Observation of magnetic supercooling of the transition to the vortex state

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Abstract. The magnetic hysteresis of an individual magnetic disc switching in and out of the vortex state has been exhaustively measured using nanomechanical torsional resonator torque magnetometry. Each individual hysteresis loop pinpoints two sharp events, a single vortex creation and an annihilation, with a bias field precision of 0.02 kA m\(^{-1}\). Statistical analysis of thousands of hysteresis loops reveals a dramatic difference in the sensitivity of the vortex creation and annihilation field distributions to the measurement conditions. The data sets measured at different magnetic field sweep rates demonstrate that the transition from the high-field state to the vortex state is not well modeled as a conventional thermal activation process in which it is assumed that the ‘true’ nucleation field is lower than any of the observed switching fields. Instead, the results are suggestive of the classic supercooling signature of a first-order phase transition, or more specifically here, its magnetic equivalent. This phenomenological evidence is consistent with a theoretical picture of the vortex nucleation process as a modified Landau first-order phase transition.

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1. Introduction

There are a wide variety of excellent techniques for the study of nanomagnetism, but generally they all involve some form of averaging. They either measure an array of discs [1], average over multiple runs for adequate signal-to-noise ratio [2], or are stroboscopic [3]–[5]. These types of measurements all have an implicit assumption. The assumption underlying the interpretation of array results has been that each element has a well-defined switching distribution, narrower than that of the entire array, and the measured distribution from the array is inhomogeneously broadened because the individual elements are not identical. More insightful than bulk-averaged array measurements have been microscopy studies (magnetic force microscopy (MFM) [6] and Lorentz transmission electron microscopy (TEM) [7]) in which the switching events of each element are individually recorded during each hysteresis cycle. This has led to the detection of unique behaviors within arrays, and even a correlation of coercivity with crystallite orientation for polycrystalline elements [8]. One motivation for moving beyond these microscopy-based studies is that they are no longer a direct magnetometry and therefore do not report precisely the same physical information as the traditional array experiments. Micro-Hall bar [9] and micromechanical torque magnetometries [10]–[12] are well suited among existing techniques for measuring actual magnetic hysteresis loops of individual nanomagnetic elements. The Hall-based approach is yet to yield sufficiently rapid operation for the acquisition of statistically significant switching data sets from individual elements. However, increased sensitivity from scaling down the dimensions of micromechanical devices enables access to the regime where individual elements can be examined via torque magnetometry, in detail comparable to previous results from entire arrays. The unambiguous result from this work is that individual polycrystalline elements themselves, in effect, manifest an entire ‘array’s worth’ or distribution of hysteresis behavior, sensitively dependent on factors such as in-plane orientation of the applied magnetic field (on an angular scale comparable to the crystalline grain size divided by the lateral dimension of the element) and magnetic field sweep rate. Most surprisingly, in measurements of the high-field state to vortex state transition of a single permalloy disc, field orientations are found for which an anomalously large downward shift of the vortex nucleation field distribution occurs with increasing sweep rate. We present this as phenomenological evidence calling for further development of a recent theoretical description of this state change as a type of first-order phase transition [13]. In contrast, no such behavior is observed for the reverse process, vortex annihilation, in which the sweep rate dependence is well modeled by a model for the thermal activation over an energy barrier [14].

The micro/nano magnetic disc that we study in this paper has a large diameter-to-thickness ratio in zero applied magnetic field and its ground state is that of a magnetic vortex [15]–[17]. The magnetization curls in the plane of the disc and there is a vortex core associated with the singularity of curling magnetization, where the magnetization points out of the plane of the disc. At large applied in-plane magnetic fields, the disc’s energy is minimized by the parallel-spin state, in which the magnetization is aligned with the applied field. At intermediate fields another magnetic state exists, the C-state, which can be thought of as a buckled form of the parallel-spin state. The magnetization curls in the plane of the disc and a virtual vortex core lies outside of the disc [18]. Other buckled states are possible, such as the S-state and onion state, but for simplicity we limit the discussion to the C-state since the exact details of the high-field state are not crucial for the physics that we discuss here, as we explain further below.

In the theory of [13], vortex creation is described by a modified Landau first-order phase transition, where the applied field plays the role typically reserved for temperature [19] and
Figure 1. Scanning electron microscopy (SEM) micrographs of (a) the silicon nitride membrane on the silicon frame, (b) the array of magnetic discs visible through the silicon nitride with the nano-torsional resonators fabricated around the discs (just barely observable in (a)) and (c) the resonator used in this paper (also the lower right resonator in (b)). (d) Finite element model of the primary torsional mode, in which all three paddles oscillate in phase.

there is a critical magnetic field, $H_c$, instead of a critical temperature. As the magnetic field is lowered, $H_c$ is the highest magnetic field that results in vortex creation. The order parameter associated with this phase transition is $\psi = s^{-1}$, where $s$ is the normalized distance between the center of the disc and the ‘rigid’ vortex core [13]. $\psi$ is zero in the parallel-spin state and describes a continuous transition to the C-state and a discontinuous transition to the vortex state. The free energy of the system can be expanded in even powers of $\psi$ and the transition from the parallel-spin state to the C-state is second order, whereas the transition from either the parallel-spin state or the C-state to the vortex state is first order. The broken symmetry associated with the first-order phase transition is the out-of-plane magnetization of the vortex core. The classic signature of a first-order phase transition is supercooling, associated with the nucleation of one state from the other [20]. The amount of supercooling is dependent on the intrinsic energy barrier to nucleation, and extrinsic factors such as the surface roughness [21] and the rate at which the temperature (here the magnetic field) is lowered. Experimental tests of this phase transition theory have not been undertaken until now, because typical experiments on nanoscale magnetic elements involve averages over arrays of elements, or averages over many runs. Ours does not.

2. Experiment and results

Our measurement is based on torque magnetometry, in which a magnetic element with magnetization $M$, in the presence of an applied magnetic field, $H$, experiences a torque, $\tau = V M \times H$. In the geometry shown in figure 1(c), we are sensitive to the magnetization in the $\hat{x}$-direction, the torquing magnetic field is in $\hat{z}$ (out of the plane) and the torsion rod is oriented along $\hat{y}$. The torque is detected as an out-of-plane displacement of the torsion paddle [11]. We apply a constant AC magnetic field, $H_e$, at the resonance frequency of the torsional resonator. The primary torsional mode, as shown in figure 1(d), is at 2.81 MHz and has a mechanical $Q$ of 500. We detect an optical interferometric signal through one of the large paddles at the
resonant frequency, which is proportional to the out-of-plane displacement. This is sensitive \((6 \times 10^7 \mu_B)\) to changes in the magnetization, \(M_x\), of the magnetic element on the torsional resonator, which is varied by controlling the applied magnetic field along \(\hat{x}\), \(H_x\). Further details can be found elsewhere [11].

Device fabrication starts with a commercially available low-stress silicon nitride membrane on a silicon frame (Norcada, Alberta, Canada) (figure 1(a)). We deposit \(42 \pm 2\) nm thick (as measured by calibrated atomic force microscopy) permalloy discs via collimated electron beam deposition at \(10^{-9}\) mbar on the nitride using a stencil mask [22] with \(1 \mu m\) diameter holes and \(12 \mu m\) spacing. A \(2\) nm gold anti-charging layer is sputtered onto the opposite side of the nitride membrane, and the frame is mounted, magnetic element side down, as in figure 1(a). The magnetic discs are visible through the silicon nitride with a scanning electron microscope, accurately aligned to be coincident with a focused-ion-beam mill. This allows fabrication of a torsional resonator around a magnetic disc (figures 1(b) and (c)). The resonator shown in figure 1(c) is used for all the measurements in the present work. After fabrication, the device is mounted \(31 \mu m\) from a silicon wafer, which acts as the back mirror of a low-finesse Fabry–Perot cavity, and the entire structure is mounted directly on a 2D Hall probe \((H_x, H_y)\) in a vacuum chamber [11]. All measurements were performed at room temperature and the laser (HeNe, 632.8 nm, \(3 \mu W\) hitting the resonator) for optical detection was kept at constant power for all measurements.

There are two applied magnetic fields in the experiment. The first is the AC magnetic field at 2.81 MHz along \(\hat{z}\). Increasing this field increases the amplitude of the detected interferometric signal, but has no effect on the statistics of vortex creation and annihilation for our accessible field range. Nonetheless, it is kept constant in all measurements (\(0.81\) kA m\(^{-1}\) peak-to-peak). The second magnetic field is the bias field that is applied along \(\hat{x}\) and varies \(M_x\) in the disc. Sweeps of \(H_x\) are shown in figure 2. At large bias fields, \(M_x\) is saturated in the parallel-spin state. This has been confirmed by micromagnetic simulations and is shown by the red disc in figure 2, although it can be seen that this is not a perfect parallel-spin state. As the bias field is lowered, there is a jump upwards and then a small plateau, which we do not understand at this time, but does not affect in any way our conclusions. Next there is a downward kink and then a sharp drop as the magnetic vortex enters the disc and the magnetization drops abruptly. This kink has been correlated with the buckling of the parallel-spin state to the C-state [23]. The disc is relatively large at \(1 \mu m\) diameter, so it is difficult to state with certainty that this is the parallel-spin to C-state transition, but that does not affect in any way our conclusions. The exact nature of the high-field state is not relevant for our conclusions on the statistics of the vortex nucleation, as long as the high-field state is one with in-plane magnetization. The reason for this is that the magnetic supercooling results from the breaking of a symmetry (the formation of out-of-plane magnetization) upon vortex creation, which is identical for all transitions from states with entirely in-plane magnetization to the vortex state. We can also be confident that our disc is not undergoing a double vortex transition. This state has a very different remnant net magnetization [24], which would show up as a large non-zero signal at zero applied bias field.

At zero field the vortex sits near the center of the disc, which has been confirmed by Lorentz TEM microscopy [25] and micromagnetic simulations, shown as Insets to figure 2. The Lorentz TEM microscopy is unequivocal evidence that the remnant state of our disc is the single vortex state. As the applied field is increased from zero, the vortex is displaced from the center of the disc and is eventually annihilated, which corresponds to a smaller, but also abrupt
Figure 2. Interferometric response at 2.81 MHz versus the applied bias magnetic field $H_x$, resulting in magnetic hysteresis loops. We show two such hysteresis loops, measured starting at saturation and following the dashed arrows, with different creation fields but identical annihilation fields. The inset is a Lorentz micrograph [25] of the disc on the central paddle of the resonator used in these measurements. The white spot in the center of the disc originates from the chirality of the vortex state, confirming the magnetic state of the exact disc used in the torque magnetometry. We also show micromagnetic simulations of the in-plane magnetization. The color code is as follows: red is $\hat{x}$, blue is $-\hat{y}$, green is $-\hat{x}$ and yellow is $\hat{y}$.

change in $M_x$ [15, 16]. All of these features can be seen in figure 2. The vortex creation and annihilation events occur over a small magnetic field interval, $\sim 0.02 \text{ kA m}^{-1}$. Experimental techniques that measure arrays of elements or average over multiple events would observe broadened transitions.

Figure 2 displays two individual hysteresis sweeps with their corresponding vortex creation and annihilation fields. Note that vortex creation occurs at two distinctly different magnetic fields, while the vortex annihilation fields are indistinguishable. Repeated measurements of the events can be performed to learn about the statistics governing them. In figure 3 we show a series of histograms showing the applied fields at which the vortex creation and annihilation events occur. Figures 3(a) and (d) correspond to low sweep rates, figures 3(b) and (e) to intermediate sweep rates and figures 3(c) and (f) to high sweep rates. The sweep rate is field dependent, since we sweep the position of a permanent magnet at a constant rate.

We observe that the vortex creation and annihilation field distributions are bimodal. If during a field sweep down, a vortex is created below $7 \text{ kA m}^{-1}$, then that same vortex annihilates during the subsequent field sweep up at a lower bias field than any vortex created above $7 \text{ kA m}^{-1}$. We are not entirely confident of the origin of this phenomenon, but it is not sweep rate dependent and is not the focus of this paper. We intend to explore this further in the future, but it does not bear on our current conclusions. Instead, we focus on the events (93% of the total number of events we have measured) that correspond to vortex creation above $7 \text{ kA m}^{-1}$ and the main annihilation peak.
It is seen in figure 3 that vortex creation occurs over a broader range of fields than vortex annihilation. In the first-order phase transition model this spread in vortex creation fields occurs because the vortex is energetically favored to be inside the magnetic disc at the highest field at which creation occurs (8.5 kA m\(^{-1}\)). But there is a barrier to nucleating the vortex core with out-of-plane magnetization. This leads to supercooling of the high-field state and the spread of vortex creation fields. It is the formation of the out-of-plane magnetization from a high-field state that is entirely in-plane magnetized that requires this symmetry breaking and results in the supercooling. No such symmetry breaking is required for vortex annihilation, since there exists in-plane magnetic moments in both the vortex state and the high-field state. There is nonetheless a discontinuous transition, because of the finite amount of magnetization associated with the annihilation of the vortex core.

Figures 3(a)–(c) show that vortex creation occurs at lower bias fields for faster bias field sweep rates. This rate dependence demonstrates that vortex nucleation occurs on laboratory time scales (the dynamics associated with the magnetization changes occur on picosecond time scales, but the vortex nucleation itself requires much longer waits). The kink in figure 2 that we associate with the buckling transition from the parallel-spin state to the C-state [23] always occurs at 8.55 ± 0.06 kA m\(^{-1}\); it does not show supercooling or sweep rate dependence. This is as expected from the theory of [13], in which this transition is of second order and should not demonstrate supercooling, although confirmation of the second-order nature of the transition will require improvements in signal-to-noise ratio. In figure 4(a), we plot the mean vortex creation field as a function of sweep rate. Sweep rate dependence is a standard effect in supercooled first-order phase transitions [20, 21]. Figures 3(d)–(f) reveal that there is also a
Figure 4. (a) Mean creation and (b) annihilation fields versus sweep rate. The dashed curves show the sweep rate dependence of the mean of the probability distribution calculated from the Arrhenius relation. We can fit the vortex annihilation data well over the full range of sweep rates. For vortex creation this model does not capture the sweep rate dependence of the mean creation field. In addition, the model does not fit the values for vortex creation at low sweep rates, since the shape of the creation data is not well fitted as is seen in figures 3(a) and (b). (c) TEM image of the magnetic disc on the resonator, with the direction of bias-field rotation. The dark flecks are the remnants of the gold on the opposite side of the nitride from the permalloy disc. (d) Rotation of the bias field can eliminate the supercooling with all else the same (sweep rate 0.36 kA (m s)$^{-1}$): the dark (purple) histogram is 175 vortex creation events at 0$^\circ$ and the light (orange) histogram is 175 events with the bias field rotated by 2$^\circ$. The inset shows a TEM micrograph of the $\sim$5 nm crystalline grains at the edge of the disc. The large dark spots are the gold remnants.

small amount of rate dependence to the vortex annihilation field distribution, which we plot in figure 4(b). The rate dependence of vortex creation is more than an order of magnitude larger than vortex annihilation.

To analyze the sweep rate dependence, we compare our data with an Arrhenius relation for the probability per unit time of nucleating or annihilating a vortex, $dN/dt = f_0 e^{-E(H)/k_B T}$ [26, 27] with a field-dependent energy barrier [14] and thermal activation over the barrier. $f_0$ is an attempt frequency given by the order of magnitude of the frequency of spin waves at the edge of the magnetic disc [28], 10$^9$ Hz for vortex nucleation, and by the gyrotropic frequency for annihilation, 10$^8$ Hz [29]. $E(H)$ is the energy associated with the magnetic volume $V$ involved in the switching event, given by $E = (1 - H/H_a)^{3/2} \mu_0 M_s^2 V/2$. 

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for annihilation and \( E = (H/H_a - 1)^{3/2} \mu_0 M_s^2 V/2 \) for creation \([14]\). \( M_s \) is the saturation magnetization for permalloy (800 kA m\(^{-1}\)) and \( \mu_0 \) is equal to \( 4\pi \times 10^{-7} \) N A\(^{-2}\). There are three possible parameters in the model: \( f_0 \), \( V \) and either \( H_a \) or \( H_s \) for nucleation or annihilation, respectively. We fix \( f_0 \) as the values above (the fit is not very sensitive to \( f_0 \)) and vary \( V \) and \( H_a \) (\( H_s \)) to match the bias field, width and sweep rate dependence of the distributions. Hence, the parameters are overdetermined.

Probability distributions calculated from this model are shown as dashed curves in figure 3. Vortex annihilation is well modeled with \( H_a = 19.4 \) kA m\(^{-1}\) and a volume corresponding to a cylinder of 12.3 nm radius and 42 nm height. This volume is slightly larger than expected, but reasonable, since the expected volume is the volume of the vortex core \([15, 30]\). Our best fit to the creation data is shown in figures 3(a)–(c), with \( H_a = 7 \) kA m\(^{-1}\) and a cylinder of 4.3 nm radius and 42 nm height. We note that in many thousands of runs we have never observed a vortex creation event above 8.5 kA m\(^{-1}\). This, along with the fact that an \( H_a \) greater than 7 kA m\(^{-1}\) is unphysical for this model with respect to the data, places additional constraints on the fit parameters. The thermal nucleation model does not capture the physics of vortex creation, in contrast to the model’s good fit to vortex annihilation. Therefore, that a smaller volume is extracted for vortex creation than annihilation is unimportant. Only the fit parameters, including the volume, for annihilation are considered relevant. We conclude that the critical field for vortex creation is instead 8.5 kA m\(^{-1}\) with corresponding supercooling to lower fields. The fundamental difference between vortex nucleation and annihilation is that there is a broken symmetry, the formation of an out-of-plane magnetization, that must occur to form the vortex state. This is not the case for vortex annihilation, because there is not such a symmetry breaking. This transition is discontinuous since the annihilation of the vortex takes some finite out-of-plane magnetization associated with the vortex core and converts it into in-plane magnetization. But there are already in-plane magnetic moments surrounding the vortex core, so there is no required symmetry breaking and no first-order phase transition. Unfortunately, the modified Landau theory \([13]\) has not yet been developed to compare probability distributions with our results as quantitatively as the thermal model.

Nonetheless, to further test the physical agreement with the theory \([13]\), we rotate the applied bias field by small angles \( \theta \), shown in figure 4(c). Such a rotation changes the location of vortex creation and annihilation along the magnetic disc’s perimeter, which affects the amount of supercooling possible at a given temperature and sweep rate, due to the effective magnetic roughness of the disc’s edge. In figure 4(d), we show two histograms. The purple histogram shows significant supercooling and corresponds to 0°, the same angle as all measurements discussed above. The orange histogram is for the bias field direction rotated by 2° in the plane of the resonator, and swept in magnitude while oriented in this new direction. The supercooling effect is entirely removed and vortex creation becomes narrowly peaked, even at fast sweep rates. Rotation back to 0° consistently recovers the supercooling effect. Moreover, the supercooling persists over angles between 0° and 2° and beyond 2°, although the amount of supercooling is altered on the \( \sim 0.5° \) scale.

2° corresponds to moving the vortex creation site along the disc circumference by \( \sim 17 \) nm, and 0.5° corresponds to moving it by \( \sim 4 \) nm. These numbers are consistent with the relevant length scales in the problem, which are: the diameter of a vortex core, \( \sim 10 \) nm \([15, 30]\), and the permalloy crystalline grain roughness, \( \sim 5 \) nm, as measured by transmission electron microscopy (TEM) (inset to figure 4(d)). Note that there are no significant defects in the disc, as can be seen in a TEM micrograph of the disc (figure 4(c)). It is by tuning the vortex entry
position that we access the regime of sweep-rate-dependent supercooling on time scales that are experimentally obtainable. One or more polycrystalline grains together act as a local magnetic roughness for vortex creation. This has not been previously explored in measurements on single discs that should be sensitive to the local magnetic roughness [9]. Landau–Lifshitz–Gilbert micromagnetic simulations of magnetic discs show that notches and bumps on the perimeter of a disc, along the diameter perpendicular to the applied magnetic field direction, significantly affect the vortex creation field as compared to a disc with a smooth edge. This confirms the crucial role of edge roughness in nucleating a vortex. In essence we have access to a range of physical behaviors in a single magnetic disc. We can tune from magnetically rough to magnetically smooth by small rotations of the applied field, which dramatically alters the transition from the high-field state to the vortex state. In contrast, rotation of the applied field has little effect on vortex annihilation. We have not observed any change in the width or sweep rate dependence of the probability distribution at any accessible angle. We have observed small variations in the absolute value of the mean of the probability distribution, consistent with variations in the local magnetic energy for different polycrystalline grains.

It is interesting to note that not only can one not measure an array of magnetic discs and assume that one is observing all of the physics in the system, but also one cannot even measure a single location in a high-quality magnetic disc and expect to observe all of the physics. We believe that there is an entire realm of physical behavior that has been previously overlooked that is finally going to be accessible because of techniques that allow sensitive measurements of single nanomagnetic objects.

3. Conclusions

In conclusion, we have observed a spreading and lowering of the vortex creation field in response to increasing magnetic field sweep rates. This originates from supercooling of the high-field-to-vortex-state transition, due to a barrier to forming a vortex with out-of-plane magnetization. This is phenomenological evidence that the transition to the vortex state in a single magnetic disc can be described in terms of a modified Landau first-order phase transition [13]. The vortex supercooling in a polycrystalline disc can be eliminated by a small-angle rotation of the applied bias field, which causes vortex creation and annihilation at a different position along the magnetic disc’s perimeter with a different vortex nucleation barrier, associated with different local regions of magnetic crystalline grains. These observations show previously unobserved physics in magnetic nanodiscs [13], and will affect their possible use as information storage media because of the long time scale for vortex core nucleation [27]. Finally, we suggest that future measurements on magnetic discs with very low edge roughness and controlled localized defects for vortex creation and annihilation [31, 32] will shed even more light on this topic.

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