Dipolar ferromagnets and glasses (invited)

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What is the ground state and what are the dynamics of 10^{23} randomly distributed Ising spins? We have attempted to answer these questions through magnetic susceptibility, calorimetric, and neutron scattering studies of the randomly diluted dipolar-coupled Ising magnet $LiHo_xY_{1-x}F_4$. The material is ferromagnetic for dipole concentrations at least as low as $x = 0.46$, with a Curie temperature obeying mean-field scaling relative to that of pure $LiHoF_4$. In the dilute spin limit, an $x = 0.045$ crystal shows very unusual glassy properties characterized by *decreasing* barriers to relaxation as $T \to 0$. Its properties are consistent with a single low degeneracy ground state with a large gap for excitations. A slightly more concentrated $x = 0.167$ sample, however, supports a complex ground state with no appreciable gap, in accordance with prevailing theories of spin glasses. The underlying causes of such disparate behavior are discussed in terms of random clusters as probed by neutron studies of the $x = 0.167$ sample. In addition to tracing the evolution of the glassy and ferromagnetic states with dipole concentration, we investigate the effects of a transverse magnetic field on the Ising spin glass, $LiHo_{0.167}Y_{0.833}F_4$. The transverse field mixes the eigenfunctions of the ground-state Ising doublet with the otherwise inaccessible excited-state levels. We observe a rapid decrease in the characteristic relaxation times, large changes in the spectral form of the relaxation, and a depression of the spin-glass transition temperature with the addition of quantum fluctuations.

INTRODUCTION

Dipolar interactions are always present in paramagnets and paraelectrics, and because of their long range, they can influence collective behavior even when the concentration of magnetic ions or paraelectric centers is small. Thus, the problem of interacting dipoles is a fundamental issue in classical statistical mechanics. Even so, there exist few definite results beyond those concerning the (anti)ferromagnetism of regular arrays of dipoles.[1,2] Most effort on random arrays has been devoted to systems which cannot be modeled simply in terms of interacting dipoles, but where other degrees of freedom, notably elastic, play important roles.[3] It is therefore of considerable interest to study more perfect realizations of the model of randomly situated interacting dipoles. In the present paper, we describe the new phenomena found in such a study, performed on the dipolar-coupled Ising magnet $LiHo_xY_{1-x}F_4$.

$LiHo_xY_{1-x}F_4$ is an isostructural dilution series where magnetic Ho^{3+} and nonmagnetic Y^{3+} ions randomly occupy the rare-earth (R) sites in the body-centered tetragonal $LiRF_4$ lattice.[4] The single-ion anisotropy is Ising with the moments ($\mu_{\text{eff}} = 7\,\mu_B$) derived from the ground-state doublet of Ho^{3+} lying parallel to the c axis. The dominant interaction between moments is dipolar, as directly demonstrated by neutron diffraction.[5,6] The pure compound, $LiHoF_4$, is a ferromagnet with an essentially perfect mean-field transition[7] at $T_c = 1.53$ K.

It is not even obvious what the ground state should be for a regular array of dipoles, such as in $LiHoF_4$. The anisotropy of the dipole-dipole interaction necessarily introduces competition between ferromagnetic interactions (e.g., one dipole under another) and antiferromagnetic interactions (e.g., one dipole next to another). Moreover, the long-range nature of the dipolar coupling means that many more than nearest-neighbor interactions must be taken into account. Luttinger and Tisza[1] solved this problem in the 1940s, showing that the ground-state ordering is determined by the lattice structure.

We wish to extend this conceptual puzzle by introducing the physics of disorder to the problem. Now, the crystal structure alone cannot be sufficient to determine the ground-state properties because only a fraction of the lattice sites are (randomly) occupied. In fact, for sufficient dilution, the long-range order, either ferromagnetic or antiferromagnetic, might no longer be viable.[8] We do indeed observe the passage from (long-range ordered) ferromagnet to (short-range ordered) spin glass with decreasing dipole concentration. However, the phase diagram for randomly distributed Ising dipoles turns out to be more complicated. At the lowest spin concentrations, the magnetic spin glass is replaced by a qualitatively different ground state which does not appear to freeze at finite temperature[9] and, in view of the long-range nature of the dipole interaction, is least expected.

Finally, we report what we believe to be the first studies of the Ising spin glass in transverse magnetic field. The first excited crystal-field level in $LiHo_xY_{1-x}F_4$ is 9.4 K above the ground-state doublet. At the low temperatures of

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our experiments, 0.025 K$\leqslant T \leqslant$0.25 K, only the Ising doublet is appreciably populated. As the states in the doublet are eigenstates of $J_{zz}(z\|c)$, the dipoles have no first-order response to a magnetic field H_t applied perpendicular to the c (Ising) axis. H_p however, can mix the eigenfunctions of the ground-state doublet, the first-excited-state singlet, and, to lesser extent, the higher level states. Hence, we can introduce the effects of quantum mechanics to an essentially classical problem in a controlled fashion.

EXPERIMENTAL METHODS

We obtained our single-crystal samples of $LiHo_xY_{1-x}F_4$ from Sanders Associates (Nashua, NH) or from Howard Guggenheim (AT&T Bell Laboratories). Compositions were determined by an air-water differential weighing technique.[10] Calorimetry using both pulse and relaxation methods was performed on approximately 30-mg single-crystal platelets. The addendum was $<1\%$ of the total signal at 1 K and negligible at the lowest T.

We used a variety of phase-sensitive inductance bridge techniques[6] to measure the real and imaginary parts of the ac magnetic susceptibility $\chi(f,T)=\chi'(f,T)+i\chi''(f,T)$ for a series of different concentration crystals at millikelvin temperatures. A broad frequency range (10^{-1}–10^5 Hz) was required to characterize the spectroscopic response of the glassy samples, where we used a combination of conventional and computer-based digital lock-in amplifiers.[11] The magnetic response for all samples was always in the linear regime, with excitation fields of order 10^{-3} G. For the transverse field experiment, we suspended a single crystal of $LiHo_{0.167}Y_{0.833}F_4$ of dimensions $1.6\times1.6\times5.1$ mm^3 from the mixing chamber of a helium dilution refrigerator inside the bore of an 80-kOe superconducting magnet, with its Ising (long) axis oriented perpendicular to the field direction. A compensation coil, oriented parallel to the sample's Ising axis, was used to null out any longitudinal field component.

We performed neutron-scattering experiments at the cold neutron facility of the Brookhaven high flux beam reactor. A single crystal of $LiHo_{0.167}Y_{0.833}F_4$, with dimensions $6\times6\times6$ mm^3, was placed in a dilution refrigerator mounted on a triple-axis neutron spectrometer. With incident and final neutron energies of 5 meV, we measured the diffuse, magnetic scattering near the (2,0,0) Bragg peak for temperatures between 0.15 and 1.5 K, as given by a carbon thermometer mounted next to the sample.

THE PHASE DIAGRAM

The parent compound, pure $LiHoF_4$, is a ferromagnet with a Curie temperature $T_c = 1.53$ K. The low-energy scale for the spins to order is a consequence of the relatively weak dipolar interaction. Random dilution of the magnetic Ho^{3+} sites with nonmagnetic Y^{3+} for dipole concentrations down to at least 46% preserves the ferromagnetism, but with a depressed characteristic energy scale. The critical temperature follows[6,12] a simple mean-field scaling with x: $T_c(x) = xT_c(x=1)$. Neutron-scattering studies[13] of $LiHo_{0.3}Y_{0.7}F_4$ find a ferromagnetic ground state, but with a T_c 20% below the mean-field prediction and with a saturation magnetic moment per rare-earth ion only 60% of that observed in the undiluted material. Not all the dipoles are able to participate in the long-range order. For $x\leqslant0.167$, the long-range order is completely destroyed. Magnetic glassiness ensues, but with what appear to be fundamentally different regimes of glassy response simply as a function of dipole concentration. We will focus our attention on a comparison between these magnetic glasses in the remainder of this section.

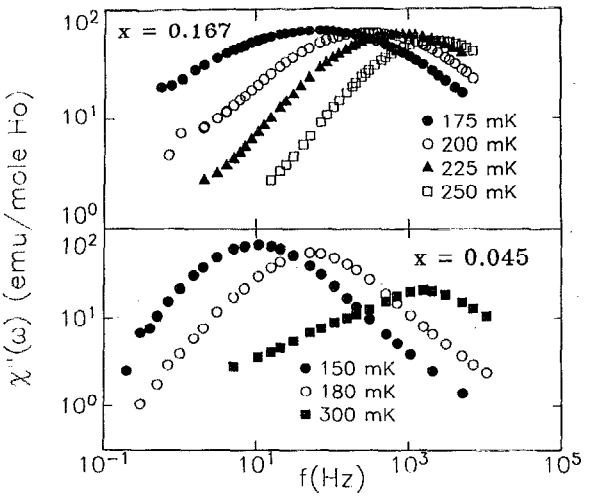

FIG. 1. Comparison of the broad magnetic response at a series of temperatures for $LiHo_xY_{1-x}F_4$ with $x=0.167$ and $x=0.045$.

We show in Fig. 1 log-log plots of the imaginary part of the magnetic susceptibility over many decades in frequency for $LiHo_xY_{1-x}F_4$ with $x=0.167$ and $x=0.045$. The low-frequency susceptibility essentially obeys a power-law frequency dependence, $\chi''(f)\sim f^{\alpha}$. The exponent α (the slope of χ'' as $f\to0$) decreases with decreasing T for $LiHo_{0.167}Y_{0.833}F_4$, as is typically observed in spin-glass systems approaching the freezing temperature from above.[14] By comparison, α increases with decreasing T for $LiHo_{0.045}Y_{0.955}F_4$.

The differing evolution with temperature of the magnetic dynamics in these two glasses is most easily illustrated in a scaling plot of $\chi''(f)$. Figure 2 is such a plot, where the data have been scaled at each temperature by dividing the frequency by the peak frequency f_p and the magnitude by $\chi''(f_p)$. The symmetrical broadening with *decreasing* temperature for $x=0.167$ in the top half of the figure is in marked contrast to the asymmetrical broadening with *increasing* temperature for the more dilute sample, $x=0.045$, shown in the bottom half of Fig. 2.

The magnetic dynamics of Ising systems are relaxational and, in the simplest case, dominated by a single relaxation time τ. The corresponding complex susceptibility is $\chi(\omega)=\chi_0/(1-i\omega\tau)$, with a fixed width of 1.14 decades of frequency. A glassy system has multiple routes to relaxation as reflected in a much wider dynamic frequency response. It is then appropriate to think in terms of a distribution of energy barriers to relaxation, $\rho(E_B)$. Each

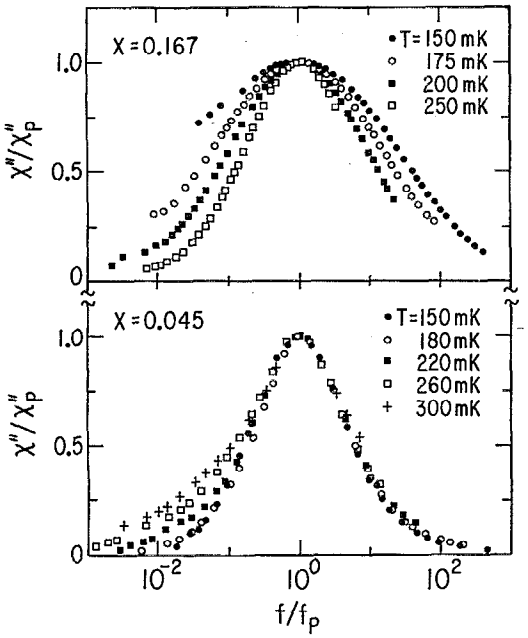

FIG. 2. Scaling plot where the imaginary part of the susceptibility broadens (narrows) with decreasing T for the more concentrated (dilute) sample.

barrier gives rise to a relaxation time via the Arrhenius form, $\tau(E_B) = \tau_0 \exp(E_B/kT)$, where τ_0 is a characteristic microscopic time and k is Boltzmann's constant. This gives for the susceptibility:

$$\chi(\omega)=\chi_0 \int_0^\infty \rho(E_B) \frac{dE_B}{1-i\omega\tau(E_B)}. \tag{1}$$

The results of Figs. 1 and 2 translate into a $\rho(E_B)$ which must evolve with T in an opposite manner for the two spin concentrations, independent of the details of the fitting forms. We compare in Fig. 3 the width of the distribution of energy barriers to relaxation, ΔE, vs T for the two magnetic glasses. The widths are determined from fits to $\chi(f,T)$ which are discussed at length in Refs. 6 and 9.

While $\chi(f,T)$ can be analyzed to yield the spectrum of energy barriers, the specific heat $C(T)$ is determined by the spectrum of excited states. We compare in Fig. 4 the temperature dependence of the specific heat for $x = 0.045$ and $x = 0.167$, normalized per mole of holmium. We have subtracted out a background term due to the nuclear hyperfine interaction. The data for the two samples essentially coincide at higher T, where C is dominated by the Schottky contribution of excitations from the ground-state Ising doublet to the first-excited-state singlet 9.4 K above. This high-temperature term should scale, as observed, with holmium concentration. At lower T, however, the thermal responses of the two glasses differ. For $x = 0.167$, $C(T)$ is typical of spin glasses with a broad maximum centered at 0.18 K, 40% above its transition temperature $T_g = 0.13$ K. Its amplitude is sufficiently large that for $0.13 < T < 1.0$ K, C accounts for 85% of the entropy ($R \ln 2$) associated with the (electronic) ground-state doublets. In contrast, in

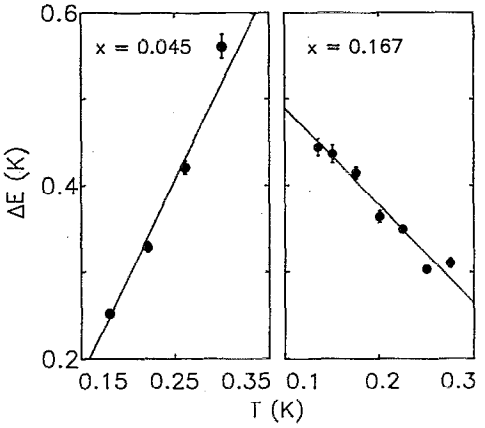

FIG. 3. The width of the distribution of energy barriers to relaxation evolves with temperature in an opposite manner for the two glasses.

the same temperature range, an extraordinarily sharp Schottky-like (see below) peak dominates $C(T)$ for $x = 0.045$. Its amplitude is well below that of the broad maximum for $x = 0.167$, and indeed accounts for only 20% of the electronic entropy.

Just as we generalized the simple Debye description of magnetic dynamics to analyze our susceptibility data, here we generalize the simple Schottky form for the specific heat. We take an excitation spectrum consisting of a discrete level with energy E_1 and degeneracy n relative to that of the ground state. The corresponding specific heat is

$$C/R=\frac{nE_1^2}{(kT)^2}\exp\left(\frac{-E_1}{kT}\right)\Big/\left\{1+n\exp\left(\frac{-E_1}{kT}\right)\right\}^2. \tag{2}$$

Narrower peaks are associated with higher excited-state degeneracies. We find $E_1/k = 0.39$ K and $n = 0.8$ for $x = 0.167$ ($T \lesssim 0.6$ K), while $E_1/k = 2.15$ K and $n = 890$ for the $x = 0.045$ peak. For $x = 0.167$, the spectrum of low-lying states is characterized by a typical energy of order $xkT_c(x = 1)$ and a degeneracy similar to that of the ground state. In contrast, the sharp feature in $C(T)$ for

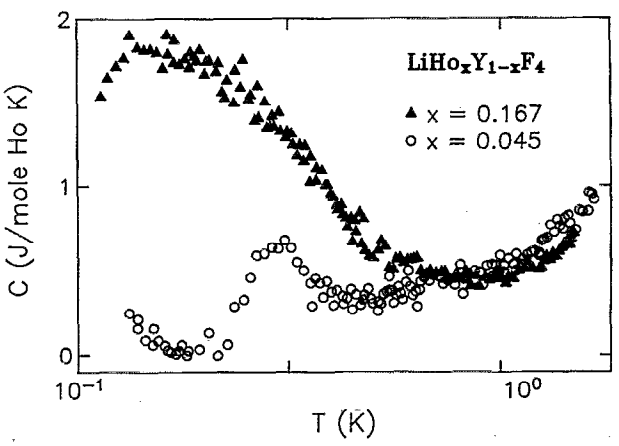

FIG. 4. Comparison of the electronic specific heats of the $x = 0.167$ spin glass and the $x = 0.045$ "decoupled cluster" glass.

$x = 0.045$ is due to excitations with an energy of order $kT_c(x = 1)$, i.e., the energy required to break a ferromagentic bond between near neighbors. That such clusters are rare is apparent from the low entropy associated with the peak.

We can combine our dynamical and calorimetric results to paint a qualitative picture of the evolution of the glassy state with dipole concentration. The ac susceptibility data for $LiHo_{0.045}Y_{0.955}F_4$ indicate that the distribution of energy barriers to relaxation narrows with decreasing temperature. If this behavior continues smoothly as the system approaches its $T = 0$ ground state, spin relaxation will be dominated by a single barrier. This is consistent with a ground state made up of spin clusters with zero net dipole moment.[9] The molecular field in which spin reorientation occurs is therefore effectively zero. As T is increased, some clusters are excited into states with nonzero moments, which then act on spins in other clusters. Thus, the distribution of molecular fields, and hence the distribution of energy barriers to spin relaxation, will broaden with increasing temperature.

The reorientation of the isolated clusters involves flips of individual dipoles, an energy of order the nearest-neighbor dipole-dipole interaction energy $E_B/k \sim 2$ K from the Arrhenius law.[12] Indeed, we find from our analysis of the specific heat of the $x = 0.045$ sample an energy $E_1/k = 2.15$ K, the energy of a spin flip in a nearest-neighbor pair. In contrast, the specific heat of $LiHo_{0.167}Y_{0.833}F_4$ is characterized by a lower energy, $E_1/k = 0.39$ K, presumably due to collective motions of many spins within an extended cluster where frustrated regions can reorient into spatially distinct configurations of roughly equivalent energies. Here, the splitting between "ground" and "excited" states is of order T and it is difficult to make a distinction between both highly degenerate levels.

How do we get from decoupled clusters to global freezing at finite temperature? Neutron scattering studies of the $x = 0.167$ spin glass provide a possible explanation. We plot in Fig. 5 elastic scans as a function of ζ in $\mathbf{Q} = (\zeta,0,0)$ for $LiHo_{0.167}Y_{0.833}F_4$ at a series of temperatures. The solid lines are fits to the mean-field theory for a uniaxial dipolar-coupled magnet.[5] The width of a peak gives the correlation length ζ in the plane *perpendicular* to the Ising axis.

Figure 5 shows that the $(\zeta,0,0)$ scan becomes both more intense and narrower as T is reduced, indicating that the ferromagnetic correlations are growing even in the basal plane. We find that ξ^2 increases from 2.6 ± 1.7 Å^2 at $T = 0.5$ K to 20 ± 4 Å^2 at $T = 0.15$ K. The spin-glass state in $LiHo_{0.167}Y_{0.833}F_4$ then would involve collective freezing of sizeable ferromagnetic clusters elongated along the Ising axis. The strong interactions between such clusters presumably give rise to the glassy behavior and the apparent freezing at this spin concentration. Sufficient interlocking inhibits the reorientation of the clusters into different spatial configurations which have essentially the same energy but opposite net moments. Furthermore, since the in-plane ferromagnetic correlations require assistance from out-of-plane neighbors, the formation of clusters should be concentration dependent. We believe that the $x = 0.045$ material is too dilute to have substantial in-plane ferromagnetic ordering (although the appropriate neutron experiments remain to be performed), and hence does not have the same type of cluster growth leading to freezing.

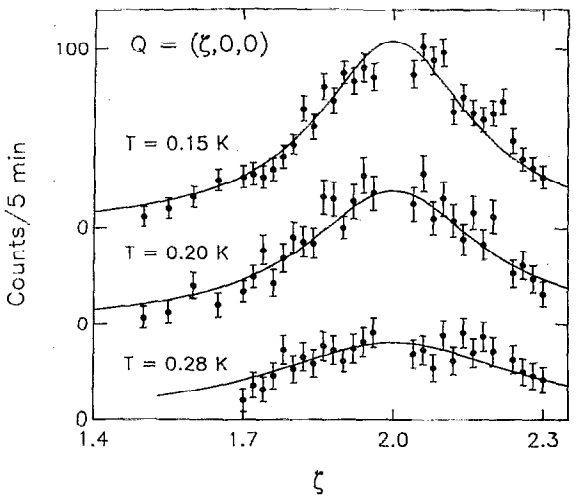

FIG. 5. Elastic neutron scattering shows the development of ferromagnetic correlations perpendicular to the Ising axis in $LiHo_{0.167}Y_{0.833}F_4$.

INTRODUCING QUANTUM MECHANICS

Until this point we have treated the dipoles as classical degrees of freedom. Quantum effects need only be considered at the lowest temperatures, well below the glass transition, where tunneling levels can contribute to the physics. However, if present in sufficient strength, quantum fluctuations could have an enormous impact on the spin-glass transition itself.

The Ising model for N interacting spins in transverse field Γ, with Hamiltonian

$$H = -\sum_{i,j}^{N} J_{ij}\sigma_i^z\sigma_j^z - \Gamma \sum_{i}^{N} \sigma_i^x, \tag{3}$$

is a theoretical construct which introduces quantum mechanics to a classical problem in a natural way. Here, spins i and j are connected by a random exchange J_{ij} and the σ's are Pauli spin matrices. Various authors[15–17] have considered spin glasses in transverse fields, with predictions ranging from the destruction of the spin-glass state[16] to an enhancement in the transition temperature[17] with the introduction of quantum fluctuations.

We plot in Fig. 6 the evolution with transverse magnetic field H_t of the lowest eigenvalues of the Hamiltonian for the single ion in the $LiRF_4$ crystal field. The principal effect of H_t is to introduce a splitting $\Gamma \propto H_t^2$ (at low H_t) of the doublet ground state. It is Γ and not H_t which appears in the model Hamiltonian of Eq. (3). In this context, the eigenstates of σ_i^x correspond to the two lowest lying states of the single-ion Hamiltonian.

We use the dynamic magnetic response of the $LiHo_{0.167}Y_{0.833}F_4$ spin glass as a whole to probe the evolution of the superposition of eigenstates with H_t, performing, in essence, a *collective* electron-spin resonance (ESR)

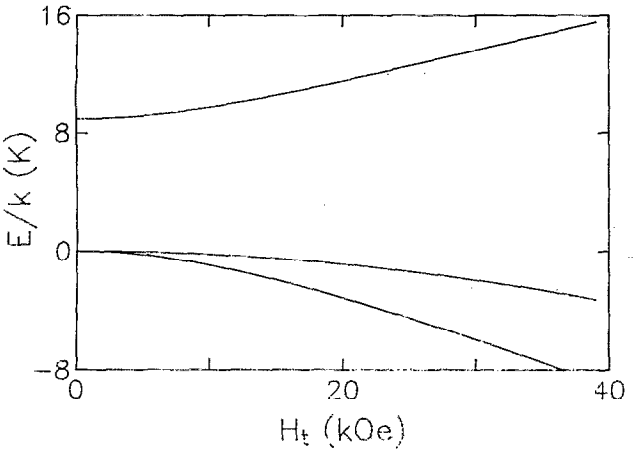

FIG. 6. Effect of transverse magnetic field on the single-ion energy levels, reflecting the mixing of the Ising doublet and excited-state singlets.

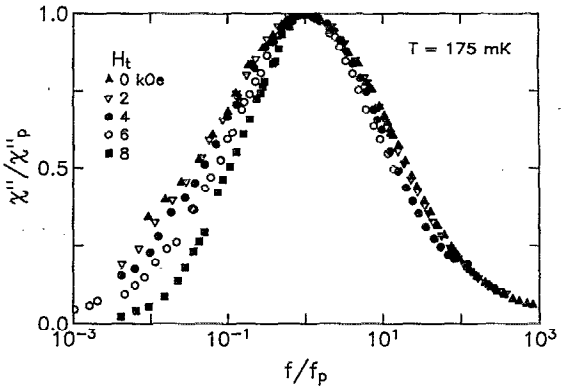

FIG. 8. Scaling plot showing that the long-time modes (low-frequency tails) are most dramatically affected by H_t.

experiment. We show in Fig. 7 $\chi''(f)$ for various H_t at $T = 0.175$ K, a temperature still above $T_g(H_t = 0)$. The application of a transverse field radically affects the time scale of this Ising system's response. The frequency of the peak f_p of χ'' increases by two orders of magnitude in only 6 kOe. Moreover, the low-frequency tails of $\chi''(f)$ are greatly suppressed, an effect which is most clearly seen in the scaling plot, Fig. 8. The narrowing of the low-frequency response with increasing H_t indicates that the new quantum routes to relaxation most profoundly affect the long-time modes.

Below $T_g(H_t = 0)$ the frequency dependence of the magnetic susceptibility is most unusual. We plot in Fig. 9 $\chi''(f)$ for various H_t at $T = 0.05$ K. In sufficient transverse field, $\chi''(f)$ mimics the data for $T > T_g(H_t = 0)$ (see Fig. 7). As H_t is decreased, the spectral response also moves to lower f. However, it becomes enormously broad and essentially flat over decades in frequency as $f \to 0$.

The $f \to 0$ limit of $\chi''(f)$ can be described by the power-law form f^{α}. We have used this form to fit the low-frequency portion of our data and we show in Fig. 10 the results for α vs H_t at several T. In other spin glasses, most notably $Eu_xSr_{1-x}S$, T_g is associated[14] with α approaching and then saturating at a small value, $\leqslant 0.1$. Furthermore, it is generally believed that the spin-glass state itself is characterized by $1/f$ noise in the magnetization,[18] which implies, via the fluctuation-dissipation theorem, that $\alpha = 0$. We therefore identify the temperature at which α approaches zero with T_g. Given that the effect of the transverse field is to introduce quantum fluctuations which aid the relaxation process, shifting the spectral response to higher f and increasing α, we expect T_g to decrease with increasing H_t. In addition, the application of H_t moves the spin-glass signature, $\alpha = 0$, into an accessible frequency window. At $T = 0.15$ K, α never reaches zero, indicating that $T_g(H_t = 0) < 0.15$ K. As T is lowered, we find increasingly robust regions of flat response, with α remaining zero to progressively higher H_t.

We show in Fig. 11 T_g as a function of H_t, where the open circles are determined by plotting α vs H_t at constant T (as in Fig. 10), and the filled circles are determined by plotting α vs T at constant H_t. The solid line is a least-squares fit to the form $T_g(H_t) = T_g(0)[1 - (H_t/H_c)^{\beta}]$, with $T_g(0) = 0.133 \pm 0.005$ K and $\beta = 1.7 \pm 0.1$. If this functional form continues below our lowest temperature

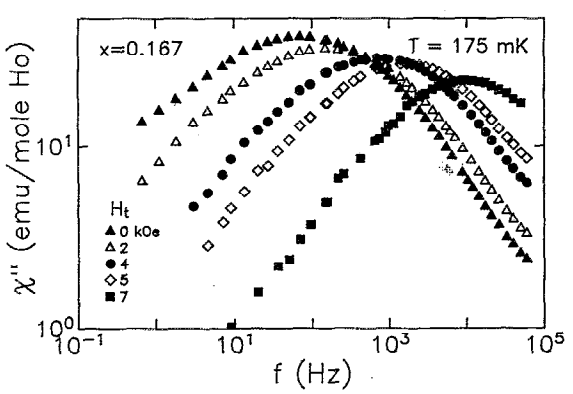

FIG. 7. The glassy response at $T > T_g$ speeds up dramatically as the transverse field introduces new quantum routes to relaxation.

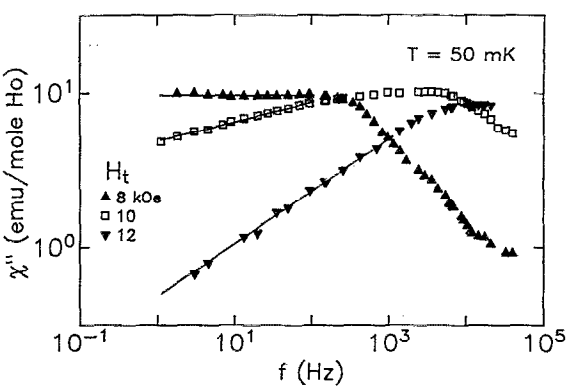

FIG. 9. Counterpart to Fig. 7 for $T < T_g(H_t = 0)$. Flat response at low f is characteristic of spin glasses and is suppressed by sufficient H_t.

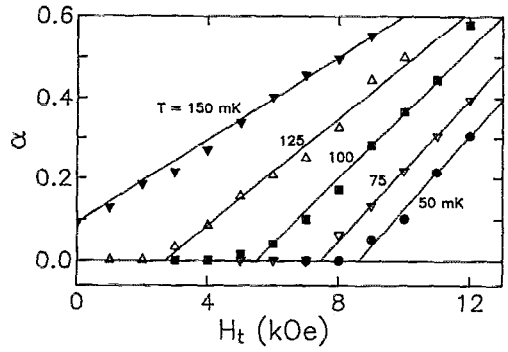

FIG. 10. The power in the form f^{α} fit to $\chi''(f \to 0)$ vs transverse field at five temperatures. We define spin-glass freezing when $\alpha \to 0$.

data at $T = 25$ mK, then the critical transverse field to completely suppress freezing is $H_c = 12.0 \pm 0.4$ kOe.

We argued earlier that the development of in-plane ferromagnetic correlations with increasing dipole concentration should aid in the process of finite temperature spin-glass freezing. One of the effects of the transverse magnetic field is to induce a basal plane g factor, which, similarly, could promote the interlocking of randomly situated spin clusters. It is clear from the data, however, that this effect is completely dominated by the additional pathways to relaxation provided by the mixing of eigenstates.

CONCLUSIONS

We have studied ensembles of randomly distributed Ising dipoles both in the classical and quantum limits. The parent compound, $LiHoF_4$, is a ferromagnet with an essentially perfect (due to the long range of the dipolar interaction) mean-field transition at $T_c = 1.53$ K. Random dilution of the Ho^{3+} spins with nonmagnetic Y^{3+} down to at least 46% holmium preserves the ferromagnetism with a mean-field scaling of the transition $T_c(x) = xT_c(x = 1)$. Given that the dipolar interaction falls off as $1/r^3$ and that the sample volume goes as r^3, one might expect continued simple scaling to the lowest dipole concentrations. However, we find spin-glass behavior preempting the ferromagnetism by $x = 0.167$, and even more surprising, a different type of magnetic glass in the dilute spin concentration limit. It should be possible to trace the development of the spin glass from a "precursor" state which acts as if reducing the temperature reduces the interactions of the dipoles. Moreover, we are able to introduce quantum fluctuations to the Ising spin-glass problem in a controlled fashion through the application of a transverse magnetic field. Modest H_t, of order 1 to 10 kOe, profoundly modify the classical (zero-field) dynamical behavior of randomly distributed dipoles and can completely suppress the freezing process.

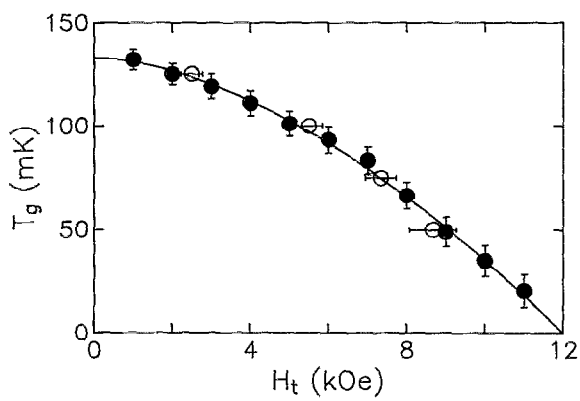

FIG. 11. Depression of the spin-glass transition temperature with H_t. Solid line is a least-squares fit with $T_g(0) - T_g(H_t) \sim H_t^{1.7 \pm 0.1}$.

ACKNOWLEDGMENT

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