Elastic and inelastic neutron scattering measurements have been carried out to investigate the magnetic properties of superconducting ($T_c \sim 8$ K) HoNi$_2$B$_2$C. The inelastic measurements reveal that the lowest two crystal field transitions out of the ground state occur at 11.28(3) and 16.00(2) meV, while the transition of 4.70(9) meV between these two levels is observed at elevated temperatures. The temperature dependence of the intensities of these transitions is consistent with both the ground state and these higher levels being magnetic doublets. The system becomes magnetically long range ordered below 8 K, and since this ordering energy $kT_N \approx 0.69$ meV $\ll 11.28$ meV the magnetic properties in the ordered phase are dominated by the ground-state spin dynamics only. The low temperature structure, which coexists with superconductivity, consists of ferromagnetic sheets of Ho$^{3+}$ moments in the $a$-$b$ plane, with the sheets coupled antiferromagnetically along the $c$-axis. The magnetic state that initially forms on cooling, however, is dominated by an incommensurate spiral antiferromagnetic state along the $c$-axis, with wave vector $q_c \sim 0.054 \text{ Å}^{-1}$, in which these ferromagnetic sheets are canted from their low temperature antiparallel configuration by $\sim 17^\circ$. The intensity for this spiral state reaches a maximum near the reentrant superconducting transition at $\sim 5$ K; the spiral state then collapses at lower temperature in favor of the commensurate antiferromagnetic state. We have investigated the field dependence of the magnetic order at and above this reentrant superconducting transition. Initially the field rotates the powder particles to align the $a$-$b$ plane along the field direction, demonstrating that the moments strongly prefer to lie within this plane due to the crystal field anisotropy. Upon subsequently increasing the field at constant T the antiferromagnetic and spiral states are both observed to decrease in intensity, but at modest fields the spiral state decreases much less rapidly. Approaching the superconducting phase boundary from high fields, we find that the spiral state is strongly preferred, in deference to the superconductivity, again demonstrating a direct coupling between these two cooperative phenomena. The magnitude of the spiral wave vector $q_c$, on the other hand, shows very little field dependence. A magnetic moment of $8.2 \pm 0.2 \mu_B$ for the Ho$^{3+}$ is obtained from the observed field dependence of the induced moment at high fields ($7T$).

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I. INTRODUCTION

The new classes of quaternary intermetallic superconductors, namely the borocarbide series, RNi$_2$B$_2$C, where R is a rare-earth ion, and more recently the boronitride series, R$_3$Ni$_2$B$_2$N$_3$, exhibit relatively high transition temperatures (up to 23 K for YPd$_2$B$_2$C) while possessing the possibility of long-range magnetic order of the rare-earth subsystem over the same temperature range. In particular, the interplay between superconductivity and magnetism, previously limited to the ternary Chevreul-phase and related systems, is realized in a dramatic fashion in these new materials, with the most interesting system being HoNi$_2$B$_2$C. This material becomes superconducting at $T_C$ $\approx$ 8K while developing incommensurate long range magnetic order at about the same temperature. At $\sim$ 5K the superconductivity is reentrant as evidenced by a deep minimum in $H_C$2 near 5 K, below which the incommensurate magnetic order is suppressed in favor of a simple commensurate antiferromagnetic structure, which allows the return of superconductivity and a coexistence at low $T$.

Following the initial discovery of these materials, and the bulk measurements by Eisaki et al. that revealed a competition between magnetism and superconductivity in HoNi$_2$B$_2$C, we reported the first magnetic diffraction data and solution of the magnetic structures in this material. We also carried out complete profile refinements of both the nuclear and magnetic structures. Fig. 1(a) shows the body-centered tetragonal unit cell (I4/mmm), which consists of Ho-$\cdot$C planes separated by Ni$_2$B$_2$ layers stacked along the c-axis. The lattice parameters at room temperature are $a$ = 3.5170(1) Å and $c$ = 10.5217(3) Å as obtained from the profile refinement. The Ho moments order at $\sim$ 8K, with two types of magnetic ordering being observed. The first is shown in Fig. 1(b), where we have ferromagnetic sheets of spins in the a-b plane which are coupled antiferromagnetically along the c-axis. However, satellite peaks are also observed along the c-axis and correspond to the direction of the spins in each sheet being rotated $\sim$ 16.6° away from the antiferromagnet compensation. Thus there is an antiferromagnet spiral that forms along the c-axis. If this angle were exactly 15° as suggested in the figure then this would be a long wavelength commensurate spiral, with a period of 24 Ho layers (12 unit cells). However, the actual spiral that forms is incommensurate with the underlying lattice.

Intensity for both types of peaks are observed below $\sim$ 8K, and they increase with decreasing temperature. However, we have found that the relative intensities are different for different samples, and thus we believe that these two magnetic phases are coming from separate regions of the samples; further work on these aspects, including studies of doped systems, will be reported elsewhere. The intensities for both magnetic phases increase with decreasing temperature, but in the vicinity of the reentrant superconducting transition at $\sim$ 5K the intensity of the spiral state suddenly begins to rapidly decrease while the intensity for the commensurate antiferromagnetic peak rapidly increases, until it saturates at low temperatures. This low temperature commensurate antiferromagnetic state coexists with superconductivity.

The behavior of HoNi$_2$B$_2$C contrasts sharply with that for ErNi$_2$B$_2$C, the only other Ni-containing magnetic-superconductor system that has been investigated so far with neutrons. Here an a-axis spin density wave state is observed at all temperatures, and this state readily coexists with superconductivity over the full temperature range where these two cooperative states are observed. Subsequent to our initial work on HoNi$_2$B$_2$C, Goldman et al. reported similar data on single crystal samples, and also found a small a-axis modulation above the reentrant transition similar to the large a-axis peaks observed in the Er material. We have also found a small a-axis modulation, but over a temperature shifted and narrower T range than what Goldman et al. found; these results will be reported elsewhere. However, since only the Ho system exhibits a giant anomaly in $H_C$2 and reentrant superconductivity, and only the Ho system possesses the c-axis spiral, it is clear that these two phenomena are directly related to each other.

II. EXPERIMENTAL CONFIGURATIONS

The HoNi$_2$B$_2$C polycrystalline sample was prepared by arc-melting and subsequent annealing using the $^{11}$B isotope to reduce nuclear absorption. Elastic neutron scattering was performed on the BT-2 and BT-9 triple-axis spectrometers at the National Institute of Standards and Technology (NIST) Research Reactor. A pyrolytic graphite monochromator was used in each case, with a pyrolytic graphite filter to remove higher order wavelength contaminations. For the diffraction measurements typical collimations of 60°-20°-20° were employed, with no analyzer crystal (double-axis mode), with a neutron wavelength of 2.35 Å. For the field-dependent measurements a vertical-field 7T superconducting magnet was utilized. For the inelastic measurements we used a pyrolytic graphite (002) analyzer crystal to select a fixed scattered neutron energy $E_F$ of 14.8 meV.

III. INELASTIC NEUTRON SCATTERING

In order to understand the nature of the magnetic order and the ground-state spin dynamics, it is important in these heavy rare earth systems to determine the single-ion crystal-field splittings of the Ho$^{3+}$ ions. We therefore carried out inelastic neutron scattering measurements to
investigate the low-lying crystalline electric field (CEF) energy levels of HoNi$_2$B$_2$C. For tetragonal D$_{4h}$ point group symmetry the 17-fold degenerate $^5$I$_8$ energy levels of Ho$^{3+}$ are split into nine (non-magnetic) singlets and four (magnetic) doublets. The heavy rare earths typically have CEF energies $\ll$ spin-orbit J-splittings so that J-mixing may be neglected to a good approximation and the ground state may be taken to be due to the CEF perturbation of the lowest J-multiplet. As the holmium ions order magnetically we anticipate that the crystal field ground state is very likely a doublet.

The delta function serves to conserve energy by equating the transition and the ground state may be taken to be due to the Fourier transform of the magnetization density, indicated by the dashed lines in Fig. 3. It is significant that the magnetic form factor $f(Q)$ is the (powder-averaged) spin wave scattering, while at higher temperatures this is just the paramagnetic diffuse scattering. The strong elastic scattering is due to the elastic crystal field scattering plus nuclear incoherent scattering. This type of measurement, taken at a series of temperatures, reveals two crystal field excitations from the ground state to the first CF level at 11.28(3), and from the ground state to the second level at 16.00(2) meV. We also observed some additional quasielastic scattering below $\sim 2$ meV, which we attribute to the spin dynamics within the crystal field ground state. Below the ordering temperature of $\sim 8$ K ($\rightarrow 0.69$ meV) this is the (powder-averaged) spin wave scattering, while at higher temperatures this is just the paramagnetic diffuse scattering. We note that since the energetics associated with the ordering is much less than the energy of the first crystal field level (0.69 11.28), below the ordering temperature only the crystal field ground state is significantly populated. Hence the symmetry of the magnetic order parameter and the spin dynamics in the ordered state will be controlled by the properties of the crystal field ground state.

At low temperature we can only observe excitations out of the ground state, but at elevated temperature the levels at 11.28 and 16.00 meV will become populated and we should then see an additional excitation at 4.72 meV. This transition is indeed observed at 4.70(9) meV. Fig. 3 shows the observed temperature dependence of the intensities of the two transitions out of the ground state and the transition between them, each point of which is the result of the fit with Gaussian lineshapes such as shown in Fig. 2. Over the range of temperatures up to 150 K the energies of the two transitions out of the ground state are temperature-independent within experimental error. The temperature dependence of the scattering intensities of each transition has been modeled with Boltzmann statistics, indicated by the dashed lines in Fig. 3. It is clear that the CF ground state is a magnetic doublet, and indeed all three levels were found to be best fit by doublets. The 11.28 and 16.00 meV transitions follow the thermal population of the ground state, which de-

\[ I \sim C \sum_i P_i g_i Z \] (5)

where all of the temperature-independent factors have been collected into a single constant, $C$, except for the degeneracy of each level, $g_i$, and where the (small) Debye-Waller factor has been ignored. The ratio of matrix elements for particular transitions may then be formed as the ratio of their $C$ values since all other terms cancel to a good approximation.

Fig. 2 shows an example (at 6 K) of the observed inelastic spectra. We chose to make these measurements at a momentum transfer of $Q \approx 2\AA^{-1}$, which avoids any Bragg peaks at the elastic position while keeping $Q$ small to obtain a strong magnetic signal and avoid significant phonon scattering. The strong elastic scattering is due to the elastic crystal field scattering plus nuclear incoherent scattering.
creases with increasing temperature as the excited levels are thermally populated, while the intensity of the 4.7 meV transition increases with temperature and appears to saturate near 150 K (the highest temperature measured). The matrix element ratios are found to be

$$\frac{|\langle 0|J_\perp|11.28\rangle|^2}{|\langle 0|J_\perp|16.00\rangle|^2} = 3.04$$

and

$$\frac{|\langle 0|J_\perp|11.28\rangle|^2}{|\langle 11.28|J_\perp|16.00\rangle|^2} = 0.99$$

using equation [3]. The elastic magnetic scattering intensity was also found to decrease with increasing temperature.

The field-dependent data we present below reveal that the moments strongly prefer to lie in the $a-b$ plane, while the crystal field data demonstrate that the ground state is doubly degenerate. The exchange interactions are three dimensional, but they are clearly different along the c-axis then in the $a-b$ plane. Thus we expect that the magnetic Hamiltonian will correspond to a $S = \frac{1}{2}$, three dimensional $xyz$ system. It is also likely that a four-fold in-plane anisotropy will be needed to adequately describe the system. The indirect (RKKY) exchange interactions, on the other hand, must be relatively complicated as evidenced by the interesting series of magnetic phase transitions and magnetic structures that are observed in this material.

IV. ZERO-FIELD MAGNETIC STRUCTURES

The nature of the magnetic phase transitions and temperature dependence of the order parameters have already been given in our earlier work [4], so here we will just briefly describe the evolution of the magnetic scattering as a function of temperature while providing some further experimental details. Fig. 4 shows the evolution of the magnetic scattering at six different temperatures. A three-peak structure is already evident for temperatures just above 8 K, indicating that long range order has set in as the scattering intensity initially becomes observable. The peak in the center belongs to the commensurate antiferromagnetic structure as given in Fig. 1(b), while the satellites at $q_c = 0.0543$ Å$^{-1}$ on either side originate from the spiral magnetic structure [Fig. 1(c)]. We see that initially the three peaks have about the same intensity—after taking into account the combination of the magnetic form factor, $f(Q)$, and the powder Lorentz factor, $(\sin\theta\sin2\theta)^{-1}$—and increase in intensity at the same rate. However, below ~5.3 K the intensity of the antiferromagnetic peak begins to grow more rapidly while the intensities of the spiral peaks plateau, and then drop sharply in intensity, as shown in Fig. 5. This drop in intensity for the spiral peak coincides with the reentrant superconducting transition temperature observed in this material; the growth of the spiral amplitude forces the system back to the normal conducting state, but when the spiral amplitude suddenly drops then the superconducting state is restored. This behavior suggests that the magnetic ordering is controlling this sequence of phase transitions. Thus we believe the lock-in transition to the low temperature antiferromagnetic state is being controlled by the magnetism, and this allows the superconductivity to re-establish itself. Note in Fig. 1(c) that in the spiral state there is a net uncompensated magnetization/exchange on the (superconducting) Ni layers, and we believe this is what is causing the magnetic/superconducting coupling and the reentrant behavior.

The temperature dependence of the intensity for the spiral state, shown in Fig. 5, indicates that there is some irreversibility on warming and cooling. It also shows that even well below the reentrant transition there remains some vestige of the spiral intensity, of the order of a few per cent. However, Fig. 6 shows that the widths of these spiral peaks become quite large at low T, indicating that these regions become quite small in size. We therefore attribute this remaining scattering either to domain walls in the crystalline particles or on their surface. Higher resolution studies show that there is also a small intrinsic width for the commensurate antiferromagnetic peaks, as the magnetic peaks are slightly broader than the nuclear diffraction peak linewidths, and correspond to a magnetic domain size of ~2175 Å at 5.3 K.

V. MAGNETIC FIELD DEPENDENCE

Field-dependent elastic neutron scattering measurements were performed in order to further probe the interactions among the superconducting, antiferromagnetic, and spiral magnetic states as functions of temperature and field. We found that for the initial application of the field the spiral, antiferromagnetic, and (001)-type nuclear reflections all increased in intensity, demonstrating that the powder crystallography were reorienting in the field. Fig. 7 illustrates the initial observed field dependence of the spiral and antiferromagnetic intensities, after cooling in zero field to 5.3 K. The intensities first increase sharply to a peak near 0.25 T, indicating that the crystallites orient in a relatively modest field, while at higher fields both types of peaks decrease in intensity gradually as the magnetic moments rotate into a ferromagnetic alignment. The reorientation is caused by the $a-b$ planes of the crystallites rotating to become parallel with the externally applied magnetic field, showing that the Ho$^{3+}$ magnetic moments strongly prefer the $a-b$ plane. After the initial field alignment, no further reorientation of the
particles was observed, and the sample remained aligned for all subsequent measurements.

The intrinsic field dependence of the intensities of the spiral and antiferromagnetic peaks is shown in Fig. 8. These data were taken at our lowest field-dependent temperature of 4.5 K, which is below the peak temperature for the spiral state so that the commensurate (001) antiferromagnetic peak is roughly three times as strong in intensity at zero field. The behavior we show here is quite typical of that observed for all higher temperatures; the data for 5.3 K have been previously presented. We see that the initial response is for both components to start slowly and monotonically decreasing with increasing field, and then they decrease more rapidly above \sim 0.3 T. The positions of the peaks, on the other hand, are essentially field independent as shown in Fig. 9. Here we see that the positions of the spiral peaks might suggest a slight decrease in \( q_c \) at the higher fields, but it should be noted that this is in the regime where the intensities are becoming small and the corresponding statistical (and systematic) errors large. We have included the zero-field temperature dependence of \( q_c \) in the figure for comparison, where we note that above the reentrant transition \( q_c \) is also observed to be temperature independent.

While the spiral and antiferromagnetic states both lose intensity with increasing field strength due to the forced rotation of the spins to a ferromagnetic alignment, the data in Fig. 8 show that they have different functional dependencies, as well as clear evidence for hysteresis. In particular, the spiral satellites are observed to have increased relative intensities on returning the field towards zero as compared to increasing field, while the commensurate peaks exhibit the opposite behavior. The magnetic scattering is therefore being redistributed. The relative scattering strengths are shown Fig. 10, where we plot the ratio of the spiral to antiferromagnetic intensities at this temperature as a function of field. We see that on increasing field the ratio has a maximum at \sim 0.5 T, which maximum is a consequence of the antiferromagnetic intensity decreasing more rapidly with field than the spiral intensity. After ramping to high field, we see that we have a much larger peak (at \sim 0.4 T) on returning towards zero field, and indeed there is a peak in the spiral intensity itself as shown in Fig. 8. Note that the competition and hysteresis are only evident at non-zero field values; the scattering intensity (essentially) returns to its initial value at zero field, as shown in Fig. 8. Thus the application of modest magnetic fields clearly favors the spiral state over the antiferromagnetic state; on increasing the field some of the antiferromagnetic phase appears to transform to the spiral phase, while on decreasing from high fields the spiral state preferentially forms and resists transforming to the antiferromagnetic/superconducting phase. We note that this field-dependent hysteresis is identical in origin to the temperature-dependent hysteresis we observe in zero field as shown in Fig. 5. Here we see that on cooling from above \( T_c \) the spiral state preferentially forms, while on warming from low temperatures the antiferromagnetic/superconducting state is preferentially maintained.

Finally we consider the magnetic field-induced response at the (002) nuclear reciprocal lattice position. In Fig. 11 the vertical axis is labelled as magnetization (\( \mu_B \)), induced by the application of \( H \); the magnetization is directly related to the square root of the integrated intensity of the magnetic Bragg peak. Based upon the crystal field results, at 5.3 K only the ground state doublet will be significantly involved. Thus to a first approximation the magnetization is given by Boltzmann statistics for a single spin-1/2 ion,

\[
M = \mu \tanh\left( \frac{\mu B}{k_B T}(H - H_c) \right),
\]

where \( \mu \) is the net magnetic moment in Bohr magnetons at the Ho\(^{3+} \) site, and \( H_c \) is the exchange field. This exchange field represents the apparent reduction in strength of the external magnetic field at the internal lattice sites due to the antiferromagnetic exchange interaction between neighboring planes.

For very low fields, \( M \sim \chi H \), and the curve should be nearly linear. However, for all fields below \( H_{c2} \) screening currents will reduce the field inside the superconductor, reducing the induced moment, while decreasing the field from above \( H_{c2} \) to below \( H_{c1} \) can result in significant trapped flux rather than the ideal internal field of zero. To avoid these problems the data were modelled over a constrained field range, empirically determined to be above \sim 0.7 T. Above this value the single-ion model works relatively well.

The results of the fit shown in Fig. 11 yield a net induced magnetic moment of \( 8.25 \pm 0.2 \mu_B \) at 7T. The effective exchange field was found to be \( 0.37 \pm 0.06 \) T. Although the moments and fields obtained for opposite field sweep directions are within the error range of each other, the data themselves indicate that there is a small amount of hysteresis, possibly due to a decreased superconducting fraction from trapped flux on reducing field.

VI. DISCUSSION

The c-axis spiral state, the antiferromagnetic arrangement of ferromagnetic a-b planes at low \( T \), and the superconductivity have all been observed to coexist in every neutron scattering study thus far on \( \text{Ho}_3\text{Ni}_2\text{B}_2\text{O}_{8}\). The coincidence of the giant (reentrant) anomaly in \( H_{c2} \) and the maximum in the intensity of the spiral magnetic component demonstrates that the spiral state is unfavorable to the formation of Cooper pairs which undergo increased pair-breaking as the spiral component grows to a maximum. This pair-breaking likely originates from the net exchange field/magnetization on the Ni layers as the magnetization vectors on each a-b layers plane rotate away from the totally compensated antiferromagnetic arrangement.
to form the spiral. From this viewpoint, which is supported by the temperature and field-dependent hysteresis effects observed, the spiral state forms naturally and is the preferred magnetic structure at intermediate temperatures and fields. At lower temperature the incommensurate spiral state locks-in to the commensurate antiferromagnetic structure, and thereby superconductivity returns and coexists. This is quite different from the situation in the ferromagnetic superconductors ErRhB$_2$(S,Se)$_6$ wherein the oscillatory state is formed as a compromise between the superconductivity and the ferromagnetism.

The related ErNi$_2$B$_2$C compound does not exhibit a c-axis spiral, but instead orders as a transversely polarized spin density wave along the a-axis. Bulk magnetization measurements also show no anomalous minimum in the upper critical field of ErNi$_2$B$_2$C and no significant hysteresis in the magnetic order parameter; there is only a small anomaly in H$_c2$ near T$_N$ which is typical for antiferromagnetic superconductors. A small a-axis modulation has been observed above the reentrant transition in HoNi$_2$B$_2$C, but it is clear that the c-axis spiral is the component that competes with the superconductivity while the a-axis modulation (if it exists in the pure phase of HoNi$_2$B$_2$C) is not strongly coupled to the superconducting state.

Recent band theory calculations have indicated that an unusual combination of states, resulting in a peak in the density of states (DOS) at the Fermi level of the Ni(3d) conduction band, is responsible for the elevated values of $T_c$ in the borocarbide series. The calculations also show the compounds to be three-dimensional metals despite their layered structure, unlike the quasi-two-dimensional high- $T_c$ cuprates. Evidence supporting the peak in the DOS is seen in $^3$He pressure studies, specific heat experiments, and the dependence of $T_c$ upon substitution and alloying, but is not supported by photoemission measurements, perhaps due to exchange correlation effects. More recently evidence has been seen for two-dimensional ferromagnetic spin wave contributions to the specific heat and the observation of a cascade of magnetic phase transitions in the range $0 \leq H \leq 1.5$ T. The transitions were identified as being consistent with the formation of a fan structure and its response to an applied magnetic field. All of these observations are consistent with the magnetic structure reported in our earlier papers as shown in Fig. 1.

VII. ACKNOWLEDGMENTS

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Fig. 1. HoNi$_2$B$_2$C (a) Crystal structure; (b) Commensurate antiferromagnetic structure; (c) Spiral magnetic structure.

Fig. 2. Inelastic intensities at 6 K of crystal field transitions out of the ground state at 11.28 and 16.00 meV. The “extra” scattering on the high-energy side of the elastic paramagnetic peak near zero energy transfer likely originates from the ground state spin dynamics.

Fig. 3. Temperature dependence of the crystal field transitions; the dashed lines are the fits to Boltzmann statistics. The two upper transitions out of the ground state decrease with ground state depopulation as the temperature increases while the intermediate transition between them increases.

Fig. 4. Temperature dependence of the magnetic diffraction pattern of the spiral satellite peaks on either side of the (001) commensurate antiferromagnetic peak at Q $\cong$ 0.59Å$^{-1}$. The central commensurate antiferromagnetic (001) peak and the adjacent spiral peaks develop near 8 K as in (a) and increase in intensity with decreasing temperature as shown in (b) and (c). Below $\sim$5.3 K the (001) commensurate antiferromagnetic peak grows rapidly in intensity (d-f) and dominates at low T.

Fig. 5. (a) Integrated intensity of the commensurate antiferromagnetic reflection as a function of temperature, showing thermal hysteresis. The small dots are the peak counts versus T. (b) Temperature dependence of the spiral satellites, observed on warming and cooling.

Fig. 6. Observed widths of the spiral and antiferromagnetic peaks versus T. The rapid broadening of the spiral magnetic peaks below $\sim$5 K suggests that the remnant spiral scattering at low T is due to a surface effect or to domain walls.

Fig. 7. Reorientation of the powder to place the a-b plane of each crystallite parallel to the externally applied vertical magnetic field during the initial field ramp to 3 T. Both the (001) commensurate and the (001)$^+$ high-angle satellite and (001)$^-$ low-angle satellite peaks, as well as the (00$\ell$) nuclear reflections, show increased intensities upon returning to zero field.

Fig. 8. Intrinsic field dependence of the intensities of the antiferromagnetic and spiral peaks, for a temperature of 4.5 K and the field applied in the a-b plane. Note that there is considerable hysteresis, with the spiral state being preferred as the superconducting state is approached from high fields.

Fig. 9. Temperature dependence (top) and field dependence (bottom) of the positions of the spiral and antiferromagnetic peaks. There is little if any field dependence to the satellite positions, and above $\sim$5K they are temperature independent as well.

Fig. 10. Ratio of the spiral satellite intensity to the antiferromagnetic peaks, as a function of magnetic field.

Fig. 11. Field-induced magnetic moment at the (002) nuclear reciprocal lattice position; at fields above 0.7 T the two-level ground state single-ion model (solid line) fits the data well.

VIII. FIGURE CAPTIONS