Static magnetic moments revealed by muon spin relaxation and thermodynamic measurements in the quantum spin ice $Yb_2Ti_2O_7$

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We present muon spin relaxation (μSR) and specific-heat versus temperature $C(T)$ measurements on polycrystalline and single-crystal samples of the pyrochlore magnet $Yb_2Ti_2O_7$. $C(T)$ exhibits a sharp peak at a T_C of 0.21 and 0.26 K for the single-crystal and polycrystalline samples, respectively. For both samples, the magnetic entropy released between 50 mK and 30 K amounts to $R\ln 2$ per Yb. At temperatures below T_C we observe a steep drop in the asymmetry of the zero-field μSR time spectra at short time scales, as well as a decoupling of the muon spins from the internal field in longitudinal magnetic fields of $\leqslant 0.25$ T for both the polycrystalline and single-crystal samples. These muon data are indicative of static magnetic moments. Our results are consistent with the onset of long-range magnetic order in both forms of $Yb_2Ti_2O_7$.

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I. INTRODUCTION

Quantum spin ice has highlighted the quantum dynamics of emergent magnetic monopoles and the associated gauge fields [1] in frustrated pyrochlore magnets [2]. In particular, the ground state of $Yb_2Ti_2O_7$ may be described as a U(1) quantum spin liquid hosting gapped bosonic spinon excitations carrying the monopole and fictitious gapless photon excitations [1] or a ferromagnetically ordered Higgs phase achieved by a Bose condensation of spinons [3]. In the latter case, the U(1) gauge invariance associated with the global phase of spinon wave functions is spontaneously broken and the gauge fields and thus the fictitious photons acquire a mass through the Anderson-Higgs mechanism [4,5]. This provides a magnetic analog of superconductivity on the lattice problem as an ideal laboratory for addressing the lattice gauge theory [6] in the U(1) case. In the early study, a first-order phase transition was observed around $T_C \sim 0.24$ K on powder samples of $Yb_2Ti_2O_7$ [7]. However, the nature of the transition and the low-temperature phase have been the subject of intense debate [3,8–13].

An emergence of ferromagnetic (FM) order has been reported for a single-crystal sample studied by neutron diffraction measurements [9]. Polarized neutron-scattering experiments on the same single crystal have shown a full depolarization of the incident neutron spins indicating the formation of ferromagnetic domains [3]. The paramagnetic state is well described with a magnetic Coulomb liquid behavior characterized by the growth of a broadened pinch-point singularity [14,15] in the energy-integrated diffuse scattering profile with decreasing temperature [3], and the observed ordered structure well below the transition temperature $T_C \sim 0.21$ K [9] is consistent with the theoretical prediction of a formation of nearly collinear ferromagnetic moments parallel to [100] in the Higgs phase [3,10].

Long-range magnetic order, however, has only been detected in the single crystals of $Yb_2Ti_2O_7$ studied in Refs. [3] and [9]. There is a sharp anomaly in the temperature dependence of the specific heat, $C(T)$ at T_C in these crystals, while other single-crystal samples have either a broader peak or no peak at all in $C(T)$ [3,10–13]. A possible crystallographic and/or chemical disorder of the crystals including the effects of strain and stuffing, i.e., an excess of Yb ions occupying the Ti sites, that has been detected from EXAFS [3] and neutron-scattering studies [12], may explain this discrepancy.

Given these contrasting results on single crystals, experiments on polycrystalline samples may provide key information to solve this problem. Most authors report a sharp peak in $C(T)$ at low temperature for powder samples of $Yb_2Ti_2O_7$ [7,11–13], while the peak temperature ranges from 0.24 to 0.265 K [Fig. 1(a)]. Muon spin relaxation (μSR) experiments on different powder samples have provided contradictory data: A short-time decay of the μSR asymmetry was observed at 0.2 K in an early study [8] indicating at least a near freezing of the magnetic moments, while this behavior was not observed in more recent work [13]. Moreover, long-range magnetic order has not been observed by neutron diffraction [8] and spin echo [16] experiments on polycrystalline $Yb_2Ti_2O_7$, although magnetic Bragg scattering at reciprocal lattice vectors is much harder to detect on powders than in single crystals. Comprehensive investigations on both powder and single-crystal $Yb_2Ti_2O_7$ are therefore required in order to resolve these controversies and to determine whether the ground state of quantum spin ice $Yb_2Ti_2O_7$ belongs to a magnetic Higgs phase or a U(1) quantum spin liquid.

In this paper, we present μSR and specific-heat measurements on both polycrystalline and high-quality single-crystal samples of $Yb_2Ti_2O_7$. A sharp maximum in the temperature dependence of the specific heat reveals the presence of a magnetic transition at a T_C of 0.26 and 0.21 K for the powder and the single-crystal samples, respectively, with a magnetic entropy released between 50 mK and 30 K that amounts to $R\ln 2$ per Yb. Zero-field (ZF) and longitudinal-field (LF) μSR

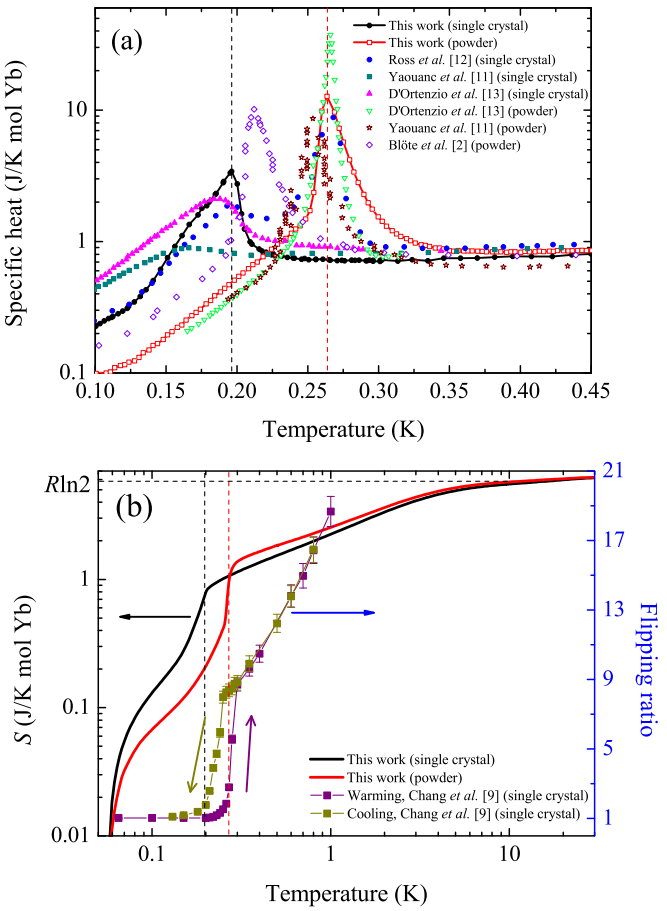

FIG. 1. (Color online) (a) Specific heat versus temperature $C(T)$ for our single crystal [3] (closed symbols) and powder (open symbols) samples of $Yb_2Ti_2O_7$ on a semilogarithmic scale. The lines are a guide to the eye. Data from Ross *et al.* [10], Yaouanc *et al.* [11], D'Ortenzio *et al.* [13], and Blöte *et al.* [7] are also shown for comparison. (b) Left-hand y axis: magnetic entropy versus temperature $S(T)$. The phonon contribution, estimated from $C(T)$ data for nonmagnetic $Y_2Ti_2O_7$ and corrected for the difference in the atomic mass of Y and Yb [17], and a nuclear Schottky contribution [7], have been subtracted from the data. Right-hand y axis: the flipping ratio of the neutron spins at the (111) Bragg peak as a function of temperature for the single-crystal sample of $Yb_2Ti_2O_7$ used in this work (see Ref. [3]). The vertical black and red dashed lines indicate the temperature of the specific heat peak for the crystal and powder, respectively.

measurements show that below T_C, static magnetic moments exist in both samples. The magnetic volume fractions are estimated to be 100% and 80% for the powder and single crystal, respectively. Our results are consistent with the onset of a magnetic order in $Yb_2Ti_2O_7$ in both forms of this material.

II. EXPERIMENTAL DETAILS

A. Sample preparation and heat capacity measurements

Polycrystalline samples of $Yb_2Ti_2O_7$ were synthesized using a solid state reaction method. Stoichiometric quantities of Yb_2O_3 and TiO_2 powders were repeatedly ground, pressed into pellets, and sintered at 1300 °C for several days. The method used for the growth of the single crystal is described in Ref. [3]. Heat capacity measurements were carried out using a relaxation method in a Quantum Design Physical Property Measurement System (PPMS) equipped with a ^{3}He-^{4}He dilution refrigerator insert. Figure 1(a) shows the specific-heat data of this new powder sample and the single-crystal used in Refs. [3] and [9], as well as data published by other groups. From the peak in the specific heat, the transition temperature T_C for the powder and the single crystal are estimated to be 0.26 and 0.21 K, respectively. Note, the T_C for our new powder sample is higher than that of Ref. [7] and as high as those of Refs. [11] and [13]. For both samples, the magnetic entropy released between 50 mK and 30 K amounts to $R\ln 2$ per Yb ion, indicating no residual entropy for the lowest-energy magnetic doublet of the Yb $4f$ electrons [7], as shown in Fig. 1(b). Although the relaxation method used here cannot correctly extract the latent heat, the first-order nature of the ferromagnetic transition at least in the case of our single crystal is clear from a thermal hysteresis of the flipping ratio of the neutron spins at the (111) Bragg peak, which decays to unity below T_C [3] [see Fig. 1(b)].

B. μSR experiments

The μSR measurements on our powder and single-crystal samples were performed using the MuSR spectrometer at ISIS where a pulse of muons with a full width at half maximum (FWHM) of ∼70 ns is produced every 20 ms. The samples were mounted on pure Ag plates using General Electric (GE) varnish and covered with silver foil. The samples were cooled to 50 mK in an Oxford Instruments ^{3}He-^{4}He dilution refrigerator and the data were collected on heating. The 100% spin-polarized muons are implanted into a sample and after coming to rest the muon spin precesses in the local magnetic environment. The muons decay with a half-life of 2.2 μs, emitting a positron preferentially in the direction of the muon spin at the time of decay. Two sets of detectors, up- and downstream of the sample, count the number $F(t)$ and $B(t)$ of decay positrons emitted from the sample. The asymmetry of the μSR time spectrum is then obtained as $A(t) = [F(t) - \alpha B(t)]/[F(t) + \alpha B(t)]$, where α represents a relative counting efficiency of the forward and backward detectors [18,19]. The data were normalized after subtracting a well characterized constant background signal in the asymmetry time spectra that arises from the high-purity silver sample holder. For the ZF muon experiments, stray fields at the sample position were compensated to within 1 μT by an active compensation coils system. LF spectra were collected with magnetic fields up to 0.25 T applied along the direction of the incident muons.

III. EXPERIMENTAL RESULTS AND DISCUSSION

Let us start with the temperature dependence of the ZF asymmetry time spectra, which are shown in Fig. 2 for the powder and the single crystal. Both data sets have been normalized by the value $A(t = 0,\ T = 0.6\ \mathrm{K})$. In the case of the powder sample, the asymmetry shows an exponential decay with time at 0.5 K. This indicates that the Yb spins are totally fluctuating in time, yielding a slow relaxation of the muon spins. The relaxation becomes faster with decreasing

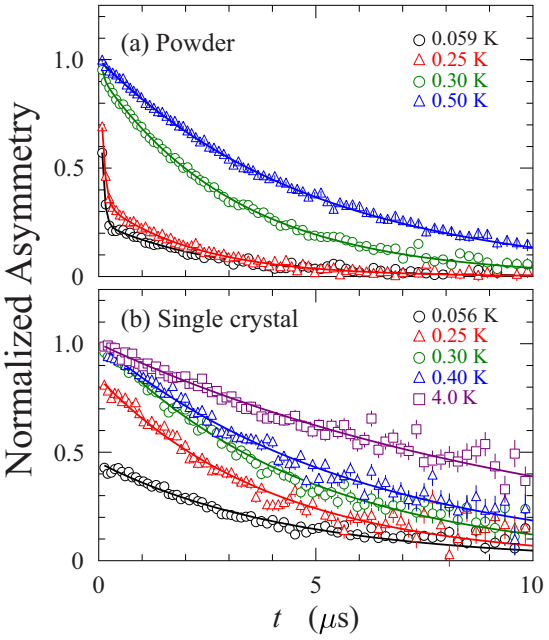

FIG. 2. (Color online) ZF μSR asymmetry time spectra for (a) the powder and (b) the single-crystal samples of $Yb_2Ti_2O_7$ between 0.05 and 4 K.

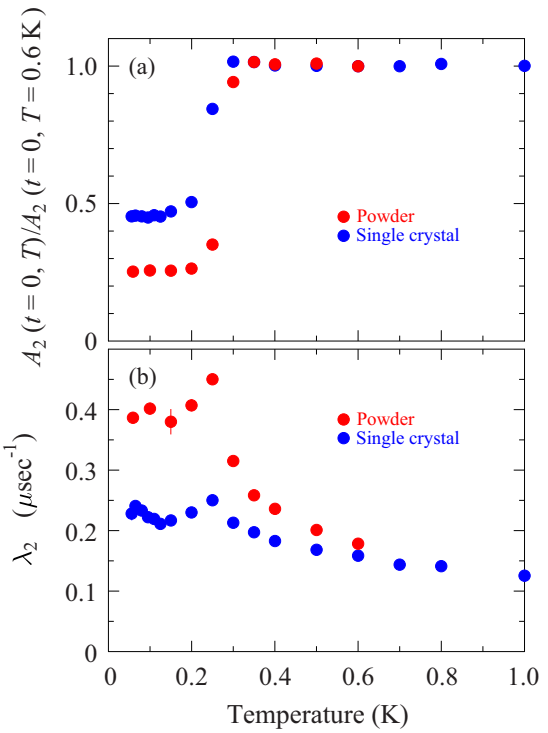

FIG. 3. (Color online) Temperature dependence of the normalized ZF μSR asymmetry, $A_2(t=0,T)/A_2(t=0,T=0.6\,\mathrm{K})$, and the depolarization rate, λ_2, of the slowly depolarizing component for (a) powder and (b) single-crystal samples of $Yb_2Ti_2O_7$.

temperature down to 0.3 K. Eventually, at 0.25 K, slightly below the T_C determined from the $C(T)$ data, the asymmetry exhibits a kink, which is characterized by a steep initial drop in a fast time domain within 0.5 μs, followed by a slow relaxation. The observation of an initial drop in the asymmetry indicates that the muon spins are depolarized more quickly than in the pulse duration of 70 ns. This provides strong evidence that the sample contains magnetic moments which are either static or quasistatic within the μSR time window 10 ps to 1 μs [20]. Our results on this powder sample are comparable with previous results on other polycrystalline samples [8]. The single crystal shows similar behavior to our powder sample, but the relaxation of the muon spins is slightly slower at both 0.4 and 0.3 K. At $T \leqslant 0.25$ K, we again observe a rapid initial drop in the asymmetry followed by a slow relaxation, although the size of the initial decrease in asymmetry is reduced from that of the powder.

These μSR time spectra can be analyzed successfully by using a two-exponential relaxation function,

$$A(t) = A_1 e^{-\lambda_1 t} + A_2 e^{-\lambda_2 t}, \quad (1)$$

with the first and second terms representing the fast and slow relaxation components, respectively, i.e., the relaxation rate $\lambda_1 > \lambda_2$. This gives a minimal effective model for treating magnetic transitions and a magnetically ordered volume fraction in ordered phases [20]. Figure 3(a) shows the value of the initial asymmetry, $A_2(t=0)$, normalized by the value at 0.6 K, for the slowly relaxing component of both the powder and single-crystal samples. $A_2(t=0)$ is nearly temperature independent at high temperatures, starts to decrease below 0.35 and 0.3 K for the powder and the single crystal, respectively, and falls to an almost constant value below 0.2 K. Note that $A_2(t=0)$ reduces to about $\frac{1}{4}$ and $\frac{1}{2}$ of its high-temperature values for the powder and the single crystal, respectively. This means that the volume fraction of static or quasistatic magnetic moments is estimated to be nearly 100% for the powder, since it is close to an ideal value $\frac{1}{3}$, and about 80% for the single crystal [20]. Figure 3(b) shows the relaxation rate λ_2 for the slowly relaxing component of both the powder and single-crystal samples. The relaxation rates are almost the same at around 1 K, are gradually enhanced with decreasing temperature, reach the maximum around 0.25 K, and are nearly saturated at lower temperatures. However, λ_2 for the powder data exhibits a stronger enhancement by a factor of about 2, as compared with the single-crystal data. The peak of λ_2 around 0.25 K indicates that the time scale of magnetic fluctuations is slowing down and crosses the μSR time window, 10 ps to 1 μs, around this temperature. Although it is thought that the transition here is first order, it is accompanied by a moderately high intensity of slow magnetic fluctuations.

To confirm that the "nearly static magnetic moments" observed with the ZF μSR results are indeed static, we have also measured the asymmetry with longitudinal fields applied along the direction of the initial muon spin polarization. Figures 4(a) and 4(b) show the LF μSR time spectra observed at 0.1 and 0.7 K on the powder sample and Figs. 4(c) and 4(d) those at 0.075 and 0.7 K for the single crystal. Again, all the curves are normalized by the value $A(t=0,\ T=0.6\,\mathrm{K})$ at zero field and then fitted using Eq. (1). Clearly, the slower relaxation rate λ_2 decreases monotonically with increasing longitudinal field both well below and well above T_C. In particular, at 0.7 K, the asymmetry still shows a single slow exponential time decay even in a magnetic field of 0.25 T.

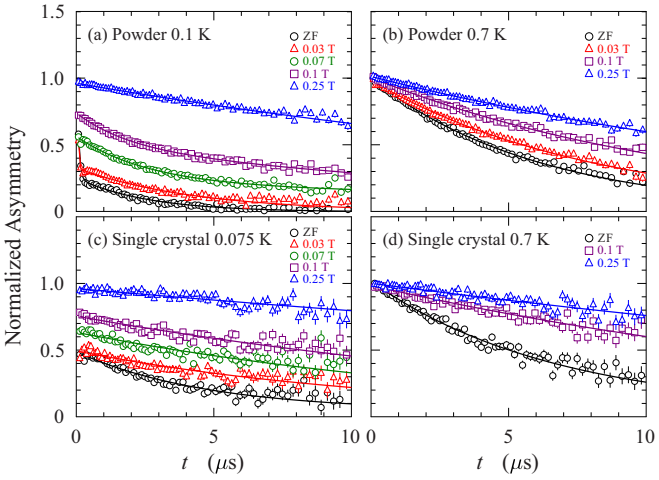

FIG. 4. (Color online) LF μSR asymmetry time spectra for polycrystalline $Yb_2Ti_2O_7$ at (a) 0.1 and (b) 0.7 K and single-crystal $Yb_2Ti_2O_7$ at (c) 0.075 and (d) 0.7 K.

This indicates that all the Yb spins are fluctuating and that some of the magnetic excitation spectra lie in the μSR time window in this field range. On the other hand, well below T_C (at 0.1 K for the powder and at 0.075 K for the single crystal), with increasing field, the whole asymmetry spectra shows an upward parallel shift with no crossing. This shift is accompanied by a recovery of the initial loss in the asymmetry and a reduction of the long-time depolarization rate λ_2. This decoupling behavior seen in the time spectrum is typical for a case in which a static internal magnetic field appears at a muon site. Namely, this provides evidence that the magnetic moments inside both the powder and single-crystal samples are indeed static. Note that an observation of the muon spin precession is not necessary to draw this conclusion [21].

We can try to estimate the magnitude of the ordered internal magnetic field. To do this we follow a method used previously [21] and discussed in detail in Ref. [22], The equation adopted is

$$A_2 = A_0\left\{\frac{3}{4} - \frac{1}{4x^2} + \frac{(x^2-1)^2}{16x^3}\ln\frac{(x+1)^2}{(x-1)^2}\right\}, \qquad (2)$$

where $x = H_{\rm LF}/H_{\rm int}$, $H_{\rm LF}$ is the applied external field, and $H_{\rm int}$ is the internal field at the muon site. There are certain conditions for this function to be applicable. The internal fields at muon sites must have the same amplitude but their directions at different sites throughout the sample may be random. These conditions can be realized in powder samples. Adopting this method the magnitude of the ordered internal magnetic field for the polycrystalline sample can be estimated from the initial drop in the asymmetry at zero field to be 65 ± 3 mT. A similar calculation for the single-crystal sample gives a value of 71 ± 4 mT. This latter value must be treated with some caution given the limitations of the analysis discussed above. Nevertheless, we included it here for completeness. We also note that the initial drop in the asymmetry eventually disappears at 0.25 T, which is actually of the order of T_C.

We now compare our results with previous work. Hodges *et al.* [8] reported the results of ZF μSR measurements on a powder sample. Their results are in reasonable agreement with ours, but are insufficient to conclude that the magnetic moments in the sample are either static or quasistatic, because they did not perform LF μSR measurements. Our LF μSR data for the powder sample are quite similar to those obtained by de Réotier *et al.*, although the presence of a clear minimum in their relaxation data at short times led them to suggest that the muons were detecting a field distribution influenced by disorder, and that this disorder probably resulted from the geometrical frustration of the magnetic interactions [23]. They used a Gaussian-broadened Gaussian function that assumed that the muons see different environments, each of them characterized by a dynamical Kubo-Toyabe function, to fit their data and concluded that $Yb_2Ti_2O_7$ presents dynamical short-range correlations at low temperature. There is no strong evidence for a shallow minimum at short times in the LF relaxation data for our powder sample, although there is a hint of such a dip in the data collected in 0.03 T [see Fig. 4(a)]. We see no dip the LF asymmetry spectra for the single crystal. As discussed above, our data are consistent with the moments being static, while a disordered but static scenario is precluded by the absence of any residual magnetic entropy at the lowest temperature in our heat capacity data.

Recently, D'Ortenzio *et al.* also performed ZF and transverse-field μSR experiments on different powder and single-crystal samples [13]. However, their asymmetry time spectra never show any significant difference in the initial ($t = 0$) value between 1 K and 16 mK, which are strikingly different from those obtained by Hodges *et al.* [8] and in our work.

The differences between our μSR results and those of Ref. [13] could be ascribed to a sample dependence issue. In particular, the specific heat of single crystals strongly depends on the sample. For the single crystals studied in Refs. [11] and [13], $C(T)$ only exhibits a broad bump followed by a large tail continuing down to 0.1 K, at which temperature the value of $C(T)$ is about five times larger than that of our powder sample (see Fig. 1). The single-crystal $C(T)$ data of Ref. [12], in which long-range magnetic order was not observed with neutron scattering [10], has a peak at 0.26 and broad shoulder around 0.20 K. To the best of our knowledge, μSR experiments have not been reported on this sample. Our single-crystal exhibits a single sharp peak in $C(T)$ at $\sim$0.21 K. The magnitude of $C(T)$ at 0.1 K is still double that of our powder sample. This is qualitatively consistent with the estimated 80% for the static magnetic volume fraction of our single-crystal sample compared with an almost perfect (100%) magnetic volume fraction for our powder sample.

In the case of powder samples, the differences between our μSR results and those of Ref. [13] cannot simply be understood from the behavior of the specific heat, since the different powder samples used for our work, by Yaouanc *et al.* [11], and by D'Ortenzio *et al.* [13] all exhibit similar anomalies in $C(T)$ at the temperature of $\sim$0.26 K which is higher than the $C(T)$ anomaly reported in the early powder work [7] as well as for our single crystal; see Fig. 1. Finally we note that in the case of the first-order phase transition expected here, measurements of $C(T)$ made using a heat pulse-relaxation method may not fully determine the true peak height, since one needs to measure the latent heat.

IV. CONCLUSIONS

Our results provide compelling evidence that in both our powder and single-crystal samples of $Yb_2Ti_2O_7$, the Yb moments undergo long-range magnetic order below T_C ($\sim$0.26 K for the powder and $\sim$0.21 K for the single crystal). The μSR results indicate the formation of static magnetic moments, while the loss of magnetic entropy from high temperature to well below T_C indicates that these static magnetic moments are ordered.

The data are consistent with our previous observations on the same single-crystal sample of a ferromagnetic order with an extremely slow relaxation by neutron diffraction [9] and polarized diffuse neutron scattering experiments [3]. The suppression of the low-frequency magnetic excitation spectral density within the μSR time window below T_C is compatible with a Higgs mechanism by which pyrochlore "photons" acquire an energy gap due to a coupling to spinons that carry emergent magnetic monopoles [1].

Uncertainties in estimating both the magnetically static volume fraction using μSR and the magnetic entropy associated with order from heat capacity data leave open the possibility that either a fraction of the magnetic moments throughout the bulk of the sample, or all the moments in some macroscopic portion of the samples (especially for the single crystal), remain dynamic or disordered. Using the data presented here, it is problematic to comment on the structure of the magnetic order, i.e., whether it is a simple ferromagnet or has a ferromagnetic component. Why some samples, and particularly some single crystals, appear to exhibit long-range magnetic order while others do not also remains an open issue. Clearly more research, and especially work which correlates the low-temperature magnetic properties with the structure and chemistry of $Yb_2Ti_2O_7$, will be required, before the properties of this fascinating and important material are fully understood.

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