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## Magnetometry of single ferromagnetic nanoparticles using magneto-optical indicator films with spatial amplification

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We present a magneto-optical technique to spatially amplify and image fringe fields from single ferromagnetic nanorods. The fringe fields nucleate magnetic domains in a low-coercivity, perpendicularly magnetized indicator film, which are expanded by an applied out-of-plane field from the nanoscale to the microscale for measurement with polar Kerr microscopy. The nucleation location and therefore magnetic orientation of the sample nanorod are detected as spatially dependent field biases in locally measured hysteresis loops of the indicator film. We first discuss our method to fabricate the high-sensitivity indicator film with low energy argon ion irradiation. We then present a map of the amplified signal produced from a single nanorod as measured by the indicator film and compare it with a simultaneously obtained, unamplified fringe field map. The comparison demonstrates the advantage of the amplification mechanism and the capability of the technique to be performed with single-spot magneto-optical Kerr effect magnetometers. Our signal-to-noise ratio determines a minimum measureable particle diameter of tens of nanometers for typical transition metals. We finally use our method to obtain hysteresis loops from multiple nanorods in parallel. Our technique is unperturbed by applied in-plane fields for magnetic manipulation of nanoparticles, is robust against many common noise sources, and is applicable in a variety of test environments. We conclude with a discussion of the future optimization and application of our indicator film technique. © 2015 AIP Publishing LLC. [http://dx.doi.org/10.1063/1.4916205]

Magnetic nanoparticles are increasingly useful for emerging applications in medicine,<sup>1–4</sup> imaging,<sup>5–7</sup> and nanoscale manipulation.<sup>8–10</sup> However, it is difficult to measure the magnetic properties of single nanoparticles, which determine the distribution of properties in ensembles. Bulk measurements are typically complicated by particle interactions and small signal strengths.<sup>11,12</sup> Microscopic techniques such as electron microscopy,<sup>13,14</sup> scanning probe magnetometry,<sup>15</sup> micro-superconducting quantum interference device (SQUID)<sup>16–18</sup> magnetometry, and magneto-optical Kerr effect (MOKE)<sup>19</sup> magnetometry can yield useful information about individual particles, but typically require low temperatures, high vacuum environments, or long measurement times.

In the past few decades, fringe field measurement techniques have been developed in which a sample is placed near a magnetic film with large zero-field susceptibility and a convenient detection mechanism. The magnetic state of this indicator film is used as a proxy for the fringe fields produced by the sample under test. Recent examples of the indicator film technique implement the Hall effect,<sup>20,21</sup> anomalous Hall effect,<sup>22</sup> or giant magnetoresistance<sup>23</sup> as detection mechanisms for single nanoparticle samples. While sensitive, these techniques are optimized for small numbers of particles and must be calibrated for the positions and orientations of the nanoparticles with respect to the nanostructured transducers. More commonly, indicator films have been applied to larger samples using MOKE or Faraday effects as detection schemes.<sup>24–28</sup> These magneto-optical indicator films (MOIFs) have been designed for large optical polarization changes, enabling observation with conventional optical microscopes. However, the thickness of the MOIFs and optical diffraction obstruct local fringe field measurements that enable analysis of single nanoparticles.

Such MOIFs exhibit sensitive hysteresis, which is typically achieved by tuning magnetic anisotropy with film growth conditions. In contrast, a simple ion milling technique<sup>29,30</sup> has been developed for modifying the anisotropy of ultrathin, perpendicularly magnetized multilayers. This technique has been applied for technological devices<sup>31–34</sup> and studies of magnetic phase transitions.<sup>35–37</sup> Despite the ultrathin structure and sensitivity to magnetic fields of these films, they have not been applied as MOIFs.

In this letter, we develop ion-irradiated ultrathin Co/Pt films as MOIFs for single magnetic nanoparticles. Fringe fields from the nanoparticles nucleate nanoscale domains in the indicator films, which are expanded by applied out-of-plane field into microscale domains, spatially amplifying the signal of the fringe fields. We image the amplified domains with polar MOKE microscopy with a charge-coupled device (CCD) video camera, mapping hysteresis loops over the surface of the film, and detecting the bias  $\Delta B_z$  from zero field of the hysteresis loops (Fig. 1(a)). In this figure, hysteresis loops with zero bias are shown in black for reference.  $\Delta B_z$  indicates the domain nucleation position, and therefore the z

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FIG. 1. (a) Experimental concept. A magnetic nanorod (yellow) produces fringe fields that bias the hysteresis loops of the underlying magneto-optical indicator film (MOIF). The bias  $\Delta B_z$  is either positive (red, left inset) or negative (blue, right inset). Hysteresis loops with zero bias are shown in black for reference. The measurement is performed on a polar magneto-optical Kerr effect (MOKE) microscope, with electromagnets in the *z* direction for exciting the film, and *y* direction for switching the magnetization of the nanorod. (b) A nanorod is visible in a representative raw image as an area of bright and dark contrast due to light irregularly reflected from its surface. (c) and (d) Background-subtracted MOKE images of the indicator film with a positive out-of-plane field  $B_z$  applied, showing preferential positive domain nucleation in (c) under the left of nanorod with negative  $B_y$  application, and in (d) under the right of the nanorod with positive  $B_y$  application.

component of the fringe field induced by the nanoparticle. Specifically, we demonstrate fringe field amplification of single ferromagnetic nanorods, compare amplified and unamplified fringe field signals, and measure hysteresis loops of multiple nanorods simultaneously with in-plane magnetic field application.

We prepare the indicator films, which we have previously investigated,<sup>38</sup> by sputtering and subsequent modification by 50 eV argon ion irradiation. As deposited, the films are Pt40 nm/Co0.5 nm/Pt1 nm/SiO2 on an undoped silicon substrate. After deposition, we ion mill the film while moving it beneath a static tantalum edge mask, removing the SiO<sub>2</sub> and producing a shallow gradient of coercive fields. The maximum sensitivity of the films to magnetic field occurs where the zero-moment susceptibility is the largest, which for these films occurs where the coercive field is approximately 1 mT for similar measurement conditions.<sup>38</sup> We perform measurements on an area of the film with this coercive field, which is nearly constant over the field of view. Since the film thickness is only  $\approx 0.5$  nm, we assume that it does not significantly influence the magnetic state of the much larger nanorod samples.

As test samples, we use Ni<sub>90%</sub>/Fe<sub>10%</sub> nanorods fabricated using a template electrodeposition process.<sup>39</sup> Scanning electron microscopy shows that the nanorods are approximately cylindrical, with diameters of 220 nm  $\pm$  40 nm and lengths of 3.9  $\mu$ m  $\pm$  0.3  $\mu$ m (average  $\pm$  standard deviation). We disperse the nanorods in deionized water and deposit a small volume of dilute solution onto the indicator film. We concentrate the nanorods at the most sensitive area of the film using an electromagnet with a sharp iron pole tip positioned beneath the film. We remove this iron tip from the electromagnet after concentration to ensure uniform magnetic fields for the fringe field measurement. To roughly align the nanorods with the axis of the in-plane magnet for measurement, we then apply a large in-plane field and let the aqueous solution evaporate, depositing the nanorods on the surface of the indicator film.

We locate a nanorod, which is visible in our MOKE microscope as an elongated area of bright and dark contrast due to light irregularly reflected from its surface (Fig. 1(b)). We apply an excitation field  $B_z$  in the out-of-plane, z direction to measure hysteresis loops over the entire image frame. We apply  $B_z$  at 250 mHz, which is slow enough to resolve domain wall motion in the indicator film. We concurrently apply a  $-10 \,\mathrm{mT} B_y$  field in the in-plane, y direction, to magnetize the nanorod. We record videos at a frame rate of 40 Hz and store the videos for later analysis. The raw MOKE frames show preferential nucleation of positive (dark) domains on the left side of the nanorod (Fig. 1(c), subtracted from a saturated film background), and preferential nucleation of negative (bright) domains on the right side of the nanorod (not shown). The differences in domain nucleation indicate that the nanorod is magnetized along its long axis, due to shape anisotropy.

To verify that this nucleation difference is indicating the stray fields from the nanorod, we apply a  $+10 \text{ mT } B_y$  field to switch the magnetic configuration of the nanorod. With this in-plane field, we confirm that the nucleation of dark domains now occurs on the right side of the nanorods, demonstrating easy axis switching of the nanorod (Fig. 1(d)).

We then obtain spatially resolved hysteresis loops of the indicator film with  $B_v = 0$ . Our image pixel size is 80 nm, and we average 2 pixel  $\times$  2 pixel areas into bins to reduce measurement uncertainty, as discussed below. These bins are too small to show to scale, and therefore are represented schematically as white squares in the example Kerr frames at the top of Fig. 2(a). A plot of  $M_{z(film)}$  as a function of time for this  $160 \text{ nm} \times 160 \text{ nm}$  bin (Fig. 2(a), middle, gray trace) shows a square wave characteristic of ferromagnetic switching of the film in response to the applied out-of-plane magnetic field  $B_z$  (Fig. 2(a), middle, purple trace). For these measurements, we do not record the phase of  $B_{z}$  but infer it from the phase of the MOKE response and the estimated coercive field of the film. This simplification does not affect the subsequent measurement of  $\Delta B_z$ , as an incorrectly determined  $B_z$  phase would only impose a uniform shift on  $\Delta B_z$ . We obtain hysteresis loops from the MOKE videos by first plotting the MOKE signal as a function of  $B_z$  (Fig. 2(a), bottom, gray trace). We then average the resulting hysteresis loops over many  $B_z$  cycles (Fig. 2(a), bottom, black trace). Despite this averaging, this measurement was performed in only 180 s, which is faster than other techniques for fringe field observation at remanence.<sup>18,40</sup>

We extract  $\Delta B_z$  by fitting the two ferromagnetic transitions composing each hysteresis loop to offset error functions.<sup>38</sup>  $\Delta B_z$  is defined as the average of the two offsets. The error function model is a good approximation for a diffraction-limited image of a sharp domain wall under our Kerr microscope and is imposed by the central limit theorem over multiple  $B_z$  cycles, as performed here. Typical adjusted  $R^2$  values for this fit are >0.99. For unaccelerated domain wall motion, the measured transition fields are unchanged by



FIG. 2. Measurement of fringe fields from single nanorods. (a) Data processing. The film magnetization  $M_{z(film)}$  extracted from binned regions of the MOKE videos (white squares, not to scale, top frames) and plotted as a function of time (top graph, gray line) alternates between up and down in response to the applied field (top graph, purple line). When plotted as a function of field,  $M_{z(film)}$  shows many cycles of a hysteresis loop (bottom graph, gray line) which is averaged to obtain a low noise hysteresis loop (bottom graph, black line). (b) Representative grid of hysteresis loops from which a measurement is obtained. Only 1/16th of the measured hysteresis loops in this area are shown for clarity. Experimental hysteresis loops with a positive field bias  $\Delta B_z$  are colored in red by the amount of bias, and loops with a negative field bias  $\Delta B_z$  are colored in blue by the amount of the bias. A sample nanorod (yellow) is located in the image center and illustrated to scale. An example asymmetric hysteresis loop for direct fringe field measurement is circled in gray. (c) Contour plot of  $\Delta B_z$  measured locally, with contours separated by  $30 \,\mu\text{T}$ . This contour plot informs the estimated orientation of our yellow schematic nanorod in this figure. (d) Nanorod fringe field  $B_{z(fringe)}$  in arbitrary units deduced from differential domain wall velocity as measured by hysteresis loop asymmetry highlighted in (b). The fringe field is just resolvable near the nanorod. The data set for this figure was obtained in <180 s of measurement time.

optical diffraction, allowing  $\Delta B_z$  maps at length scales below the diffraction limit. The bias from accelerated domain wall motion is insignificant, as discussed below.

We show a selection of these hysteresis loops at the spatial location from which they were obtained (Fig. 2(b)). We also plot centered hysteresis loops constructed from the two curve fits, with  $\Delta B_z$  set to zero. The space between the experimental and centered hysteresis loops is colored by the direction of the bias, showing positive (red)  $\Delta B_z$  bias on one side of the nanorod and negative (blue)  $\Delta B_z$  bias on the other. A map of  $\Delta B_z$  shows its microscale extent around the nanorod (Fig. 2(c)). In this figure, the contours are separated by  $30 \,\mu\text{T}$ , and the white area represents  $\Delta B_z = 0$ . This data demonstrates that the technique could be performed with single-spot MOKE magnetometers. Furthermore, the  $\Delta B_z$ measurement allows us to estimate the orientation of the nanorod, which we use to position the yellow nanorod schematic in Fig. 2.

The *z* component of the fringe field of the nanorod  $B_{z(fringe)}$  is not directly measured by  $\Delta B_z$ . Since the indicator film has an intrinsic domain size that is much larger than the nanorod,  $\Delta B_z$  instead reflects the difference in nucleation site and domain wall velocity for the positive  $B_z$  and negative  $B_z$  sweeps, which is then amplified by coherent domain wall expansion. In this way, the nanoscale ferromagnetic domain nucleated by the sample nanorod during each  $B_z$  cycle is

amplified into a microscale reversed domain, which is readily resolved by optical microscopy. This amplification, which is not present in a traditional MOIF, allows measurements at microscale distances away from the nanorod, unperturbed by reflected light. For comparison, we can qualitatively determine the unamplified spatial extent of  $B_{z(fringe)}$  by noting that the domain wall velocity near the nanorod is different for positive  $B_z$  sweeps and negative  $B_z$ sweeps, due to the fringe field of the nanorod. This influence is manifested as an asymmetry in the zero moment susceptibility of the hysteresis curves, as exemplified by the hysteresis loop circled in gray in Fig. 2(b). This asymmetry, measured by the difference in the widths of the error function fits (Fig. 2(d)), is to first order insensitive to residual inhomogeneity of the indicator film, due to the differential nature of the measurement. With this technique, the fringe fields are resolved near the ends of the nanorod with a signal-to-noise ratio of  $\approx$ 5. Micromagnetic factors may reduce the amplitude of the measured asymmetry due to the curved shape of the newly nucleated domains, but this influence must be smaller than the readily observed fringe fields. A comparison of Figs. 2(c) and 2(d) shows that the intrinsic amplification of domain wall expansion in the film strongly increases the detectable signal. Therefore, the spatially amplified  $\Delta B_{z}$ measurement enables a much more sensitive and therefore rapid detection of the magnetic state of the nanorod than a direct, unamplified  $B_{z(fringe)}$  measurement.

The  $\Delta B_z$  measurement contains two types of uncertainty. The lesser uncertainty arises from transduction of the magnetic state of the film to the digital MOKE signal. It is dominated by illumination instability, camera read noise, and biases from accelerating domain walls, as discussed above. Uncertainty from these factors is obtained from the curve fits and determined to be  $<5 \,\mu$ T. The greater uncertainty arises from transduction of the fringe field to the magnetic state of the film. This uncertainty is dominated by pinning of the magnetic domain walls in the indicator film. Since pinning varies spatially (Fig. 2(c)), its influence on the  $\Delta B_z$  maps is systematic and cannot be reduced by signal averaging. By comparing the measured  $\Delta B_z$  map in Fig. 2(c) to one which would be produced by deterministic domain wall motion, we characterize the total uncertainty as  $\approx 15 \,\mu\text{T}$ , although this may be improved<sup>41</sup> by optimizing the  $B_z$  excitation frequency.

This 15  $\mu$ T uncertainty determines a signal-to-noise ratio of  $\approx 60$  for the  $\Delta B_z$  measurement, indicating applicability to smaller particles. In our experiment,  $\Delta B_{\tau}$  is proportional to the difference in propagation time  $\Delta t$  between the two different polarity domain walls from the nucleation site to the measurement site.  $\Delta t$  arises from two additive factors: differences in domain nucleation position and differences in domain wall propagation speed from the fringe fields of the nanorods. The time difference due to differences in nucleation position is proportional to nanorod length, as a shorter nanorod has closer nucleation positions. However, the time difference due to differential domain wall propagation speed does not depend on nanorod length. Since  $\Delta B_z$  is proportional to the sum of these two time differences, we can conservatively estimate the minimum measureable particle length by assuming that the  $\Delta t$  is due *only* to differential



FIG. 3. Simultaneous measurement of hysteresis loops from six nanorods. An in-plane field  $B_v$  is applied along the long axes of the nanorods. (a)–(c)  $\Delta B_z$  maps from a single nanorod under  $-20 \,\mathrm{mT}$ ,  $10 \,\mathrm{mT}$ , and  $20 \,\mathrm{mT} B_v$  field application. Contours are separated by 100  $\mu$ T, and uncertainties are  $\approx$ 40  $\mu$ T for each data point. (b) Clockwise rotation of the nanorod magnetization during switching is apparent. (d)–(i) Hysteresis loops extracted from  $\Delta B_z$ maps, obtained simultaneously through wide-field imaging. The ensemble average of the six hysteresis loops is shown in panel (i) as a black line. Uncertainties are  $\approx 10\%$  of the saturation value of  $M_{v(rod)}$ . All of the data for this figure required  $\approx 200$  s of measurement time.

nucleation. This minimum length is therefore estimated as the length of our nanorods divided by our experimental signal-to-noise ratio, allowing measurement of nanoparticles only 65 nm long. In support of this analysis, we have resolved fringe fields from Fe<sub>2</sub>O<sub>3</sub> nanoparticles with nominal diameters of 100 nm (not shown).

Because the indicator film exhibits perpendicular anisotropy, the applied in-plane fields do not significantly affect its magnetization. We can therefore obtain  $\Delta B_{z}$  maps with inplane field application to measure hysteresis loops of the sample nanorods themselves. To do so, we slowly ramp  $B_{y}$  at 10 mHz between large positive and negative saturation values while cycling  $B_z$  at 500 mHz. For this measurement, we use a lower magnification objective lens to image multiple nanorods simultaneously. These wide-field measurements preclude direct field maps such as measured in Fig. 2(d), which require high-resolution mapping of hysteresis loops. Additionally, since high-precision  $\Delta B_z$  maps as shown in Fig. 2(c) are not required, we assemble each map (Figs. 3(a)-3(c)) from a single cycle of  $B_z$ . This increases the uncertainty of a single measurement to  $\approx 40 \,\mu\text{T}$ . Despite the increased uncertainty, these plots show the magnetic orientation of the nanorods, and reveal, in some cases, the direction of magnetization rotation during switching (Fig. 3(b)). We extract the y component of the nanorod magnetization  $M_{\rm v(rod)}$  by convolving the  $\Delta B_z$  data from an area of the film centered on a nanorod with an idealized dipole kernel. With this technique, we obtain hysteresis loops from six nanorods in a parallel measurement (Figs. 3(e)-3(i)) lasting 200 s, which compares favorably with MOKE magnetometry performed on similarly sized nanorods.<sup>19</sup>

The measurement uncertainty is again dominated here by domain wall pinning, as the error function fit uncertainty is less than the noise present in the data. The pinning uncertainty is estimated from the noise on the saturated areas of the nanorod hysteresis loops, which corresponds to  $\approx 10\%$  of the saturation value of  $M_{y(rod)}$ . This noise value translates to a nanorod coercive field uncertainty of <1 mT. These nanorods have coercive fields that vary by approximately 50%, which is far larger than the measurement uncertainty. This heterogeneity, arising from field misalignment and variation in nanorod properties, would appear in an ensemble average (Fig. 3(i), black line) as significantly different hysteresis behavior, not directly representing the properties of single nanorods. For example, the saturation behavior of the ensemble average differs from the complete switching measured from single nanorods.

In conclusion, we have developed a fast and flexible technique for spatially amplifying and measuring magnetic fringe fields from single nanorods based on domain nucleation and expansion in ultrathin magneto-optical indicator films. We have compared data from this measurement technique to unamplified fringe field maps and used it to obtain hysteresis loops of multiple nanorods in parallel. The ability to measure the in-plane angles between the applied magnetic field, the physical nanorod orientation, and the magnetic nanorod orientation will enable future measurements of magnetic anisotropy and torque magnetometry. Single nanoparticle hysteresis measurements could be performed with a single-spot MOKE magnetometer, by positioning the spot within a few micrometers of the nanoparticle under measurement. Furthermore, since the domain wall pinning energy can be arbitrarily lowered,<sup>38</sup> our technique is additionally applicable over a broad range of temperatures, potentially allowing, for example, cryogenic measurements.<sup>42</sup> Finally, the technique can be implemented with magnetic nanoparticles in solution, allowing the integration of fluidic devices with indicator films for nanoparticle transport. We therefore expect our method to be useful in the future manufacture and application of nanoparticles.

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