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Nonlinear dynamic susceptibilities of interacting and noninteracting magnetic nanoparticles

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Abstract

The linear and cubic dynamic susceptibilities of solid dispersions of nanosized maghemite γ-Fe_2O_3 particles have been measured for three samples with a volume concentration of magnetic particles ranging from 0.3% to 17%, in order to study the effect of dipole–dipole interactions. Significant differences between the dynamic response of the samples are observed. While the linear and cubic dynamic susceptibilities of the most dilute sample compare reasonably well with the corresponding expressions proposed by Raikher and Stepanov for noninteracting particles, the nonlinear dynamic response of the most concentrated sample exhibits at low temperatures similar features as observed in a Ag(11 at% Mn) spin glass.

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Keywords: Maghemite; Nanoparticles; Nonlinear dynamic susceptibility

1. Introduction

The magnetic response of single-domain magnetic particles has been in focus since the pioneering work of Néel [1] and Brown [2]. In the case of *noninteracting* particles, experiments on both individual particles [3] and assemblies of particles (see, for instance, Ref. [4,5]), reasonably well support the existing models [2,6–11]. These have mainly concentrated on the study of relaxation times and the *linear* dynamic susceptibility.

For *interacting* magnetic particles it has recently been shown that dipole–dipole interactions introduce collective behavior, as evidenced by the appearance of magnetic aging and a significantly broadened magnetic relaxation at low temperatures [12,13]. Moreover, the dynamics of a magnetic particle system of monodispersive nature is indicative of critical slowing down at a finite temperature [14], implying the existence of a low-temperature spin glass like phase.

Much less work has focused on the *nonlinear* response of magnetic particles. Bitoh and coworkers studied experimentally the cubic dynamic susceptibility of (nominally) *noninteracting* cobalt nanoparticles [15–17].

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On the theoretical side, expressions for the dynamic nonlinear susceptibility in the simplest case — noninteracting particles without magnetic anisotropy — can be found by simply translating the expressions obtained for the analogous dielectric relaxation problem [18] (a rederivation of $\chi_3(\omega)$ for isotropic dipoles is presented in [19]). However, the inclusion of magnetic anisotropy, even at the level of the equilibrium response, is more involved and it is only recently that this has been done for systems with the simplest uniaxial anisotropy [20–22]. Of special interest is the work of Raikher and Stepanov [21], who solved the Fokker–Planck equation, in the overdamped case, to obtain numerically exact results for the linear and cubic dynamic susceptibilities. They also suggested approximate analytical expressions for these quantities.

In the case of *interacting* magnetic particles, experimental work related to the *nonlinear* magnetic response are even more scarce [23,24]. However, in a recent study it was shown that the *equilibrium* nonlinear response of an interacting magnetic particle system of monodispersive nature indicates that the cubic equilibrium susceptibility diverges at a finite temperature [24], thus providing further evidence for a low-temperature spin glass like phase. To the best of our knowledge, no experimental work has been reported for the *dynamic* nonlinear response of *interacting* particles.

In this paper, we study the linear and cubic dynamic susceptibilities of a magnetic particle system consisting of nanosized maghemite particles. The effects of inter-particle interactions are investigated by studying three samples with different volume concentrations of particles.

2. Experimental

The experiments were performed on samples consisting of nanosized maghemite (γ-Fe_2O_3) particles, with a mean diameter of 7 nm and almost spherical shape (observed in TEM analysis) [25][3]. The particles were suspended in a hydrocarbon oil and coated with a surfactant layer preventing the particles from agglomerating. Since the measurements were performed at low temperatures, the oil was frozen and the particles fixed randomly in the sample. Three samples with different volume concentration of particles, 0.3%, 3%, and 17%, were used. Experiments were also performed on a Ag(11 at%)Mn spin glass sample exhibiting long-range spin–spin interactions of RKKY type.

Two different experimental equipments have been used: (i) a commercial AC-susceptometer[4] was used to measure the first and third harmonics of the magnetization for different AC-field amplitudes in the range 100–2000 A/m; (ii) a noncommercial low-field superconducting quantum interference device (SQUID) magnetometer [26] was used to perform studies of the frequency dependence of the linear and cubic dynamic susceptibilities. Frequencies in range of 2–200 Hz were used.

The magnetization, M, can be expanded with respect to an applied field H as

$$M = \chi H + \chi_3 H^3 + \cdots, \tag{1}$$

where χ is the linear susceptibility and χ_3 the cubic susceptibility. The dynamic response can be probed by applying an AC-field, $H = h_0 \cos(\omega t)$. The linear susceptibility is then obtained from the magnetization measured at the fundamental frequency as $\chi(\omega) = M_\omega/h_0$ and the cubic dynamic susceptibility is obtained from the third harmonic of the magnetization as $1/4\chi_3(\omega) = M_{3\omega}/h_0^3$. These expressions are only valid if the applied AC-field is sufficiently low, so that contributions from higher order terms in the expansion of M_ω and $M_{3\omega}$ are negligible. For the measurements of the linear and cubic dynamic susceptibilities we used, in contrast to Refs. [15–17], different AC-fields to ascertain that no mixing with higher order susceptibilities occurred.

3. Theoretical background

3.1. Equilibrium susceptibilities

The linear and cubic *equilibrium* susceptibilities of a monodispersive system with uniaxial anisotropy

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and randomly distributed anisotropy axes, are given by [20,21]

$$\chi^{\mathrm{eq}}(T) = \frac{\mu_0 M_s^2 V}{3k_B T}, \qquad \chi_3^{\mathrm{eq}} = -\frac{\mu_0^3 M_s^4 V^3}{(k_B T)^3}\frac{(1+2S_2^2)}{45}, \tag{2}$$

where V is the particle volume, M_s the spontaneous magnetization, and

$$S_2(\sigma) = Z^{-1}\int_{-1}^{1} P_2(z)\exp(\sigma z^2)\,\mathrm{d}z. \tag{3}$$

Here $P_2(z) = (3z^2-1)/2$ is the second Legendre polynomial, $\sigma = KV/k_B T$, where K is the uniaxial anisotropy constant, and $Z = \int_{-1}^{1}\exp(\sigma z^2)\,\mathrm{d}z$ is the partition function in the absence of applied magnetic field. (In Ref. [27], Eq. (2) has been generalized for arbitrary anisotropy.)

3.2. Dynamic susceptibilities

Raikher and Stepanov [21] have studied theoretically the linear and cubic *dynamic* susceptibilities of noninteracting single-domain particles with uniaxial anisotropy, by solving numerically the Fokker–Planck equation in the overdamped case. It is difficult to derive analytical expressions for these quantities, but for a particle system with randomly distributed anisotropy axes, they suggested the following simple formula for the linear dynamic susceptibility:

$$\chi(T,\omega) = \chi^{\mathrm{eq}}\left[\frac{1}{3}\frac{1+2S_2}{1+\mathrm{i}\omega\tau} + \frac{2}{3}(1-S_2)\right]. \tag{4}$$

This expression has been shown to be a good approximation to the exact linear dynamic susceptibility for frequencies below ferromagnetic resonance (see, for instance, Refs. [28,29]. In the above expression τ is the relaxation time, for which various analytical expressions are available (see, for instance, Ref. [30]). In the moderate-to-high energy-barrier case, the relaxation time is given by an Arrhenius law as $\tau = \tau_0\exp(\sigma)$, where τ_0 is approximately a constant. Raikher and Stepanov also proposed an expression for the cubic dynamic susceptibility in the overdamped case, namely

$$\chi_3(T,\omega) = \chi_3^{\mathrm{eq}}\frac{(1-\mathrm{i}\omega\tau)}{(1+\mathrm{i}\omega\tau)(1+3\mathrm{i}\omega\tau)} \tag{5}$$

which they showed [21] to be a good approximation of the exact χ_3 in the low frequency regime.

4. Experimental results

The linear susceptibilities for the three maghemite samples measured at the frequency $\omega/2\pi = 125$ Hz and with an AC-field amplitude of 100 A/m are shown in Fig. 1. Because of the low particle concentration, the magnetic response of the most dilute sample is close to that of noninteracting particles. Dipole–dipole interactions shift the susceptibility peaks to higher temperatures, lower the magnitude of the peaks, but produce a slightly higher equilibrium susceptibility.

The frequency dependence of the out-of-phase component can be used as an indicator for the importance of including dipole–dipole interactions

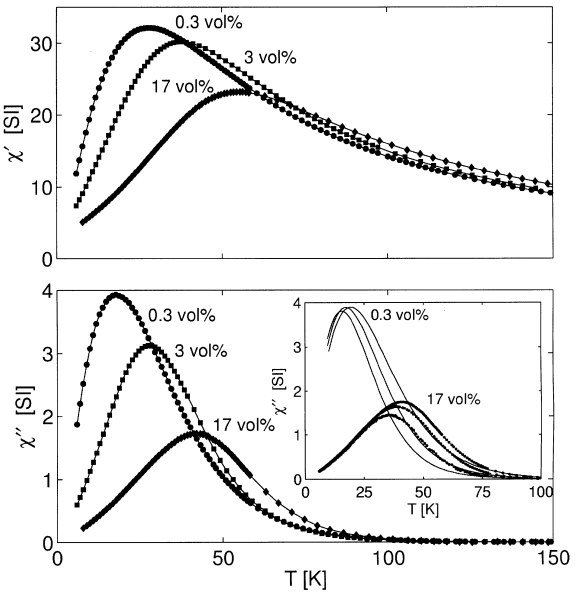

Fig. 1. The real (a) and the imaginary (b) parts of the linear susceptibility vs. temperature for three samples with different volume concentration of magnetic particles. $\omega/2\pi = 125$ Hz and $h_0 = 100$ A/m. Inset: the imaginary part of the linear susceptibility vs. temperature for the 0.3 and 17 vol% samples. The different curves correspond to different frequencies; for the 17 vol% sample $\omega/2\pi = 15$, 125 and 320 Hz and for the 0.3 vol% sample $\omega/2\pi = 2$, 20 and 200 Hz.

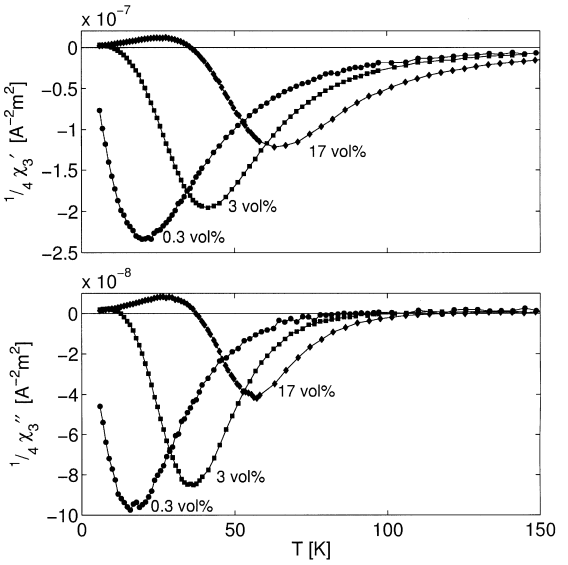

Fig. 2. The real (a) and the imaginary (b) parts of the cubic susceptibility vs. temperature for three samples with different volume concentration of magnetic particles. $\omega/2\pi = 125$ Hz and $h_0 = 200$ A/m.

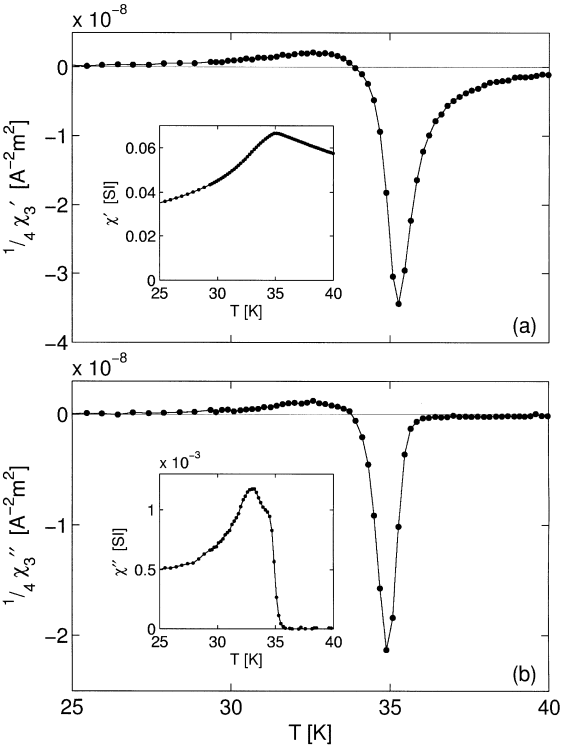

Fig. 3. The real (a) and the imaginary (b) parts of the cubic dynamic susceptibility vs. temperature for the Ag (11 at% Mn) spin glass sample. The linear susceptibility is shown in the insets. $\omega/2\pi = 125$ Hz and $h_0 = 1600$ A/m.

to explain the behavior of a nanoparticle system. In Fig. 1 it is seen that the height of the peak in χ'' for the 0.3 vol% sample is almost constant with frequency, as expected for a noninteracting system [4,5], while the height of the peak increases with increasing frequency for the 17 vol% sample.

Fig. 2 shows the corresponding cubic susceptibilities measured with an AC-field amplitude of 200 A/m. Similar to the linear susceptibility case, dipole–dipole interactions shift the susceptibility peaks to higher temperatures and reduce their magnitudes. The sample with the highest concentration of particles exhibits a second positive peak at low temperatures in both the real and imaginary components of the cubic susceptibility.

Finally, Fig. 3 shows the susceptibility of a Ag(11 at% Mn) spin glass measured at the frequency $\omega/2\pi = 125$ Hz and with an AC-field amplitude of 1600 A/m. The real part of the linear susceptibility has a sharp cusp at about the same temperature as where there is a sudden rise from zero of the imaginary part. The two components of the cubic susceptibility have sharp negative peaks at high temperatures followed by broad positive peaks at low temperatures.

5. Discussion

5.1. Noninteracting particles

It is important that effects of interaction are negligible in the experimental system, in order to be able to compare the measured susceptibilities with theoretical expressions for noninteracting particles. In an earlier study [12], the linear susceptibility of the most dilute sample used in the present work (0.3 vol%) was compared to that of an even more dilute sample (0.03 vol%). The linear susceptibility is identical for the two samples above 30 K, and below it differs only by a few percent. A second evidence for the 0.3 vol% sample being close to noninteracting is that the height of the peak in the linear out-of-phase component is approximately frequency independent (Fig. 1). In the only previous

published work on nonlinear susceptibility for 'noninteracting' particles the peak height of the linear out-of-phase component (see Fig. 2 in Ref. [15,16]) had a similar strong frequency dependence as that of the 17 vol% sample used here, which gives clear evidence that the particle system was interacting and therefore not suitable for comparison with theoretical expressions for noninteracting particles. This assertion is also confirmed by the poor agreement found between calculated and measured $\chi''(T,\omega)$ curves in Ref. [17, Fig. 7] in this reference).

To compare the measured susceptibilities with the theoretical expressions in Section 3, the polydispersivity of the particle system needs to be taken into account. The conventional approach is to choose a trial volume distribution and to determine its parameters by fitting theoretical curves to experimental data. We used the Nelder–Mead simplex method to perform nonlinear fitting to the measured linear and cubic susceptibilities. The fitting parameters apart from the volume distribution were the anisotropy constant and the pre-exponential factor (τ_0) in the expression for the relaxation time. To get the temperature dependence of the spontaneous magnetisation, we used the result from Ref. [4,5], namely, $M_s(T) = M_s(0)(1 - 1.8\times10^{-5}T^{3/2})$, with $M_s(0) = 4.2\times10^5$ A/m.

The best fit was obtained using a volume distribution of gamma type, given by

$$g(V) = \frac{1}{\Gamma(1+\beta)V_0}\left(\frac{V}{V_0}\right)^{\beta}\exp(-V/V_0), \qquad (6)$$

where $V_0 = 210\,\mathrm{nm}^3$ and $\beta = 0.34$. This corresponds to a mean particle diameter of 8 nm, which is slightly larger than the mean particle diameter of 7 nm observed in TEM studies [25] (see foot note 3). The uniaxial volume anisotropy constant was $K = 1.2\times10^4\,\mathrm{J/m}^3$ and $\tau_0 = 2.1\times10^{-10}$ s. The fits are shown in Fig. 4 for two frequencies of the AC-field. We also used a log-normal volume distribution, surface anisotropy, and expressions for the relaxation time different from the simple Arrhenius law, but none of these changes improved the quality of the fits significantly. Better fits are only obtained when fitting the linear and cubic susceptibilities separately, but the extracted parameters in the two cases are then different.

The discrepancies between the calculated and the measured susceptibilities may have several possible origins. One obvious origin is that the models used for the polydispersivity of the system are too simple and that the real situation is more involved (distributions in particle shapes, spontaneous magnetizations, etc.). It may also be that the symmetry of the magnetic anisotropy is significantly different from uniaxial (because of the geometrical shape of the particles, the magnetocrystalline anisotropy or even conflicting shape/magnetocrystalline anisotropy). However, the lack of theoretical expressions for the *dynamic* susceptibilities of systems with nonuniaxial anisotropy, does not allow us to investigate this possibility.

Another possible cause for the observed differences relies on details of the Fokker–Planck equation used in the work of Raikher and Stepanov [21], who studied the overdamped ($\lambda \gg 1$) case (λ is the damping constant in the stochastic Landau–Lifshitz equation from which the Fokker–Planck equation is derived). However, the most reliable values of the damping constant reported for magnetic nanoparticles are in the range 0.05–0.5 [31]. Although for uniaxial particles, the form of the dynamic linear susceptibility curves is not very sensitive to the value of λ (it essentially changes the value of τ_0), the same does not hold for the nonlinear response, and a significant dependence of $\chi_3(\omega)$ on the damping parameter has recently been predicted [32].

5.2. Interacting particles

In a model often used to analyze the dynamics of interacting particles [33,34], the effect of inter-particle interactions is accounted for by shifting the energy barrier distribution to higher energies and thereby increasing the relaxation times. For the particle system investigated here, it has previously been shown that the low-temperature magnetic relaxation of the most interacting sample is qualitatively different from that of the noninteracting sample [13]. For instance, the relaxation time spectrum broadens significantly with the interactions and *cannot be reduced to that of noninteracting*

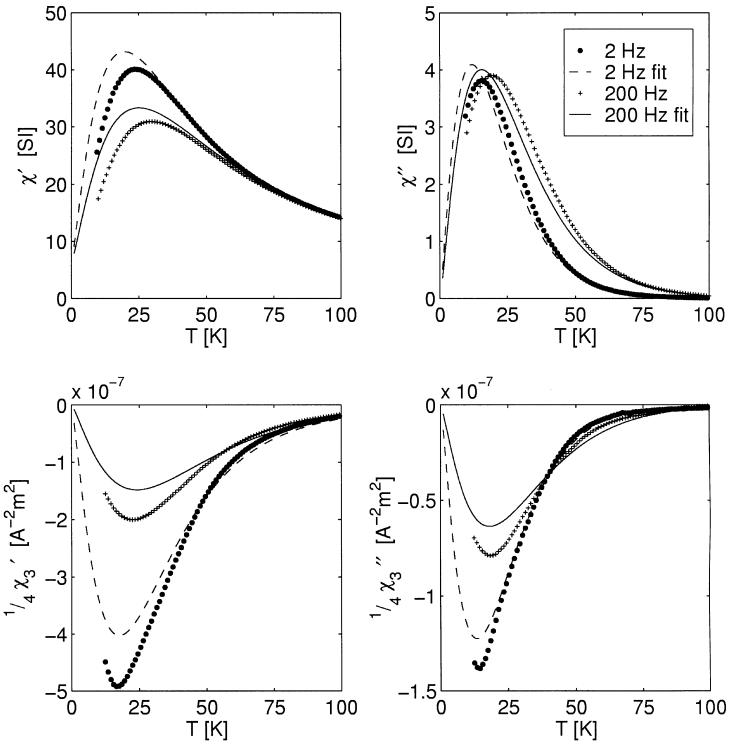

Fig. 4. The linear and cubic susceptibilities vs. temperature for the sample with a volume concentration of 0.3 vol%. Solid and dashed lines give the calculated susceptibilities using a gamma volume distribution, while the symbols give the corresponding experimental results.

particles shifted to longer time scales. This together with the observed aging effect [12] clearly shows that the low-temperature magnetic relaxation is dominated by collective particle dynamics. This makes it interesting to compare the low-temperature behavior of the most interacting sample to that of a typical spin glass with long-range interactions.

The cubic susceptibility of the AgMn spin glass (Fig. 3) shows a low-temperature positive peak similar to that of the most concentrated particle sample (Fig. 2). To explain this low-temperature positive peak, we will make use of the phenomenological droplet scaling theory [35,36] developed to describe both equilibrium and nonequilibrium behavior of spin glasses. In this model, following a quench to low temperature, the spin configuration can be decomposed into fractal domains of many length scales belonging to either of two spin glass ordered equilibrium states. The system will lower its energy by decreasing the amount of interface between the equilibrium states. This process begins by removing small domains and involves creation and/or annihilation of droplet excitations with a typical size similar to the domain sizes removed. Energy barriers for creation and annihilation of droplet excitations of length scale L, thus opposing the equilibration process, scale as $B(L) \sim L^{\psi}$, where the barrier exponent ψ depends on the dimensionality of the spin system. With time, larger and larger domains are removed and the characteristic length scale of domains after a time t is $R \propto [T\ln(t/\tau_0)]^{1/\psi}$.

In an AC susceptibility experiment, the polarization of droplets of size $L_\omega \propto [-T\ln(\omega\tau_0)]^{1/\psi}$ is probed. The time dependence of the AC susceptibility observed at low temperatures is closely connected to the aging process; at constant temperature,

both χ' and χ'' decrease with time. For droplets close to interface regions, the excitation energy is lower, yielding a correction to the density of active droplets. With equilibration time at constant temperature, the amount of interface region decreases and at the same time the density of active droplets decreases.

According to the droplet model, there is no spin glass transition in a field; the magnetic correlation length, ξ_H, is the length scale above which the field destroys the zero-field equilibrium spin glass states. Zero-field droplets of size $\gg \xi_H$ will, when the field is applied, be broken up into many smaller droplets of size ξ_H. These droplets are inactive on the time scale of the AC susceptibility measurement, having a size $\xi_H \gg L_\omega$, but will increase the amount of interface regions and thus, on average, decrease the excitation energy of active droplets. Applying a 'large' field, we thus expect an increase of both χ' and χ'' and a positive nonlinear susceptibility. The effect is expected to be largest in the temperature range where the influence of magnetic aging is largest, which for the most concentrated particle sample corresponds to temperatures $T < 40$ K.

At higher temperature, the behavior of the interacting nanoparticle sample is qualitatively different from that of the spin glass. For the nanoparticle sample, with increasing temperature, the time scale of the collective dynamics is gradually shifted to shorter time scales and the slow magnetic relaxation remaining is due to single-particle relaxation of large particles (still influenced by smaller superparamagnetic particles surrounding them). This gradual change of the magnetic relaxation with increasing temperature, from collective to single-particle dynamics, obstructs the observation of critical slowing down in this particle system. For the same reason, it will not be possible to observe a divergent cubic equilibrium susceptibility and all features are broader in temperature as compared to the corresponding features observed for the spin glass sample.

The high-temperature equilibrium susceptibility of the 17 vol% sample is larger than that of the noninteracting sample (cf. Fig. 1). This takes place because the local field acting on any particle moment contains a contribution from the dipole moments of all other particles in the system. For a random system, like a frozen ferrofluid, this contribution can be decomposed into two parts; the demagnetising field and the Lorentz cavity field. The implication is that the local field will be larger than the applied field for elongated samples when magnetised along the long axis. This is consistent with the shape of the studied sample, which is elongated along the probing field direction.

As for the increase of the equilibrium nonlinear susceptibility with increasing interaction we do not have such a simple argument as in the linear case. However, we believe that it is again related to the shape of the sample, and that either an increase or a decrease of the equilibrium nonlinear susceptibility could be attained by just changing the geometry of the system.

6. Summary and conclusions

In this work, detailed studies of the dynamic nonlinear susceptibility on magnetic nanoparticle systems exhibiting different degrees of interparticle interactions have been performed. The results for the most dilute sample enable for the first time a reliable comparison with theoretical expressions for the nonlinear susceptibility of noninteracting particle systems. Comparing separately the linear and nonlinear dynamic susceptibilities to the corresponding theoretical expressions, good fittings are obtained but with different values of the fitting parameters. This is also consistent with the differences observed between calculated and measured susceptibilities when the linear and nonlinear dynamic susceptibilities are taken into account simultaneously in the fitting procedure. The discrepancies thus found call for further theoretical developments, for instance by generalizing the theoretical expression for the dynamic nonlinear susceptibility to arbitrary symmetry of the magnetic anisotropy or to arbitrary values of the damping constant λ [32].

The dynamic nonlinear response of the most interacting sample is compared to that of a AgMn spin glass. In the low-temperature regime, both samples display the same characteristic behavior with a broad positive peak in both components of the nonlinear susceptibility. This behavior of the

nonlinear susceptibility, which has not been observed previously, is closely connected to the appearance of magnetic aging and is therefore attributed to the nonequilibrium behavior associated with disordered and frustrated spin systems. At higher temperature, the behavior of the interacting nanoparticle sample is qualitatively different from that of the spin glass. While the results of the spin glass sample indicate a divergent nonlinear susceptibility, the features observed in the nonlinear susceptibility of the nanoparticle sample are much broader in temperature. This is an effect caused by the polydispersivity of the particle system investigated here; with increasing temperature, there is a gradual change from collective to single-particle response.

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