Aging and memory in a superspin glass

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Magnetization relaxation measurements show that aging occurs in a discontinuous metal-insulator multilayer (DMIM) $[Co_{80}Fe_{20}(0.9\ nm)/Al_2O_3(3\ nm)]_{10}$ below the spin-glass transition temperature, $T_g \approx 44$ K. Furthermore, the DMIM system memorizes the structure of a quasiequilibrium state reached after an intermittent stop-and-wait protocol during cooling. These results unambiguously corroborate the collective nature of the low-temperature spin dynamics. The correlation length achieved at $T=0.95T_g$ after a wait time of 10^4 s is estimated to extend to 10^5 superspins, which seems to imply a crossover from three- to two-dimensional growth of the correlation length.

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The interest to understand the behavior of an ensemble of ferromagnetic nanoparticles has been growing due to both the richness of their experimental properties and their technological applications, for example, in magnetic recording industry (see Ref. 1 for recent reviews). One of the most challenging questions in such systems concerns the dynamics at low temperatures. In a dilute system, the magnetic dipole-dipole interaction between the particles is negligible compared to the anisotropy energy of an individual nanoparticle. In this case, the dynamics follows the predictions of the Néel-Brown model[2] and the system is considered as superparamagnetic (SPM). However, in a dense system the dipole-dipole interaction is of the order of the particle anisotropy energy and strongly affects the low-temperature dynamics. Three-dimensional (3D) random distributions and random orientation of anisotropy axes of such nanoparticles in an insulating matrix with high enough packing density and sufficiently narrow size distribution will create a competition of different spin alignments. The nature of the low-temperature state of such a frustrated system is currently discussed in literature based on experimental results[3–7] as well as on Monte Carlo (MC) simulations.[8,9] Despite sophisticated experimental work[3–6] and MC simulations[9] supporting collective dynamics at low temperature, there are also contradictory results in favor of SPM behavior and noncollective blocking[8] or of modified energy barriers within the SPM ensemble.[7] At this juncture, it becomes crucial to investigate the low-temperature dynamics more carefully. If a low-temperature collective superspin-glass (SSG) state is formed, typical properties of an ordinary atomic spin glass should be observed in this phase.

It is well known that phenomena like aging, memory, and rejuvenation are the inherent characteristics of a spin-glass (SG) phase.[10,11] In fact, aging in dc magnetic relaxation[12] and memory in ac susceptibility[13] have recently been observed in frozen ferrofluids containing nanosized particles of γ-Fe_2O_3, $Fe_{1-x}C_x$, and ϵ-Fe_3N. In this paper, we report on the occurrence of aging and memory effects of the discontinuous metal-insulator multilayer (DMIM) system $[Co_{80}Fe_{20}(0.9\ nm)/Al_2O_3(3\ nm)]_{10}$ from zero-field-cooled (ZFC) magnetization relaxation measurements. The fact that CoFe superspins ($\mu \approx 3000\mu_B$) freeze cooperatively has previously been evidenced from the divergence of the relaxation time[14] and of the nonlinear susceptibility[15] at the glass transition temperature $T_g \approx 44$ K and stretched-exponential relaxation[16] in the SSG phase.

Understanding the complete mechanism behind the nontrivial aging, memory, and rejuvenation phenomena in spin glasses has imposed a big challenge to the SG scientific community.[11,17] The observations of aging are, however, most often interpreted within the framework of existing models. One is the droplet model suggested by Fisher and Huse[18] based on scaling arguments, while the other one is the hierarchical model inspired by Parisi's solution of the Sherrington-Kirkpatrick Hamiltonian.[19] Recent wide experimental[20] and numerical[21–23] investigations show a general agreement with the idea that a certain coherence length grows when a spin glass is aged. Although this concept was originally proposed for the droplet model, also hierarchial phase space models account for many observed aging and memory effects.[24] However, the existence of an observable correlation length also demands for real-space interpretations. That is why we will interpret our results primarily within the concept of the droplet model.

Magnetic relaxation and magnetization M vs temperature T measurements have been performed on the DMIM system $[Co_{80}Fe_{20}(0.9\ nm)/Al_2O_3(3\ nm)]_{10}$ by use of a noncommercial low-field superconducting quantum interference device (SQUID) magnetometer.[25] Details of the sample preparation and characterization are described in Refs. 26 and 27. It is worth mentioning that transmission electron micrographs obatained on a similar sample with nominal thickness $t_n = 0.9$ nm show that CoFe forms well separated and nearly spherical particles with an average diameter $d \approx 3$ nm within

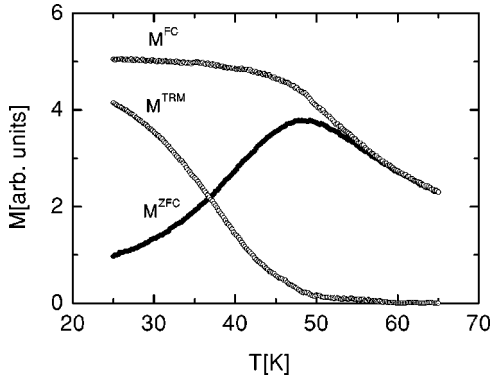

FIG. 1. Various magnetizations (M^{ZFC}, M^{FC}, and M^{TRM}) vs temperature involving an applied field of $\mu_0 H = 0.01$ mT.

a log-normal distribution width of $\sigma_d = 0.35$. Furthermore, transmission electron microscopy studies on a sample with $t_n = 1.3$ nm reveal that the average particle size increases approximately linearly with t_n, while their average clearance monotonically decreases. In accordance with the previously observed transport properties, i.e., a transition from tunneling to Ohmic conductance, percolation is expected to occur at $t_n = 1.8$ nm.[27] At the nominal thickness, $t_n = 0.9$ nm, the particles are almost equidistant $D = 6$ nm from each other. The average volume fraction of the magnetic particles is about 23%, hence similar as in densely packed frozen ferrofluids with superspin-glass properties.[12] Using an average moment of one CoFe particle $\mu = 3000\mu_B$ (obtained from a Langevin-type fit of the M vs $\mu_0 H$ curve in the high-temperature SPM regime, e.g., at $T = 100$ K) and a mean in-plane interparticle distance of $D = 6$ nm the mean value of dipole-dipole interaction between two neighboring particles, $E_{d-d}/k_B = (\mu_0/4\pi k_B)\mu^2/D^3$, is estimated to be 30 K.

All magnetization (zero-field cooled, M^{ZFC}, field cooled, M^{FC}, and thermoremanent, M^{TRM}) vs T data are recorded on heating the sample while using conventional experimental protocols. The sample is cooled in zero field (for M^{ZFC}) or in a field $\mu_0 H = 0.01$ mT (for M^{FC} and M^{TRM}) from 65 K, where it shows a reversible SPM behavior, to 25 K. In M^{ZFC}, a field step of $\mu_0 H = 0.01$ mT is applied and in M^{TRM} the field is cut to zero at 25 K, while in M^{FC} the field is kept on all the time. In a stop-and-wait protocol, the sample is ZFC from the SPM phase to a stop temperature $T_s < T_g$ (T_{s1} and $T_{s2} < T_{s1}$ in a double stop-and-wait protocol), where the system is aged for a certain duration before further cooling down to 25 K. For conventional aging experiments the sample is ZFC to a constant measurement temperature, $T_m < T_g$, where after a wait time t_w, a small probe field $\mu_0 H = 0.04$ mT is applied and magnetization is recorded vs time t. It should be noted that all measurements are done in quite low magnetic fields in order to avoid nonlinear effects.

Figure 1 shows the magnetization curves M^{ZFC}, M^{FC}, and M^{TRM} vs T of our DMIM sample according to the above protocols. The data were collected at a heating rate of 0.4 K/min. M^{ZFC} and M^{FC} vs T reveal irreversibility, the characteristic feature of a spin-glass system. The peak of M^{ZFC} occurs a few K below the onset temperature of the irreversibility, which can be attributed to spurious blocking of larger particles whose blocking temperatures exceed T_g.[15] M^{ZFC} exhibits a characteristic peak at $T \approx 48$ K, a few Kelvin above the estimated static glass transition temperature $T_g \approx 44$ K.[14,15]

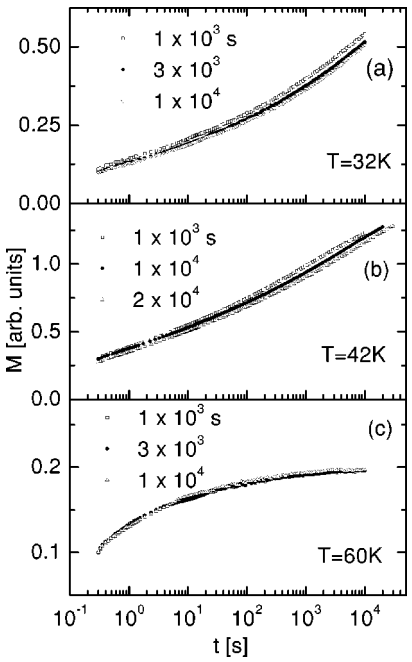

FIG. 2. Relaxation curves M vs $\log t$ at temperatures $T = 32$ K (a), 42 K (b), and 60 K (c) recorded at $\mu_0 H = 0.4$ mT after different wait times t_w as indicated.

Figures 2 and 3 show the relaxation of the magnetization M and its rate $S = (1/\mu_0 H)(\partial M/\partial \ln t)$ vs $\log t$, respectively, obtained at three different temperatures in the SSG phase.

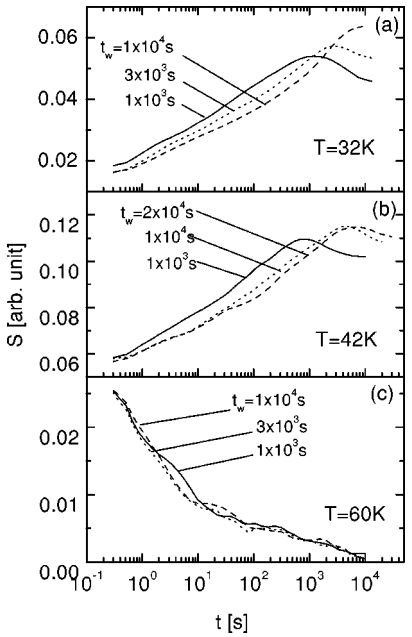

FIG. 3. Relaxation rate S vs $\log t$ at $T = 32$ K (a), 42 K (b), and 60 K (c) corresponding to the relaxation curves of Fig. 2. Different wait times t_w are indicated in each plot.

The employed wait times t_w are indicated in each plot. At 32 and 42 K, i.e., at $T < T_g$, the curves show a clear wait time dependence with inflexion points (Fig. 2) and peaks (Fig. 3), respectively, appearing at times close to the corresponding wait times, i.e., the system ages. In contrast, at $T = 60$ K the relaxation is independent of the wait time, implying that the observed relaxation is governed by noncollective thermally activated dynamics of individual particles and the system behaves like a SPM one.

The characteristic aging observed in the SSG phase implies that the correlation between the particle magnetic moments develops in the same way as the correlation between the spins of an atomic spin glass. This nonequilibrium dynamics can be dealt within the context of the droplet model,[18] according to which the approach towards equilibrium after a quench from above T_g to $T_m < T_g$ is governed by the growth of equilibrium domains during aging in the SG phase. In the droplet model it is assumed that a twofold degenerate ground state is formed when a spin glass is quenched in zero magnetic field. Let us call one of these states Ψ and its global spin reversal $\overline{\Psi}$. With the progress of time at T_m the system lowers its energy by decreasing the amount of interfaces, i.e., domain walls between Ψ and $\overline{\Psi}$, thereby growing larger and larger domains of either size. This growth is governed by the lowest-energy excitations called droplets.[18] The free-energy cost for creating a droplet of size L scales as $F \propto \gamma(T) L^{\theta}$, where $\gamma(T)$ is the stiffness constant and θ is a stiffness exponent satisfying $0 \leqslant \theta \leqslant (d-1)/2$ for a d dimensional system. Moreover, an energy barrier $B \propto \Delta(T) L^{\psi}$, where $\Delta(T)$ sets the free energy scale of the barriers and ψ is a barrier exponent which satisfies $\theta \leqslant \psi \leqslant (d-1)$, must be surmounted in order to move a section of domain wall. The free-energy barriers B are overcome by thermally activated dynamics as time evolves according to $B \propto T \ln(t/\tau^*)$, where τ^* corresponds to a microscopic flip time. Hence in an aging time $t_a = t_w + t$ after the quench the characteristic size of equilibrated domains is given by

$$R_{t_a} \sim \left(\frac{T \ln(t_a/\tau^*)}{\Delta(T)} \right)^{1/\psi}. \quad (1)$$

When a small dc field is applied, the equilibrium system is probed via the polarization of droplets which grow in time as

$$L_t \sim \left(\frac{T \ln(t/\tau^*)}{\Delta(T)} \right)^{1/\psi}. \quad (2)$$

Two limiting cases can be considered: $\ln t \ll \ln t_w$ and $\ln t \gg \ln t_w$. In the first case, we have $L_t \ll R_{t_a}$, i.e., the probed length scale occurs within the equilibrated domains and sees no domain wall. This quasiequilibrium dynamics leads to an increase of the observed magnetization with time. In the second case, we have $L_t \approx R_{t_a}$ (since $t \approx t_a = t_w + t$) so that the probed length scale involves domain walls and yields a nonequilibrium response. Within this picture, an often observed point of inflexion in the magnetization vs $\ln t$ or, equivalently, a peak in the relaxation rate vs $\ln t$ at the corresponding wait time is interpreted to reflect the crossover from equilibrium to nonequilibrium response. Applying this model to a nanoparticle system requires, e.g., that one takes into account that the atomic spins are substituted by particle moments with strongly temperature dependent Néel-type relaxation times. These might in addition be affected by the dipolar magnetic fields from the neighboring particles.[1]

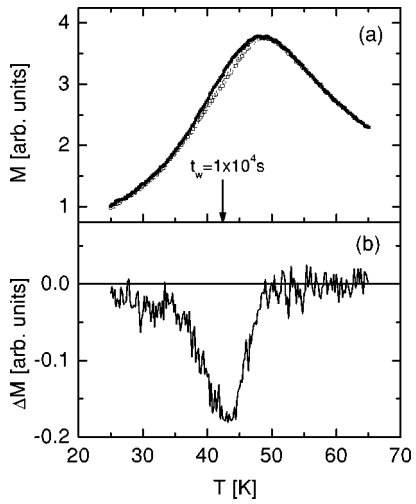

FIG. 4. (a) Temperature dependence of the reference magnetization M_{ref} (solid squares) and of the magnetization with a stop and wait protocol, M (open squares), at a magnetic field of $\mu_0 H = 0.1$ mT. (b) $\Delta M = M - M_{ref}$ vs T.

Figure 4 illustrates the memory and rejuvenation effects of the ZFC dc magnetization after a stop-and-wait at $T_s = 42$ K ($= 0.95\ T_g$) for a duration of 10^4 s. As can be seen in Fig. 4(a), the data corresponding to the intermittent stop-and-wait (open squares) lie significantly below the reference curve (solid squares) at temperatures close to T_s. This difference indicates that the magnetic moment configuration spontaneously rearranges towards equilibrium via growth of equilibrium domains, when the system is left unperturbed at constant temperature T_s. These equilibrated domains become frozen in on further cooling and retrieved on reheating. In other words, the system shows a memory effect, which is observed as a minimum in $\Delta M = M(T) - M_{ref}(T)$ at about T_s in Fig. 4(b). The fact that reference and the stop-and-wait curves coalesce at low temperatures and only start to deviate as T_s is approached from below clearly indicates that rejuvenation[17] of the system occurs as the temperature is decreased away from T_s in the stop-and-wait protocol. Similar memory and rejuvenation effects have recently been reported in ordinary atomic spin glasses in both dc magnetization[28] and ac susceptibility.[11,17]

Within the droplet model the rejuvenation occurs as a consequence of the chaotic nature of the equilibrium spin-glass phase,[29] i.e., the equilibrium states at two different temperatures are different. However, in an experimental situation, the size of the equilibrium domain is at every temperature below T_g set by the cooling rate or/and the stop-and-wait time and the influence of the overlap length scale, $l(\Delta T)$. The overlap length is the maximum size up to which the equilibrium states $\Psi(T)$ and $\Psi(T + \Delta T)$ cannot be

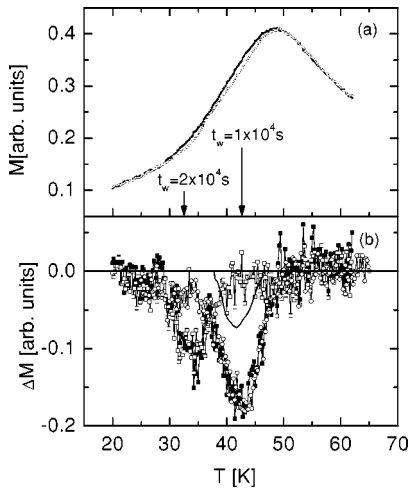

FIG. 5. (a) Comparison of the reference magnetization (solid cirles) with the magnetization (open circles) employing a double stop-and-wait protocol. The data are recorded at $\mu_0 H = 0.1$ mT. (b) ΔM vs T for the double stop-and-wait protocol (solid squares) in comparison with two independent stop-and-wait protocols at 42 and 32 K for durations of 10^4 and 2×10^4 s (open circles and squares, respectively). The solid curve corresponds to smoothed ΔM data obtained after a wait time of 10^3 s at 42 K.

distinguished,[18] i.e., when the sample is cooled from T_s, it appears rejuvenated once the overlap length becomes smaller than the domain size reached at T_s. Translated into the observation of a memory dip in the ZFC magetization curve (or ac-susceptibility curve[11]), the width of the dip gives an indirect measure of the dependence of $l(\Delta T)$ on T_s. Experimentally, memory dips have been found to be broader in Ising than in Heisenberg spin glasses.[30] However, an even more significant difference is given by the observation time of the employed probe. In MC simulations, where the observation time is some ten orders of magnitude shorter than in experiments, the memory dip is very broad or even extends outside the observed temperature window. In a nanoparticle system, the individual relaxation times of the average individual particle is some five to six orders of magnitude larger than the atomic relaxation time at the measurement temperature, implying that the observation time is correspondingly shorter than in an ordinary spin glass. This difference in time scales is probably the main reason why the observed memory dip is much broader than in atomic spin glasses—Ising or Heisenberg like.

Figure 5 presents a double memory and aging experiment: the cooling process is interrupted twice at $T_{s1} = 42$ K ($=0.95\ T_g$) and $T_{s2} = 32$ K ($=0.72\ T_g$) for durations of 10^4 and 2×10^4 s, respectively. Upon subsequent reheating M^{ZFC} shows memory anomalies at the corresponding T_s's as seen in Fig. 5(a) and ΔM as seen in Fig. 5(b) shows two distinct minima. For comparison, a single-dip smoothed ΔM curve obtained after a wait time of 10^3 s at 42 K is also shown (Fig. 5(b); solid curve). The double memory curve (solid squares) can be regarded as a superposition of two independent stop-and-wait experiments as shown by two ΔM curves obtained consecutively at $T_s = 42$ and 32 K (open circles and squares, respectively). One also sees that the stop-and-wait magnetization curves coalesce with the reference curve except at temperatures close to T_s's which, as discussed above, signals rejuvenation. It is expected and has been shown[28] that the depth of a memory dip should approximately increase linearly with logarithmically increasing stop times. Associating an effective observation time of the ZFC magnetization curve of about 100 s, the expected linear increase of the memory dip with the logarithm of the stop time is supported by the data.

It is of interest to derive some rough numbers for the size of spin-glass correlation length, $\xi(t^*,T)$,[23] evolving at T_s during aging. We will adopt the simplest possible growth law and account for the fact that our particles have temperature-dependent relaxation times, i.e., $t^* = t/\tau^*$ with t being the time at constant temperature and $\tau^* = \tau_0 \exp(E/k_B T)$. Joh *et al.*[20] have found that $\xi(t,T)$ might either follow a power-law dynamics, $\xi(t^*,T) \propto (t^*)^{\alpha(T)}$, where $\alpha(T)$ is a temperature-dependent exponent, or an activated dynamics, $\xi(t^*,T) \propto (\ln t^*)^{1/\psi}$. Komori *et al.*[22] have numerically found that $\xi(t,T) \simeq (t^*)^{\alpha(T)}$, where $\alpha(T) = 0.17$ T^* and $T^* = T/T_g$, these values are rather similar to what was found experimentally.[20] The number of correlated spins $N_s(t^*,T)$ corresponding to the parameters t^* and T can then be derived from $\xi(t^*,T) \approx [N_s(t^*,T)]^{1/3}$. Inserting $\tau_0 = 10^{-10}$ s and $E/k_B = 410$ K (Ref. 31) for our DMIM system and following the power-law dynamics, we obtain at $T = T_s = 42$ K (32 K), $\xi(t^*,T) = 38(12)$ for $t = 10^4$ s (2×10^4 s), hence $N_s = 5.6 \times 10^4$ (1.7×10^3) correlated superspins. Considering activated dynamics and using $\psi = 0.85$ in close agreement with Refs. 21 and 32 and the prefactor as unity, we find that $\xi(t^*,T)$ has similar values as obtained with power-law dynamics at these temperatures.

Since our DMIM consists of only ten stacked monolayers of magnetic particles, these rough values of the correlation lengths imply that the growth at $T = T_s = 42$ K on our experimental time scales could have experienced a crossover from 3D nature on short timescales to 2D character on time scales of order seconds and longer. Since a 2D spin glass does not have a finite-temperature spin-glass transition, this encourages us to shortly reconsider and discuss the analysis of the critical slowing down and the earlier derived glass transition temperature.[14] The critical slowing down anlysis was made on experimental data in the time window 10^{-1}–10 s, at reduced temperatures $(T - T_g)/T_g$ in the range 0.2–0.3. The exponent $z\nu$ is from the scaling analysis found to be 10 (Ref 14) implying a value of ν of around 1.6 [if z is assumed to be about 6 (Ref. 5)]. Using these numbers we find a critical correlation length covering no more than 12 interparticle distances at the lowest reduced temperature and the longest observation time (10 s) of the analysis. This means that the system to a good approximation should remain 3D on the observation times and corresponding temperatures employed in the earlier reported critical slowing down analysis. It would, however, be of interest to see if indications of a crossover behavior to a 2D character could be found, if longer observations times and thus lower reduced temperatures were included in the analysis. It is in this connection worthwhile to mention that apart from such time-consuming ex-

periments also studies on single layered nanoparticle systems of the same materials as our current DMIM system are underway—and if the experimental resolution permits—interesting comparisons as to the influence of the dimensionality will be obtained.

In summary, we have presented some crucial characteristics of a collective superspin-glass state which may thus be considered as counterparts of conventional spin glasses. Presumably this is a consequence of random orientations of the easy axes of the nanoparticles and the long-range character of the dipolar interactions involved in the SSG ordering process. We expect that the present results will stimulate further analytical investigations in order to understand the dynamics of interacting magnetic nanoparticle systems which may also play a crucial role in applications.

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