

Comparing magnetization fluctuations and dissipation in suspended magnetic nanoparticle ensembles

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Recent research has demonstrated that thermal fluctuations on the net zero magnetization of a magnetic nanoparticle (MNP) ensemble can serve as a valuable tool for characterizing the sample's magnetic properties. These spontaneous fluctuations are intrinsically linked to the MNP system's response to small perturbations, as described by the fluctuation-dissipation theorem. We experimentally compare fluctuations and dissipation in both the linear and non-linear response regimes. Notably, a strong correspondence between the power spectral density of the fluctuations and the out-of-phase dynamic susceptibility in the linear response regime was observed over a 500 kHz frequency range, facilitating interchangeability between these two characterization methods. The work contributes to the advanced characterization of MNPs for biomedical applications.

Index Terms—Magnetic nanoparticles, Fluctuation-dissipation theorem, Thermal noise magnetometry, AC susceptibility, AC hysteresis

I. INTRODUCTION

The characterization of magnetic nanoparticles (MNPs) is an important prerequisite for their safe and efficient use in biomedical applications. A crucial part of the characterization of MNP ensembles is the determination of their magnetic properties over a frequency range as wide as possible. Several established magnetic characterization techniques exist [1], [2], such as static magnetization measurements (DCM) [3], AC susceptibility (ACS) [4], magnetic particle spectroscopy (MPS) [5], magnetorelaxometry (MRX) [6], and AC hysteresis measurements (ACH) [7], covering a wide frequency range. Recently, thermal noise magnetometry (TNM) has been developed as an alternative characterization method [8], [9]. In this purely observational method, the spectrum of the spontaneous magnetization fluctuations in thermal equilibrium is measured directly without the need of applying an external magnetic excitation to the MNP sample.

The magnetization of MNPs can switch by overcoming the energy barrier set by their shape and magnetocrystalline anisotropy. These magnetization fluctuations are called Néel fluctuations and have a characteristic timescale

$$\tau_N = \tau_0 \exp\left(\frac{KV_c}{k_B T}\right),$$

for a large anisotropy barrier KV_c , where τ_0 is the inverse of the attempt frequency, K the anisotropy constant, V_c the core volume of the MNPs and $k_B T$ the thermal energy. There is an additional fluctuation mechanism when the MNPs are suspended in a fluid due to Brownian rotations with timescale

$$\tau_B = \frac{3\eta V_h}{k_B T},$$

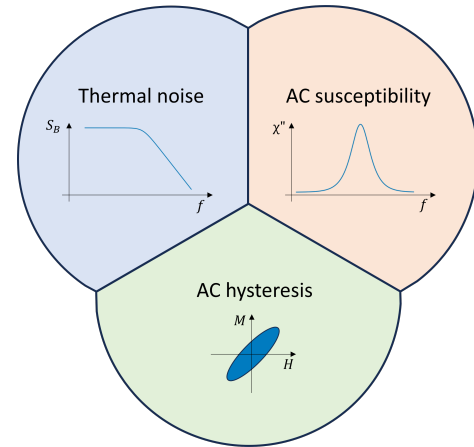


Fig. 1. Overview of compatible quantities for examining the magnetization dynamics of MNP ensembles within the linear response regime.

with V_h the hydrodynamical volume of the MNPs and η the viscosity of the suspension. An effective fluctuation time can then be defined as

$$\tau = \frac{\tau_N \tau_B}{\tau_N + \tau_B} \quad (1)$$

The thermal fluctuations observed in TNM are fundamentally related to the dissipation resulting from dynamics induced by a small AC magnetic field, as employed in ACS and ACH. In this case, the relationship between the fluctuations and the dissipative response is described by the Fluctuation Dissipation Theorem (FDT) [10]. This makes TNM, ACS and ACH compatible characterization techniques within the linear response regime, see Fig. 1. In this work, we experimentally compare the fluctuations with the out-of-phase component of the dynamic susceptibility for a linear magnetization response,

and the hysteresis losses in the case of a non-linear response at large excitation field amplitudes.

II. MAGNETIC NANOPARTICLES

Synomag-D (Micromod Partikeltechnologie, Rostock, Germany) particles with an average hydrodynamic diameter of 70 nm and an iron concentration of 8.25 mg/mL were used in the experiments. Synomag-D are multi-core particles composed of maghemite crystals forming a *nanoflower* structure in a matrix of dextran. They are suspended in a water solution, and have a plain surface. The particles are stable in the suspension, and no aggregation behaviour was observed. Experiments were conducted at room temperature, where the contribution of Néel fluctuations is negligibly small compared to the Brownian fluctuations for this MNP system, and this single relaxation mechanism is sufficient to describe the dynamics in small external fields.

III. THERMAL FLUCTUATIONS

In the absence of external magnetic excitations, thermal fluctuations maintain a zero net magnetization for a MNP ensemble. The Power Spectral Density (PSD) S_M of these fluctuations in the magnetization, measurable in one dimension (here z), takes the form of a Lorentzian¹:

$$S_M(f) = \langle M_z^2 \rangle \frac{2\tau}{1 + (2\pi f\tau)^2} \quad (2)$$

Here, τ is the effective fluctuation time of Eq. (1) and $\langle M_z^2 \rangle$ is the variance of the fluctuations. For an isotropic magnetization probability density, the variance has a value of $M^2/3$, with M the amplitude of the magnetization vector. The thermal fluctuations in the net magnetization of the MNP sample are measured experimentally as fluctuations in the magnetic flux density B with PSD

$$S_B(f) = \langle B^2 \rangle \frac{2\tau}{1 + (2\pi f\tau)^2} \quad (3)$$

so that $S_B \propto S_M$. The variance of the magnetic flux density fluctuations $\langle B^2 \rangle$ is therefore defined by $\langle M_z^2 \rangle$, the geometry of the experiment, and the magnetic volume of the MNPs [12]. For a polydisperse MNP ensemble, the PSD is a superposition of the Lorentzian in Eq. (2), weighted by the size distribution of the ensemble.

To perform TNM measurements with a frequency bandwidth spanning 5 orders of magnitude, we employed two different in-house developed SQUID systems. System 1 (S1) consist of a SQUID magnetometer in a superconducting magnetic shield [13]. System 2 (S2) consist of an ultrasensitive SQUID magnetometer with a bandwidth up to 1 MHz in a magnetically shielded room [14]. The stochastic time trace of the ensembles magnetization is recorded for several minutes, from which the PSD is calculated as reported in Ref. [9].

¹in the assumption that the autocorrelation function, i.e. the relaxation function, can be described by a single exponential decay, which is the case for a pure Brownian relaxation [11].

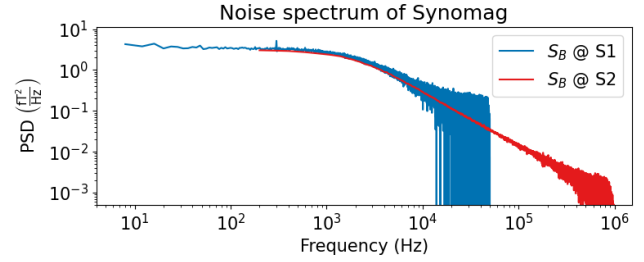


Fig. 2. Thermal noise spectrum of Synomag measured in system 1 (S1) and system 2 (S2).

Fig. 2 shows the PSD of the Synomag MNP ensemble measured in the two SQUID systems. A small offset in PSD amplitude is present due to the difference in the geometries (SQUID pickup coil geometry, sample-sensor distance etc.) of the two setups. TNM as method for determining magnetization in its presented form covers a frequency range from 8 Hz to 1 MHz. Here, the lower boundary on this range is dictated by the total duration of the measurement, and can thus be extended for longer measurements.

IV. MNP RESPONSE AND DISSIPATION

In the presence of an AC excitation field of the form $H(t) = H_0 \cos(2\pi ft) = \Re(H_0 \exp(i2\pi ft))$, the magnetization response $M(t)$ of the MNPs has the form:

$$M(t) = \Re(\chi H_0 \exp(i2\pi ft)) \quad (4)$$

with χ the magnetic susceptibility $\chi = \chi' - i\chi''$. Here, χ' is the real part of the susceptibility, denoting the in-phase component of the response signal, and χ'' is the imaginary part of the susceptibility, denoting the out-of-phase component of the response signal. The out-of-phase component is responsible for the dissipative hysteresis behaviour.

A physical quantity related to MNPs heat dissipation under an AC excitation field is given by the Specific Absorption Rate (SAR), which can be calculated as

$$\begin{aligned} \text{SAR} &= \frac{f}{c} \mu_0 \oint M(t) dH(t) \\ &= \frac{f}{c} \mu_0 A \end{aligned} \quad (5)$$

Here, f is the frequency of the applied field, c the MNP concentration, and μ_0 the vacuum permeability. The integral denotes the area A of the magnetic hysteresis loop. SAR is an important parameter for magnetic hyperthermia, as it assess the efficiency of MNPs to transfer magnetic energy into heat [15].

A. Dissipation in the linear magnetization response regime

For small excitation amplitudes², the MNPs' magnetization response is linear:

$$M(t) = H_0 (\chi' \cos(2\pi ft) + \chi'' \sin(2\pi ft)) \quad (6)$$

²e.g. when H fulfills the requirement $\frac{\mu_0 M_S V H}{k_B T} < 1$ [16]

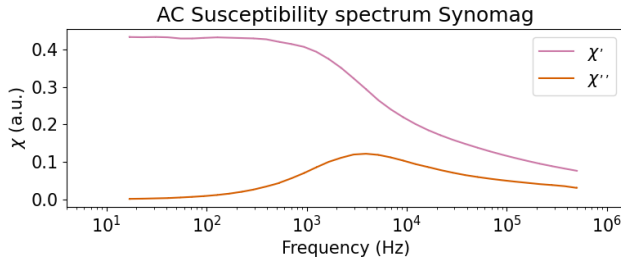


Fig. 3. In-phase (χ') and out-of-phase (χ'') dynamic susceptibility spectrum of Synomag for an excitation field with frequencies running from 17 Hz - 500 kHz and field amplitudes below 400 A/m.

In this regime, linear response theory (LRT) applies. The dynamic susceptibility χ of MNPs can be described by the Debye model:

$$\chi(f) = \frac{\chi_0}{1 + i2\pi f\tau}, \quad \chi_0 = \frac{\mu_0 n m^2}{3k_B T} \quad (7)$$

Here, χ_0 is the static susceptibility with n the MNP density, m the magnetic moment of a single MNP. The in-phase $\chi'(f)$ and out-of-phase $\chi''(f)$ susceptibility are then given by

$$\chi'(f) = \frac{\chi_0}{1 + (2\pi f\tau)^2}, \quad \chi''(f) = \chi_0 \frac{2\pi f\tau}{1 + (2\pi f\tau)^2} \quad (8)$$

In addition to calculating the integral as in Eq. (5), the dissipated energy per unit volume ΔU at excitation frequency f can be calculated in the linear response by [17], [18]:

$$\Delta U(f) = \mu_0 A(f) = \frac{c}{f} \text{SAR} = \pi \mu_0 H_0^2 \chi''(f) \quad (9)$$

The largest dissipation is thus found when χ'' is maximal; i.e. for the frequency $f = 1/(2\pi\tau)$.

AC susceptibility measurements

The dynamic susceptibility $\chi(f)$ of the Synomag MNP sample was measured using a commercial AC susceptometer [19] (Rise Research Institutes, Sweden). The excitation field has frequencies running from 17 Hz - 500 kHz and field amplitudes below 400 A/m; small enough to ensure the linear response of the MNPs. The in-phase and out-of-phase susceptibility spectra of the Synomag MNPs are shown in Fig. 3.

B. Dissipation in the non-linear magnetization response regime

For large excitation amplitudes, higher order susceptibilities ($k > 1$) contribute to the MNP AC response (with $\omega = 2\pi f$):

$$M(t) = H_0 \sum_{k=1}^{\infty} (\chi'_k(H_0, \omega) \cos(\omega kt) + \chi''_k(H_0, \omega) \sin(\omega kt)) \quad (10)$$

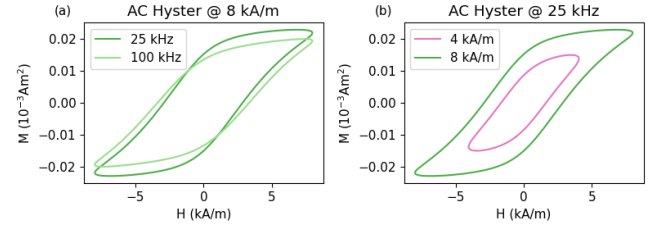


Fig. 4. AC hysteresis loops of Synomag at (a) different frequencies and (b) different field amplitudes. Note that for both field amplitudes, there is a non-linear response.

The LRT cannot be applied anymore, and the dissipation can only be calculated by integrating the hysteresis loop [20]:

$$\begin{aligned} \Delta U &= -\mu_0 \oint M(t) dH(t) \\ &= \mu_0 H_0^2 \sum_{k=1}^{\infty} \chi'_k \int_0^{2\pi/\omega} \cos(\omega kt) \sin(\omega t) \omega dt \\ &\quad + \mu_0 H_0^2 \sum_{k=1}^{\infty} \chi''_k \int_0^{2\pi/\omega} \sin(\omega kt) \sin(\omega t) \omega dt \\ &= 0 + \pi \mu_0 H_0^2 \chi''_1 \end{aligned} \quad (11)$$

From the out-of-phase response, only the linear component remains after the integration over the excitation field. This means that the dissipation is fully determined by the linear component of the out-of-phase non-linear response for a given excitation amplitude H_0 and vice versa. Note that the response variables (χ'_k , χ''_k) depend both on field frequency and amplitude, and thus that χ''_1 is different from the linear χ'' in Eq. (9).

AC hysteresis measurements

AC magnetization responses in the non-linear regime were measured using a commercial system (AC Hyster, NanoTech Solutions) at nominal field strengths of 4 kA/m and 8 kA/m and frequencies of 10, 25, 50, 75, and 100 kHz. The $\Delta U(f)$ values are subsequently calculated by numerically integrating the AC hysteresis loops for all excitation field frequencies. Examples of the AC hysteresis loops are displayed in Fig. 4. The shape of the loops deviate from an ellipse, thus it is clear that the magnetization response is non-linear.

V. COMPARING FLUCTUATIONS WITH DISSIPATION

The FDT [10] relates the out-of-phase dynamic susceptibility in Eq. (8) with the PSD of the fluctuations in Eq. (2):

$$S_B(f) \propto S_M(f) = \frac{k_B T}{\pi^2 V \mu_0 f} \chi''(f) \quad (12)$$

The noise spectrum $S_B(f)$ from the TNM measurements and the *frequency weighted* out-of-phase AC susceptibility spectrum $\frac{\chi''(f)}{f}$ from the AC susceptibility measurements (i.e. the calculated PSD from ACS spectrum) are compared with each other in Fig. 5 (a)³. For clarity, the PSD measured

³As the prefactors of the spectra do not match each other due to different experimental geometries, only the comparison in terms of spectral dependency is possible

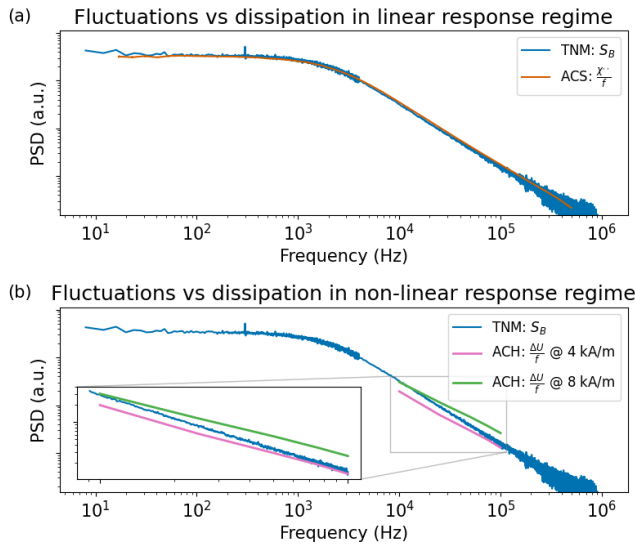


Fig. 5. Comparison of the dissipation in magnetization response measurements of suspended Synomag MNPs with the fluctuations in the magnetization in thermal equilibrium. The combined PSD of Fig. 2 is plotted against (a) the frequency weighted out-of-phase AC susceptibility of Fig. 3 and (b) the frequency weighted dissipation of the AC hysteresis of Fig. 4. The inset highlights that the slopes of the TNM and ACH-based data do not coincide, indicating a non-linear response for the ACH measurement.

in S1 is only shown up to 4 kHz, and the PSDs of S1 and S2 were scaled to produce a combined PSD from the two TNM measurement setups⁴. A good agreement between the TNM and ACS spectra is found over the full frequency range, implying identical magnetization dynamics in both methods. It is the first time that this agreement of the two magnetization dynamics measurement techniques has been shown for suspended MNPs over a 500 kHz bandwidth.

The measure of dissipation ΔU from the AC hysteresis measurement in the excitation frequency range [10-100] kHz can also be compared with the noise measurements. In this case, the response is non-linear and the FDT is not valid. Therefore, ΔU does not satisfy the relation

$$S_B(f) \propto \frac{\Delta U(f)}{f}, \quad (13)$$

which is clear on Fig 5 (b). As extensively discussed in earlier work [16], [18], [21], linear response theory is not suited to describe MNP responses with high excitation amplitudes, which are most relevant to magnetic hyperthermia. A less steep decrease of $\frac{\Delta U(f)}{f}$ is found than the decrease in the PSD in the same frequency range for both field amplitudes, suggesting the largest dissipation per unit volume $\Delta U(f)$ will be found at a smaller frequency than the one predicted by LRT $1/(2\pi\tau)$. However, due to the small frequency range of the hysteresis measurements compared to the noise measurements, conducting a comprehensive comparison between the two methods is challenging.

⁴the scaling factor solely depends on the geometrical differences between S1 and S2, as mentioned in Fig. 2

VI. CONCLUSION

We compare the thermal fluctuations in the magnetization with the dissipation in two magnetization response regimes for suspended MNPs at room temperature. For the non-linear response, the FDT cannot be applied, and the noise measurements cannot be unified with the hysteresis losses. In the linear response, a good agreement over the wide frequency range of 500 kHz between the PSD of the fluctuations and the out-of-phase AC susceptibility is found, showing for the first time that the magnetization dynamics are accessible by both methods. ACS and TNM can now be used interchangeably, from noise measurements to response measurements and vice versa.

The TNM measurements prove the claim of the ACS method that the small fields applied in ACS do not manipulate the sample for the considered MNP system. This implies that no interparticle structures are formed as a result of the applied field — an assumption that cannot be made in other measurements methods, for instance M(H) measurements. TNM and ACS are thus both methods with minimal impact on the MNPs ensemble. However, while ACS measurements of MNP ensembles take several hours to complete, the presented TNM measurements only take a few minutes. The out-of-phase component of the susceptibility is proposed as a means to monitor changes in MNP properties over an extended period, spanning several months [22], [23]. Likewise, TNM can be utilized to monitor MNP properties during processes occurring on the hourly timescale, such as cellular uptake [24], as well as over longer durations.

Finally, incorporating MRX measurements into this array of magnetization dynamics characterization methods would be interesting for future studies [25]–[27]. The MNPs are exposed to a large DC magnetic field during the MRX experiment, but the method still complies with LRT. The characteristic curve in MRX is represented by the magnetization autocorrelation function, which is obtained by measuring after the DC field is switched off. As a result, it serves as the direct Fourier inverse of the PSD. Thus, comparing the PSD in TNM, measured in the total absence of external excitation, to the characteristic relaxation curve in MRX can unveil the effects of the DC field on the formation of interparticle structures [28].

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