Power spectrum of magnetic relaxation in spin ice: Anomalous diffusion in a Coulomb fluid

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Magnetization noise measurements on the spin ice $\mathrm{Dy_2Ti_2O_7}$ have revealed a remarkable "pink noise" power spectrum S(f,T) below 4 K, including evidence of magnetic monopole excitations diffusing in a fractal landscape. However, at higher temperatures, the reported values of the anomalous exponent b(T) describing the high-frequency tail of S(f,T) are not easy to reconcile with other results in the literature, which generally suggest significantly smaller deviations from the Brownian motion value of b=2, that become negligible above T=20 K. We accurately estimate b(T) at temperatures between 2 and 20 K, using ac susceptibility measurements that, crucially, stretch up to the relatively high frequency of $f=10^6$ Hz. We show that previous noise measurements underestimate b(T) and we suggest reasons for this. Our results establish deviations in b(T) from b=2 up to about 20 K. However, we confirm that b(T) is sample dependent: The details of this dependence agree in part, though not completely, with previous studies of the effect of crystal defects on monopole population and diffusion. Our results establish the form of b(T) which characterizes the subtle, and evolving, nature of monopole diffusion in the dense Coulomb fluid, a highly correlated state, where several dynamical processes combine.

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Introduction. Noise with a nonclassical exponent—so-called "pink" noise—has been proposed as a ubiquitous occurrence in physics that has been observed in dynamical systems as diverse as star luminosity, river flow, resistivity [1,2], and models of self-organized criticality [3]. Intermediate exponents generally reflect the influence of an underlying fractal structure or critical behavior, comprising many length scales or timescales [4], but identifying such fractals can be challenging. In this context, it is of particular interest that magnetization noise measurements on the spin ice $\mathrm{Dy_2Ti_2O_7}$ have connected a pink noise power spectrum S(f,T) below 4 K, to magnetic monopole excitations diffusing in an emergent fractal landscape [5].

The spin ice material $Dy_2Ti_2O_7$ has long been of principal interest among highly frustrated magnets. Large crystal field splittings combine with the large (10 μ_B) fields of the magnetic Dy^{3+} ions to create an excellent approximation to classical Ising spins. These occupy a pyrochlore lattice of corner-sharing tetrahedra, whose triangular faces lead to geometrical frustration in which the spins cannot simultaneously

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minimize their energies. Polarised neutron scattering at $T < 1~\rm K$ reveals spin correlations characteristic of "ice rule" states in which two spins point in, and two out, of each tetrahedron in agreement with a residual entropy measured by specific heat [6]. Part of the excitement about spin ices is that the minimal excitations would take the form of mobile, localized sources and sinks of magnetization, resembling classical analogs of magnetic monopoles [7,8]. Spin ice may be pictured as a dilute Coulomb gas of magnetic monopoles below $\sim 1~\rm K$ and an increasingly dense Coulomb fluid of both single- and double-charge magnetic monopoles above that temperature [9].

A new tool to probe monopole dynamics came from theoretical proposals to use magnetic noise spectroscopy [10,11], which passively probes the magnetic field fluctuations outside the sample. This technique, which has previously been applied to spin glasses, for example Ref. [12], allows a direct measurement of the noise spectral density S(f, T), and was used to confirm the fractal hopping environment in spin ice [5]. The function S(f, T) can alternatively be inferred from neutron scattering or from response measurements such as ac susceptibility, by means of the fluctuation-dissipation theorem [13]. Methodology based on the response function is the standard approach, but it can be valuable to compare the two different measurements of S(f, T) in cases of slow and complex dynamics, as afforded by spin glasses [12] and spin ice [14–16] where the fluctuation-dissipation theorem may not apply [17]. Here, we focus on a temperature regime where we expect the fluctuation-dissipation

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theorem to apply, so the two measures of S(f, T) should be identical [18].

In the case of spin ice, numerical simulations suggested that magnetic monopoles would lead to a noise spectral density as a function of frequency $S(f,T) \sim 1/f^{b(T)}$, with $1 \leq$ $b \leq 2$ varying with temperature T, at frequencies above about 1 kHz [11]. This "pink noise" would therefore be measurably distinct from the b=2 "red noise" expected for a simple paramagnet. This prediction was quickly confirmed experimentally using superconducting quantum interference device (SQUID) magnetometry on Dy₂Ti₂O₇, with pink noise measured at temperatures between 1 and 4 K, and frequencies up to 2.5 kHz. The anomalous b(T) was found to decrease with increasing temperature, from b = 1.5 at T = 1 K to b = 1.2at T = 4 K [19]. A subsequent noise measurement in the temperature range 0.8–3.8 K, with frequencies up to 10⁵ Hz, found close agreement, with b = 1.6 at T = 1 K again decreasing to b = 1.2 at T = 3.8 K [20]. Recent theoretical work has proposed a mechanism by which b = 1.50 can arise from the motion of isolated magnetic monopoles, relevant at the lowest temperatures of the spin ice regime (around 650 mK), as a result of their seeing an effectively fractal hopping environment [5].

However, these measurements have revealed a central mystery. Above 20 K it is well established from neutron spin echo experiments that the classical spin ice materials based on Dy and Ho have b=2 [21,22]. While the data are more complete for $\text{Ho}_2\text{Ti}_2\text{O}_7$ [22], it is clear that $\text{Dy}_2\text{Ti}_2\text{O}_7$ behaves in essentially the same way and on a similar temperature scale. Hence we can certainly expect a crossover to b=2 around T=20 K. Yet the noise-measured b(T) are seen to *decrease* from b(1 K)=1.5 with increasing temperature, with no sign of the expected approach to b=2 [19,20].

The aim of the present Letter is to accurately identify the exponent b(T), and to understand how it reaches b(20 K) = 2. To obtain unbiased estimates of S(f,T) we argue that frequency-domain measurements are required (here, ac susceptibility). This is in contrast to noise measurements, which are taken in the time domain and which effectively require a Fourier transform.

The problem is subtle, but errors in noise-based estimates of b(T) could be generic. First, the Nyqvist sampling theorem states that continuous signals can be accurately reproduced from periodic samples provided the sampling rate is at least half of the maximum frequency in the signal [23]—but physical signals are never truly band limited. This problem becomes of concern when sampling pink noise spectra (with powerlaw correlations), which are characterized by their significant weight at high frequencies. In order to apply a Fourier transform to a real-time pink noise signal to obtain S(f, T), a high-frequency cutoff must be introduced. A point that has yet to be discussed in spin ice studies is that this will create significant aliasing across the full spectrum, not just at high frequencies. The effect of such aliases is well known: they reduce the apparent b(T). This problem was discussed in detail by Kirchner [24], who introduced an unbiased method of spectral filtering to remove pink noise aliases. A second possible reason is the absence of a demagnetizing correction: Assuming the fluctuation-dissipation theorem, zero-field noise should concur with low-field ac susceptibility measure-

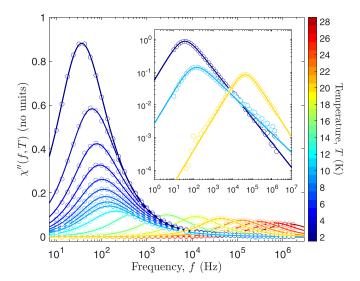


FIG. 1. Imaginary part of the intrinsic, differential magnetic susceptibility, $\chi''(f,T)$, of single-crystal Dy₂Ti₂O₇ along the [111] direction for various temperatures as a function of frequency. Inset: $\chi''(f,T)$ for selected temperatures $(T=2,\ 10,\ \text{and}\ 20\ \text{K})$ on a log-log scale to highlight the asymmetric deviation from Debye relaxation. The solid lines are fits to a Biltmo-Henelius function for $\chi''(f,T)$ [28].

ments for any given sample [18], but to compare with any idealized model, the noise [24], as the susceptibility [25], will need to be corrected for sample-shape-dependent (demagnetizing) effects.

Using ac susceptibility, we confirm that, while showing some sample variation, b(T) is significantly closer to 2 than implied by previous noise measurements. Nevertheless, our results confirm the key observations of earlier studies: that b < 2 at low temperatures, decreasing with increasing temperature; but that b = 2 by 20 K. We find that b(T) reaches a minimum at around 11 K, raising the question of which physical processes occur at this temperature.

Methods and results. Our ac susceptibility measurements were performed in a Quantum Design physical properties measurement system (PPMS). For measurements between $10^1 \le f \le 10^4$ Hz we employed the alternating current magnetic susceptibility (ACMS) option of the PPMS. For measurements in the range $f > 10^4$ Hz we employed a unique, high-frequency magnetic susceptometer developed to operate within the PPMS cryostat up to frequencies $f \sim$ 10⁶ Hz [26]. The extension to much higher than usual frequencies gives a crucial handle on the asymptotic power-law behavior of S(f) at higher temperatures. In all cases data can be overlapped in the common frequency regime and demonstrated to be in the linear response regime for excitation fields between 0.04 and 0.4 mT. The sample was zero-field cooled to the base temperature of the PPMS cryostat, T=2 K, and ac susceptibility measurements were performed with no static magnetic field as a function of increasing temperature for $2 \leqslant T \leqslant 28$ K. The Dy₂Ti₂O₇ single-crystal sample used in this study is the same as that studied previously in Ref. [27]. For all measurements a demagnetization factor N = 0.367was applied and the crystal was aligned along the [111] direction with respect to the excitation field. The complex

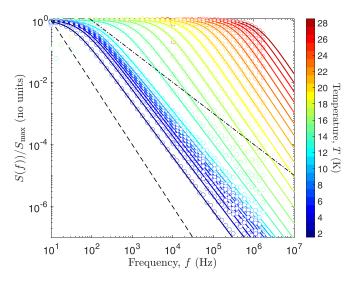


FIG. 2. Normalized power spectrum of magnetic noise, $S(f,T)/S_{\max}(T)$ [from fitting Eq. (2)] as a function of frequency, for a single crystal of $\mathrm{Dy_2Ti_2O_7}$ along the [111] direction for various temperatures as a function of frequency on a log-log scale. Solid lines are fits to Eq. (1). The dashed-dotted and dashed lines show $S(f) \propto f^{-1}$ and $\propto f^{-2}$, respectively.

susceptibility was measured up to frequencies of 3 MHz, an order of magnitude higher than the highest frequency that could be resolved in the noise measurements at all measured temperatures (2–22 K).

Starting with the complex intrinsic susceptibility $\chi' + i\chi''$, we obtained the function S(f, T) by means of the classical fluctuation-dissipation theorem [13]

$$S(f,T) = \frac{2k_B T}{hf} \chi''(f,T), \tag{1}$$

where h and k_B are Planck's and Boltzmann's constants. respectively. Figure 1 shows the frequency dependence of $\chi''(f,T)$ measured for a series of temperatures for the $\mathrm{Dy_2Ti_2O_7}$ sample. The inset shows a few temperatures for clarity on a log scale and the asymmetry demonstrates that, at the lower measured temperatures, the distribution is not well described by a single Debye relaxation process (which behaves as ω^1 and ω^{-1} in the low- and high-frequency limits, respectively). This phenomenon has been well studied in the context of spin glasses, where, as the spin-freezing temperature is approached from above, $\chi''(f,T)$ often exhibits appreciable asymmetry with respect to logarithmic frequency. Such behavior is also observed in the dilute dipolar Ising magnet $\mathrm{LiY}_{1-x}\mathrm{Ho}_x\mathrm{F}_4$ (x=0.045) [28,29].

The measured curves of S(f,T) are shown in Fig. 2 where it is clear that the high-frequency slope indicates an exponent b(T) much closer to 2 than to 1 at all temperatures. We determined the exponent b(T) roughly by fitting the high-frequency gradient, and in more detail by fitting $\chi''(f,T)$ to a Biltmo-Henelius (BH) [28] function and then transforming this to S(f,T) to enable extraction of the exponent as described by

$$S(f,T) = \frac{A\tau(T)}{1 + [f\tau(T)]^{b(T)}}. (2)$$

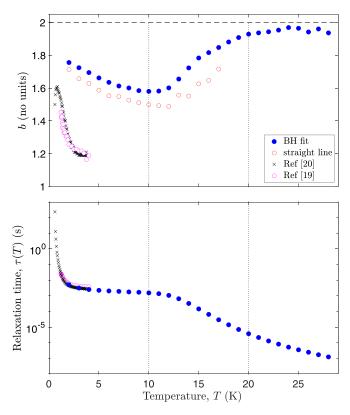


FIG. 3. Top: Temperature dependence of b(T) characterizing the distribution of relaxation times from the ac susceptibility with the exponents obtained from Eq. (2) and a straight line fit. Also plotted are the data obtained from the previous noise measurements. Bottom: Relaxation time as a function of temperature, $\tau(T)$, extracted from fits of $\chi''(f,T)$ to Eq. (2) compared with $\tau(T)$ from the published noise data on Dy₂Ti₂O₇ [19,20].

This function can be derived from a restricted version of the phenomenological Biltmo-Henelius $\chi''(f,T)$ originally introduced for spin glasses [28] to describe any asymmetry in the relaxation process around the response peak. It is seen to fit the data well. It is likely to give more accurate estimates of b(T) than the simple gradient method, particularly at high temperatures, where a limited frequency range above 100 kHz means that the estimate of b(T) will suffer from systematic errors.

Results for the two estimates of b(T) and the single estimate of τ are shown in Fig. 3, where they are compared with results estimated from the noise measurements of Refs. [19,20]. It is clear that the noise measurements severely underestimate b(T) while the susceptibility measurements connect the low-temperature regime with the neutron scattering results [21]. As explained above, we expect estimates of b(T) based on the curve fitting method [Eq. (2)] to be more accurate than those based on the gradient method, but the two follow the same trend and their comparison at lower temperatures gives some idea of how the observed slope on the log-log plot differs from its asymptotic value.

Discussion and conclusion. In comparison to the estimates of b(T) derived from the noise measurements [19,20], the accurate b(T) determined here remains anomalous but is always significantly closer to the paramagnetic value of b=2

than the noise-estimated value of b=1.2, and agrees with the crossover to b=2 at ~ 20 K observed by neutron scattering. The higher values of b(T) are consistent with the fact that monopole dynamics can be represented, on average, as Brownian motion in this regime. For example, based on measurements on the same crystal as used here, the mean mobility and mean diffusion constant (as defined with respect to the grand canonical ensemble) have been shown experimentally to accurately obey the Nernst-Einstein relation [27].

The thermal evolution of the relaxation rate derived from our fits suggests that effectively Arrhenius activated dynamics start to become relevant only above about 11 K. This agrees with the early susceptibility study of Matsuhira [30] and is consistent with neutron spin echo measurements, which found evidence for Arrhenius activation in $\mathrm{Dy_2Ti_2O_7}$ at temperatures of the order 20 K and above [21]. This is understood to be an Orbach-like phonon assisted spin flipping (or monopole hopping) associated with higher crystal field states [31] as noted in other work [20].

The absolute values of b(T), however, show significant sample variation. Reference [30] reported $b \approx 1.5$ at 17 K, which contrasts with our value of ≈ 1.8 . We measured b(T) on several samples of varying quality and isotopic composition. Samples showed a similar temperature variation but with absolute values of b(T) dipping as low as 1.48, and varying by no more than ≈ 0.3 from our reported values at $T \approx 2$ K. The values of b(T) previously derived from noise measurements remain anomalously low, but they may be influenced by sample variation, in addition to the systematic error identified above.

Sala *et al.* [32] showed how the precise relaxation rate and exponent b(T) can be affected by small concentrations of defects in the crystal structure, as these can affect the diffusion and density of monopoles. However, while they found that slower diffusion correlates with lower values of b(T), our measurements indicated the opposite, suggesting that there is still a lot to understand about the detailed role that defects play in monopole diffusion.

Our results have implications for the measurement of anomalous noise exponents in magnetic systems, as well as for the physical processes in spin ice itself.

For the estimation of the exponents b(T), we have demonstrated the value of measuring susceptibility to high frequencies and we have identified possible systematic errors in the analysis of the noise experiments for b(T). An obvious question is whether such errors also apply in the regime at T < 1 K where the anomalous value of b(T) is believed to be a signature of the effectively fractal hopping landscape for monopoles. In this regime the relaxation timescale slows considerably, and a number of processes are thermally frozen out.

Indeed, Morineau *et al.* speculate the measured susceptibility means smaller corrections to the noise measurements at low temperature [18], and measure a value of b(T) in accordance with the picture of monopole hopping within a fractal landscape [5]. However, for noise measurements the filtering method of Kirchner [24] should be applied even when standard antialiasing methods, which involve a defined experimental cutoff, have been implemented [5,20]. The noise should be compared directly to ac susceptibility, including shape-dependent demagnetizing corrections [25,33,34], to extract an accurate value of b(T) to compare with theory [18].

There remains a good chance that b(T) extracted from noise at the lowest temperatures remains correct, in accordance with the picture of monopole hopping within a fractal landscape [5]. However, the point remains that any pink noise data sampled in the time domain (numerical or experimental) require spectral filtering to avoid significant aliasing across their spectra.

With regard to physical processes, it is important to clarify that the power spectral density, whether estimated by noise or susceptibility measurements, is not a spectroscopic probe of any individual dynamical process, but instead reflects the combination of all processes present: It can only distinguish these if they are well separated in frequency. In spin ice the magnetization relaxes by means of the thermal excitation and diffusion of both single- and double-charge monopoles [35]. The actual dynamics involved include correlated quantum tunneling [36] related to details of the local magnetic field, and phonon-assisted spin flipping [31]. In the dense monopole regime, monopole motion is highly correlated, partly as a result of local constraints [35] and partly as a result of the manybody Coulomb interaction [9]. A result of this complexity is that while, on average, monopole diffusion can be approximated as Brownian motion, in detail it differs from that. In the lower-temperature regime (T < 1 K) and at high temperature (T > 20 K), the picture considerably simplifies as a more limited type of dynamics dominates in each case. While theoretical models [5,31,37] have success in accounting for aspects of the dynamics in these limits, the evolution of the anomalous relaxation with temperature in the regime of 1-20 K remains to be understood in detail. In accurately characterizing the magnetic relaxation in this regime, we hope to facilitate future theories of this fascinating highly correlated state.

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Data availability. The data that support the findings of this article are openly available [38].

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