

# Zero-temperature spin-glass freezing in self-organized arrays of Co nanoparticles

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**Abstract.** - We study, by means of magnetic susceptibility and magnetic aging experiments, the nature of the glassy magnetic dynamics in arrays of Co nanoparticles, self-organized in  $N$  layers from  $N = 1$  (two-dimensional limit) up to  $N = 20$  (three-dimensional limit). We find no qualitative differences between the magnetic responses measured in these two limits, in spite of the fact that no spin-glass phase is expected above  $T = 0$  in two dimensions. More specifically, all the phenomena (critical slowing down, flattening of the field-cooled magnetization below the blocking temperature and the magnetic memory induced by aging) that are usually associated with this phase look qualitatively the same for two-dimensional and three-dimensional arrays. The activated scaling law that is typical of systems undergoing a phase transition at zero temperature accounts well for the critical slowing down of the dc and ac susceptibilities of all samples. Our data show also that dynamical magnetic correlations achieved by aging a nanoparticle array below its superparamagnetic blocking temperature extend mainly to nearest neighbors. Our experiments suggest that the glassy magnetic dynamics of these nanoparticle arrays is associated with a zero-temperature spin-glass transition.

**Introduction.** – Dense arrays of magnetic nanoparticles contain the physical ingredients of spin-glasses [1]. Disorder in the positions and orientations of the particles leads to disorder and frustration of the dipolar interactions, usually dominant, between their magnetic moments. In contrast with "canonical" spin-glasses, the slow magnetic relaxation introduced by interactions coexists with the slow magnetization reversal associated with the high anisotropy energy barriers. Many experiments performed on dense nanoparticulate materials show phenomena, such as magnetic aging [2] and the slowing down of the ac susceptibility [3], which are *typical* of spin-glasses [4–11]. However, some of these phenomena are not *exclusive* of the spin-glass phase [12, 13]. The question is, then, whether real nanoparticulate materials show a true (super)spin-glass phase.

Experimental studies are often hindered by the lack of control over the sample parameters that determine the nature and strength of dipolar interactions, such as interparticle distances, spatial organization, etc. This usually makes it difficult to know *a priori* if a particular system is expected to show a spin-glass phase. Perhaps the most clear-cut situation to discuss the existence of a phase transition and its experimental manifestations is offered by the study of a single layer of nanoparticles. In contrast with three-dimensional systems [14], it is generally accepted [15, 16] that the transition temperature  $T_g$  vanishes in two-dimensions. Results of tempered Monte Carlo simulations seem to confirm the same conclusion also for Ising spins coupled by dipolar interactions [17].

Based on these considerations, our work was aimed to elucidate the nature of the glassy magnetic dynamics, i.e.

whether it is associated with a superspin-glass phase at  $T_g > 0$  or if, by contrast,  $T_g = 0$ , in self-organized nanoparticle arrays. For this, we compare results obtained on very well-characterized three- and two-dimensional arrays of Co nanospheres. Previous experiments reveal that the superparamagnetic blocking temperature  $T_b$ , defined as the temperature of the in phase  $\chi'$  ac susceptibility cusp, increases as additional layers are deposited on a two-dimensional sample [18, 19]. Since the number of layers modifies the number of nearest neighbors in the nanoparticle array, that result indicates that dipolar interactions slow down the magnetic relaxation processes. In the present study, we have investigated how the number of layers modifies the critical slowing down and the magnetic aging, properties that are usually associated with the spin glass behavior [20]. Our results show that no qualitative changes in these quantities occur as the two-dimensional limit is approached. The control over the number of layers and their separation has also enabled us to directly probe the magnetic correlation length and show that it is mainly restricted to a first shell of nearest neighbors and, in any case, shorter than what would be expected for a conventional spin glass.

**Experimental details.** – Samples made of  $N$  layers of Co nanoparticles with average diameter  $D \simeq 2.6$  nm were prepared by the sequential sputtering of  $N = 1, 2, 3, 4, 5, 7, 10, 15,$  and  $20$  Co and  $\text{Al}_2\text{O}_3$  layers on silicon substrates [18, 19, 21, 22]. The particle's shape and average size (thus also the average magnetic moment  $\mu_p$  per particle), as well as the width of the size distribution ( $\sigma_D = 0.26D$ ) are approximately independent of  $N$  [19]. Nanoparticles deposited on adjacent layers tend to self-organize in a structure that resembles a closed-packed hexagonal lattice of nanospheres [22]. The separation between the Co layers is determined by the thickness  $t_{\text{Al}_2\text{O}_3} = 3$  nm of the alumina layer. Nearest neighbors separations are  $d_{\text{nn},\parallel} \simeq 4.6$  nm, within a given layer, and  $d_{\text{nn},\perp} \simeq 4.2$  nm, between adjacent layers. They correspond to dipolar energies  $E_{\text{dip}} = \mu_p^2/d_{\text{nn}}^3 \approx 13$  K and 17 K, respectively. As described in [23], the anisotropy energy barrier  $U_0$  for the magnetization reversal was estimated from ac susceptibility experiments performed under sufficiently strong magnetic fields, which dominate over dipolar interactions. This method gives  $U_0 \simeq 430$  K. In the same way, we estimate an attempt time  $\tau_0 \sim 10^{-13}$  s, of the same order of that found for samples of very small Co nanoparticles ( $D \sim 1$  nm), prepared by the same technique [18], for which interactions are expected to become negligible. A multilayer with  $N = 20$  layers but a larger interlayer separation  $t_{\text{Al}_2\text{O}_3} = 10$  nm, and thus also a much smaller interlayer  $E_{\text{dip}} \approx 1.6$  K, was prepared under identical experimental conditions.

Ac susceptibility and magnetization measurements were performed with a commercial SQUID magnetometer. Samples were rectangular plates with approximate dimensions  $9 \times 3 \times 0.5$  mm<sup>3</sup>. Ac and dc magnetic fields were

parallel to the plane of the sample to minimize demagnetizing effects. In our study of aging [6,7], we measured the time-dependent relaxation of the zero-field cooled (ZFC) magnetization on samples aged, at zero field, for a time  $t_w$  at temperatures  $T_w < T_b$ . In addition, we employed a different method which consists on measuring magnetization curves (zero-field and field cooled (FC), and remanence) using the waiting time protocol described in [24, 25].

**Results and discussion.** – A typical method to characterize the spin-glass behavior is by measuring the frequency-dependent ac magnetic susceptibility [3–5]. At any fixed frequency  $\omega$  we define a characteristic relaxation time  $\tau_c$  such that  $\omega\tau_c(T) = 1$  at  $T = T_b(\omega)$ . For spin glasses  $\tau_c$  diverges at  $T_g$  according to a power law, reflecting the growth of magnetic correlations [4]

$$\tau_c = \tau_* |1 - T/T_g|^{-z\nu} \quad (1)$$

In Fig. 1, we plot  $\tau_c$  versus the reduced temperature for  $N$  ranging from 1 to 20 layers. The experimental data are compatible with a critical slowing down of the magnetization dynamics at a finite  $T_g$ . In order to limit the number of fitting parameters, we took  $T_g$ , for each sample, as the temperature of the ZFC susceptibility cusp (i.e. equal to the  $T_b$  corresponding to a typical timescale of the order of 170 s). The microscopic time scale  $\tau_*$  and the dynamical critical exponent  $z\nu$  are found to be nearly the same for all samples. The critical exponent is close to typical values found for spin glasses [5] as well as for some nanoparticle materials [26]. The present results are remarkable because it is generally believed that  $T_g = 0$  in two dimensions. Notice however that, as often happens with plots of this type obtained for nanoparticles [3, 26, 27], experiments do not explore the close vicinity of the critical region. For this reason, the data are relatively easy to fit. Fits of similar quality can be obtained by scaling all  $T_g$ 's by a factor in between 1 and 0.75. The characteristic  $\tau_*$  increases then from about  $10^{-6}$  s to  $10^{-4}$  s while, at the same time, the exponent  $z\nu$  increases from 7.3 to 14. In fact, if one wishes to include also in the analysis the freezing temperature extracted from ZFC susceptibility data (getting closer to  $T_g$ ), the best fits with Eq. (1) are obtained then for the largest  $z\nu$  and  $\tau_*$  values (thus also for the lowest  $T_g$ ). Such large  $z\nu$  values are not uncommon in systems of magnetic nanoparticles [3, 27] but they are significantly larger than what it is expected for a canonical spin glass phase transition (of the order of  $z\nu = 7$  [28]).

An alternative theoretical framework to describe the frequency-dependent susceptibility, which seems very appropriate in the case of a layered material with a markedly two-dimensional character, is the activated dynamics characteristic of glassy systems undergoing a phase transition at  $T_g = 0$  [4, 16]. In the latter situation, the critical slowing down of  $\tau_c$  obeys the following expression

$$\tau_c = \tau_0 \exp(E_a/k_B T)^\sigma \quad (2)$$

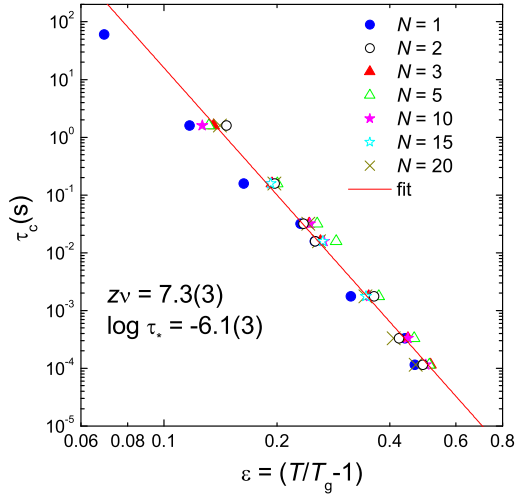


Fig. 1: (Color online). Critical slowing down of the characteristic relaxation time extracted from ac susceptibility experiments. Results are shown for samples with varying number of layers  $N$ .

where  $E_a$  is an effective activation energy and  $\sigma$  is a critical exponent. As it is shown in Fig. 2, we find a good agreement with our data, including also the temperature of the ZFC magnetization cusp, for  $\sigma = 1.3$  (to be compared with  $\sigma = 3.2$  found for 2- $D$  spin-glasses [16]) and  $E_a$  gradually increasing with the number of layers from 345 K up to 471 K. From these frequency-dependent susceptibility experiments, we conclude that the nature of the slow magnetic dynamics of two-dimensional (i.e. with  $N$  equal to or close to unity) and three-dimensional (with large  $N$ ) nanoparticle arrays is the same. The description based on a zero-temperature phase transition is appealing, because it is consistent with the behavior expected for a single layer. By themselves, however, these experiments cannot discriminate between the two alternatives, i.e., whether the underlying physics corresponds to the existence of a second-order phase transition at a finite  $T_g$  or if, by contrast,  $T_g = 0$ .

Aging experiments can shed some light and help deciding between these two alternatives, since they probe how dynamical magnetic correlations grow with time [6, 8, 11]. We have carried out two different experiments, which measure the magnetic memory effects associated with the aging of the sample at a given temperature. In the first of these, the quantity of interest is the difference  $\Delta M = M - M_w$  between the magnetizations (ZFC, FC or remanent) measured after cooling the sample without or with a pause at an intermediate temperature  $T_w < T_b$  [24]. Results measured for  $t_w = 10^4$  s are shown in Fig. 3.  $\Delta M_{ZFC}$  shows a peak centered near  $T_w$ . If  $T_w$  is varied, the peak shifts accordingly. In addition, the relationship

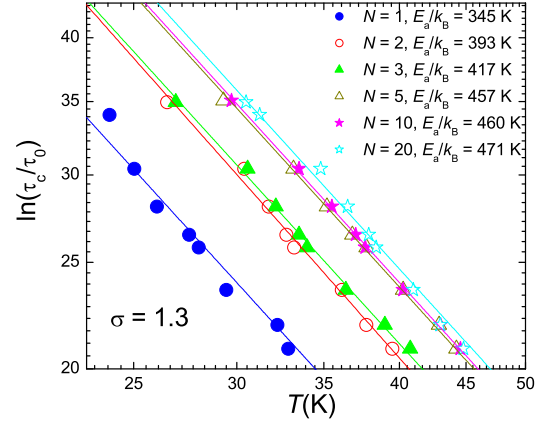


Fig. 2: (Color online). Log-log plot showing the variation with temperature of  $\ln(\tau_c/\tau_0)$ , where  $\tau_c$  is a characteristic relaxation time extracted from ac susceptibility data and  $\tau_0 = 10^{-13}$  s. The lines are fits of the law  $\tau_c = \tau_0 \exp(E_a/k_B T)^\sigma$ , characteristic of a spin-glass transition at  $T_g = 0$

[24]  $\Delta M_{FC} = \Delta M_T + \Delta M_{ZFC}$  is fulfilled, showing that they are associated with the aging of the sample at  $T_w$  and not with experimental artifacts.

Figure 3 compares results obtained on a single layer  $N = 1$  with those measured on a multilayer made of  $N = 20$  layers. The aging was performed at  $T_w = 0.7T_b$  for the two samples. Besides the obvious difference in the signal-to-noise ratios, they look qualitatively the same. The maximum in  $\Delta M_{ZFC}$  vs  $T$  is just about 25 % larger in the case of the multilayer. A first conclusion is, therefore, that the magnetic memory induced by aging a nanoparticle array does not show any abrupt change as the two-dimensional (2D) limit is approached. Notice also that, as we have seen with the critical slowing down, the analogy is not restricted to aging. The FC curves measured on the two samples show also the same degree of “flattening” below  $T_b$ , a property that has been considered as a signature of the superspin glass phase [25].

By gradually changing the number of layers  $N$  we can study how magnetic correlations grow. In Fig. 4, we show, as a function of  $N$ , the relative amplitude of the magnetic memory effect  $\Delta M_{ZFC}/M_{ZFC}$  measured after aging at  $T_w = 15.8$  K for  $t_w = 10^4$  s. Within the droplet picture of the spin-glass phase [29], this quantity is connected with the size that domains of correlated spins attain after time  $t_w$  [11]. We see that  $\Delta M_{ZFC}/M_{ZFC}$  increases rapidly when one or two layers are added to a two-dimensional sample, nearly saturating as  $N$  increases further. The right-hand panel of Fig. 4 shows that, within the relatively large experimental uncertainties,  $\Delta M_{ZFC}/M_{ZFC}$  is approximately proportional to the increase in the average number of nearest neighbors  $N_\perp = 6(N-1)/N$  that is associated with the addition of extra layers. The same linear dependence was also found for the blocking temperature [19]. This behavior suggests that the enhancement in the amplitude of the magnetic memory is provided mainly by correlations with

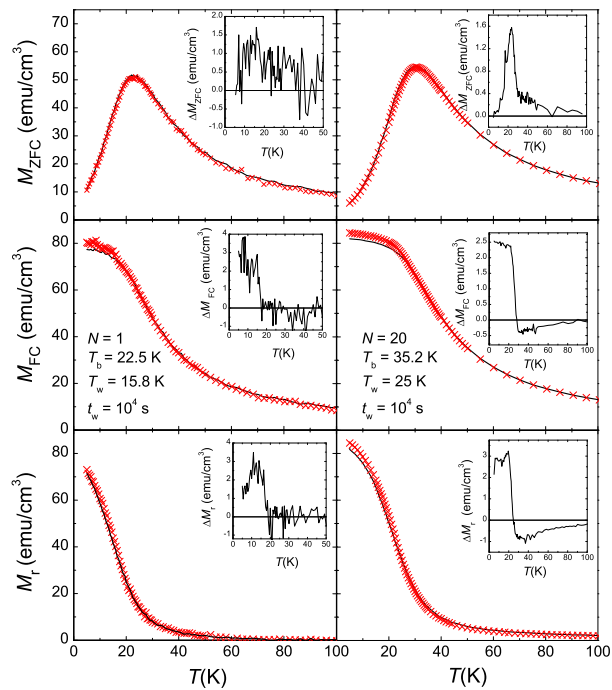


Fig. 3: (Color online). ZFC, FC and thermoremanence curves measured with (red crosses) and without (black solid line) pause. The insets show absolute values of  $\Delta M_{ZFC}$ ,  $\Delta M_{FC}$  and  $\Delta M_T$ . Left: sample with  $N = 1$ ;  $T_w$  was 15.8 K. Right: sample with  $N = 20$ ;  $T_w$  was 25 K. The waiting time was  $10^4$  s and the applied magnetic field was 10 Oe.

the first one or two nearest layers. Another result suggesting that magnetic correlations remain rather short-ranged is shown in Fig. 5. There, we compare the magnetic memory  $\Delta M_{ZFC}$  obtained for a single layer of 2.6 nm particles with that obtained for a  $N = 20$  multilayer in which the interlayer separation is  $t_{Al_2O_3} = 10$  nm, i.e. more than twice  $d_{nn,\parallel} \sim 4.6$  nm. Within their respective experimental uncertainties, these two quantities are found to be the same. Also  $T_b$  and other quantities agree. It seems then that no measurable magnetic correlations are established between layers of nanoparticles located 10 nm far from each other.

We also studied the effects of aging using a different experimental method, which enables a more quantitative determination of magnetic correlation lengths. For this, we measured the magnetic relaxation of the ZFC magnetization of a  $N = 15$  multilayer at  $T_w = 15.8$  K. The sample was first cooled from 100 K to  $T_w$  in zero field. After aging the sample for  $t_w = 10^4$  s, a magnetic field  $H$  was applied and the ensuing magnetization measured as a function of time. This method has been applied to estimate the number of correlated spins in spin glasses [30, 31], and recently applied also to investigate the slow dynamics of frozen ferrofluids [26]. Its basic idea is as follows. During the waiting time  $t_w$ , at zero field, magnetic correlations between nanoparticles grow [6]. Typical free energy barriers  $\Delta(t_w)$  for the flip of  $N_s(t_w)$  cor-

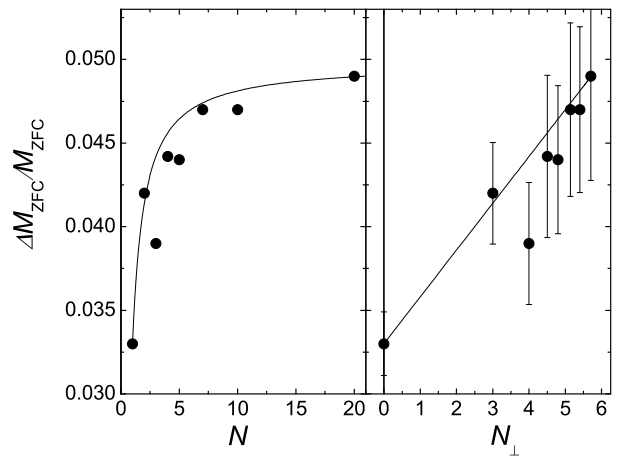


Fig. 4: (Color online). Left: Variation with the number  $N$  of layers of the memory  $\Delta M_{ZFC}/M_{ZFC}$  measured after aging the sample for  $t_w = 10^4$  s at  $T_w = 15.8$  K ( $\bullet$ ). Right: Same data as a function of the average number of nearest neighbors  $N_{\perp} = 6(N - 1)/N$  that a nanoparticle has in adjacent layers [19]. The lines represent  $0.033 + 0.015N_{\perp}$ .

related spins increase also with the age  $t_w$  of the system. This growth of dynamical correlations reflects itself in the appearance of a maximum in the relaxation rate, defined as  $\partial M_{ZFC}/\partial \log(t)$ , when the experimental time  $t$  approaches the age of the system  $t_w^{\text{eff}} \sim t_w$  (see Fig. 6). A magnetic field  $H$  reduces the free energy barriers, from its zero-field value to  $\Delta(t_w) - E_Z[H, N_s(t_w)]$ , where  $E_Z[H, N_s(t_w)] = \mu[H, N_s(t_w)]H$  and  $\mu$  is the magnetic moment of a "drop" of  $N_s$  correlated spins. The energy shift induced by this Zeeman term effectively reduces the "age" of the system according to

$$t_w^{\text{eff}}(H) = t_w^{\text{eff}}(H = 0) \exp \left\{ -\frac{E_Z[H, N_s(t_w)]}{k_B T} \right\} \quad (3)$$

therefore shifting the relaxation rate maximum towards shorter times with increasing  $H$ , as it is indeed observed experimentally (Fig. 6). From a series of experiments performed at different fields, ranging from 5 Oe up to 100 Oe, we have extracted the Zeeman energy  $E_Z[H, N_s(t_w)]$ , which we plot in the main panel of Fig. 6. The field dependence of this energy can be fitted using a quadratic function of  $H$ , compatible with the following expression

$$E_Z[H, N_s(t_w)] = N_s(t_w) \chi_{ZFC} H^2 \quad (4)$$

which was found to agree also with experiments performed on frozen ferrofluids [26]. Inserting in Eq. (4) the measured FC susceptibility per particle  $\chi_{FC}$ , we estimate the number of correlated spins  $N_s$  to be approximately 17, i.e., rather close to the average number of nearest neighbors (12) in a multilayer of nanoparticles [19].

Our experimental findings suggest therefore that magnetic correlations achieved after aging the sample at low  $T$  extend mainly to nearest neighbors. The detailed characterization of our samples enables us to make a quantitative

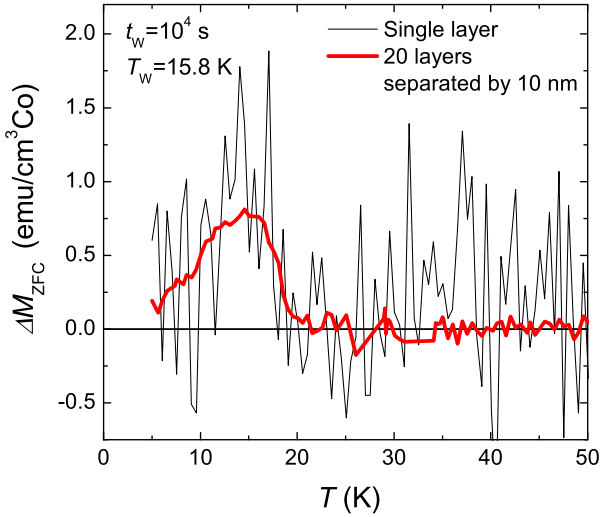


Fig. 5: (Color online). Magnetic memory for a single layer of 2.6 nm particles (black thin line) compared with data obtained for a  $N = 20$  multilayer in which the interlayer separation is  $t_{\text{Al}_2\text{O}_3} = 10$  nm (red thick line).

comparison of the present results with predictions for the growth of correlations in spin-glasses. Theoretical considerations as well as experiments support the idea that correlations grow approximately as a power law of time [26, 30, 32]:

$$\xi(t^*, T_w)/d_{\text{nn}} \sim (t^*)^{\alpha(T_w)} \quad (5)$$

where  $d_{\text{nn}} \sim 4.4$  nm is the distance to nearest neighbors,  $t^* = t_w/\tau(T_w)$  is a dimensionless timescale, the exponent  $\alpha = 0.17(T_w/T_g)$ , and  $\tau$  is the relaxation time of individual spins at the given temperature. We have estimated  $\tau = \tau_0 \exp(U/k_B T)$  using parameters estimated, as described above, for the noninteracting case:  $\tau_0 \sim 10^{-13}$  s and  $U \simeq 430$  K [23]. For  $T_w = 15.8$  K and  $t_w = 10^4$  s, Eq. 5 gives  $4.2d_{\text{nn}} < \xi < 7d_{\text{nn}}$ , i.e., between 19 and 31 nm for a single layer and  $3d_{\text{nn}} < \xi < 4.2d_{\text{nn}}$  (13 – 19 nm) for a multilayer. The upper and lower limits of  $\xi$  correspond to, respectively, the lower and upper limits of the freezing temperatures  $T_g$  that are compatible with the ac susceptibility experiments described above. Our magnetic memory experiments point to significantly shorter correlation lengths  $\xi \sim 4.4$  nm.

**Conclusions.** – The central result of the present study is that we observe the same magnetic memory and critical slowing down in two-dimensional nanoparticle arrays, as well as in multilayers, suggesting that the underlying physical behavior is also the same. The slowing down of the ac (and dc) susceptibility curves measured on all these samples can, in fact, be accounted for using the activated law [Eq.(2)] that is typical of two-dimensional spin-glasses. These results suggest, therefore, that the glassy magnetic dynamics observed in these materials is associated with a phase transition occurring at  $T_g = 0$ ,

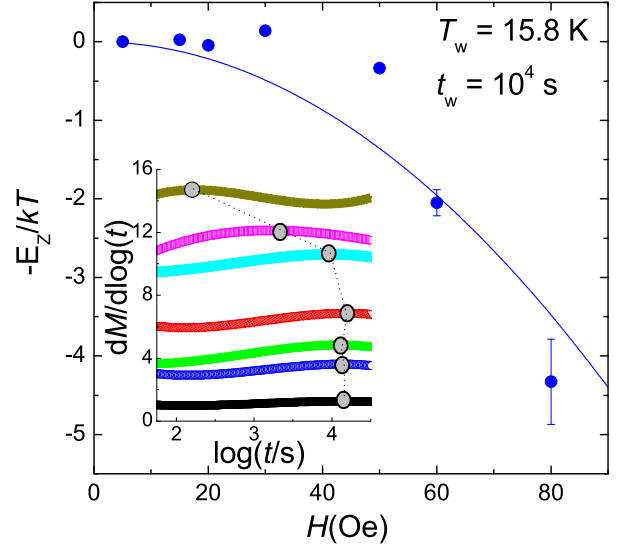


Fig. 6: (Color online). Zeeman energies estimated from the relaxation of ZFC magnetization curves measured after aging the sample at zero field and at  $T_w = 15.8$  K for a waiting time  $t_w = 10^4$  s, before the application of a magnetic field  $H$ . The inset shows the time dependence of the magnetic viscosity, defined as  $\partial M/\partial \log(t)$  with  $M$  being the sample's magnetization, measured for (from bottom to top curves)  $H = 5, 15, 20, 30, 50, 60,$  and  $80$  Oe. This quantity shows maxima, marked by grey dots, at the effective age of the system that decreases with increasing  $H$ . The dotted line is a guide to the eye.

rather than with a conventional spin-glass transition with a finite  $T_g$ . This conclusion is supported by the results of magnetic memory experiments, which show that dynamical magnetic correlations are rather short ranged and, in any case, shorter than expected for canonical spin glasses.

As mentioned in the introductory section above, the observed facts disagree with the prediction, derived from Monte Carlo simulations, that Ising-like spins interacting via dipole-dipole interactions should undergo a spin-glass transition below a finite temperature [17]. Establishing the origin for this discrepancy is beyond the scope of the present work. Here, we content ourselves with discussing possible deviations of real materials from the ideal conditions set by such models. In our opinion, an important aspect to be considered is the, unavoidable, distribution in particle sizes. In our multilayers, the distribution is properly described by a Gaussian function of width  $\sigma_D \simeq 0.7$  nm, which is equivalent to roughly  $\pm 1$  atomic layer and provides an indication of the good homogeneity of these samples. This narrow size distribution leads, however, to an extremely large dispersion in the relaxation times  $\tau$  associated with the magnetic anisotropy of the nanoparticles. Using the parameters  $U$  and  $\tau_0$  given above (see also the second reference in [19] for further details), it follows that intrinsic timescales separated by more than 13 orders of magnitude can coexist at temperatures near or below  $T_b$ . A possible consequence of this enormous dispersion

is the following. Smaller, and therefore faster, relaxing nanoparticles are able to immediately react to spin flips of larger (slower) ones, minimizing their mutual interaction energy. We have previously shown that this effect accounts for the modification of the average relaxation times by interactions, at temperatures close to  $T_b$  [19]. We might speculate with the possibility that the disorder in relaxation times also hinders the growth of magnetic correlations at lower temperatures. For instance, the formation of negatively polarized magnetic clouds surrounding the largest nanoparticles can screen dipolar interactions between them. Clearly, further theoretical studies that include effects of disorder and nonequilibrium dynamics are required to clarify the nature of the collective magnetic response in nanoparticle arrays.

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