Dynamic susceptibility of magnetic liquids

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The initial susceptibility of magnetic liquids was measured and calculated in the one-particle approximation for a wide range of frequencies from 10 to 10^9 Hz. A detailed analysis was made of the reasons for an analogy between the dynamics of magnetic liquids and that of dipole glasses. The interparticle magnetic-dipole interactions in ferromagnetic colloids were found to have a secondary role. Their influence on the dynamic susceptibility can be allowed for using the one-particle approximation by renormalization of the effective anisotropy constant.

INTRODUCTION

Intensive investigations of systems with a dipole–dipole interaction have drawn attention to the physics of magnetic liquids. These liquids are stable colloidal solutions of ferromagnets or ferrimagnets in any normal liquid and they have been investigated by many research teams for more than twenty years. This interest is due to the unique combinations of flow and high magnetic permeability of ferromagnetic colloids. For example, typical magnetite colloids with single-domain particles of 5–10 nm size and a saturation magnetization $M_0 \approx 50$ G have an effective viscosity of $\eta \sim 10^{-1}$ $\text{g}\cdot\text{cm}^{-1}\cdot\text{s}^{-1}$ (i.e., only an order of magnitude higher than that of water and two orders of magnitude less than that of glycerin) and an initial permeability $\mu \lesssim 10$. Liquids with record-high magnetizations of $M_0 \approx 100$ G are characterized by $\eta \sim 1$ $\text{g}\cdot\text{cm}^{-1}\cdot\text{s}^{-1}$ and $\mu \sim 10$–10^2, respectively.

For the nanometer particle size the magnetic moment of the particles is $m \sim 10^{-16}$ erg/G, i.e., $\sim 10^4$ Bohr magnetons. The magnetization curves of dilute or moderately concentrated ferromagnetic colloids can be described satisfactorily by a Langevin function with an argument mH/kT. This circumstance suggests that it should be possible to use the one-particle model to describe also dynamic properties of magnetic liquids. According to this model the dispersion of the initial susceptibility χ (at frequencies low compared with the ferromagnetic resonance frequency) is governed by the Debye expressions with two characteristic relaxation times corresponding to two independent fluctuation mechanisms of reorientation of the magnetic moment of the particles.[1] One of them is the Brownian mechanism associated with rotational diffusion of a particle in a viscous liquid and the other is the Néel mechanism with rotational diffusion of the magnetic moment inside a particle, i.e., relative to its crystallographic axes. (This mechanism is known to result in superparamagnetism of small particles of a ferromagnet embedded in a solid matrix.[2]) However, the experimental results usually fail to confirm the Debye nature of the dispersion of the susceptibility χ. Magnetic liquids exhibit a continuous and extremely wide (7–8 orders of magnitude!) spectrum of relaxation times, a quasilinear reduction in the real part of the susceptibility χ_1 on increase in the logarithm of the frequency, and a constant imaginary part χ_2 in a wide (4–6 orders) frequency range. Such a susceptibility spectrum is characteristic of spin and dipole glasses.

The hypothesis of the possibility of a transition of a magnetic liquid to a dipole glass state has been considered on a number of occasions.[3–7] In addition to the above-mentioned nature of the dispersion of χ, this hypothesis is supported also by the existence of a temperature maximum of the susceptibility.[5,8–10] The temperature of this maximum increases linearly on increase in the frequency logarithm and decreases on reduction in the concentration of particles in a solution. Our aim will be to show that the analogy between magnetic liquids and dipole glasses is purely superficial. In our opinion, both the dispersion of the susceptibility and the temperature maximum can be explained and described quantitatively by the one-particle model. The wide spectrum of the relaxation times is due to the natural polydisperse nature of colloidal particles and a strong (exponential) dependence of the Néel relaxation time on the particle size. A similar strong temperature dependence of the relaxation times is responsible for the observed temperature maximum of the susceptibility.[10] The collective effects due to the interaction of particles play a secondary role. These interactions create many-particle clusters and this can be allowed for primarily by renormalization of the effective magnetic anisotropy constant. The new experimental results obtained by us cover practically the whole range of dispersion of the susceptibility from 10 to 10^9 Hz.

1. EXPERIMENTAL METHOD AND RESULTS

We investigated highly concentrated colloidal solutions of magnetite in kerosene stabilized by oleic acid. These were the liquids used in the majority of the investigations cited above. Measurements of the real χ_1 and imaginary χ_2 parts of the susceptibility at low frequencies (up to 2×10^5 Hz) were made using a mutual inductance bridge employing a method described in Ref. 11. The experimental error did not exceed 2–3% for χ_1 and 4–6% for χ_2. At high frequencies (2×10^5–4×10^7 Hz) the susceptibility was determined from the change in the resonance frequency and in the loss resistance in a series-connected circuit. In the microwave range (up to 10^9 Hz) the measurements were made using a coaxial waveguide filled with a magnetic liquid.[12]

Images of colloidal particles obtained with the aid of an electron microscope indicated that their shape was nearly spherical. According to our data, the particle sizes could be described satisfactorily by the following gamma distribution:.

$$\varphi(x)=x^\beta \exp(-x)/\Gamma(\beta+1), \qquad (1)$$

where $\varphi(x)$ is the distribution density; $x = d/d_0$; d is the

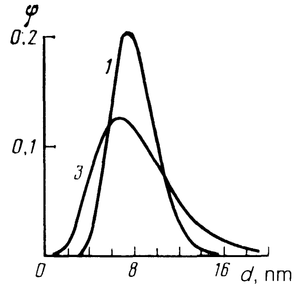

FIG. 1. Size distribution of magnetite particles in samples Nos. 1 and 3 (curves 1 and 3, respectively).

diameter of a magnetic (without a stabilizer coating) core of a colloidal particle; $\Gamma(Z)$ is the gamma function; d_0 and β are parameters. Experimental histograms[13,14] can also be approximated satisfactorily by the distribution of Eq. (1): the rms error does not exceed 2–4% of the maximum value of $\varphi(x)$. In the present study the parameters d_0 and β were determined from a static magnetization curve (by a variant of a magnetic granulometric analysis) making corrections for the interparticle magnetic-dipole interaction. The corrections for weak fields were calculated in the average sphere approximation[15,16] which made it possible to allow for the effects associated with the formation of clusters. In strong fields the interaction was allowed for using the model of the effective Lorentz force. The validity of this approach is demonstrated in Ref. 15.

We investigated three samples of magnetic liquids differing primarily in respect of the width of the particle size distribution. The average diameters of the particles were practically identical for all the samples. Figure 1 shows the distribution curves for samples Nos. 1 and 3 plotted on the basis of Eq. (1). The curve representing sample No. 2 is not included: it would occupy an intermediate position. The average magnetic moment $\langle m \rangle$ and the average diameter $\langle d \rangle$ of the particles, the saturation magnetization M_0, the static susceptibility in weak fields χ_0, and the density ρ of magnetic liquids are all listed in Table I. The frequency dependence of the susceptibility reduced to the static value is shown in Fig. 2. The points are the experimental results. Although the distributions of the particle sizes in the investigated magnetic liquids quite similar (Fig. 1 and Table I), the susceptibility spectra were very different. The reason for this effect was the above-mentioned feature of magnetic liquids, i.e., the existence of two mechanisms of relaxation of the magnetization and an exponential dependence of the Néel relaxation time on the particle size.

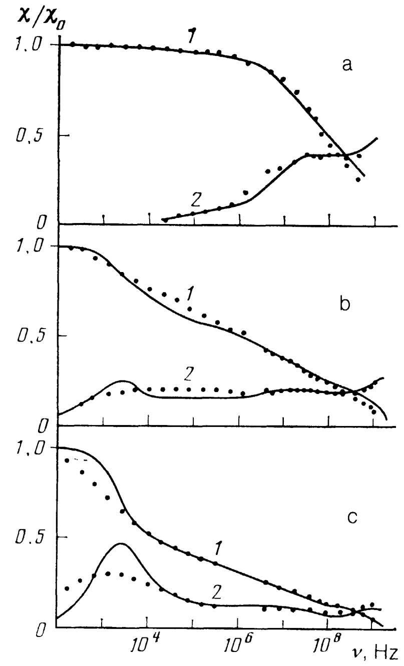

FIG. 2. Frequency dependences of the initial susceptibility of magnetic liquids: a) sample No. 1,; b) sample No. 2; c) sample No. 3. Curves labeled 1 represent the real part of the susceptibility and curves labeled 2 represent twice the imaginary part. The continuous curves are calculated.

2. ANALYSIS OF EXPERIMENTAL RESULTS

The Brownian τ_B and Néel τ_N relaxation times of the magnetization depend in different ways on the particle volume. The condition $\tau_N = \tau_B$ determines the characteristic size of a particle d_* and the characteristic frequency $\nu_* = 1/2\pi\tau_B(d_*)$. (Ref. 1). If $d < d_*$, the Néel relaxation mechanism predominates ($\tau_N < \tau_B$), whereas for $d > d_*$ the Brownian mechanism is stronger. It should be pointed out that generally speaking the value d_* is not equal to the limiting size of superparamagnetic particles: the Brownian fraction includes magnetically hard particles and some superparamagnetic particles with $\tau_N > \tau_B$. In the case of magnetic colloids, we have

$$d_* = 16-18 \text{ nm}, \quad \nu_* \sim 10^4 \text{ Hz},$$

$$\tau_N \sim 10^{-10}-10^{-5} \text{ s}, \quad \tau_B \sim 10^{-5}-10^{-3} \text{ s}.$$

Therefore, the high-frequency part of the susceptibility spectrum is formed by the "Néel" fraction of the smaller particles. The Brownian mechanism of the magnetization relaxation is then inactive. At low frequencies the magnetization

TABLE I.

Sample No.	$\langle m \rangle$, 10^{-16} erg/G	$\langle d \rangle$, nm	M_0, G	$4\pi\chi_0$	ρ, g/cm^3
1	1.54	8.0	55	2.07	1.61
2	1.81	7.8	51	4.88	1.44
3	2.23	8.2	47	6.63	1.40

reversal in small particles is quasistatic and the susceptibility dispersion is determined by the larger Brownian particles.

Our first sample had the narrowest particle size distribution and did not contain Brownian particles. For this reason the susceptibility dispersion was observed only at high frequencies in the range $10^6 \lesssim \nu \lesssim 10^9$ Hz (Fig. 2). The presence of particles of size $\approx d$ in the second sample increased immediately the range of dispersion to seven orders of magnitude because of the exponential dependence of τ_N on the particle volume V. These are the liquids that exhibited the closest similarity to dipole glasses: the susceptibility χ_2 remained constant in a wide frequency range and χ_1 decreased quasilinearly on increase in the frequency logarithm. The third sample had the widest particle size distribution. The relative number of Brownian particles this sample was about 3% but their contribution to the static susceptibility reached 50%. (This contribution was disproportionately high, because it rose as the sixth power of the particle diameter.) Consequently, a strong dispersion of χ_1 and a maximum of χ_2 at $\nu \sim 10^3$ Hz were observed.

The value of τ_B depended relatively weakly (linearly) on the particle volume; the natural polydisperse nature of ferromagnetic colloids simply increased (by one or two orders of magnitude) the broadening of the range of the Brownian times. A further rise of the number of large Brownian particles was accompanied by a fast increase of their contribution to the lf susceptibility and a reduction in the width of the spectrum of the magnetization relaxation times. In the final analysis the $\chi_1(\nu)$ and $\chi_2(\nu)$ curves became of the quasi-Debye type with a strong maximum of χ_2 in the frequency range 10^3–10^4 Hz. (The magnetic liquids with predominance of Brownian particles were investigated earlier[11,17] and the results were similar.) These results were in conflict with the hypothesis of a transition of magnetic liquids to a dipole glass state. According to this hypothesis the dispersion of the susceptibility typical of dipole glasses should be observed primarily for liquids with larger particles, i.e., cases characterized by a higher energy of the dipole–dipole interaction. However, the experimental results demonstrated that these liquids exhibited quasi-Debye behavior. The wide spectrum of the relaxation times was observed for ferromagnetic colloids with a predominant contribution of superparamagnetic particles to the susceptibility and a small value of the magnetic-dipole interaction parameter $\lambda = m^2/d^3kT$.

In estimating the influence of the magnetic-dipole interactions on the susceptibility spectra of ferromagnetic colloids we compared the experimental results with calculations carried out using the one-particle approximation.

3. CALCULATION METHOD

Cubic crystals of magnetite have a very weak magnetocrystalline anisotropy: the first anisotropy constant is $K_1 = -1.1 \times 10^5$ erg/cm^3. The main contribution to the effective anisotropy constant K comes from the shape anisotropy. This predominates when particles depart from spherical shape by just ≈ 1.5–2%. For this reason, the behavior of colloidal particles of magnetite in an external magnetic field is similar to that of uniaxial crystals, which is supported in particular by experimental studies of birefringence.[18,19]

The magnetic susceptibility of superparamagnetic particles in a periodic field, parallel and perpendicular to the easy magnetization axis, is discussed in Ref. 20. In a magnetic liquid subjected to a very weak (~ 1 Oe) measuring field the particle anisotropy axes are oriented at random. The contribution of the Néel particles to the susceptibility of a liquid is a linear combination of the longitudinal $\chi_\parallel$ and transverse $\chi_\perp$ susceptibilities:

$$\chi_N = {}^1/_3(\chi_\parallel + 2\chi_\perp).$$

The transverse susceptibility $\chi_\perp$ is found analytically in Ref. 20 for the full range of values of the ratio of the magnetic anisotropy to the thermal energy, $\sigma = KV/kT$, but there are no general solutions for $\chi_\parallel$ and the corresponding relaxation time $\tau_\parallel$. We shall use below an interpolation expression for $\tau_\parallel$, which gives reliable asymptotes for the limits of low and high values of σ, and agrees well with the numerical solution[20] for $\sigma \sim 1$:

$$\tau_\parallel = \tau_0 \sigma \exp(\sigma)(1+\sigma^3)^{-1/2} \cdot \tag{2}$$

Here, $\tau_0 = M_s/2\alpha\gamma K$; M_s is the saturation magnetization of the particle material; γ is the gyromagnetic ratio for an electron; α is the dimensionless damping coefficient. The contribution of Brownian particles can be calculated using familiar expressions (see, for example, Ref. 1).

Using the results given in Refs. 1 and 20, the density of the particle distribution described by Eq. (1), and the comments made above, we find that the dynamic susceptibility of a magnetic liquid can be represented by

$$\frac{\chi}{\chi_0} = \frac{1}{\Gamma(\beta+7)} \int_0^\infty G(\omega, x) x^{\beta+6} \exp(-x)\, dx. \tag{3}$$

In the case of Brownian particles ($x \gg d_*/d_0$) the function $G(\omega,x)$ in the integrand is a Debye factor:

$$G(\omega, x) = (1 + i\omega\tau_B)^{-1}, \tag{4}$$

where the Brownian relaxation time is

$$\tau_B = 3\eta(V + \Delta V)/kT, \tag{5}$$

where $\omega = 2\pi\nu$; η is the viscosity of the magnetic liquid; ΔV is the volume of the protective coating of a particle. In the case of Néel particles ($x \ll d_*/d_0$), the function $G(\omega,x)$ is described by

$$2G(\omega, x)F = \frac{(F+F')(R_1 + i\alpha W)}{R_1R_4 - W^2 + iWR_2} + \frac{2F'}{1 + i\omega\tau_\parallel}, \tag{6}$$

where

$$F(\sigma) = \int_0^1 \exp(\sigma Z^2)\, dZ,$$

$$R_1 = \sigma/R_3 + \alpha^2(\sigma R_3 - 2), \quad R_2 = \alpha(R_4 + \sigma R_3 - 2),$$

$$R_3 = \frac{F+F'}{3F'-F}, \quad R_4 = \frac{F+F'}{2(F-F')}, \quad W = \frac{\omega M_s V}{2\gamma kT}.$$

A prime denotes a derivative with respect to σ. In the range $x \approx d_*/d_0$ both relaxation mechanisms are important, so that in calculations we have to use Eq. (4) replacing the Brownian relaxation time τ_B with the reduced time[1]

$$\tau = \tau_B \tau_\parallel (\tau_B + \tau_\parallel)^{-1}.$$

Equations (3)–(6) describe the susceptibility dispersion throughout the experimentally investigated range of frequencies including the natural ferromagnetic resonance frequency ($\nu \gtrsim 10^9$ Hz).

The integral of Eq. (3) was calculated numerically using quadratures of the lowest degree of precision[21] on the assumption that $T = 295$ K, $M_s = 480$ G, and $\alpha = 0.1$. The viscosity of the investigated liquids was determined in additional experiments and was found to be within the range 0.11–$0.13\ \mathrm{g \cdot cm^{-1} \cdot s^{-1}}$. The thickness of the protective coating was assumed to be equal to the length of oleic acid molecules amounting to $\Delta d = 2$ nm. The values of χ_1 and χ_2 found from Eq. (3) were averaged on the assumption that a magnetic liquid is characterized by a uniform distribution of particles in respect of the effective anisotropy constant. The average value of the anisotropy constant $\langle K \rangle$ was found from the best fit to the experimental values of χ_1 and χ_2 near the frequency $\nu \sim 10^7$ Hz.

Therefore, the value of $\langle K \rangle$ was the only fitting parameter in our calculations. In this connection it should be mentioned that an independent determination of the particle shape anisotropy using electron micrographs is practically impossible, because one would then have to carry out measurements with a precision exceeding the resolution of the electron microscope employed. Moreover, the effective anisotropy constant is influenced strongly by particle aggregation processes, i.e., by the formation of many-particle chains or clusters in a magnetic liquid. Therefore, this constant can vary within a wide range depending on the degree of aggregation and its maximum value is $K_m \approx \pi M_s^2 = 7.2 \times 10^5$ erg/cm^3. The existence of many-particle aggregates containing 10 to 30 particles in ferromagnetic colloids can be regarded as firmly established. This follows from the experimental studies of the scattering of light[22] and polarized neutrons,[23] from investigations of diffusion processes,[24] from numerical modeling of the effects of the magnetic dipole interactions,[25] etc.

4. COMPARISON OF EXPERIMENTAL AND CALCULATED DATA

The results of a susceptibility calculation are presented in Fig. 2 in the form of continuous curves. We can see that the one-particle model describes well the high-frequency ($\nu = 10^4$–10^9 Hz) dispersion range of the susceptibility of all the samples. The influence of the magnetic dipole interactions can be allowed for by renormalization of the average anisotropy constant. The effective values of this constant for samples Nos. 1–3 are 1.3×10^5, 2.0×10^5, and 2.9×10^5 erg/cm^3. As expected, the effective magnetic anisotropy is much stronger than the crystallographic anisotropy and its magnitude rises on increase in the fraction of large particles, tending to form aggregates. The value $\langle K \rangle = 0.9 \times 10^5$ erg/cm^3 obtained in Ref. 26 by the rotating cell method for strongly dilute magnetite colloids is in agreement with our data.

A serious quantitative discrepancy between the experimental and calculated values of the susceptibility was observed only at low frequencies for sample No. 3 with the greatest proportion of large particles. This discrepancy is to be expected because the calculation model ignores the formation of ferromagnetic colloidal aggregates with the Brownian relaxation mechanism. The appearance of such aggregates alters the effective particle size distribution and shifts the dispersion region toward lower frequencies. Our estimates show that in order to ensure the observed shift each aggregate should consist of 3–10 particles, which is in agreement with the results of Refs. 22–25.

Equations (3)–(6) allow us to calculate the change in the dynamic susceptibility with temperature on condition that the temperature dependences of the viscosity $\eta(T)$ and of the static susceptibility $\chi_0(T)$ are determined independently. (The latter varies monotonically, but it is not generally described by the Curie law.[10,15]) In the present study the $\eta(T)$ and $\chi_0(T)$ dependences were obtained by direct measurements and smoothing out of the experimental data. The results of numerical modeling confirmed the conclusion[10] that the temperature maximum was of dynamic nature. The main reason for its formation was the blocking of the rotational degrees of freedom as a result of an exponential rise of the viscosity and of the Brownian relaxation time due to cooling.

Figure 3 shows, by way of example, the temperature dependence $\chi_1(T)$ for a magnetic liquid with a particle size distribution similar to that in sample No. 3, but with a somewhat lower concentration of the magnetic particles. Clearly, the one-particle model provided a qualitatively correct description and predicted quite accurately the temperature T_* of the maximum. This temperature rose linearly on increase in the frequency logarithm, as in the case of dipole glasses. The imaginary part of the susceptibility $\chi_2(T)$ also had a maximum, but it was shifted toward lower temperatures relative to T_*. This shift is sometimes regarded as one further example of transition magnetic liquids to a dipole glass state.[5] In reality, it is not associated with the magnetic dipole interactions [and this is true also of the maxima of the dependences $\chi_1(T)$ and $\chi_2(T)$] and is predicted even by the one-particle model. The blocking of the Néel relaxation mechanism can also give rise to a strong temperature maximum of the susceptibility, but in the case of ferromagnetic colloids with a distribution which is narrow in terms of the parameter σ.

It follows therefore that dynamic properties of ferromagnetic colloids (temperature and frequency dependences of the initial susceptibility) can be explained satisfactorily and quantitatively even by the one-particle model, provided we allow for the naturally polydisperse nature of colloidal particles. It is the particle size distribution that determines primarily the general nature of the susceptibility spectra. The magnetic dipole interactions do not give rise to qualita-

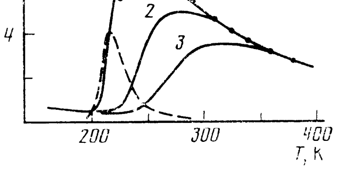

FIG. 3. Temperature dependences of the initial susceptibility. The continuous curves represent χ_1 calculated in the one-particle approximation: 1) $\nu = 40$ Hz; 2) 640 Hz; 3) 2.5 kHz. The dashed curve is χ_2 at $\nu = 40$ Hz. The points are the experimental values of χ_1 at $\nu = 40$ Hz.

tively new effects. Their influence may be allowed for in the one-particle model by renormalization of the effective anisotropy constant.

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