULTS AND DISCUSSION

A. Transition temperatures

We determined the magnetic transition temperature T_M of our $(Er_{0.16}Ho_{0.84})Rh_4B_4$ sample by measuring the temperature dependence of the (101) Bragg reflection. Above T_M, the (101) reflection is very weak, due to the small (101) nuclear structure factor. Below T_M, strong magnetic Bragg scattering is superimposed on the weak nuclear peak. The temperature dependence of the (101) Bragg reflection is shown in Fig. 2(a). Background counts have been subtracted at each data point. Only data on cooling the sample are plotted; the data on warming show no evidence of hysteresis. The nearly linear dependence of intensity on temperature makes it easy to determine the magnetic transition temperature in this material.[10] For our sample of $(Er_{0.16}Ho_{0.84})Rh_4B_4$, we obtain $T_M=(5.97\pm0.04)$ K. Only the Ho^{3+} moments order at this temperature. Magnetic order of the Er^{3+} moments at lower temperatures is expected in our sample, but has not been investigated in this work.

Zero-field ac magnetic susceptibility data taken on the same crushed sample at ORNL are shown in Fig. 2(b). Magnetic susceptibility data taken at UCSD appear identical to the data shown in Fig. 2(b). We find $T_{c1}=6.27$ K and $T_{c2}=5.58$ K at the midpoints of the transitions (ORNL thermometry), with no significant different between data taken on cooling and on warming. The UCSD magnetic-susceptibility data yield transition temperatures that agree with the ORNL transition temperatures to within 0.01 K. The temperature at which we see the first indication of the destruction of superconductivity is $T_{des}=5.67$ K (ORNL thermometry). This temperature is more useful than T_{c2} for comparison with the neutron-diffraction data, because the neutrons are sensitive to the first indication of magnetic order in the sample, and not to an average of the bulk behavior. From our neutron-diffraction and ac magnetic-susceptibility data, it is evident that there is a temperature range between T_M and T_{des} where both magnetic order and superconductivity are present in this sample.

The heat-capacity data for a slab taken from the ingot that was later crushed for the neutron-diffraction experiment is displayed in Fig. 2(c). Similar data were also observed for the crushed sample using Crycon grease for thermal contact, indicating that crushing does not significantly alter the heat-capacity data. A mean-field-shaped magnetic transition is observed, and superimposed at the maximum of the sawtooth is a small spike at 5.54 K, due to a weak first order superconducting to ferromagnetic transition.[14] These data are similar to those for other samples in this system with $x=1$ (Refs. 11 and 12) and 0.912, 0.813, and 0.6.[12] The smallness of the spike is expected because its size scales with the degree to which ferromagnetic order is suppressed by superconductivity.[15] In $(Er_{0.16}Ho_{0.84})Rh_4B_4$, we are so close to the meeting of the ferromagnetic and superconducting phase boundaries that only a small suppression is expected. The two heat-capacity data sets agree well, although there is some broadening of the magnetic transition in the neutron-diffraction sample, perhaps due to strains induced by crushing. This broadening makes it difficult to define T_M, but we estimate $T_M = 5.72$ K (bulk slab) and $T_M = 5.81$ K (neutron-diffraction sample). These values are again the temperatures at the onset of the transition to ferromagnetism.

We also measured the ac magnetic susceptibility for the neutron-diffraction sample using coils mounted in the calorimeter and found excellent agreement (±0.01 K) with the ORNL ac susceptibility measurements shown in

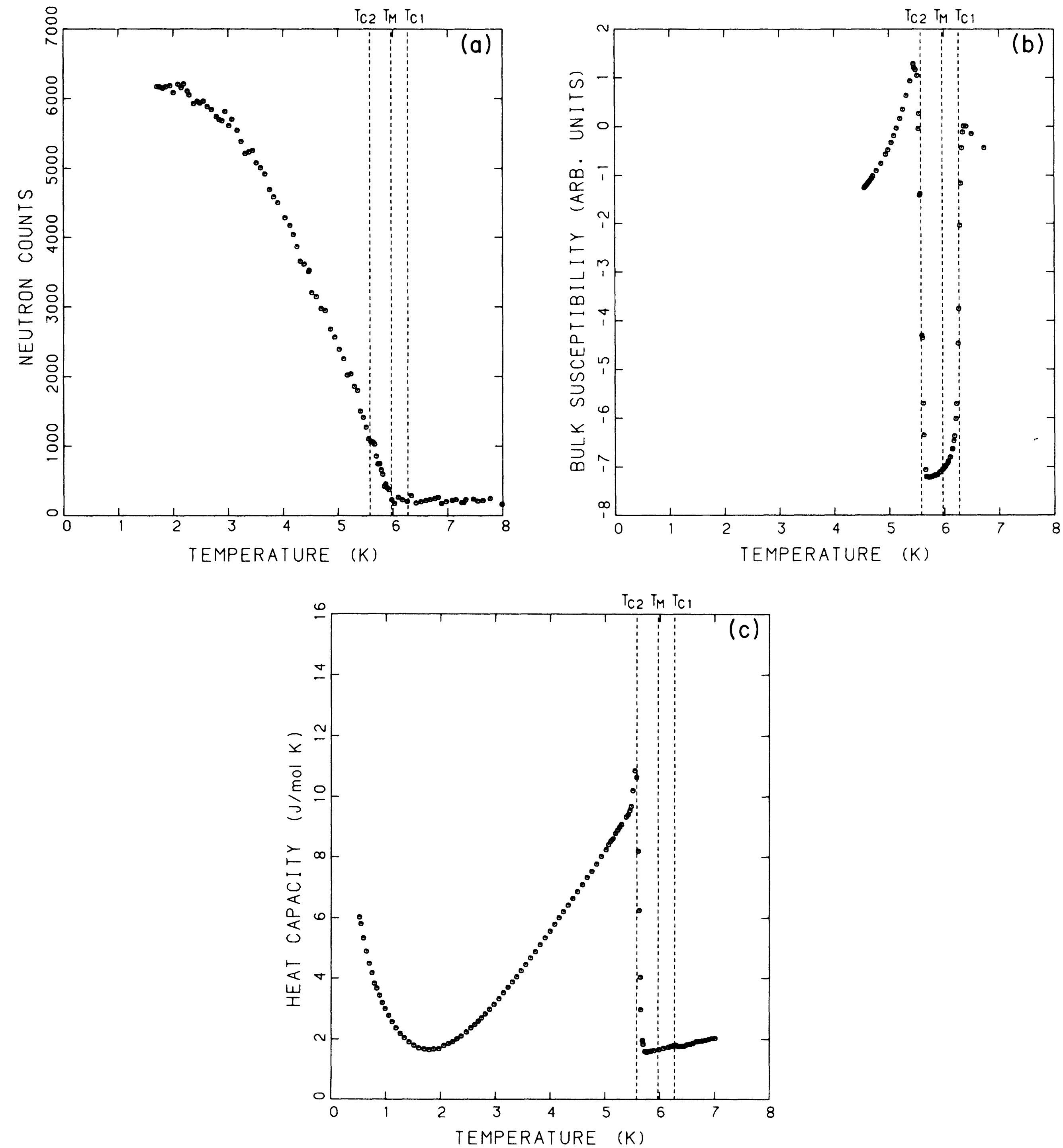

FIG. 2. (a) Observed (101) peak intensity as a function of temperature for $(Er_{0.16}Ho_{0.84})Rh_4B_4$. The counting time at each point was approximately 2.5 min. The temperature-dependent background has been subtracted from each point. The dashed lines indicate the superconducting and magnetic transition temperatures. (b) Bulk magnetic susceptibility of $(Er_{0.16}Ho_{0.84})Rh_4B_4$, measured at ORNL using the same sample, cryostat, sample container, and thermometer as the neutron-diffraction measurements. (c) Heat-capacity measured at UCSD for a bulk ingot from the neutron-diffraction sample of $(Er_{0.16}Ho_{0.84})Rh_4B_4$.

TABLE I. Measured transition temperatures for $(Er_{0.16}Ho_{0.84})Rh_4B_4$. For comparison, the transition temperatures obtained by Lynnm *et al.* (Ref. 10) are also shown, as well as our extrapolation of Johnston's result (Ref. 3). T_{des} refers to the temperature at which the first evidence for the destruction of superconductivity is seen (see the text).

Source	T_{c1} (K)	T_{c2} (K)	T_M (K)	T_{des} (K)
Neutron diffraction	...	...	5.97	...
Magnetic susceptibility	6.27	5.58	...	5.67
Heat capacity[a]	...	...	5.81	...
Heat capacity[b]	...	5.54	5.72	...
Reference 3	6.33	5.57	...	...
Reference 10	5.95	4.95	5.30	...

[a]Neutron-diffraction sample. T_M is for the onset of ferromagnetism.
[b]Slab sample.

Fig. 2(b). This demonstrates that our heat-capacity thermometry is consistent with our magnetic susceptibility and neutron-diffraction thermometry. Table I summarizes our neutron-diffraction, ac susceptibility, and heat-capacity results. The transition temperatures obtained by neutron diffraction and magnetic susceptibility suggest the simultaneous presence, but not necessarily microscopic coexistence, of ferromagnetism and superconductivity in this sample over a temperature range of 5.67–5.97 K. Our heat-capacity results for T_M differ from our neutron-diffraction results by more than the combined uncertainties. This may be due to the greater difficulty of discerning a small change in heat capacity on a temperature-dependence background.

B. Mean-field behavior in $(Er_{0.16}Ho_{0.84})Rh_4B_4$

The magnetic properties of $HoRh_4B_4$ can be described by a mean-field model with $S=\frac{1}{2}$.[11] The magnetic neutron scattering from $(Er_{0.4}Ho_{0.6})Rh_4B_4$ shows mean-field-like behavior well below T_M, but the mean-field transition near T_M is preempted by a first-order transition due to the presence of superconductivity.[4] Neutron diffraction from the intermediate compounds $(Er_{1-x}Ho_x)Rh_4B_4$ with $x=0.11$ and 0.16 also exhibits mean-field-like behavior,[10] while the heat capacity of the compounds with $c=0.912$, 0.813, and 0.6 shows the sawtooth-shaped feature associated with mean-field behavior.[12]

We have used mean-field theory to fit our neutron-diffraction data for $(Er_{0.16}Ho_{0.84})Rh_4B_4$. We consider first the temperature region near T_M. In a material which can be described by a spin-$\frac{1}{2}$ mean-field model, the intensity of the magnetic neutron scattering near T_M is given by

$$I_{mag}=I_0 t^{2\beta} , \quad (1)$$

where $t=(1-T/T_M)$ is the reduced temperature, I_0 is the magnetic intensity at 0 K, and $\beta=\frac{1}{2}$. A least-squares fit of our neutron-diffraction data to Eq. (1) for $t<0.05$ gives $\beta=0.51\pm0.02$. Figure 3 shows a plot of $\ln(I_{mag})$ versus $\ln(T_M-T)$ for our neutron-diffraction data for $t<0.05$. The linearity of these data indicates that it is valid to use Eq. (1) for this temperature range; i.e., $t<0.05$ is "near" enough to the transition. The solid line in Fig. 3 is calculated from Eq. (1) with $\beta=0.51$. Over the full range of temperature below T_M, the magnetic neutron scattering for a spin-$\frac{1}{2}$ mean-field model is given by

$$(I_{mag}/I_0)^{1/2}=\tanh[(I_{mag}/I_0)^{1/2}(T_M/T)] . \quad (2)$$

The total measured peak intensity in the neutron-diffraction experiment is $I_{mag}+I_{nuc}$, where I_{nuc} is the nuclear-only contribution. A least-squares fit of our data to Eq. (2), with I_0, T_M, and I_{nuc} as adjustable parameters, yields values for I_0 and T_M that clearly disagree with the measured data. Our interpretation of this disagreement is given in the next subsection.

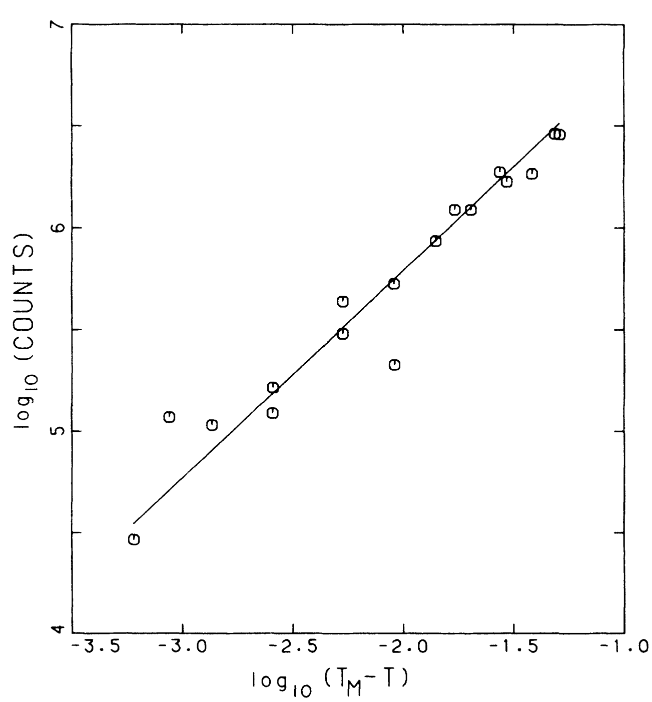

FIG. 3. log-log plot of the (101) magnetic intensity from $(Er_{0.16}Ho_{0.84})Rh_4B_4$ vs (T_m-T) for $(1-T/T_M)<0.05$. The solid line is a fit of the data to Eq. (1) with $T_M=5.97$ K and $\beta=0.51$.

C. Discussion

The mean-field behavior of Ho^{3+} moments in the $(Er_{1-x}Ho_x)Rh_4B_4$ system is well established.[4,10–12] It is therefore surprising that our neutron-diffraction data for this sample with $x=0.84$ show significant deviations from the expected temperature dependence, especially since an excellent fit to Eq. (2) can be obtained for the data of Lynn *et al.*[10] for a different sample of the same composition. One possible explanation is that superconductivity is destroyed and ferromagnetic ordering begins in different parts of the sample over a range of temperatures. The transition to the superconducting state at T_{c1} observed by ac magnetic susceptibility on a crushed sample takes place over a temperature interval of 0.16 K (10%–90%), and a "tail" is observed to even lower temperatures as the remaining 10% of the sample becomes superconducting. It would not be surprising if a similar distribution of transition temperatures broadened the transition at T_{des} over a range of ≈ 0.2 K. Calculations using Eq. (2) with a distribution of values of T_M from 5.7 to 5.97 K yield reasonable agreement with the data. Figure 4 shows one such fit to Eq. (2). Our lack of knowledge of the precise distribution of transition temperatures permits only a qualitative discussion. Most of the intensity corresponds to $T_M=5.97$ K, and about 80% of the sample appears to have this T_M. The data in Fig. 4 correspond to the square of the magnetization, so a square root must be taken to determine the relative proportion of the sample having each T_M. We note that it has been proposed[16] that strain could significantly

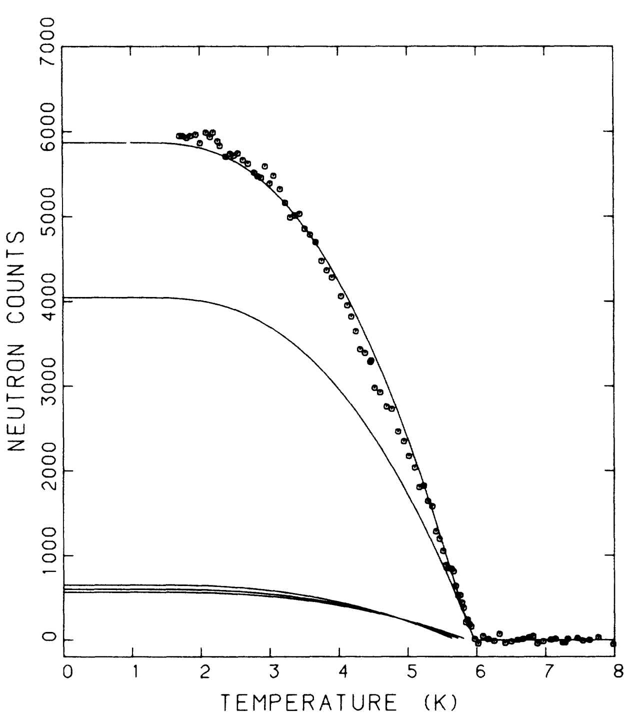

FIG. 4. Fit to mean-field theory for the $(Er_{0.16}Ho_{0.84})Rh_4B_4$ neutron-diffraction data, taken on cooling. The (101) nuclear intensity I_{nuc} has been subtracted from the neutron counts. See the text for a description of the fit.

enhance the magnetic ordering temperature in $ErRh_4B_4$. However, these authors show[16] that the same mechanism is not important for ions such as Ho^{3+} substituted for Er^{3+} in $ErRh_4B_4$, because Ho^{3+} ions have uniaxial anisotropy, rather than the basal plane anisotropy of Er^{3+} ions.

A more important question is the nature of the coexistence of ferromagnetic order and superconductivity. An analysis of heat-capacity and neutron-diffraction data for $(Er_{1-x}Ho_x)Rh_4B_4$ shows that competition between superconductivity and magnetic order is responsible for a pronounced spike in the heat-capacity data for $T\approx T_{c2}$.[15] This feature is caused by an abrupt increase in the ferromagnetic order parameter when superconductivity is destroyed, i.e., a transition that is thermodynamically of first order.[14] A state exhibiting the coexistence of superconductivity and ferromagnetic order could change the size of this spike, or eliminate it completely depending on the nature of the magnetic order in the coexistent state. Consequently, observation of a spike in the specific heat such as the one shown in Fig. 2(c) does not eliminate the possibility of a coexistent state. In fact, some models predict a first-order transition from a coexistent state to the ferromagnetic state (see, for example, Ref. 17).

An analysis of the upper critical magnetic field $H_{c2}(T)$ does argue against microscopic coexistence. To demonstrate this point, we use the $H_{c2}(T)$ curve determined for the paramagnetic compound YRh_4B_4.[18] The slope dH_{c2}/dT is 2.24 kG/K at $T_{c1}=6.27$ K, yielding a hypothetical critical field for $(Er_{0.16}H_{0.84})Rh_4B_4$ at $T_M=5.97$ K estimated to be 717 G in the absence of magnetic moments. The only source of magnetic field in our measurements is ferromagnetic ordering of the Ho^{3+} ions, so we assume a saturation moment of $8\mu_B$ per Ho^{3+} ion and calculate the temperature dependence of the internal field using

$$[M(T)/M_{\text{sat}}]^2=1-T/T_M \;, \tag{3}$$

which is the spin-$\frac{1}{2}$ mean-field result in Eq. (1) rewritten in terms of the magnetization M. We find $(T_M-T)=0.055$ K for $4\pi M=717$ G. Thus, an internal field strong enough to destroy superconductivity develops only 55 mK below T_M. Furthermore, 55 mK is an upper bound, since other effects, such as the exchange interaction between the superconducting electron spins and the magnetic moments, can further reduce $H_{c2}(T_m)$.[18] We conclude that the coexistence of ferromagnetism and superconductivity observed in $(Er_{0.16}Ho_{0.84})Rh_4B_4$ must be inhomogeneous.

IV. CONCLUSIONS

We have measured the superconducting transition temperatures T_{c1} and T_{c2} and the magnetic transition temperature T_M of a sample of $(Er_{0.16}Ho_{0.84})Rh_4B_4$. Neutron-diffraction and zero-field ac magnetic-susceptibility measurements at ORNL were made using the same sample, cryostat, sample container, and thermometer. Additional ac susceptibility and heat-capacity measurements were made on the same sample at UCSD.

Temperature calibrations at ORNL and UCSD were compared by measuring the ac magnetic susceptibility of the same $(Er_{0.16}Ho_{0.84})Rh_4B_4$ sample. The resulting transition temperatures agree within 0.01 K, indicating that differences in thermometry at the two laboratories are negligible for the data presented here.

We obtain $T_{c1}=6.27$ K and $T_{c2}=5.58$ K for the two superconducting transition temperatures. Our results are in good agreement with the values $T_{c1}=6.33$ K and $T_{c2}=5.57$ K obtained from the $(Er_{1-x}Ho_x)Rh_4B_4$ low-temperature phase diagram of Johnston *et al.*[3] We also obtain $T_M=5.97$ K, which supports the observations of Lynn *et al.* that ferromagnetism and superconductivity coexist over a narrow temperature interval in $(Er_{0.16}Ho_{0.84})Rh_4B_4$.[9,10] Calculations of the possible temperature range of coexistence, based on the upper critical field necessary to destroy superconductivity, indicate that the coexistence in this material is probably inhomogeneous rather than microscopic.

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