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Sensing magnetic nanoparticles using nano-confined ferromagnetic resonances in a magnonic crystal

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We experimentally demonstrate the use of the magnetic-field-dependence of highly spatially confined, GHz-frequency ferromagnetic resonances for the detection of magnetic nanoparticles using an anti-dot-based magnonic crystal. The stray magnetic fields of nanoparticles within the anti-dots modify nano-confined ferromagnetic resonances in the surrounding periodically nanopatterned magnonic crystal, generating easily measurable resonance peak shifts. The shifts are comparable to the resonance linewidths for high anti-dot filling fractions with their signs and magnitudes dependent upon the mode localization, consistent with micromagnetic simulation results. This is an encouraging result for the development of frequency-based nanoparticle detectors for nano-scale biosensing. © 2015 AIP Publishing LLC. [<http://dx.doi.org/10.1063/1.4922392>]

Magnetic biosensors, in which biological analytes are tagged, typically *in-vitro*, with magnetic nanoparticles (MNPs), have excellent potential for solid-state point-of-care medical diagnostics.^{1–3} The technique is intrinsically matrix-insensitive,¹ can compete with industry-standard immunoassays,⁴ and can be combined with magnetic separation.⁵ The central element of many miniaturized magnetic biosensors is a detector for the stray or “fringing” magnetic fields generated by the magnetized MNP tags. Previously used sensors include SQUIDs,⁶ Hall sensors,⁷ ferromagnetic rings,^{8,9} and magneto-impedance devices.¹⁰ There are also other detection methods for MNPs in fluids using, e.g., micro-NMR¹¹ or coils.¹² However, one of the most widely used MNP detection methods employs magnetoresistive (MR) magnetic field sensors^{1–5,13–17} which typically have at least one lateral dimension on the order of a few μm or greater. The stray magnetic fields of nearby MNPs modify the quasi-static magnetic configuration in ferromagnetic MR devices, changing their resistances and thus enabling electronic MNP detection.

It can, however, be challenging to minimize noise in conventional MR sensors when reducing their size to nanometric scales due to thermal instabilities of the magnetic configuration.^{18–20} A suggested approach to overcome this issue is to use intrinsically frequency-based detection methods exploiting magnetic-field-dependent resonant magnetization dynamics in ferromagnetic nanostructures.^{21–23} These (typically GHz frequency) dynamics can also be driven electrically in spin torque oscillators (e.g., Refs. 21 and 24–27, and references therein) which have been predicted to retain high sensing signal to noise ratios at sub-100 nm dimensions.^{21,22} The possibility of real time electrical detection of these dynamics^{28–30}

may open a route to high speed,²² solid-state, magnetic flow cytometry (e.g., Refs. 31–33) using micro- or nano-fluidics.

In this work, we use a large area^{34,35} magnonic crystal (MC)³⁶ to macroscopically probe resonant GHz-frequency magnetization dynamics which are spatially confined to nanometric regions and experimentally demonstrate their use for MNP sensing. In contrast to continuous ferromagnetic layers, nanostructured MCs [e.g., Fig. 1(a)] exhibit a number of distinct ferromagnetic resonance (FMR) modes with different lateral spatial localizations within the crystal’s nano-periodic structure.^{37–39} A previous study demonstrated that FMR modes within anti-dot-based MCs were sensitive to the presence of magnetic nanostructures fabricated within the anti-dots, leading to a proposal for a magnonic biosensor.⁴⁰ Here, we demonstrate that stray magnetic fields generated by captured MNPs within an anti-dot-based MC do indeed generate clear FMR peak shifts which are mode-dependent and which approach the resonance linewidth when the fraction of MNP-filled anti-dots is high. Note that resonances in small collections of MNPs can also be detected directly; however, only broad, relatively weak signals have been seen previously.⁴¹ Rather, here we detect MNP-induced changes to very well defined resonances within a periodically nanostructured, high quality ferromagnetic film.

FIG. 1. (a) Scanning electron micrograph of a portion of a magnonic crystal. The scale bar is 1 μm long. (b) Schematic of the experimental setup showing the MC placed face-down above a micro-stripline (the side of the wafer covered by the patterned magnetic layer (the MC) is facing the stripline).

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Large area ($4 \times 4 \text{ mm}^2$) ferromagnetic MCs consisting of arrays of $0.3 \mu\text{m}$ wide anti-dots with edge-to-edge spacings of $0.15 \mu\text{m}$ were fabricated from a $\text{Ni}_{80}\text{Fe}_{20}$ (30 nm)/ Au (8 or 10 nm) layer using deep ultraviolet lithography^{34,35} [Fig. 1(a)]. The MCs' FMR modes were probed at room temperature using broadband, stripline-based, magnetic field modulated FMR spectroscopy [Fig. 1(b)]. This technique measures the derivative of finite width FMR peaks with respect to the external magnetic field, H_{ext} , at a fixed GHz frequency. MC spectra are measured both before and after the addition of cluster-shaped MNPs (diameters $\sim 0.1 - 0.3 \mu\text{m}$). Two micromagnetic simulation methods have also been used, both employing periodic boundary conditions:⁴² (i) a time domain (ringdown) simulation using MuMax3,⁴³ which subjects the MC to an in-plane magnetic field pulse which is perpendicular to H_{ext} and exploits Fourier analysis on the resulting dynamics to extract frequency-resolved information (e.g., Ref. 44), and (ii) an eigenmode method (e.g., Ref. 45) using the FinMag package (successor to Nmag⁴⁶), which directly calculates the FMR mode frequencies and profiles from the system's equilibrium configuration at a given H_{ext} . Additional details are given in the supplementary material.⁴⁷

An experimental FMR spectrum for a bare MC (i.e., no adsorbed MNPs) obtained at 11.5 GHz is shown in Fig. 2(a) in which two high amplitude resonances near the extremes of the measured H_{ext} range can be identified. The frequency (f) dependence of the resonance fields, H_{res} , of these two FMR modes compare well with those predicted from time domain simulations for the extended (E) and side (S) modes³⁷⁻³⁹ [Fig. 2(b)]. In Fig. 2(c), we show Fourier transformed data from the time domain simulation obtained at $\mu_0 H_{\text{ext}} = 200 \text{ mT}$, showing the E and S modes together with their spatial localizations inside the MC's unit cell.

These modes will be the focus of this letter. The E mode is concentrated in bands between rows of anti-dots oriented perpendicular to H_{ext} , while the S mode is localized between neighboring anti-dots [see also the schematic in Fig. 2(d)]. In both simulation and experiment, a number of lower amplitude modes lie between the side and extended modes, demonstrating good agreement in terms of the overall mode structure. We also note that the simulation tends to slightly overestimate the frequency of the S mode (as observed previously for a similarly localized mode³⁹).

MNPs were adsorbed onto the MCs at $H_{\text{ext}} = 0$ by placing $12 \mu\text{l}$ of diluted MNP suspension on the MC's surface, which then was allowed to dry in ambient conditions before re-measurement. Scanning electron microscopy of dried MNPs on a MC reveals irregularly shaped MNPs with the majority lying inside the anti-dots [see Fig. 3(a) and caption]. The MNPs generate an upward shift in H_{res} for the side mode resonance [Fig. 3(b)] and a downward shift for the extended mode resonance [Fig. 3(c)]. Both shifts exceed experimental uncertainty (see the caption of Fig. 3).

The observed shifts can be qualitatively understood by considering the field generated by an idealized magnetized MNP, located at the center of an anti-dot and magnetized along H_{ext} [Fig. 2(d)]. Treating the MNP as a dipole, the y-component of its stray magnetic field will, to a first approximation, reinforce H_{ext} at the E mode's region but oppose H_{ext} at the S mode's region. Thus, for a given experimental measurement frequency, a larger H_{ext} must be applied to attain the S mode resonance condition when MNPs are within the anti-dots. In the same way, the E mode should be observed at a lower H_{ext} . These predictions are consistent with the experimental results [Figs. 3(b) and 3(c)] as well as analogous numerical results obtained for anti-ring structures.⁴⁰ Time domain simulations were also repeated with a

FIG. 2. (a) Experimental, field-resolved FMR trace ($f = 11.5 \text{ GHz}$) showing differential absorption peaks corresponding to FMR modes in the MC. (b) Comparison of experimental and simulated resonant frequencies versus $\mu_0 H_{\text{ext}}$ for the side (S) and extended (E) modes. (c) Frequency-resolved, Fourier transformed time domain simulation data at $\mu_0 H_{\text{ext}} = 200 \text{ mT}$ for a single unit cell of the MC with and without a 150 nm MNP at the center of the anti-dot. The Fourier amplitude has been differentiated with respect to f to facilitate comparison with experimental spectra. Insets show the localization of the resonant dynamics for the S and E modes with lighter shading indicating a stronger dynamic magnetization component perpendicular to H_{ext} . The anti-dot boundary is shown as a blue circle. (d) Schematic showing the spatial localization of the E and S modes together with a MNP and a sketch of its stray magnetic field.

FIG. 3. (a) SEM image of MNPs on the MC with a 300 nm wide scale bar. Out of 33 holes counted, 30 contain MNPs. In seven of the thirty filled holes, the “captured” MNP extends outside of the anti-dot. There are also 5 isolated nanoparticles on the MC’s upper surface. FMR traces obtained at 11.5 GHz showing the S mode (b) and E mode (c) before (bare) and after the addition of MNPs. Multiple traces have been taken following repeated removal and replacement of the MC, a requirement for MNP application, enabling an estimation of the associated experimental uncertainty (~ 0.5 mT). Traces have been vertically scaled and vertically shifted to locally normalize the differential absorption signals to that obtained with a bare MC.

150 nm wide spherical MNP within the anti-dot (see supplementary material⁴⁷ for further details including the H_{ext} -dependent MNP moment in Fig. S1). The result for the MC in the presence of a MNP with $\mu_0 H_{\text{ext}} = 200$ mT is shown as a red dotted line in Fig. 2(c). The S mode’s frequency is indeed decreased, consistent with that part of the MC experiencing a lower net field. Likewise, an increased resonance frequency is predicted for the E mode.

The experimentally observed peak shifts will, however, depend on the MNP coverage. The effects of this were measured on a second MC with an equivalent anti-dot lattice geometry. We carried out consecutive applications of diluted solutions of MNPs with increasing concentration, c , obtaining FMR spectra and SEM micrographs before and after solution applications. Representative SEM images are shown in Figs. 4(a)–4(d) for each c . Increases in c visibly increase the MNP coverage, resulting in an increased peak shift for both the E and S modes. For the lowest coverage, a shift of the E mode is measurable and just higher than the experimental uncertainty [Fig. 4(e)]. At the highest coverage however, the shift is much larger and approaches the peak-to-peak resonance linewidth. There was some variation in the measured shifts for different f ; however, no clear, widely applicable trends could be determined (except at low f , and thus low H_{ext} , for the S mode where a smaller shift presumably resulted from a lower H_{ext} -induced MNP moment). In Fig. 4(f), we have averaged the S and E modes’ peak shifts over the measured f range (11.5–16 GHz) and plotted the averaged shifts versus c . Reliably measurable shifts of the E mode are observed over the entire frequency range. However, a higher c must be used to register a consistent shift for the S mode. We also note that the five intermediate modes closest to the E mode exhibited MNP-induced shifts consistent in sign with those predicted from Fig. 2(c).

FIG. 4. (a)–(d) $2.25 \mu\text{m}$ wide images of the MC following consecutive applications of aqueous MNP solutions of increasing concentrations, c ($\mu\text{g}/\mu\text{L}$). (e) Differential absorption peak for the E mode at 12 GHz in the bare MC and following adsorption of MNPs for increasing values of c . (f) E and S mode peak shifts and (g) peak-to-peak linewidth increase relative to the bare MC (1 = no change, 2 = linewidth doubled) as a function of MNP solution concentration. Error bars combine the uncertainty in the shift measurement at each f and the spread of shifts over the measured f range (11.5–16 GHz). The horizontal dotted lines in (f) show the predicted field shifts calculated from the eigenmode simulations for the S mode (2 mT) and E mode (−6.9 mT).

As an aside, we note that equivalent experiments were carried out on an unpatterned $\text{Ni}_{80}\text{Fe}_{20}$ (30 nm)/Au (10 nm) layer for which a single FMR mode was seen. At the highest concentration, a weak shift of -0.6 mT was obtained at 12 GHz, which is comparable to the experimental uncertainty and about ten times less than the shift observed for the E mode in Fig. 4(e). This highlights the importance of MNP capture within the anti-dots and the use of nano-confined modes for high sensitivity FMR-mode-based MNP detection. We also note that there was more MNP agglomeration on the unpatterned film.

Eigenmode simulations were used to estimate numerical values for these MNP-induced shifts. Simulations were carried out at $\mu_0 H_{\text{ext}} = 37$ mT [to probe the side mode, referencing the trace in Fig. 3(b)] and at 206 mT [to probe the extended mode, referencing the trace in Fig. 3(c)]. The simulations yielded MNP-induced frequency shifts of $+0.311$ GHz for the E mode and -0.085 GHz for the S mode, in good agreement with time domain simulations. The local gradient of the f_{res} vs. H_{res} data [Fig. 2(b)] was then used to convert the

frequency shifts into equivalent field shifts. This yielded -6.9 mT for the extended mode (45 GHz/T) and $+2.0$ mT for the side mode (43 GHz/T). These values compare well with the shifts observed in Fig. 4(f) at $c = 0.225$ $\mu\text{g}/\mu\text{l}$. Notably, this is the concentration which is closest to the simulation condition where we have almost every anti-dot containing a MNP [Fig. 4(d)]. The frequency-field duality is further discussed in the supplementary material,⁴⁷ Fig. S2.

The presence of the MNPs results in a coverage-dependent linewidth increase with a ~ 1.5 times increase on average in the peak-to-peak resonance linewidths at the highest c [Fig. 4(g)]. However, the linewidth broadening does not dominate the observed shifts in that the minimum of the differential absorption line moves in the direction of the peak shift [Fig. 4(e)]. This is qualitatively consistent with that expected for a collection of MNPs with different sizes (and thus different moments) acting in unison due to their localization within the anti-dots.

In summary, we have used an anti-dot-based MC to experimentally demonstrate MNP detection via MNP-induced changes to spatially confined, high frequency, ferromagnetic resonance modes. MNPs are preferentially captured within the holes which, depending upon a mode's spatial localization, lead to an increased or decreased resonant frequency. The resonance shifts are reproduced well by micromagnetic simulations, observable even for quite low anti-dot fillings [$\sim 12\%$ based on Fig. 4(a)] and are comparable to the resonance linewidths when the proportion of filled anti-dots is high. In this latter situation, a non-dominant linewidth broadening of ~ 1.5 is observable. The shift of the extended mode is approximately 10 times that of the fundamental FMR mode in a continuous film at the highest studied MNP concentration. Our results are also applicable to nano-confined modes in isolated spintronic²¹ or magnonic⁴⁸ nanostructures. This is encouraging for the development of frequency-based spintronic devices such as spin torque oscillators for nano-scale magnetic biosensing²¹ in applications such as flow cytometry.^{32,33} Notably, our observed frequency shifts are significantly larger than measured spin torque oscillator linewidths;^{30,49,50} however, oscillator-based electrical detection will rely on close proximity of the MNP to the sensing layer as well as high GHz/T field sensitivities.

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