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A THESIS SUBMITTED FOR PARTIAL FULFILLMENT OF THE
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Abstract

The magnetization reversal process in a number of lithographically-patterned NiFe magnetic thin film structures has been investigated, using experimental characterization techniques and micromagnetic simulation. The patterned nanomagnets were fabricated using conventional electron beam lithography technique, as well as unconventional nanolithography techniques. A longitudinal magneto-optical Kerr effect magnetometry (MOKE) was constructed during the course of my PhD study and it was used to probe the magnetic hysteresis behaviour of the nanomagnet arrays. The spin configurations of the nanomagnets were imaged by using magnetic force microscope with in situ magnetic fields.

A technique to trap domain walls at exact position in sub-micron asymmetric magnetic ring was demonstrated. Detailed studies on the domain wall trapping behavior were performed both numerically and experimentally. When the film thickness was controlled to a range of 8 ~ 24 nm, a 360° domain wall was trapped at the narrow arm of the asymmetric ring as the ring adopted a magnetic vortex configuration. The trapped domain wall stability was strongly dependent on the vortex chirality of the asymmetric ring.

The magnetization reversal process in sub-100 nm asymmetric rings was found to be dependence on the applied field orientation. The results were carried out via MOKE measurement, MFM scanning and were confirmed by micromagnetic simulations. A highly stable and chirality controllable flux-closure state was formed when the reversal field was applied along the asymmetric axis of the asymmetric ring. The flux-closure state was considered highly stable as the measured coercivity was relatively large, *i.e.* 1.2 kOe, which may have high potential for patterned media

application. The flux-closure state was persistently stable even when the diameter of the ring was reduced to as small as 30 nm.

Using an unconventional nanosphere lithography technique an array of sub-100 nm triangles was successfully fabricated. Micromagnetic simulation showed that the magnetization reversal mechanism of 45 and 80 nm nanotriangles was dominated by coherent rotation process, rather than the usual domain wall nucleation and annihilation process. Moreover, in 100 nm nanotriangle the magnetisation reversal was completed via the formation of a V-like intermediate state, resulting a double-switched behavior in the hysteresis loop. All the simulated reversal behavior can be accounted by the measured MOKE hysteresis loops.

Fe_3O_4 thin film was grown on thermally oxidized Si substrate at different growth temperatures. The film was prepared using DC reactive sputtering technique on a Fe target in $\text{Ar}+\text{O}_2$ environment. Fe_3O_4 was a high spin polarization material that has high potential for spin injection application. The presence of the Verwey transition in our measurement indicated that an ordered crystallographic structure was effectively formed in the Fe_3O_4 thin film.

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Publication list

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4. **X. H. Wang**, S. Goolaup, W. S. Lew, "Highly stable flux-closure state in sub-100 nm nanomagnets", *Applied Physics Express* 5 (2012) 053001.
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6. **X.H. Wang**, S. Goolaup, W. Z. Wang and W. S. Lew, "High stability flux-closure in sub 100 nm asymmetric rings", Oral presentation at 11th MMM-Intermag Joint Conference

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List of symbols and abbreviations

2-D	Two Dimentional
3-D	Three Dimentional
AFM	Atomic force microscope
AMR	Anisotropic magnetoresistance
Au	Gold
ASR	Asymmetric ring
AC	Alternating Current
Al	Aluminum
Ar	Argon
BNC	Bayonet Neill Concelman
Co	Cobalt
Cr	Chromium
Cu	Copper
CHCl ₃	Chloroform
CoFeB	Cobalt Iron Boron
DC	Direct current
DW	Domain wall
EBL	Electron beam lithography
Fe	Iron
Fe ₃ O ₄	Magnetite
Fe ₂ O ₃	Hematite
GMR	Giant magneto resistance
HeNe	Helium neon
HDD	Hard disk drive
LED	Light emitting diode
IPA	isopropanol
MRAM	Magnetic Random Access Memory
MR	Magnetiresistance

MOKE	Magneto Optical Kerr Effect
MFM	Magnetic force microscopy
MIBK	Methyl isobutyl ketone
NA	Numerical aperture
Ni	Nickel
NiFe	Permalloy
N ₂	Nitrogen
NSL	Nanosphere lithography
O ₂	Oxygen
OOMMF	Object Oriented MicroMagnetic Framework
PPMS	Physical Property Measurement System
PMMA	Para-Methoxymethamphetamine
PS	Polystyrene
RT	Room temperature
RF	Radio frequency
SEM	Scanning electron microscope
SR	Symmetric ring
Si	Silicon
SPM	Scanning probe microscopy
VSM	Vibrating sample magnetometer
XRL	X-ray lithography
XRD	X-ray diffraction
XPS	X-ray Photoelectron Spectroscopy

Chapter 1

Introduction

1.1 Why patterned magnetic thin film nanomagnets?

In recent decades, patterned magnetic thin film nanomagnets^{1,2,3} (artificial structures with dimensions on the nanometer scale) created by lithographic techniques or by advanced growth technique such as self organization are now attracting technological and scientific interest. In another aspect, due to the development of the processing power of computers, the magnetics of the nanomagnets are calculable, making it possible for the comparison with experimental observations. The study of the patterned magnetic thin film nanomagnets holds interest in both fundamental and application perspective.

1.1.1. Fundamental perspective

From the fundamental perspective, when the lateral sizes of the magnets are made comparable or smaller than certain characteristic lengths of a material, new properties are expected. These certain lengths are namely carrier mean free path (e.g. CoFeB at 4.2K, $l=70 \text{ nm}^4$), spin diffusion length (e.g. Ni at 4.2K, $l=21 \text{ nm}^5$), and domain wall length (e.g. for Fe, $l_d=39.5 \text{ nm}^6$). In bulk material, the magnetization states are usually divided into domains and the exact configuration is unpredictable. While the magnetic material is patterned into nanostructures, the size, number and orientation of the domains become predictable. According to Brown's fundamental theory, the competition of the magnetic energy items, such as exchange, magnetostatic, and anisotropy energy, suppresses the domain formation in very small

particles, leading the nanomagnets behaving as a single giant spin⁷. The lateral confinements of the magnets lead to both novel transport and new magnetization reversal mechanisms in the structures.

Compared with conventional magnetic material whose anisotropy arises from the configuration of the electronic Fermi surface, the anisotropy in the magnetic nanopatterns not only depends on the band structure of the material but also the shape of the nanomagnets. By designing the unique shape and the size of the nanomagnets, the anisotropy can be engineered. Moreover, the interaction between the elements may also induce new collective magnetic properties. As a result, the ordered nanomagnets array is different from the isolated elements^{8,9,10-11,12}. And the nanomagnet is well suited for testing micromagnetics and exploring new physics.

1.1.2. Applications

From an application perspective, nanomagnets are the basic elements for the future spintronic devices. As data densities increase, the characteristic dimensions for magnetic bits written in computer hard discs are already measured in nm scale. With the increasing in demand for storage capacity, the conventional recording medias are approaching their maximum recording density due to the onset of the superparamagnetic effect^{13,14}, which is defined as when the time used to measure the magnetization of the nanoparticles is much longer than the Néel relaxation time, leading magnetization appears to be in average zero, the state is in superparamagnetic state. Developing magnetic sensors for future ultra high density^{15,16,17,18} magnetic storage media and non-volatile random access memory has attracted a lot of research interest now. Therefore, the magnetic properties of sub-micron or nano structures needs to be both controlled and understood. The dynamic magnetic reversal process^{19,20} is dramatically changed as the characteristic dimensions approach the superparamagnetic stability limit (corresponding to mean bit dimensions in magnetic media of around 50-100 nm). Therefore developing nanostructured magnetic

materials whose dimension is in the nm range, whereby the shape, thickness and magnetic properties are controlled is of vital importance in order to create ultra small magnetic structures which remain stable.

A lot of research interests are also attracted to the spin dependent transport properties of nanomagnets due to the novel idea of using the ‘spin’ angular momentum^{21,22,23} of electron to operate future devices^{24,25,26,27,28}. One of the devices exploiting the ‘spin’ of electron is Magnetic Random Access Memory (MRAM). The switching fields of the two magnetic layers comprising the nanomagnets of the MRAM are different. By altering the spin of localized electrons on one of the magnetic layers using spin transfer, the changes between the “1” and “0” states are accomplished. By measuring the resistance of the elements, the states can be sensed. MRAM also offers promise for smaller, faster, cheaper, and less power consuming memories^{29,30,31}.

Recently, it has been postulated that the scope of spintronics could be greatly expanded if the domain walls and domain wall movement can be utilized in the data storage as well as in computation^{32,33}, resulting a completely magnetic computer^{34,35}. A racetrack memory³⁶ comprising an array of magnetic nanowires arranged horizontally or vertically on a silicon which controls the movement of domain walls in magnetic nanowires by short pulses of spin-polarized current has been proposed. A processing mechanism based on magnetism, in which networks of interacting mesomagnets have been used to perform logic operations and propagate information also has been reported. These kinds of systems have potentials to offer integration in density and reduction in power dissipation as compared to the current recording and micro-electronic technology.

1.2 Challenges in patterned magnetic thin film nanomagnets

The last decade has seen tremendous progress in our understanding of magnetism at the meso and nanoscale, motivated by the search for new physical

phenomena and by the potential for applications. For submicron meter and nanometer elements, the accessible magnetic configurations are mainly determined by the magnetostatic energy, which is intimately linked to the global shape of the element. The application of such nanomagnets as device elements in magnetic recording media and random access memories requires the perfect controllability of the magnetization at room temperature. For this reason, methods to achieve the required control over the magnetization in various magnetic thin film patterns. The most widely studied shape of nanomagnet to date has been rectangular, due largely to the applicability of such structures to MRAM and spin-valve magnetic field sensors³⁷. A number of experimental^{38,39,40} and theoretical^{41,42} studies have been conducted. Several other studies were carried out at square nanoelements^{43 44 45}. Ferromagnetic nanoelements developing magnetic vortex structures are attracting a great deal of interest for their potential application in high density data storage technology and because of the underlying physics governing the vortex formation/annihilation process, the vortex core displacement under the action of an external field, and its dynamic behavior^{46 47 48}. Despite the disks, the ring geometry has attracted much attention by virtue of the fact that it exhibits simple and highly symmetric equilibrium states at remanence, the vortex and the ‘onion’ states, both existing with mirror equivalents⁴⁹: the vortex state corresponding to a state of circular magnetization (such as that found in disc elements but without the energetically costly vortex core) and the onion state corresponding to the state closest to saturation that is compatible with the topology of the element, i.e., with two halves of the ring magnetization each adopting opposite circulations, each half separated by 180° domain walls^{50 51}. A small number of studies exploited the shape anisotropy such as rings with notches and elliptically shaped ring structures.

The magnetic patterns described above have limitations of “writing” the desired magnetization circulation in the element. There is need to explore new structures to control the magnetization circulation. In this thesis, by shifting the inner circle of a symmetric ring (SR), asymmetric ring (ASR) structure was obtained. The ASR is unique, since it retains most of the properties of the SR. There is less possibility inducing fringing field. The asymmetry ratio is controllable by changing the distance

between the two centers. Thus the ASR is promising structure for carrying out systematic studies on magnetic properties of nanomagnets.

In the other new application, which aims at combining the electron charge degree of freedom with that of the electron spin, magnetoelectronics or spintronics, good performance is expected to partially depend on the ratio of spin injection. Thus, material with high- spin polarization is needed in the spin injection process. In this thesis, the half metallic material Fe_3O_4 was studied. Due to the completely spin-polarized conduction band, Fe_3O_4 ferromagnets are promising spin injectors.

1.3 Scope of this Thesis

This thesis carried out a comprehensive investigation on the effect of various patterns and geometrical parameters on the magnetization reversal process of a soft magnetic thin film (NiFe). Ordered homogeneous nanomagnets were fabricated using electron beam lithography technique as well as nanolithography technique. The effects from geometrical parameters such as film thickness, lateral dimensions as well as asymmetric ratios were systematically studied. Magnetic properties of the nanomagnet arrays were obtained using a home-built longitudinal Magneto Optical Kerr Effect magnetometry (MOKE). The spin configurations of the nanomagnets were imaged using magnetic force microscope (MFM) with magnetic field in situ. In the initial part of this thesis, domain wall behaviors in the sub-micron sized asymmetric ring have been investigated as they hold potential applications in the domain-wall recording and domain wall logic devices. From the ultrahigh density recording media application perspective, the magnetic properties in sub-100 nm magnets were also investigated. Finally fabrication process of the potential spin injection material, Fe_3O_4 , using reactive sputtering technique was explored.

The main objectives of this thesis are listed as follows:

- (a) Mapping spin configuration and switching process of asymmetric nano-ring structure with the inner circle purposely off-centered.
- (b) Investigating of the magnetic reversal process of nanomagnets with the lateral size going close to supermagnetic limit.

- (c) Exploring the fabrication process of Fe_3O_4 , which is a promising spin injector conquers the conductance mismatch.

1.4 Organization of this thesis

In chapter 2, the fundamentals in thin film magnetism was presented to aid the understanding of the experimental work in this thesis. Chapter 3 describes the fabrication and characterization techniques that were used in this work. In chapter 4, magnetization reversal process in submicron NiFe ASR was studied. In chapter 5, the effect of the magnetic field orientation on the magnetisation switching mechanism was studied. The vortex state was found to be able to sustain in ASR with lateral size as small as 30 nm. Magnetisation reversal in nanotriangles fabricated by nanosphere lithography was studied in chapter 6. Structural, Magnetic and electrical Properties of Reactive-sputtered Fe_3O_4 Thin Film was studied in chapter 7. In the final chapter, a summary of the major observations and results were given. Possible extensions on the current work are suggested.

Chapter 2

Theoretical background

2.1 Magnetic materials

In magnetic materials, the magnetic field produces magnetization M in magnetic materials. The magnitude, the sign and the response of M to an applied field H are used to characterize the magnetic properties of a certain material. The ratio of M to H is called the susceptibility χ : Since M is the magnetic moment per unit volume, χ also refers to unit volume and is sometimes called the volume susceptibility.

The flux density B , whose change generate voltage following Faraday's law is another useful quantity in the magnetic materials. In ferro- and ferrimagnetic materials the ratio of B to H is called the permeability μ . μ is the slope of a line from the origin to a particular point on the curve. The initial permeability μ_0 and the maximum permeability $\mu_{\max}$ are often quoted. If not specified, permeability μ is taken to be the maximum permeability $\mu_{\max}$. The local slope of the B , H curve dB/dH is called the differential permeability. The magnetic behavior of various kinds of substances is thus characterized by their corresponding values of χ and μ :

1. Empty space; $\chi = 0$, since there is no matter to magnetize, and $\mu = 1$.
2. Diamagnetic; χ is small and negative, and μ slightly less than 1.
3. Para- and antiferromagnetic; χ is small and positive, and μ slightly greater than 1.
4. Ferro- and ferrimagnetic; χ and μ are large and positive, and are functions of H .

The permeability of air is about 1.000,000,37. The difference between this and the permeability of empty space is negligible, relative to the permeabilities of ferro- and ferrimagnetics, which typically have values of μ of several hundreds or thousands. We can therefore deal with these substances in air as though they existed in a vacuum. In particular, we can say that B equals H in air, with negligible error

2.2 Magnetic thin film

The early research on magnetic thin films was done due to their promising application as memory elements in digital computers. This was followed by the intense development of bubble domain memories, which required the use of patterned

magnetic thin films to control the motion of the bubbles. Afterwards other researches related to various aspects of computer technology include the original idea of magnetic memory have arise. Of all the magnetic materials, the 80 permalloy (80 wt% Ni, 20% Fe) has low anisotropy and low magnetostriction combined with reasonably high magnetization. Thin films are deposited onto a substrate by different techniques, such as chemical vapor deposition, electrodeposition from a liquid electrolyte, thermal evaporation (using one of several different heat sources), and sputtering. The surface condition and temperature of the substrate during the deposition of the film are variables to be controlled. Of all the techniques, a physical technique sputtering is the most widely used, because it permits almost any combination of elements to be deposited. Thin film production can be done in a highly reproducible way by sputtering.

The structure of thin films is complex and hard to characterize. In the thin film the grain size is of the order of $1000\text{\AA}$ or less and is about 1/100th the grain size of most bulk materials. Since the grain size is comparable to the domain wall thickness, domain walls may extend through many grains and lose their crystallographic identity. Preferred orientation (texture) may or may not be present, depending on deposition conditions and nature of the substrate. Directional structures can be produced in a film by elegant chosen deposition conditions in which the direction of the incoming atoms makes an angle with respect to the film plane. There is also stress in the film which is induced by the difference in thermal expansion coefficients between film and substrate. The film itself may also has micro stress due to various imperfections. By choosing a film composition with magnetostriction as close as possible to zero, the effect of these stresses is minimized.

2.3 Magnetic energies⁵²

The total energy^{53,54,55,56} E_{total} of a ferromagnet typically comprises of the magnetostatic E_s , the exchange E_{ex} , the anisotropy E_a , and the Zeeman energies E_z .

$$E_{total}=E_s+E_{ex}+E_a+E_z$$

57

The magnetostatic energy, which is long range magnetic interaction, is zero in the case when the magnetic film has infinite lateral extension and is uniformly magnetized in the film plane. In fact, the film size is finite where the surface charging has to be taken into account leading to a demagnetizing field. Thus domains with different orientations of magnetization are formed in order to reduce the magnetostatic energy. The boundary between these domains is magnetic domain wall. Inside the domain wall, the spins rotate gradually, leading to a certain width of the wall, which is determined by the competition between two energy terms, the exchange energy and the anisotropy energy. The exchange energy between neighboring spins tends to increase the wall width. However, a wider wall induces higher anisotropy energy, since inside the wall the direction of spins is away from the easy axis of magnetization.

2.3.1 Magnetostatic Energy

The magnetostatic energy or magnetic dipole energy originates from the classic interaction of atomic magnetic dipoles and arises from alignment of atomic magnetic dipoles. Thus the magnetostatic energy depends on the magnetization M , the magnetic-dipole moment per volume. To calculate the magnetostatic energy, the magnetic dipoles in the film, M , create the stray field, H_{stray} . However, due to the surface charging, inside the film, there exists the field which has the same amplitude as H_{stray} but directs opposite to M , the so called demagnetizing field, H_d . So the magnetostatic energy, which has the same amplitude to the demagnetizing energy in this case, will be described with M and H_d :

$$E_M = -\mu_0 \int_V M \cdot H_d dV$$

58

Where M denotes the local magnetization and H_d is the magnetizing field generated by the samples and μ_0 is the initial permeability. The effect of magnetostatic energy is to minimize the surface charge which is a consequence of energy minimization. If

the decrease in magnetostatic energy is greater than the energy needed to form magnetic domain walls, then multi-domain states will arise, as illustrated in Fig.2.1

Fig.2.1 Illustration of the formation of flux-closure in a magnetic bar to reduce the high magnetostatic energy due to surface charge at the edge

The demagnetizing field H_d of a body is proportional to the magnetization which creates it and can be expressed as:

$$H_d = -N_d M \quad \{2.3\}^{59}$$

where N_d^{59} is the demagnetizing factor or demagnetizing coefficient. The value of N_d depends mainly on the shape of the body, and has a single calculable value only for an ellipsoid⁶⁰ since the induction uniformity can be achieved only in an ellipsoid as illustrated in Fig.2.2.

Fig.2.2 Illustration of magnetic induction in an ellipsoid

The sum of the demagnetizing factors along the three orthogonal axes of an ellipsoid is a constant

$$N_a + N_b + N_c = 4\pi \text{ (cgs)}^{59} \quad \{2.4\}$$

$$N_a + N_b + N_c = 1 \text{ (SI)}$$

61

For a sphere, the three demagnetizing factors are equal, thus,

$$N_{sphere}^{62} = \frac{4}{3}\pi(cgs) \text{ or } N_{phere}^{62} = \frac{1}{3} \text{ (SI)}$$

And the magnetostatic energy can be written as a function of the magnetization direction in a prolate ellipsoid as shown in Fig 2.3

Fig.2.3 Uniformly magnetized prolate ellipsoid with the magnetization at an angle α in from the semi-major axis.

$$E_M = \frac{V}{2}(N_a - N_c)M_s^2 \sin^2 \alpha \quad 4$$

Where N_a and N_c are the demagnetizing factors along the semi-minor and semi-major axis, M_s is the saturation magnetization and α is angle between the magnetization direction and the semi-major axis and V is the volume of the ellipsoid.

However, most of the samples are not ellipsoids. The demagnetizing factors calculated according to the previous formulas will be in error to some degree. The demagnetizing field in rod, disks or rings^{63,64,65,66,67} varies from one point to another in the specimen and so has no single value. Values of the demagnetizing factor depend primarily not only on the geometry of the sample, but also on the permeability or susceptibility of the material. A table and graphs of demagnetizing factors for prolate and oblate (planetary) spheroids have been given out by Bozorth [R. M. Bozorth, Ferromagnetism, Van Nostrand (1951); reprinted IEEE Press (1993)]. Here the graph is reprinted and given out in Fig. 2.4 and Fig.2.5

Fig.2.4 Demagnetizing factors for prolate spheroids

Fig. 2.5 Demagnetizing factors for oblate (planetary) spheroids

2.3.2 Exchange Energy

The exchange interaction is a quantum effect which occurs due to the overlapping of electron wave functions⁶⁸. It is a manifestation of the Coulomb interaction⁶⁹ between electron charges and the Pauli principle, which causes the

cooperative magnetic ordering. The exchange interaction energy, E_{ex} , between two spins, $\vec{S}_i$ and $\vec{S}_j$, scales with the exchange integral,

$$E_{ex} = -J_{ij}\vec{S}_i \cdot \vec{S}_j \quad \{2.7\}^{70}$$

Where $\vec{S}_i$ and $\vec{S}_j$ are the unit vectors of interacting spins on two atoms and J_{ij} the exchange integral. For ferromagnetic material, the exchange interaction is short-range and tends to align the neighboring spins in parallel direction. To estimate the exchange energy, it is convenient to take all spins together, i.e., a continuous model of spin rotation. By doing so, the total E_{ex} can be expressed as:

$$E_{ex} = A\left(\frac{d\theta}{dx}\right)^2 \quad \{2.8\}^{70}$$

where

$$A = \frac{a^2 s^2 L J N_V}{2} \quad \{2.9\}^{70}$$

and L is the exchange stiffness constant (J/m), s is the spin quantum number ($= 1/2$), and a is the lattice constant. N_V indicates the number of nearest neighbor atoms per unit volume. θ is the angle with respect to the easy axis of magnetization. From the equations above, the exchange energy is minimum for parallel alignment of spins and maximum for anti-parallel alignment.

2.3.3 Anisotropy energy

The anisotropy, which is called magnetic anisotropy energy results from spin-orbit interaction and it depends on the orientation of the magnetization with respect to the crystallographic axes of the material. In ferromagnets with uniaxial anisotropy, the anisotropy energy density is given by:

$$E_a = K \sin^2 \theta \quad \{2.10\}^{71}$$

where K is the anisotropy constant and θ is the angle between the magnetization and the easy axis direction. In the case of polycrystalline materials such as thin films evaporated and sputtered in this thesis, the grains' easy axis are oriented randomly

and thus the material shows isotropic property in terms of magnetocrystalline energy. For the soft magnetic material used in this thesis, which is permalloy, the anisotropy energy is quite small which can be neglected compared to the other energies. However, an intrinsic uniaxial anisotropy⁷² may still be present in the polycrystalline ferromagnetic films.

2.3.3.1 Magnetic crystalline Anisotropy

Crystal anisotropy is mainly due to spin-orbit coupling of the spin orientation to the crystallographic lattice and is brought about by a change in the overlap of electron wave functions. On applying an external field, the orientations of the orbits are fixed very strongly to the lattice, while the spins change directions. This causes the overlap of the electron wave functions of neighboring atoms to change, and hence the Coulomb interaction⁷³ also changes. As such, the anisotropy symmetry reflects the lattice symmetry. To describe it phenomenologically, the symmetry of the anisotropy is defined, which most commonly are uniaxial and cubic. For a single domain state, the dependence of the energy density on spin orientation for the uniaxial case can be written as

$$E_{uniaxial}=K_u \sin^2 \theta \quad \{2.11\}^{70}$$

where K_u is the uniaxial anisotropy constant and θ is the spin orientation with respect to the symmetry direction. For the cubic case, the energy density follows

$$E_{cubic}=\frac{K_1}{4} \sin^2 \theta \quad \{2.12\}^{70}$$

where K_l is the cubic anisotropy constant. Since the zero level of energy density is not important, the $+\sin^2$ function in the previous two equations can be replaced by

$-\cos^2$. Figure 2.6 shows the polar plotted form of the equation equations {2.11} and {2.12}.

Fig.2.6 The dependence of energy density on spin orientation shown in polar form for uniaxial anisotropy (left), cubic anisotropy (right) and a mixture of $K_u/K_l=0.2$ (center)

Spin directions corresponding energy maxima are referred as hard axis while the energy minima gives easy axis. Due to the energy minimum principle, the spins will align spontaneously to the easy axis under zero applied field. Also given out in the lower panel of Fig.2.6 is the energy profile of a system comprising a mixture of uniaxial and cubic anisotropies. The plot is a linear combination of uniaxial and cubic profile for $K_u/K_l=0.2$. The easy and hard axes of the mixed anisotropy give an easy-easy axis and a hard easy- axis.

2.3.3.2 Shape Anisotropy

Shape anisotropy is originated from magnetostatic energy. The total magnetostatic energy can be calculated by summing the potential energy of each

magnetic dipole in the magnetic field induced by the other dipoles. Another way to estimate the energy is to assume that the magnetic dipoles in the film, M , create the stray field, H_{stray} . And the energy density stored in the stray magnetic field comes from the boundary of the sample. A third way is to imaging due to the surface charging, inside the film, there exists a demagnetizing field of magnitude $4\pi M_s N$ which is opposite to M . N is the demagnetizing factor and is determined by the shape of the sample. Consider a polycrystalline specimen having no preferred orientation of its grains, the magnetization will be influenced by the physical dimensions of the sample and hence the potential energy summation. A full treatment is given by Bleaney⁷⁴ and Chikasumi⁷⁵.

The magnetostatic energy is minimized when all the spins are lying in the plane in the case of a thin film. From Fig.2.7 one can see any component of out of plane magnetization will lead to surface charges on the top and bottom large surfaces. And the energy is much higher than the case that the magnetization are all aligning in plane, where the surface charging only exists across the thin edges. The demagnetizing factor is 1 for an infinite thin slab with out of plane magnetization and is 0 for in-plane magnetization. The shape anisotropy field due to an ultrathin film is $4\pi M_s$.

Fig.2.7 A diagram showing the origin of the strong shape anisotropy in thin films

2.3.4 Zeeman energy

Zeeman energy is the interaction energy of the magnetization vector field with an external magnetic field $\vec{H}_{ext}$. The Zeeman energy is given by:

$$E_Z = -\mu_0 \int_V \vec{M} \cdot \vec{H}_{ext} dV \quad 76$$

where $\vec{M}$ denotes the local magnetization. From the equation, we can calculate that the Zeeman energy is minimized when the magnetization is aligned along the external field direction.

2.4 Magnetic Domains and Domain walls

Stable magnetic configurations in a ferromagnet are determined by the minimization of the total energy. Magnetic domains are generated in order to reduce the total

energy in the ferromagnetic materials, and the size of the domain is defined by energy minimization^{77,78}. The boundary between different domains are the domain walls^{64,79}.

2.4.1 Domain structures in uniaxial crystals

For a large single crystal with uniaxial anisotropy, suppose it adopts a single domain, as shown in Fig. 2.8A, the free poles formed at the ends generate a large field. The magnetostatic energy of this crystal in this case⁸⁰ is:

$$\frac{1}{8}\pi \int H^2 dV \text{ (cgs)} \quad \{2.14\}^6$$

$$\frac{1}{2} \int H^2 dV (SI) \quad \{2.15\}^6$$

This considerable energy can be reduced by almost a factor of 2 if the crystal splits into two domains magnetized in opposite directions as shown in Fig. 2.8B. Since splitting the single domain into two brings north and south poles closer to one another, thus the spatial extent of the H field is decreased. If the crystal further splits into four domains as in Fig. 2.8C, the magnetostatic energy again decreases, and so on. But the division into smaller and smaller domains cannot continue indefinitely. Each division forms extra domain walls in the crystal and the exchange energy increases, which adds energy to the system. Eventually equilibrium between the decrease in magnetostatic energy and the increase in exchange energy will be reached so as the domain size.

Fig.2.8 The origin of domains. (A) Magnetic flux on the exterior of a single-domain structure. (B) and (C) Reduction of magnetostatic energy by dividing into domains. (D) and (E) Ideal domain arrangement resulting in zero magnetostatic energy due to flux closure within the structure

The critical size⁵⁶ of the single domain is calculated by comparing the energy between the single domain configuration and other configurations. A prolate ellipsoids which is thin compared to the wall thickness, is chosen as a model to simplify the calculations. The flux-closure configuration as shown in Fig. 2.9 was assumed in this ellipsoid with size increasing.

Fig.2.9 Flux-closure configuration assumed in this ellipsoid with size increasing

In this configuration, the exchange energy is represented by

$$E_{ex} = A \sum_i (\nabla \alpha_i)^2 \quad \{2.16\}^6$$

Substituting the direction cosines,

$\alpha_x = \sin\omega\cos\phi, \alpha_y = \sin\omega\sin\phi, \alpha_z = \cos\omega$ in equation {2.16}, one gets

$$E_{ex} = A\left[\left(\frac{d\omega}{dr}\right)^2 + \left(\frac{1}{r^2}\right)\sin^2\omega\right] \quad 57,81$$

Taking $\omega=\pi/2$, the exchange energy density would be $E_{ex} = A/r^2$, and the exchange energy ε_{ex} of the ellipsoid per unit volume is

$$(4/3)\pi R^2 b \varepsilon_{ex} = \int_{\alpha/2}^R \int_0^{b[1-(\frac{r}{R})^2]^{\frac{1}{2}}} 2\pi r^{-1} dz dr = 4\pi Ab \left[\ln\left(\frac{4R}{a}\right) - 1 \right] \quad \{2.18\}^6$$

Where $2R$ and $2b$ are the short axis and the long axis of the ellipsoid, respectively.

The magnetostatic energy is

$$\varepsilon_m = \frac{1}{2} N_d M_s^2 \quad \{2.19\}^6$$

Where N_d is the demagnetizing coefficient along the polar axis. Thus compare the equations of⁸² and {1.19} yields the critical size R_c :

$$\left(\frac{N_d}{6A}\right) M_s^2 R_c^2 = \ln\left(\frac{4R_c}{a}\right) - 1 \quad \{2.20\}^6$$

The domain structures in cubic crystals are much complicated because there are three or four easy axes, depending on the sign of the anisotropy constant K_1 . Related works can be find elsewhere^{83,84}.

2.4.2 Neel walls and Bloch walls

In magnetism, a domain wall is a separation region between magnetic domains with opposite magnetization. It is a transition between different magnetic moments and normally angular displacement between the magnetic moments is 90° or 180° . Inside a domain wall, individual moment undergoes a gradual reorientation across a finite distance. The domain wall thickness is dependent on the anisotropy of the material, but on average spans across around 100–150 atoms.”

The width of the domain wall is dominantly determined by the competition between the two energies that create it: the Magnetocrystalline anisotropy energy and the exchange energy (J_{ex}). In order to become a more favorable energy state, both energies tend to be as low as possible. The presence of anisotropy energy tries to reduce the width of the domain wall, since only when the individual magnetic moments are

aligned to the crystalline axis direction, the lowest anisotropy energy state is obtained. Whereas the lowest exchange energy state is when the magnetic moments are aligned parallel to each other. Thus due to the repulsion between the magnetic moments, the exchange energy tries to make the wall thicker.

There are two main types of spin structures inside the domain walls, Bloch⁸⁵ and Neel⁸⁶ types. In the Bloch walls, spins rotate in the plane parallel to the wall plane. The wall width of a 180° Bloch wall is most commonly determined by $\sqrt{A/K}$, where A and K are the exchange constant and anisotropy energy, respectively. The Bloch wall is usually favored in bulk materials. In thin films, a Bloch wall induces surface charges by its stray field, thus is energetically unfavorable. The Neel wall in which spins rotate in the film plane as shown in Fig.2.10, becomes preferable. The Neel wall has a narrow core and μm -long tails on both sides. The core width is of the order of the exchange length. The critical thickness for the transition between Neel wall and Bloch wall is found to be 1 μm .

Fig. 2.10 Illustration of the structure of Neel Wall and Bloch Wall.

Domain walls are also indicated with an angle representing the magnetization rotates through the wall from one domain to the other. The two kind domain walls of high interest are the 180° Néel wall in the transverse direction and the vortex domain wall. Fig. 2.11A and B give out the spin configurations of the two kind domain walls obtained by OOMMF 2D simulations. Schematic drawings of both transverse and vortex domain walls are also given out in Figure 2.11C and D respectively. Due to the energy competition, the transverse wall is stable in thin film structures with thickness lower than 200 nm ⁸⁷. When the thickness increases up to more than 200 nm , the vortex wall is more favorable. And there is another type of the Neel wall, 360° domain wall which separates two magnetic domains with the same magnetizations.

Fig.2.11 Simulated (A) “down” head-to-head transverse domain wall and (B) a clockwise vortex domain wall. The schematic drawings of the transverse and vortex domain walls can be seen in (C) and (D) respectively.

For the transverse domain walls, according to the spin structures, four classifications can be obtained due to the field history of the domain walls. Shown in Fig. 2.12 are the four spin configurations of a transverse domain wall.

Fig. 2.12 Simulated Head-to-head (A) and (B) and tail-to-tail (C) and (D) transverse domain walls.

If the magnetic field was first applied in the $+x$ direction then in the reversed direction, we can inject a domain wall of a head-to-head (HH) configuration as shown in A and B. In the opposite case, when the initial magnetic field is applied in the $-x$ direction then switched to the $+x$ direction, the injected domain wall is of a tail-to-tail (TT) configuration shown in C and D.

2.5 Magnetic switching mechanism

In nanomagnets, the magnetization configuration is determined from the Brown equation by minimizing the total free energy⁵⁶. There are two principle mechanisms for the spins in a thin film changing direction under an applied magnetic field. These two mechanisms are coherent rotation and domain wall processes^{88,89,90,91}. The driven force in both cases is the possibility of reducing the Zeeman energy of the system by increasing the alignment of spins to an applied field.

2.5.1 Coherent rotation

In the coherent rotation model, the system is assumed to remain in a single domain state during the reversal process, as shown in Fig. 2.13. This mode is first identified by Stoner-Wohlfarth⁹⁰.

Fig.2.13 Illustration of coherent rotation

The hysteresis loops are one of the most fundamental probes of a magnetic system and also can be measured experimentally. Fig. 2.14 gives out a simulated hysteresis loop on which the terminology commonly used in discussion such as coercivity⁹² and remanence⁹³ are labeled. And these concepts and phenomena are not restricted to coherent rotation reversals.

Fig.2.14 A simulated hysteresis loop. The coercive field is the field required to change the magnetization from minus saturation to zero; remanence is the magnetization under zero field.

Another important result from the plot, when the external field is applied close to the easy axis, which is $0^\circ < \theta < 45^\circ$, the hysteresis loops are relatively square. Thus the magnetization change corresponding to the coercivity occurs at the switching field. For the applied field is close to the hard axis, where $45^\circ < \theta < 90^\circ$, the hysteresis loop is sheared and the coercivity corresponds to the value when the magnetization $m=0$.

The angular variation of coercivity for coherent reversal mode is then given by:

$$h_s = (\cos^{\frac{2}{3}}\varphi + \sin^{\frac{2}{3}}\varphi)^{-\frac{3}{2}}, 0^\circ < \theta < 45^\circ \quad \{2.21\}$$

$$h_c = \sin\varphi\cos\varphi, 45^\circ < \theta < 90^\circ \quad 10$$

2.5.2 Domain wall processes

While the coherent rotation model agrees the experimental results well for the reversal jump, the irreversible jump at an external field is observed from the experiment significantly smaller than the anisotropy field. The reason responsible for

this disagreement is that the irreversible part of the switching can also be mediated by the creation and propagation of domain walls. Compared to the coherent rotation reversals, which are collective processes, the domain wall processes are sequential and are much slower⁹⁴. Fig.2.15 shows a domain wall mediating spin reversal process.

Fig. 2.15 Spin reversal by the propagation of a domain wall.

A small domain with spins oriented in the direction different from the original domain nucleates around an inhomogeneity in the sample such as surface defect and the sample edge. The domain wall separating the original rest sample and the nucleated domain advances by small rotation of its spins. It advances in a selected direction so as to remove the areas with spins unfavorable oriented and replace them with the new direction.

There are two types of domain wall process⁸⁹. The first type is just as described above; the entire reversal is achieved by a small number of domain walls moving

large distance. This is domain wall propagation reversal process. The second type is that in a sample a large number of domain walls nucleate and move a short distance before coalescing with neighboring domains. This process is domain wall nucleation reversal process.

Chapter 3

Experimental Techniques

Sample fabrication and characterization techniques used for the study of the nanomagnet structures in this thesis are presented. Electron beam lithography was used to pattern the nanomagnetic structures. Electron-beam and thermal evaporation and direct current (DC) magnetron sputtering techniques were used to deposit the ferromagnetic materials. Scanning electron microscope and scanning probe microscope were used to characterize the structural properties of the magnets. Magneto optical Kerr effect magnetometer (MOKE) and Magneto resistance (MR) measurement joint system were set up, combined with the magnetic force microscope, to measure the magnetic properties.

3.1 Introduction

The Magneto Optical Kerr Effect (MOKE) has been used in this thesis as an experimental probe of the magnetic state within the skin depth of the light of the surface of ultrathin films. This technique does not directly give the absolute value of the layer magnetic moment or the relevant anisotropies of ultrathin magnetic film; however, it is highly sensitive to the magnetic behaviour. This chapter departs into two sections, i.e. fabrication and characterization techniques. Electrical Beam Lithography (EBL) was used to pattern the nanostructures. Electron beam evaporation and direct current (DC) magnetron sputtering were used to deposit the ferromagnetic materials. The characterization section begins by describing the principle behind the magneto-optical phenomena. MOKE magnetometer which is used to characterize film properties. Finally, other characterization techniques used throughout this thesis are described. I have set up the longitudinal MOKE and magneto resistance measurement (MR) joint system and will describe that in details.

3.2 Fabrication techniques

In this thesis, the fabrications of the nanomagnets and the contacts are through the bottom up approach which is based on the idea of depositing a metal film onto the patterned resist, followed by the removal of unwanted metal. The fabrication process is divided into five steps, namely: substrates cleaning, resist coating, lithography, metallization and lift off. This is a very versatile method, which in principle is material independent and its ultimate spatial resolution is determined and resist in a final lift-off step. A typical fabrication process flow for the structure investigate in this thesis, ring arrays, asymmetric ring arrays, micron strips and contacts, is shown in Fig.3.1

Fig. 3.1 Schematic of the flow of the fabrication process for the nanomagnets

3.2.1 Lithography procedures

The lithography procedures described here employ a patterned resist mask on the sample. Depending on the geometries (micron, nanometer) of the samples, different lithography techniques such as photolithography and electron beam lithography were used. The small features defined by lithography are from 100 to 0.015 μm .

3.2.1.1 Photolithography

Photolithography is a process using microfabrication to selectively remove parts of a thin film or the bulk of a substrate. It uses light to transfer a geometric pattern from a photo mask to a light-sensitive chemical photo resist such as AZ9260 and AZ5214 on the substrate. A series of chemical treatments then engraves the exposure pattern into the material underneath the photo resist. Optical lithography is the most widely used technique to fabricate micro/nanoscale structures, and has been developed in the semiconductor industry. A quartz mask is used to transfer the pattern onto photoresist coated on a Si substrate using light. The resolution is defined as:

$$R = k \frac{\lambda}{NA} \quad 95-96$$

Where k , λ and NA stand for the Rayleigh constant, the wavelength and the numerical aperture of the lens. To achieve high resolution, one can decrease the wavelength of the light source. Short wavelengths such as Hg (g-line 436 and i-line 365 nm) light and X-ray radiation have been recently used. On another hand, increasing the numerical aperture also increases the resolution. Besides using phase shift mask and new photoresist also helps to reduce the k factor and the off-axis illumination.

3.2.1.2 Electron beam lithography, EBL

EBL was first discovered in the late 1960s. It is an advanced lithography technique containing two steps: the first step is the practice of scanning a beam of electrons in a patterned fashion across a surface covered with a film of the resist which is called "exposing" the resist; and the second step is the selective removing of either exposed or non-exposed regions of the resist called "developing". The purpose, the same with photolithography, is to create very small structures in the resist that can subsequently be transferred to the substrate material, often by etching. The main advantage of electron beam lithography is that it can beat the diffraction limit of light and make it possible to pattern features in the nanometer regime. Besides, it is a

maskless lithography technique where designs for devices can be made in a computer program and directly used to expose a sample. These advantages make it widely used in low-volume production of semiconductor components, and research & development.

All of the samples discussed in this work have been fabricated by an electron beam direct-write lithography system, capable of writing sub-50 nm patterns with an acceleration voltage of 100kV. The general principle of the sample fabrication is shown in Fig.3.2

Fig.3.2 Schematic of the process of sample fabrication.

An electron sensitive resist was first spin coated on top of a (silicon) substrate and then dried on a hot plate. Subsequently, a predefined pattern is exposed using the high energy electron beam. Due to the exposure, the polymer chains are broken and become selectively soluble in a particular developer⁹⁷. In order to obtain the best resolution of the system, two factors should be minimized: the forward scattering of the electron beam that spread the electrons in the resist; the back scattering of the electron beam that scatters the electrons in the substrate and reflects back into the resist. The latter gives rise to the secondary exposure of the resist and are especially strong near large exposed area. After the e-beam exposure, the sample is put in a

suitable developer and the exposed areas of the resist are removed. The next step is material deposition and later in this chapter the techniques used for the material deposition will be described in detail. After deposition, the sample is put in a solvent and the polymer and metal on top of it will be removed. This technique is called lift off.

In this thesis two different resist systems combining with e-beam lithography are used to fabricate our samples. The electron beam lithography in this work was performed in the local Astar research institution IMRE, using shaped beams. The system is able to write sub 50 nm structures by delicately chosen exposure dose and current: a conventional PMMA resist⁹⁸ for fabricating structures with feature size larger than 100 nm and a ZEP based for fabrication of structures with feature size ranging from 10 to 100 nm.

Conventional PMMA

This technique represents the standard and most widely used resist system for electron beam lithography. The substrates used for all experiments is single side polished P-doped Si<100> with a thermally oxide of 300 nm. Du Pont Elvacite PMMA 2041 (high molecular weight) 4% in chlorobenzene, was filtered with 0.2 μm filters and spun at 5000 rpm for 1 min. Subsequently, the resist is baked for 15 minutes on a hotplate at 170 °C.

ZEP

Zep in IPA with a ratio of 1:3 was filtered with 0.2 μm filters and spun at a speed of 4000 rpm for 1 min. The baking temperature and time were 180 °C and 2 min respectively.

The sample with feature size bigger than 100 nm is exposed using a 100k eV electron beam and the current is stabilized at 100pA. Smaller feature with size lower than 100 nm typically use a dose of 35 mJ. The stabilized current is 50Pa. The

development for the PMMA sample is dipping in a 3:1 mixture of IPA:MIBK for 70 seconds and then flushed in IPA. For the ZEP sample, the solvent is oxylene and the developing time is 30 sec. As a last step, a short anisotropic oxygen etch is done in a reactive ion etcher to remove the last residual resist on the substrate. The etching settings are: Oxygen pressure 10 mtorr, Power 20 W, etching time, 6s.

Lift off

The last step in the process is lift off⁹⁹. For delicate samples in which metals are used with relatively poor adhesion such as Cu and Al, the sample is immersed in boiling acetone until all residual material has lifted off. Then it is flushed in cold ethanol and dried with pure N₂. For more robust samples such as Co, Ni and sputter deposited Py with a tantalum adhesion layer, ultrasonic agitation in heated acetone can be used. Ultrasonic agitation in combination with the stronger heated solvent J. T. Baker PRS 3000 resist stripper can be important, especially after sputter deposition of the Py metal to remove resist ears at the edges of a structure.

3.2.1.3 Nanosphere lithography

With nanotechnology becoming widely used, there is an increasing demand for rapid, parallel fabrication strategies for nanoholes and nanopillars. Some applications that require repetitive uniform nanoholes and nanopillars over large area are photonic crystals¹⁰⁰, memory devices¹⁰¹, nanofiltration¹⁰², solar cells¹⁰³, etc. Of all the applications, towards delaying the onset of thermal effect and superparamagnetic limit in the ultrahigh density recording media, patterned magnetic thin film nanostructures are intensively investigated^{70, 104}. For optimum performance of the patterned storage medium, understanding and controlling the magnetic properties of individual and interacting elements at reduced dimension are of key importance. Different patterned magnetic nanostructures like rectangles¹⁰⁵, dots⁴⁶ and rings¹⁰⁶ have been studied. Due to the requirement of the density increase in the developing recording technology, the magnetization reversal mechanisms in sub-100 nm sized

patterns have recently attracted a lot of attention. In nanomagnets with size in this sub-100 nm range, the shape anisotropy becomes a key issue to the magnetization reversal. Using conventional lithography techniques, such as electron beam lithography¹⁰⁷, interference lithography¹⁰⁸, as well as x-ray lithography, magnetic reversal process in patterns of hundreds nm scale have been fabricated. However, the conventional lithography techniques are limited in resolution by the wavelength of light (λ) used and the system numerical aperture (NA), giving a minimum feature size of $\lambda/2NA$. Besides, in the application sense, finding lithographic processes which can pattern the thin films into nanoscale patterns at an economically acceptable cost is also a challenge. A variety of nanofabrication technologies have been developed to prepare large-area 2D or 3D colloidal patterned surfaces, such as self-assembly, spincoating, electric-field-induced electrokinetic flowing, and Langmuir-Blodgett deposition^{109,110,111,112-113}. In particular, self-assembly technique, which forming ordered periodic arrays of packed spherical configuration with uniformly sized microspheres, has been applied widely because of its unique features: it is inexpensive, inherently parallel, and enables high-throughput nanofabrication. Of all the appealing approaches to capitalize on the self-ordered structures found in nature, nanosphere lithography (NSL) is a well-known, low-cost, and quick preparation method, which makes use of self-assembly of nanospheres to fabricate nanostructure and nanostructure arrays. Fig.3.3 shows the schematic of the fabrication process

Fig. 3.3 Schematic of the fabrication process

This technique is always used for production of periodic particle arrays and it strictly requires the nanospheres to form a perfect hexagonal closed monolayer.

3.2.3 Deposition techniques

This chapter introduces the different deposition techniques employed in this thesis. During the experiments, two main deposition methods of evaporation and sputtering were routinely used.

3.2.3.1 Sputtering

Sputter deposition is a method of depositing thin films by sputtering material from a target, which then deposits onto a substrate. Sputtering usually uses argon plasma. The incident argon ions set off collision cascade in the target. When such cascades recoil and reach the target surface with energy above the surface binding energy, an atom can be ejected. The average number of atoms ejected from the target per incident ion is called the sputter yield and depends on the ion incident angle, the energy of the ion, the masses of the ion and target atoms, and the surface binding energy of atoms of the target. Sputtered atoms ejected into the gas phase are not in their thermodynamic equilibrium state, and tend to deposit on all surfaces in the vacuum chamber. A substrate such as a wafer placed in the chamber will be coated with a thin film.

The sputtering process was conducted using an in house built sputter system. This system consists of a deposition chamber interconnected with a load-lock which is used to transfer samples into the chamber. The chamber can hold six target materials and is always kept in vacuum and the base pressure in the chamber is better than 2×10^{-8} Torr. The system is capable of DC, RF and co-sputtering. A baratron valve was used to control the deposition pressure. For the permalloy patterns studied in this thesis, DC sputtering at room temperature was used to deposit $\text{Ni}_{80}\text{Fe}_{20}$. While for the study of magnetite at the final part of this thesis, the Fe_3O_4 films were reactively sputtered from pure Fe target in an Ar- O_2 mixture, with the substrate maintained at room temperature and high temperature, respectively. The sample holder is at the center of the chamber with a self rotation to ensure uniformity in the film deposition. Before deposition, reverse sputtering with a bias voltage was used to pre-clean the sample surface. A schematic of the deposition process is shown in Fig.3.4

For a particular sputtering process, the deposition power determines the growth rate of the thin film, while the film uniformity is determined to a large extent by the deposition pressure. The lower the pressure is, the more uniform the film is. However,

there is a minimum value of pressure for the sustain of the plasma. The flow rate of the injected gas also affects the film deposition but in a less-influential way. Unless the flow rate is too low to maintain the deposition, it won't play an important role.

Fig.3.4 Schematic and picture of the sputtering process.

One drawback of sputtering is the fact that it is isotropic which demands a better more over hanging resist profile. If this condition is not met, very high ears are formed at structure edge which can cause major problems if good step coverage of the sputtered Py layer is needed in a second deposition step.

3.2.3.2 Evaporation

For the evaporation process, an Edwards 306 system was used. Both thermal and focused electron beam evaporation processes are allowed in this system. It comprises one electron-beam pocket and two thermal boats, which enables the

deposition of different materials without breaking the vacuum condition. As shown in Fig.3.5, the material sources are placed at the bottom of the chamber and the samples are fixed on the sample holder mounted on the ceiling of the chamber. The sample holder is a self rotation holder which orbits around a central axis during deposition. The base pressure for the evaporation is better than 5×10^{-7} Torr.

Fig.3.5 Edwards 306 system as well as its illustration.

The material deposited in this work involves two evaporation modes. For the ferromagnetic materials such as Co and NiFe are melted by the focused electron beam since they have higher melting temperatures. While for the deposition of materials with low melting points such as Cr, Cu and Au, thermal evaporation was employed. In order to obtain spatially uniform deposition, the sample holder was rotated at a speed of 50 rad/min. A quartz crystal microbalance was employed to monitor the deposition rate.

3.3 Characterization techniques

This section describes the tools that were used in nanomagnets properties

characterizations, such as magneto optical Kerr effect magnetometry, scanning electron microscope, scanning probe microscope, room temperature magneto transport measurement system, x-ray diffraction, x-ray photoelectron spectroscopy.

3.3.1 Magneto-optic Kerr effect magnetometry (MOKE) and Magneto resistance (MR) measurement system

Magneto-optical Kerr effect (MOKE) which is first discovered by Kerr¹¹⁴, is referred to the observation that the plane of polarisation of a linearly polarised light was rotated upon reflection from the poles of an ¹¹⁵electromagnet. The polarisation change results from the interaction of the electromagnetic wave with the electrons in the material in the presence of magnetisation. The magnetisation is coupled to the electron system via the spin-orbital interaction¹¹⁶. The MOKE can be described phenomenologically by including off diagonal elements in the dielectric tensor ϵ'

$$\epsilon' = \begin{pmatrix} n^2 & -in^2Q & 0 \\ in^2Q & n^2 & 0 \\ 0 & 0 & n^2 \end{pmatrix} \quad 117$$

where n is the complex refractive index of the material such that $n^2 = \epsilon'$ and Q is the complex magnetooptic vuigt constant. Q is determined by the strength of the spin-orbit coupling, and, importantly, by the magnetisation. Freiser¹¹⁸ has shown that when Maxwell's equations are solved for this dielectric tensor, the reflected light wave can be described by a set of Fresnel reflection coefficients such that

$$E_r = \begin{pmatrix} r_{pp} & r_{ps} \\ r_{sp} & r_{ss} \end{pmatrix} E_i \quad \{3.3\}$$

where the electric field vectors of the incoming and outgoing light E_i and E_r respectively are described by the Jones notation $E = \begin{pmatrix} E_p \\ E_s \end{pmatrix}$. Freiser showed that the

Fresnel coefficients are determined by the angle of incident light and the magnetisation (via Q). Since the Fresnel coefficients are in general complex, plane polarised light is reflected with both a rotation of the polarisation plane and the introduction of ellipticity. These are known as the Kerr rotation and the Kerr ellipticity respectively.

There are three kinds of basic MOKE configurations as shown in Fig. 3.6. In the longitudinal MOKE, the magnetization vector is parallel to both the reflection surface and the plane of incidence. Therefore, the longitudinal MOKE measurement is sensitive to the component of in-plane magnetisation lying in the plane of incident light. The transverse MOKE is sensitive to in-plane magnetisation vertical to the plane of the incident light. The polar MOKE is sensitive to the component of magnetisation lying along the surface normal.

Fig.3.6 Three kinds of basic MOKE configurations.

An analyser and photodiode are used to pole the polarisation state of the reflected light. Hunt¹¹⁹ has shown that in the case of an arbitrarily oriented in-plane magnetisation, the transverse MOKE signal can be nulled by setting the incoming light to s-polarisation and setting the analyser close to extinction. In this case the r_{sp} Fresnel coefficient is poled and is proportional to the component of in-plane magnetisation during spin reversal.

Fig.3.7 shows a schematic diagram as well as the picture of the experimental MOKE set-up. The light source used is a 5 mW continuous mode HeNe laser ($\lambda = 632.8$ nm) with an intensity stabiliser. The size of the laser spot is 500 nm. The stabiliser is housed in the laser case and the outcoming beam is linearly polarised by an optical polarizer. The optical chopper is used to add frequency information into the polarized

laser, so that only the light in the frequency will be detected by the lock-in amplifier. An electromagnet is used for field ramping and the field step is controlled by the LabView program. The virtual magnetic field is measured by a Lakeshore gaussmeter (model 475 DSP). The reflected light beam is focused by a lens towards the analyser and the detector. The detector is a photodiode and is placed after the analyser. A Keithley voltmeter (model 2000) is used to detect the MOKE signal, which is then analysed using a LabView program in a computer. The maximum magnetic field of the electric magnet is 2T. While the field strength and the field step for a particular measurement can be programmed from the front panel of the labview program.

Fig. 3.7 Schematic diagram as well as the picture of the experimental MOKE set-up

3.3.2 Magneto resistance measurement

Magnetoresistance is the property of a material to change the value of its electrical resistance when an external magnetic field is applied to it. The resistance of a ferromagnetic sample has a $\cos^2$ dependence on the angle between the directions of the magnetization and of the current (the anomalous magneto resistance effect-AMR). Thus careful MR measurements can yield useful insights not only in transport properties, but also in non-transport processes like magnetization reversal. As far as AMR effect is a band structure effect, the sensitivity is retained as long as the band structure of the metal is preserved at room temperature. This high sensitivity allows us to probe both the position and the configuration of the domain wall. To investigate the AMR effect in patterned magnetic nanostructures helps to understand the spin configurations which hold much promise for the magnetic memory elements and the read heads.

Shown in Fig. 3.8 is the schematic diagram of the MR measurement system, which shares the electromagnet, lock-in amplifier and the Keithley 2000 multimeter with the MOKE system. Compared to the MOKE system, the input of the MR system is AC/DC current powered by a source meter instead of laser with programmed frequency. There are two kinds of sample holders in this system as shown in the upper panel of Fig.3.9. The sample holder shown in the left panel is for patterned device testing. Before testing, wire bonding technique is employed to bond the gold pads to the chip carrier, which is plugged into the sample holder as shown in the picture. Shown in the right panel is a sample holder with four pin in plane which is designed for giant magneto resistance (GMR) measurement. All these sample holders are made from plastic or brass.

Fig.3.8 Schematic diagram of the MR measurement system.

Shown in Fig.3.9 is a picture of the MR system.

Fig.3.9 Picture of the MR system and two examples of measured graphs at room temperature: the left panel shows a graph of organic magnetoresistance measured on an organic LED; the right panel shows giant magneto resistance in a spin valve structure.

In the measurement, the current source keeps on sourcing current with programmed frequency and magnitude. The ramping of the power supply for the electrical magnet, the signal detecting and the gaussmeter are controlled by Labview program. The lower panel show graphs of AMR and GMR measured by this system and the details on the Labview program are attached in the appendix.

3.3.3 Vibrating Sample Magnetometer (VSM)

VSM is the most widely used magnetometer for characterizing large-area magnetic materials, via hysteresis loop measurement, using the induction technique. The hysteresis loop is obtained by mapping the magnetization of the sample with respect

to an externally applied magnetic field. The hysteresis loop provides an insight into the magnetic properties of the ensemble of nanomagnets in the sample. The detection of the sample magnetic properties is based on the Faraday's Law of induction: the induced voltage is proportional to the rate of change of magnetic flux through a coil. The measurement of the hysteresis behavior of the magnetic meso-nano structures in this thesis is performed by EV5 VSM from Digital Measurement Systems. The sensitivity of the VSM is in the order of 10^{-5} emu. The sample is suspended by a temperature-insensitive glass rod in between a pair of electromagnets. The electromagnets can provide a magnetic field of magnitude up to 1.4 Tesla. The rod is vibrated at a constant frequency F ($\sim 70\text{Hz}$) in the vertical direction. This vibration disturbs the magnetic field generated by the electromagnets, and results in an alternating current with an effective voltage of

$$V = MAFS \quad \{3.4\}$$

Where M is the sample's magnetic moment, A is the amplitude of vibration, F is the frequency of vibration and S is the sensitivity of pick up coils. Therefore, the induced voltage in the pick-up coil is proportional to the magnetic moment of the sample. By detecting and amplifying the AC voltage using a lock-in amplifier, the magnetic moment is converted to DC signal, which is mapped to the magnetic moment from the calibration parameters. As the external field (H) is swept at a fixed rate, the pick-up coils measure the magnetization (M) of the sample for each applied field. The plot of the magnetic moment (M) as a function of the externally applied field (H), results in the hysteresis loop. To improve the accuracy of the results, a stepped field approach is used. The magnetic field is changed in selected step sizes and the magnetic moment is measured with the field stationary.

PPMS is generally a low temperature system, which can provide low temperature environment. The cooling process is just let He gas in to the sample chamber at above 10K. PPMS is generally a system which is able to provide variable temperature-field system, and is designed to perform a variety of automated measurements. Use the PPMS with our specially-designed measurement options, or easily adapt it to your own experiments. Sample environment controls include fields up to ± 16 tesla and

temperature range of 1.9 - 400 K.

3.3.4 Scanning electron microscope (SEM)

The scanning electron microscope (SEM) is a type of electron microscope that images the sample surface by scanning it with a beam of high-energy electrons in a raster scan pattern. The electrons interact with the atoms and produce signals that contain information about the sample's surface topography, composition and other properties such as electrical conductivity as shown in Fig.3.10. The SEM is very important and powerful in imaging structures ranging from millimeter to nanometer scale¹²⁰. SEM is capable of resolving features down to the nanometer regime due to the extremely small wavelength of the highly accelerated electrons.

Fig.3.10 Schematic of sample- electron interaction.

A SEM with a field emission gun from JEOL was used to study the profile of the fabricated structures. By detecting the secondary electrons^{121,122} which are usually emitted from the first few nanometers of sample, the profile information of the sample surface was obtained. To avoid ion bombardment of the electron gun and also to

minimize the electron scattering, the working pressure of the chamber is maintained below 10^{-7} Torr. During the measurement, the electron beam which works as the probe is controlled by scanning magnetic coils. A detector comprised by a scintillator and a photomultiplier is used to obtain the topographical information by detecting the secondary electrons emitted from the sample surface.

3.3.5 x-ray photoelectron spectroscopy (XPS)

X-ray photoelectron spectroscopy XPS is a quantitative spectroscopic technique that measures the elemental composition, empirical formula, chemical state and electronic state¹²³ of the elements that exist within a material. Chemical composition and ratio of calcium and phosphate can be obtained by X-ray photoelectron spectroscopy (XPS, Kratos-Axis Ultra System). Monochromatic Al K α X-ray source (1486.7 eV) was employed and working condition was in the vacuum of the chamber of pressure of 2×10^{-9} torr. The survey scan was recorded in the range of 0-1100 eV in 1 eV step for each sample, followed by narrow scan for each element of the coating in 0.1 eV step. Then, the coating composition and ratio of element was calculated according to the narrow scans by Kratos software. The measured binding energy¹²⁴(BE) of all XPS profiles was calibrated using C 1s (BE = 284.8 eV).

3.3.6 X-ray powder diffraction (XRD)

X-ray powder diffraction (XRD) data was obtained using a Bruker Advance D8 powder diffractometer equipped with a focusing Ge(111) incident beam monochromator (Cu K α 1 radiation) with a Bragg-Bretano geometry. The thin film-disk was placed on a glass-background quartz sample holder. X-ray diffraction data was obtained at ambient temperature in the range of $2\theta = 10-90^\circ$ with a step range of 1° and a measuring time of 2 s per set. The full width at half maximum (fwhm) and d-spacings of the peaks was determined with DIFFRACPLUS software by Bruker AXS.

3.3.7 Scanning probe microscope (SPM)

Scanning Probe Microscopy (SPM) refers to a family of microscopy that forms images of surfaces using a physical probe that scans the specimen. In the scanning, a sharp probe scans across a surface and the interaction between the probe and the sample is recorded. By doing so, various kinds of information are obtained, such as height, magnetic pole and friction. Since the atomically sharp probe is employed in the scanning, the resolution is no longer restrained by the wavelength of the light or electron. The forms of the SPM are various: the primary forms are the atomic force microscope (AFM) and scanning tunneling microscope; other members of the SPM family are the lateral force microscopy, the magnetic force microscopy (MFM), electric force microscopy etc. In our experiment, AFM and MFM measurements from a Veeco SPM system were used.

3.3.7.1 Atomic Force Microscope AFM

The Atomic Force Microscope (AFM) employs a very sharp tip (at the size of 10 atoms) at the end of a low spring constant cantilever. The spring constant is typically on the order of 0.1N/m for contact mode and 10N/m for noncontact mode imaging. The tip interacts with the sample surface through atomic forces (electric, magnetic, van der Waals, etc.) and the information about the material characteristics can be obtained. The movement of the cantilever in X-Y direction across the sample surface and its response to atomic forces are tracked by a laser beam which is focused on the tip. The reflecting beam from the tip passes through a series of optics and is finally focused on a quad photodiode detector. The signal from the photodiode is then sent into a feedback system which causes the piezoelectric tube to move in Z direction to keep the cantilever deflection constant as shown in Fig.3.11. Thus, as the tip responds to the surface during the raster scanning, the action of the piezoelectric

crystal is recorded and stored as images. The AFM has two modes — Tapping Mode and Contact Mode. In the tapping mode, the surface topography is mapped by lightly tapping the surface of the sample with an oscillating probe tip¹²⁵.

Fig.3.11 Schematic of typical AFM measurement.

3.3.7.2 Magnetic force microscopy

When a magnetic tip is magnetized in one direction, NC-AFM can be sensitive to the magnetic force^{126, 127}. The relationship between the force gradient and tip-sample distance z is $F' = \frac{\partial F}{\partial z}$. For MFM, the sharp tip is coated with a ferromagnetic thin film which is mostly CoCr with high coercivity. As such, the MFM tip has a larger curvature and is sensitive to magnetic force with $50 < z < 500$ nm. The MFM senses the force gradient between the MFM tip and magnetic sample due to the perpendicular component of the sample stray field. The force¹²⁸ acting on the tip is given by:

$$F = \int d^3w' \int d^3w'' M(w') \nabla \cdot H(w'', w') \quad \{3.5\}$$

where $M(w')$ is the tip magnetization at a position w' inside the tip and $H(w'', w')$ is the stray field generated at point w' by the magnetic moment at position w'' inside of the sample. This force causes the bending of the cantilever with an amount of $\partial Z = \frac{F_{\perp}}{k_c}$, where k_c is the spring constant of the cantilever and $F_{\perp}$ is the force normal to the cantilever. The MFM is operated in ac mode by driving the cantilever to its resonant frequency¹²⁶. In this mode, the resonance frequency f_0 is given by:

$$f_0 = \frac{(\frac{k_c}{m'})^{\frac{1}{2}}}{2\pi} \quad 129$$

where m' is the effective mass of the cantilever¹³⁰. A shift in the cantilever resonant frequency is induced by the long range magnetic forces which can be expressed as:

$$\partial f = \frac{F'_\perp}{2k_c} f_0 \quad \{3.7\}^{131}$$

where $F'_\perp$ is the force gradient along the normal direction of the cantilever.

In an MFM scanning, the tip of a magnetic probe is magnetized perpendicular to the sample surface and then mounted on a tip holder. The phase curve measures the phase lag between the driving voltage and the cantilever response¹³². Hence the vertical gradient in the magnetic force cause a shift Δf_0 in the resonance frequency. This resonance Δf_0 on one hand gives rise to a phase shift $\Delta\phi$, which provides an image of the magnetic force gradient. On the other hand, it gives variations in oscillation amplitude ΔA . This variation can be used to image the magnetic force gradient¹³³. A major problem in MFM is to separate the magnetic image from the topographical data. In order to obtain a good image, the magnetic measurements are carried out by a two-pass technique. In the first pass, the topology of the sample is determined with the cantilever oscillation amplitude as feedback using the tapping mode. Then in the same trace line, the cantilever is raised by a selected height offset using lift mode to record the magnetic influence with the stored surface topography information. In this thesis, the MFM images were taken by raising the cantilever to a height of 40 nm after the first pass.

Chapter 4

Magnetization reversal process in NiFe asymmetric rings

This chapter presents a technique for flux-closure chirality control and domain wall trapping at the narrowest position in asymmetric magnetic ring. Micromagnetic simulation works were performed on permalloy asymmetric magnetic rings to observe its magnetic switching behavior. By controlling the lateral geometric features, the ring asymmetry, and the thickness of the film, a local vortex-free reversal process and well-controlled chirality of flux-closure can be achieved. Furthermore, a domain wall trapping feature is also observed at the thinner arm of the asymmetric ring, which corresponds to the phenomenon where the magnetic domain wall do not annihilate until a relatively large magnetic field is applied. A phase diagram of the asymmetric ring switching behavior shows that the switching regimes (e.g. the domain wall propagation or nucleation-annihilation) of the asymmetric rings and the domain wall pinning mainly depend on the film thickness. In situ magnetic force microscopy was carried out to directly observe the notch-free DW trapping behavior and the field history effect on the domain wall in $\text{Ni}_{80}\text{Fe}_{20}$ asymmetric ring. We found that a 360° domain wall is trapped at the narrow arm while the ring adopts a vortex configuration. The stability of domain wall is dependent on the chirality of the vortex state and the external field direction. A 360° domain wall trapped in a clockwise vortex configuration is highly resistant to annihilation upon the application of $+x$ field; the 360° domain wall trapped in an anti-clockwise vortex breaks apart with a small $+x$ field.

4.1 Introduction

Magnetic nanostructures⁵⁴ created by controlled nanofabrication techniques are attracting intense research interest due to their potential significance in magnetic data storage¹⁰⁴, and solid state memory¹³⁴ devices. New technologies which are able to maintain the areal density growth have been pursued by the hard disk drive industry and research community. 1 Tb/in² recording has been realized by using perpendicular technology. Beyond that and towards the 10 or multi Tb/in² regime, new recording mechanisms need to be explored and implemented. The energy-assisted magnetic recording¹³⁵ and bit patterned media recording¹⁴ are two promising new technologies.

Fig. 4.1 Promising technologies in realizing 10 Tb/in² magnetic recording

Towards achievable applications of bit patterned magnetic recording devices, a stable and reproducible switching mechanism is needed for the nanomagnets. Of all the studied nanostructures, ring-shaped nanomagnets have reproducible magnetic states, that arise from a competition⁵¹ between the magnetostatic energy and the exchange energy. The ring structure also prohibits the formation of edge domains and minimizes the stray field. This makes the ring structure an ideal candidate for use in

ultra high density data storage. The switching process in ring structures is by the formation of the ‘onion’ state⁴, followed by the vortex state⁵ occurring via the movement of domain wall (DW). One difficulty associated with the ring structures during the reversal process is the inability to pin the DW in a certain position.

Fig.4.2 Domain wall trapping and vortex state with clockwise and anticlockwise chirality

An approach that is commonly used to pin the DWs is by exploiting the shape anisotropy, e.g. the use of elliptically shaped ring structures¹³⁶. Alternatively, the domain wall can be pinned via the creation of geometrical defects or notches¹³⁷ along the ring structure. Such notches introduce low energy well to interrupt the continuity of the magnetization.

In this experiment, asymmetric ring-shaped Ni₈₀Fe₂₀ nanomagnets of different thicknesses were fabricated using electron beam lithography (EBL) techniques. Details of EBL technique and process are described in Chapter 2. A silicon substrate was first coated with a 300-nm-thick PMMA photoresist layer. Electron beam lithography at 100kV beam energy was used for patterning. The *e*-beam exposed resist pattern was developed in a mixture of MIBK: IPA 1:3. Subsequently, magnetron sputtering technique was used to deposit Ta (5nm)/NiFe (*t* nm)/Ta (5nm) films. The growth rate of Ta and NiFe were calibrated before growing. The Ta film was deposited as capping and buffer layers so as to minimize the degradation of the NiFe layer. The chamber base pressure was better than 3.0×10^{-8} Torr, and the sputtering pressure was maintained at 3 mTorr. The magnetic asymmetric ring pattern was obtained after an ultrasonic agitation-assisted lift-off process¹³⁸. Lift-off completion was confirmed by inspection under scanning electron

microscope (SEM). Figure 4.1 shows the SEM image of asymmetric ring-shaped nanomagnet arrays.

Fig.4.3 Scanning electron micrograph of a 10nm thick $\text{Ni}_{80}\text{Fe}_{20}$ asymmetric ring array. The outer diameter D_o of the asymmetric ring is 1200 nm, the inner diameter D_i is 600 nm and the offset from the center is 120 nm.

The outer diameter of the fabricated asymmetric ring structure is 1200 nm while the inner diameter is 600 nm. The asymmetry was induced by offsetting the center of the inner circle by a distance ($l = 120$ nm) with respect to the center of the outer circle, as shown in the schematic diagram in Figure 4.3. The rings were patterned with a spacing of 1200 nm to avoid magnetostatic field coupling among the nanomagnets.

Fig.4.4 Geometry of the asymmetric magnetic ring used in the simulation. An example of asymmetric magnetic ring for ratio $R=0.2$ is shown here. For clarity, we refer to the asymmetric axis as AC and the symmetric axis as BD.

4.2 2D Object Oriented MicroMagnetic Framework (OOMMF) magnetic simulation

The magnetization configuration and switching properties were simulated using the Object Oriented MicroMagnetic Framework (OOMMF) simulation program¹³⁹. Given a problem description, the simulator integrates the Landau-Lifshitz equation,

$$\frac{d\mathbf{M}}{dt} = -|\bar{\gamma}|\mathbf{M} \times \mathbf{H}_{\text{eff}} - \frac{|\bar{\gamma}|\alpha}{M_s}\mathbf{M} \times (\mathbf{M} \times \mathbf{H}_{\text{eff}})$$

Where

$\mathbf{M}$ is the pointwise magnetization(A/m)

$\mathbf{H}_{\text{eff}}$ is the pointwise effective field(A/m)

$\bar{\gamma}$ is the Landau-Lifshitz gyromagnetic ratio(m/(A·s)),

α is the damping coefficient (dimensionless).

The effective field is defined as

$$\mathbf{H}_{\text{eff}} = -\mu_0^{-1} \frac{\partial E}{\partial \mathbf{M}}$$

The average energy density E is a function of M specified by Brown's equations, including anisotropy, exchange, self-magnetostatic (demagnetization) and applied field (Zeeman) terms. The micromagnetic problem is impressed upon a regular 2D grid of squares, with 3D magnetization spins positioned at the centers of the cells. The anisotropy and applied field energy terms are calculated assuming constant magnetization in each cell. The exchange energy is calculated using the eight-neighbor bilinear interpolation, with Neumann boundary conditions.

The Landau-Lifshitz ODE is integrated using a second order predictor-corrector technique of the Adams type. At each stage the spins are renormalized to M_s before evaluating the energy and effective fields. The right side of is evaluated at the predicted M , which is then combined with the value at the current step to produce a linear interpolation of dM/dt across the new interval. This is then integrated to obtain the final estimate of M at the new step.

In this simulation, the micromagnetic equilibrium equations were solved in a two dimensional square mesh with a cell size of 4 nm and the spins are free to rotate in three dimensions. A ramping magnetic field of -1500 G to 1500 G in the asymmetrical axis of asymmetric ring (AC in Fig. 1) in 60 steps was applied. Other parameters used for the permalloy were crystalline anisotropy constant $K_1 = 0$,

saturation magnetization $M_s = 8.6 \times 10^5 \text{ A m}^{-1}$, exchange stiffness

$A = 1.3 \times 10^{-11} \text{ Jm}^{-1}$ and the damping coefficient $\alpha = 0.5$. The asymmetric ratio, R of

the ring is defined as $R = \frac{l}{D_o - D_i}$, where l is the displacement distance of the

internal circle from the outer circle, while D_o and D_i is the outer and inner diameter of the ring, respectively, as depicted in Fig. 4.3. For the initial part of this work, D_o and D_i , as well as the thickness t were varied at fixed $R=0.2$ to observe their effects on the switching mechanisms and behavior. Subsequently, calculations on asymmetric ring for $R=0.1$, and $R= 0.3$ were also presented for comparison.

4.3 Flux-closure chirality control and domain wall trapping in asymmetric magnetic ring

In order to study the effect of the lateral size and thickness of the asymmetric magnetic ring on the switching processes and mechanisms, a wide range of outer diameter of the ring ($D_o=400, 600, 800, 1000, 1200, 1400$ nm) and the film thickness ($t=8, 12, 16, 20, 24, 28, 32$ nm) were performed. The effect the D_i , which bears some similarity to the influence of ring width as in the case of the symmetric ring is also studied; for a fixed $D_o=1200$ nm and for various inner diameter ($D_i=600, 800, 900$ nm). Our calculations revealed that when the inner circle is off-centered by a ratio $R=0.2$, a triple-switch loop with two plateaus can be seen. This is in contrast to the double switching that occurs in symmetric ring structures reported by F.v. Belle et al¹⁴⁰. The simulated hysteresis loop indicates a number of magnetization states along with different magnetic field strengths applied: negative saturation, bi-domain, vortex, reverse bi-domain, and positive saturation. Typically simulated hysteresis loop of the asymmetric ring (for a fixed $D_o=1200$ nm and $D_i=600$ nm) for $t=12$ nm, $t=8$ nm is shown in Fig. 4.5 and Fig. 4.6, respectively. Insets show its corresponding magnetic configurations.

Fig. 4.5 Typically simulated hysteresis loop on an asymmetric magnetic ring for $D_o=1200$ nm, $t =16$ nm and its magnetic configurations (shown in insets);

In Fig. 4.6 the first two switches in the loop are due to the propagation of the domain walls via the thinner and the thicker arm of the asymmetric ring. The third switch is caused by the domain wall annihilation in the wider arm of the asymmetric ring. The annihilation of the domain wall in the wider arm cause a magnetization drop which is similar as the symmetric ring¹⁴¹ as reported by Klaui et al. This resemblance in hysteresis loop is due to the similarity of the geometric feature since the wider arm of the asymmetric ring can be seen as a part of a wide symmetric ring. However switching behaviors of asymmetric ring with $t=8$ nm are quite different: instead of forming a flux-closure after the bi-domain state, the two domain walls in the thin arm do not annihilate, and a pair of head-to-head and tail-to-tail domain walls are trapped at the narrowest point of the ring. When the reversed magnetic field is large enough, the trapped domain wall annihilates. Fig. 4.6 shows the switching process.

Fig. 4.6 Typically simulated hysteresis loop on an asymmetric magnetic ring for $D_o=1200 \text{ nm}$, $t = 8 \text{ nm}$ and its magnetic configurations (shown in insets);

The hysteresis loop is similar to that in asymmetric ring with a thickness of 16 nm, except that there is one additional small plateau. This small plateau is caused by the trapped domain wall. The annihilation of this trapped domain corresponds to the last switch in the hysteresis loop. We further varied the inner diameter from 600 nm to 800 nm and 900 nm of the asymmetric ring for the same $t = 8 \text{ nm}$ and calculate the magnetic switching process. The switching processes are similar. Fig. 4.7 is the calculated hysteresis loop of asymmetric ring with inner diameter of 600 nm, 800 nm and 900 nm. From the hysteresis loop we can see that the switching field increases as the ring arm width decreases which is probably due to the uneven edge acting as pinning sites for the domain walls.

Fig. 4.7 Hysteresis loop of the asymmetric magnetic ring for various $D_i = 600$ nm, 800 nm and 900 nm at a fixed $D_o = 1200$ nm and $t = 8$ nm.

From the switching process of the asymmetric ring, the circulation of the flux-closure state is determined by the spin direction in the wider arm of the asymmetric ring can be seen. Since the narrowest point of the asymmetric ring is the energy minimum position for the transverse walls, the domain walls nucleated at the asymmetric axis (AC in Fig. 4.4) at large field will selectively move to the position of lower energy which is the narrowest point of the ring arm (B in Fig. 4.4). By this selectivity of the domain wall movement, the circulation of the flux-closure state can be controlled by applying a magnetic field along the symmetric axis direction of the asymmetric ring. An elegant way of controlling the circulation of flux-closure was proposed by Vavassori et al¹⁴² by introducing a void in a disk. Compare to their methods, our flux-closure control using asymmetric ring can attain a local vortex free switching process by using different inner diameters and asymmetric ratios.

4.4 Effect of geometry

In order to investigate the influence of geometric parameters such as ring width, thickness and outer diameter to the switching process, we systematically studied the switching processes and the switching field distributions. Fig. 4.8 shows the phase diagrams of the asymmetric ring switching behavior. In the phase diagram there are two significant boundaries. The first boundary is at line *I*. Below line *I*, a pair of head-to-head and tail-to-tail domain walls can be trapped at the thinner arm of the asymmetric ring. This trapped domain wall does not annihilate until a relatively large magnetic field is applied as shown previously for asymmetric ring of a $D_o = 1200$ nm. The annihilation of the trapped domain wall causes the last small switch in the hysteresis loop. The second boundary is at line *II*. This line separates the switching process of the simple domain wall propagation reversal and multi-nucleation and annihilation reversal. Above line *II*, the switching process is via multi-nucleation and annihilation. In between line *I* and line *II*, the switching process is simple domain wall propagation which corresponds to the squares in Fig. 4.8. This result shows that the film thickness is the key factor that determines the switching behavior and separating the three switching regimes.

Fig. 4.8. Phase diagram of switching as a function of the ring geometrical parameters i.e. the thickness of the film and the outer diameter of the ring for $D_i/D_o=0.5$; line I and line II are two boundaries separating following three kinds of switching;

- (i) Transverse domain wall propagation in the thin arm (■),
- (ii) Vortex nucleation in the wide arm (●), and
- (iii) Multi-nucleation switching (▲).

4.5 Effect of asymmetric ratio on the domain wall trapping

We further varied the distances between the inner and outer circle of the asymmetric rings to investigate the influence of asymmetric ratio on the switching behavior. The lateral size of the asymmetric rings used here are: $D_o=600$ nm, $D_i=300$ nm. The thickness of the rings is 16 nm. The asymmetric ratios used were $R=0.1, 0.2$, and 0.3 which means that the distances between center of the inner and outer circles were 30, 60, and 90 nm respectively. According to our calculations, with same lateral size and thickness, the switching behaviors of asymmetric rings with asymmetric ratios $R=0.1, 0.2$ and 0.3 are quite similar shown in the insets to Fig.4.9. The most

significant influence of the asymmetric ratio is that with a larger asymmetric ratio, the field to drive the domain walls nucleated at the symmetric axis of the asymmetric ring becomes smaller which means the domain walls in the asymmetric ring move easier. This easier of the movement of the domain wall corresponds to the earlier switch in the loop when applying same magnetic field. With increment of asymmetric ratio, the sensitivity of the domain wall to the magnetic field increases. The influence of magnetic field on the domain wall has the same trend as what was reported elsewhere^{143,144}.

Fig. 4.9 Simulated hysteresis loops of the asymmetric magnetic ring for various asymmetric ratio of $R=0.1$, 0.2 , and 0.3 . Other geometrical parameters of the asymmetric rings were arbitrarily chosen as $D_o=600$ nm, $D_i=300$ nm, and $t=16$ nm.

While numerical simulation provides information about the evolution processes of the magnetization in asymmetric ring nanomagnets, it is important to understand how the reversal mechanism is affected by the shape anisotropy^{145,54,146}. For a thin film magnetic nanoring, the magnetization lies in the plane of the film because of the shape anisotropy. The total energy is thus the sum of the exchange and the

magneto-static terms¹⁴⁷ and the spin configurations is determined by the minimization of the sum of the energy terms. For the asymmetric rings in this work, the energy minimum for annihilated transverse domain walls will be at point A in Fig. 4.4. Therefore when the initial field along BD decreasing, two transverse domain walls will be generated at BD with a large energy slope. In near remanence, the two domain walls will tend to slide towards the energy minimum A to lower the energy of the whole system. We can neglect the effect of magneto crystalline anisotropy energy since it plays no significant role as compared to the magneto-static term. The simulated energy diagram in Fig. 4.9 shows when the domain walls begin to move, the energy decreases. As the magnetic field increases, the Zeeman energy will dominate instead of the magneto-static energy, which explains why the domain wall propagates across the wider arm of the asymmetric ring and the wall annihilates.

Fig. 4.9 Diagram of simulated exchange energy and magnetostatic energy during the magnetic switching.

4.6 Direct observation of DW trapping

In order to directly observe of the DW trapping behavior and the motion of a

360° domain wall in asymmetric rings, Magneto optical kerr effect magnetometry was employed to measure the hysteresis behavior of the rings. Shown in Figure 4.10 and 4.11 are normalized hysteresis measurements of the asymmetric NiFe nanorings, for different film thicknesses measured by magneto-optical Kerr effect (MOKE) magnetometry.

Fig. 4.10 Normalized MOKE hysteresis behaviour measured on ASRs with thickness of 20 nm superimposed with simulated hysteresis loop. The loop shows a-triple-switch behavior with one plateau at low field and two small plateaux at high field regions.

For all measurements, the field was applied along the asymmetric axis of the ring (AC). Also shown in Fig 4.10 and 4.11 are the simulated hysteresis loops and the corresponding spin configurations, which were obtained from OOMMF simulations. The simulated hysteresis loop of a 20-nm-thick asymmetric ring, is characterized by a three-step plateau as shown in Fig. 4.10. The first plateau, results from a large increase in magnetization at 80 Oe, due the formation of a vortex configuration, where the spins are oriented circularly around the circumference, as seen from the corresponding spin state. The plateau extends until a field of around 200 Oe. The

second plateau occurs at a larger field of 250 Oe, close to the saturation field of the asymmetric ring. From the simulated spin configuration, we note that this is due to the nucleation of a local vortex wall in the wide arm region of the asymmetric ring. The switching process thus is via negative saturation-‘onion’-vortex-vortex wall at wide arm and positive saturation. The MOKE hysteresis measurement of the 20-nm-thick asymmetric ring reveals a gradual switch in magnetization in the field range of 80-150 Oe. This switch is associated with the formation of the vortex state in the ring structure, similar to the plateau region shown in the simulations. The magnitude of the MOKE signal intensity at the first switch I_{MOKE}^1 is smaller than that at the second switch I_{MOKE}^2 . The fraction of magnetization can be expressed by a phenomenological relation $I_{MOKE}^1 / I_{MOKE}^2 = 0.50$, which is close to the simulated ratio $I_{Sim}^1 / I_{Sim}^2 = 0.57$. The magnitude difference of the switch reflects the selective domain wall movement, where the domain wall will always move towards the narrowest part of the ring arm. Since the domain wall energy¹⁴⁸ $E \approx 2\pi A \log(\frac{ewt \log(w/t)}{\pi\lambda^2})$ (where λ is the magnetic length, and w, t are the ring width and thickness) is dependent on the ring arm width w , the nucleated domain walls will move selectively to the narrow arm of the asymmetric ring. This domain wall selective movement can be used to control the chirality of the vortex state in the asymmetric ring.

Shown in Fig. 4.11 is a normalized hysteresis loop of the 10-nm-thick NiFe asymmetric nanorings measured by magneto-optical Kerr effect (MOKE) magnetometry. The field was applied along the x -axis. The simulated hysteresis loop is characterized by two major steps of different magnitudes. The plateau which results from an increase in magnetization at 0 Oe, is due to the formation of a domain wall trapping vortex configuration. The spins are oriented circularly around the circumference of the ring, with a 360° domain wall at the narrow arm, as seen from the simulated spin state. The plateau extends until a field of around +200 Oe. The 360° domain wall trapped at the narrow arm does not annihilate until a large reversal

field of +300 Oe is applied. The small step at larger field region is due to the annihilation of the trapped domain wall at the narrow arm. The MOKE hysteresis measurement of the asymmetric ring reveals a gradual switch in magnetization in the field range of +50 Oe to +130 Oe. This switch is associated with the formation of the 360° domain wall trapping vortex state in the ring structure, similar to the plateau region seen in the simulation. The magnitude of the MOKE signal intensity from the first switch I_{MOKE}^1 is smaller than that of the second switch, $I_{MOKE}^1 / I_{MOKE}^2 = 0.55$ which is close to the ratio, obtained from the simulation: $I_{Sim}^1 / I_{Sim}^2 = 0.6$.

Fig. 4.11 Normalized MOKE hysteresis loop for a 10nm thick $\text{Ni}_{80}\text{Fe}_{20}$ asymmetric rings superimposed with simulated hysteresis loops. Insets show the corresponding simulated spin configurations.

The magnitude difference of the switch reflects the selective transverse domain wall movement, when the ring evolves from the onion to the domain-wall trapping state. Since the domain wall energy¹⁴⁸ is dependent on the ring arm width, the transverse wall in the onion state, will always move to the narrow arm of the

asymmetric ring. This domain wall selective movement can be used to control the chirality of the vortex state in the asymmetric ring. The chirality of the vortex configuration is determined by the initial direction of the magnetization along the wider arm of the ASR during saturation; when the wider arm has spins along the $+x$ ($-x$) direction, the resulting chirality of the vortex will be clockwise (anti-clockwise). It is worth pointing out that due to the small magnetic moment of the trapped domain wall, our MOKE measurement was not able to resolve the presence of the small step at high field prior to its annihilation.

To directly observe the magnetic states and the chirality control features in the asymmetric ring structure during the reversal process, we have carried out magnetic force microscopy (MFM) imaging. A lift scan height of 50 nm was used for all the measurements. Shown in Fig. 4.12 are the representative MFM images and the corresponding simulated states at remanence for the asymmetric ring.

Fig. 4.12 MFM images and simulated configurations of (a) clockwise and (b) anti-clockwise vortex states with the 360° domain wall at the narrow of the 10nm thick $\text{Ni}_{80}\text{Fe}_{20}$ asymmetric ring, when the external field is 0 Oe.

By applying a large saturation field along the $+x$ (or $-x$) direction and reducing to 0 Oe, two states with bright and dark contrast vortex were observed. The dark and bright contrast corresponds to clockwise and anti-clockwise chirality of the vortex respectively. In both cases a DW is trapped at the narrow arm. The MFM measurement confirms the domain wall trapping feature in the asymmetric ring predicted by our simulation.

4.7 Field effect on the trapped DWs

To study how the vortex chirality affects the stability of the 360° DW in the ASR, the magnetic states were imaged as a function of a sweeping in-situ magnetic field.

Fig. 4.13 MFM images of the field driven domain wall motion in the clockwise domain wall trapping vortex configuration, as the in-situ magnetic field is applied in the $+x$ axis. The corresponding simulated spin configurations are shown in the lower panel.

Starting from the bright/dark contrast vortex state, the in-situ magnetic field was

increased along the $+x$ direction. For the clockwise vortex state, the domain wall at the narrow arm remains trapped as the external field is increased up to +100 Oe. Fig.4.14 shows the MFM image of the asymmetric ring array when the in situ field is +100 Oe.

The dark contrast at the narrow arm corresponds to the trapped domain wall. As the in-situ magnetic field is increased to +200 Oe, we observed the formation of a second domain wall at the wider arm, as seen in Fig.4.14.(b). Increasing the field to +300 Oe, leads to the annihilation of the domain wall at the wider arm while the trapped domain wall at the narrow arm can still be observed, as seen as the dark contrast at the narrow arm in Fig.4.14(c). Simulation reveals that when the chirality is clockwise, applying a magnetic field in the $+x$ direction does not annihilate the trapped domain wall. Instead, an additional domain wall is nucleated at the wide part of the ring. As the reversal field is further increased to +300 Oe, the domain wall at the wide arm annihilates leaving the persistently trapped domain wall at the narrow arm. The simulation results are in good agreement with our MFM observation.

For the domain wall trapped in anti-clockwise vortex, the external magnetic field influences the trapped domain wall more significantly.

Applying a small reversal field of +30 Oe disrupts the trapped domain wall and an ‘onion’ state is formed. The dark contrast in the MFM image in Fig 4.15(a), measured with an in-situ field of +30 Oe, corresponds to the two domain walls in the ‘onion’ state. When a reversal field of +50 Oe is applied, the asymmetric ring array exhibits a low contrast. The micromagnetic simulation shows that when the vortex state is anti-clockwise, a field of +30 Oe breaks the trapped 360° domain wall into two transverse walls.

Fig. 4.15 MFM images of the 360° domain wall motion, trapped within a vortex configuration with anticlockwise chirality in the 10nm Ni₈₀Fe₂₀ asymmetric ring as a function of in situ magnetic field applied in the $-x$ axis. The lower panel shows the corresponding simulated spin configurations.

The separated domain walls move apart and forming a tilted ‘onion’ state. When the reversal field increases to +50 Oe, the asymmetric ring array adopts a negative saturation as shown in Fig.4.15.(b). From the MFM scanning and the simulation, the stability of the domain wall trapped in the vortex state is strongly dependent on the chirality of the vortex.

4.8 Summary

A technique to trap a domain wall at the exact position in asymmetric magnetic ring is proposed. By controlling the geometry of the nonmagnetic ring, simple and reproducible switching can be obtained. The switching field dependency of permalloy asymmetric magnetic rings were studied by means of micromagnetic simulations.

When the thickness is controlled to a range, a pair of head-to-head and tail-to-tail domain walls can be trapped at the thinner arm of the asymmetric ring. The vortex circulation can be also controlled using asymmetry. The phase diagram of the switching behavior through the asymmetric rings by different regimes and domain wall trapping is presented as a function of ring thickness, inner diameter and outer diameter. It is found that the boundary between different switching regimes depends much more on the thickness than the other two geometric parameters. The sensitivity of the domain wall nucleated at the symmetric axis of the asymmetric ring is reducing with decreasing of the asymmetric ratio. Such domain wall trapping feature has potential application for magnetic memory devices¹⁴⁹.

Further detailed studies on the domain wall trapping behavior and the field effect on the trapped domain wall in an asymmetric magnetic ring were performed. By analyzing our experimental observations with the aid of micromagnetic simulation, we have been able to identify that a 360° domain wall is trapped at the narrow arm when the ring adopts the vortex configuration. We found that the domain wall stability is strongly dependent on the vortex chirality of the asymmetric ring. This non-geometric defect domain wall trapping behavior in the asymmetric ring structure can be useful in the applications of domain wall devices.

Chapter 5

Highly stable flux-closure state in asymmetric rings structures

In this chapter, we report on the direct observation of the switching behavior of sub-100 nm $\text{Ni}_{80}\text{Fe}_{20}$ asymmetric ring. We have found that in the simulated hysteresis behavior of sub-100 nm asymmetric ring structures, a highly stable flux-closure state is formed during the magnetization reversal process when the field is applied along the asymmetric axis of the structure. Experiment demonstration shows that such flux-closure state does not annihilate until a large reverse magnetic field is applied, which is typically larger than 1000 Oe. Micromagnetic simulations results show that the flux-closure persists in the structures with a lateral size of as small as 30 nm. Such phenomenon is uncommon to any reported nanomagnets where a single domain state is usually obtained when the size shrink significantly. The high resistance of the vortex state to external field is possibly due to the high configuration stability in ASR.

5.1 Introduction

The hard disk drives areal density increased more than 100% per annum during the late 1990s¹⁵⁰. This development, however, appears to be moribund since mid-2006. The increases in the performance metrics, e.g. data density, data rate, have generally been achieved by making the read-write head smaller, the medium thinner and higher in coercivity, and the head-medium spacing smaller. By doing so, density about 1 T bit/in² is estimated. Due to the noise originating from the granular microstructure of the thin film, the performance of the medium is limited. As a result, decreasing the grain size is a necessary trend. However, this trend will eventually be limited by thermal instability (superparamagnetism¹⁵¹) of the grains comprising the medium. When the grain size becomes too small, thermal energy will be sufficient to reverse the magnetization spontaneously, which is predicted¹⁵² to occur at densities of 6–15 Gbit/cm², causing the loss of the recorded signal

To increase the recording density of hard disk drives (HDDs) beyond that of the current unpatterned perpendicular magnetic media, patterned media technologies^{153,154} including bit patterned media (BPM)^{155,156,157,158,159,160} and discrete track media (DTM)^{161,129,162,163} are being developed. For a patterned media system to be viable, significantly higher data densities than a conventional hard drive must be achieved. Using longitudinal media, the hard disk is expected to reach a density of 15 Gb/cm², so the patterned media designs must be capable to reach a density beyond. This implies a periodicity of 100 nm or smaller. Fabricating and understanding the magnetism of the Nanometre scale magnets are thus of vital importance. In the particles, due to the competition of the magnetic exchange energy and the magnetostatic energy items, the magnetic behavior of the magnetic nanostructures are highly size dependent. For small particles, the exchange energy term dominates, and the particles have uniform or near-uniform single-domain magnetization states,

whereas large particles show multidomain magnetization states at remanence. The balance between the exchange coupling and magnetostatic energy determines the size at which a particle becomes single domain. Particles are expected to be uniformly magnetized when the dimensions are near exchange length. As the dimensions increase, the magnetization distribution becomes less uniform, different magnetic states such as flower states or magnetization vortices can develop, depending on the shape and anisotropy of the particle.

Nanoscale magnetic circular disks¹⁶⁴ and rings⁵¹ have been identified as potential recording elements for next generation storage media due to the merits of simple and reproducible switching mechanisms⁴⁹. Towards achieving ultrahigh density recording, it is potentially required to shrink the size of the magnetic elements¹⁶⁵ to sub-100 nm range. The study of size-dependent magnetic switching process in thin film patterned disk structures has been well established¹⁰⁷. For instance, when a circular disk with radius above a critical value, the magnetization adopts a vortex state due to the large demagnetizing field induced by dipole interactions. When the lateral dimension is smaller than this critical value, the significant increase of the exchange interaction between magnetic dipoles renders the decrease in magnetostatic interaction, making single domain state more favorable energetically. In another aspect, the displacement and polarity switching¹⁶⁶ of vortex core in magnetic circular disk can lead to irreproducible switching. This size effect and irreproducibility can limit the disk applications as a recording element in magnetic memory devices. Removing the vortex core in the disk forms a ring vortex, and the vortex state is found to be relatively more stable^{167,96,65}. However, with the ring size reduction, two issues arise. First, the switching mechanism in the rings becomes non-controllable due to the high configuration stability¹⁶⁸ in sub 100 nm range, *i.e.* the key transition in the magnetization reversal of a symmetric ring can be either onion-vortex-reverse onion or onion-reverse onion without forming a vortex state¹⁴⁸. Second, the vortex state in the sub-100nm rings is only stable in the field range of tens of Oe. From application viewpoint, the stability of the vortex state is critical for use as a recording medium. Moreover, the ability to manipulate the vortex

state with a determined chirality is also important. In this chapter, we demonstrate a consistent formation of vortex state that can be obtained by controlling the external field direction in sub-100nm asymmetric ring. The vortex state is highly stable with a reversal field of 1200Oe. Additionally, the chirality of the vortex state can be controlled via field history due to induced geometric asymmetry. Further calculation reveals that the controllable vortex state is sustainable in asymmetric ring with a diameter as small as 30 nm.

5.2 Fabrication of sub100 nm asymmetric ring

Asymmetric ring-shaped $\text{Ni}_{80}\text{Fe}_{20}$ (NiFe) nanomagnets were fabricated using electron beam lithography and lift-off processes. Magnetron sputtering technique was used to grow a Si/Ta (5 nm)/NiFe (8 nm)/Ta (5 nm) structures. The Ta films were used as buffer and protection layers for the NiFe. The sputtering pressure was maintained at 3.5 mTorr and the base pressure was better than 3.0×10^{-8} Torr. Shown in Fig.5.1 is SEM image of an array of asymmetric ring-shaped nanomagnet.

Fig. 5.1 Scanning electron micrograph of an array of NiFe asymmetric ring. The outer diameter D_o of the asymmetric ring is 100 nm, and the inner diameter D_i is 50 nm.

The outer diameter (D_o) of the fabricated ring structure is 100 nm while the inner diameter ($D_i = 0.5D_o$) is 50 nm. The asymmetry was induced by offsetting the center of the inner circle by a distance, l ($= 0.1 D_o$) of 10 nm, with respect to the center of the outer circle. The spacing between the rings was maintained at 200 nm.

5.3 Field Orientation Dependent Measurements

Shown in Fig. 5.2(a) is the normalized hysteresis loop of the NiFe asymmetric nanoring structure arrays measured by magneto-optical Kerr effect (MOKE) magnetometry at room temperature with fields applied along the y axis.

Fig. 5.2(a) Normalized MOKE hysteresis behavior measured on an array of NiFe asymmetric ring with fields applied along the y axis. D_i and D_o is 50 nm and 100 nm respectively; thickness t is 8 nm. Only partial graph from field -3000Oe to 3000Oe was shown, since 3000Oe already saturates the magnetization in the ASR structure.

A large field of 6000Oe is applied along the $+y$ direction to saturate the magnetization. When the field is decreased to a value of +50 Oe, a sharp switch in the magnetization occurs. On reversing the field to the negative direction, the magnetization switches at -50 Oe before achieving negative saturation. The sudden switch at the relatively low field produces a near-square loop. This is an indication of the spins rotate coherently. As the field is reducing from total saturation state, the spin is expected to align along the ring shape, *i.e.* an onion configuration. Hence, the magnetization changes from onion to reverse onion, without having any intermediate state. For fields applied along the x axis, a multi-jump hysteresis loop is obtained, as shown in Fig. 5.2(b).

Fig. 5.2(b) Normalized MOKE hysteresis behavior measured on an array of NiFe asymmetric ring with fields applied along the x axis.

As the field strength is reduced from $+x$ saturation, the magnetization decreases gradually until a critical field of +100 Oe, corresponding to point A in the loop. On reversing the field to -300 Oe, the magnetization jump rapidly corresponding to

region AB in the loop. The observed magnetization jump is characteristic of the movement of transverse domain walls towards the narrow part of the asymmetric ring¹⁶⁹. Since the domain wall energy^{148,170} is dependent on the width of the ring arm, the narrow arm of the asymmetric ring forms an energy well for the domain walls. Consequently, all domain walls nucleated during the switching process move to the narrow arm of the asymmetric ring. On further increasing the magnetic field in the negative direction, the magnetization gains monotonically in the field region $-300 \text{ Oe} \sim -700 \text{ Oe}$ which is marked as region BC. Following that, the magnetization shows a plateau-like condition (region CD), indicating a stable state is present in the asymmetric nanoring. This plateau-like region signifies the formation of a vortex configuration, where the magnetization vectors align along the edge of the structure. As the field is further increased in the $-x$ direction an abrupt jump in magnetization leads to negative saturation (E).

5.4 2D simulations on the field orientation dependent switching mechanism

In order to aid our understanding of the reversal process in the sub-100 nm asymmetric ring, micromagnetic simulation was carried out with the field conditions corresponding to those in Fig. 5.2(a) and 5.2(b).

The magnetization configurations and switching properties were obtained by using the OOMMF¹³⁹ simulation program. The parameters used were similar to the previous work¹²¹ except that the chosen cell size here is 1 nm. To model the reversal process for fields applied along the y -direction, a large field of 6000 Oe was applied along the $+y$ direction to saturate the magnetization. When the field is decreased close to +300 Oe, a positive onion spin configuration is formed, with two domain walls nucleated at the symmetric axis and the spins align along the ring shape.

Fig. 5.3(a) Simulated hysteresis loops of NiFe asymmetric rings ($D_0 = 100$ nm, $t = 8$ nm) and their spin configurations (insets). A near-square loop indicates a spin switching via coherent rotation when the field is applied along the y axis. A fully saturated loop is shown in the inset.

Upon reducing the field, the magnetization gradually decreases and then switches abruptly at a field close to zero. The magnetization then progressively increases as the field is increased in the negative direction and reaches negative saturation. The simulated spin configuration confirm our interpretation of the MOKE measurement that the asymmetric ring structure adopts a switching process that all spins rotate coherently from the configurations of negative onion to positive onion without going through intermediate states. The simulated M-H loop for fields applied along the x axis is shown in Fig. 5. 3(b).

Fig. 5.3(b) Simulated hysteresis loops of NiFe asymmetric rings ($D_0 = 100$ nm, $t = 8$ nm) and their spin configurations (insets). A multi-jump loop indicates a spin switching via the formation of a vortex state when the field is applied along the x axis. Insets are MFM images scanned with in situ magnetic field. The dotted circles are inserted as reading guidance.

When the field is reduced, a significant decrease in magnetization is observed, spreading a field distribution of around 300 Oe (A'B'). This is followed by an abrupt magnetization drop (B'C') at a critical field of -1000 Oe. Our simulated magnetic configurations have revealed that the magnetization drop in the A'B' region is due to the movement of two transverse domain walls. The transverse domain walls are nucleated at asymmetric axis forming the onion state, and move to the narrow part of the asymmetric ring to form a 360° domain wall. The B'C' abrupt switch is caused by the annihilation of the 360° domain wall leading to a vortex state as shown in Fig.5.4.

Fig.5.4 Annihilation of the 360 degree domain wall in simulations

On the other hand, the spin orientation of the wide arm remains unchanged, aligning to the $+x$ direction. The interesting feature of this finding is that the chirality of the vortex state, which is determined by the spin orientation of the wide arm, can be decided by the saturation field direction. On increasing the negative field, the magnetization reaches a quasi-stable plateau of C'D', and then another sharp switch at E'. The sharp switch at D'E' is attributed to the reversal of magnetization at the wide arm of the asymmetric ring. The C'D' plateau region extends more than 1500 Oe and the simulated spin configurations show a stable vortex state is present. Thus, the switching process is a transition from onion to reverse onion state via the formation of the vortex state. The measured MOKE hysteresis loops are qualitatively confirmed by the simulation. Some minor differences such as the simulated coercivity and saturation field are relatively larger than the measured values. These are possibly attributable to thermal fluctuations and switching field distribution. As thermal fluctuation is not taken into considerations in the micromagnetic simulation, the switching fields tend to be overestimated. The broadening of the switching field in the BC region and the slanted measured loops are due to the distribution of the switching fields in the measured asymmetric nanoring samples.

In order to observe the magnetic configurations directly at the above mentioned regions, magnetic force microscopy (MFM) scanning with *in situ* magnetic field was carried out. The MFM images are shown in the insets of Fig. 5.3(a) and 5.3(b). A lift-scan-height of 50 nm was used for all the scanning. A large magnetic field of -3000 Oe was initially applied along the y or x axis to magnetically saturate the asymmetric ring structures; the field was then reduced to -300 Oe to induce a magnetic state. When the field is in the y axis direction (symmetric axis), MFM image shows that an onion state with a domain wall at the narrow and wide arm, respectively, was obtained. When the field is applied along the x axis (asymmetric axis), a relatively low contrast MFM image with a black region in the narrow arm was obtained, which is corresponding to the domain wall at the narrow arm. When the field is increased to $+750$ Oe, a lower contrast MFM image indicating a vortex state was obtained. The MFM results confirm that the plateau in our MOKE hysteresis loop was due to the formation of the vortex state.

To further investigate the magnetic configuration at smaller scales, systematic simulations were carried out on asymmetric nano rings of different sizes ($D_o = 30, 50$ and 70 nm, $D_i = 0.5D_o$ and $l = 0.1D_o$).

Fig. 5.5 Hysteresis loops simulated as a function of diameter and thickness of the NiFe asymmetric ring. For each loop the horizontal axis is field and the vertical axis is normalized magnetization. The field is applied along the x axis. The results show that the vortex state remains in the asymmetric ring with a diameter as small as 30 nm.

The chosen thicknesses t is 8 nm, 12 nm and 16 nm. Representative of simulated hysteresis loops of the ring structures as a function of D_o and t is shown in Fig.5.5. The results can be categorized into single-switched and double-switched hysteresis loops. The double-switched loop is typified by the 100 nm asymmetric ring loop with field applied along the x axis, which corresponds to the switching process via the formation of a vortex state. The double-switched loop is observed in rings with thickness larger than 12 nm and a lateral size as small as 30 nm. The single-switched loop is shown in 8-nm and 12-nm thick asymmetric ring of 30-nm- D_o , showing single domain characteristics. Moreover, the saturation field of the single-switched loop is in

the range of tens of Oe.

In order to study the effect of the asymmetry on the switching process of the ring structure, we performed the micromagnetic simulations on the switching process of symmetric rings with same lateral size and thicknesses. Shown in Fig.5.6 is the simulated hysteresis loops as a function of diameter and thickness of the NiFe symmetric ring.

Fig. 5.6 Hysteresis loops simulated as a function of diameter and thickness of the NiFe symmetric ring. For each loop the horizontal axis is field and the vertical axis is normalized magnetization. The field is applied along the x axis.

Firstly, unlike in the asymmetric rings, the flux-closure state can only survive in the symmetric ring with thickness larger than 12 nm. Secondly, in symmetric rings with lateral size smaller than 50 nm, the magnetization switches via a process of

onion -reverse onion. Compare the switching diagram of the symmetric ring with the asymmetric ring, one can find two differences. These two different switching properties shown are mainly due to two reasons. First, in the larger ($D_o > 30$ nm) and thicker ($t > 16$ nm) asymmetric rings, the introduction of asymmetry into the ring structure has caused the configuration stability of the formed onion state is smaller than that in the symmetric ring. Second, the domain wall energy well in the narrow arm has induced a selective movement of the domain walls of the onion state, resulting the vortex state more favorable. When the lateral size and film thickness in the asymmetric ring shrink further ($D_o < 30$ nm, $t < 12$ nm), the significant increase of the exchange energy renders the vortex state unfavorable.

5.5 Conclusions

In conclusion, combining MOKE measurement, micromagnetic simulations and MFM scanning on sub-100 nm asymmetric rings, we have revealed that the switching process is field orientation dependent. When the external field is applied along the y axis, the magnetization switches via a key process of onion-reverse onion with all spins rotate coherently, whereas that of x axis, the magnetization switches via a process of onion-vortex-reverse onion. The observed intermediate vortex state is of high stable and the chirality of the vortex state is controllable. The vortex state is persistently existent even when the ring diameter approaches a small lateral dimension of 30 nm. The highly stable state and its chirality control feature in the asymmetric ring holds promising applications in magnetic memory and recording devices.

Chapter 6

Magnetisation reversal in nanotriangles

This chapter presents the magnetization reversal behavior of sub 100-nm triangular shaped $\text{Ni}_{80}\text{Fe}_{20}$ dot array fabricated by nanosphere lithography. Hysteresis loops measured by magneto-optical Kerr effect magnetometry are classified into single- and double-switched loops in 45-nm, 80-nm and 100-nm triangular nanomagnets. Micromagnetic simulations show that a plateau observed in the double-switched loop was due to the formation of a metastable mediating V state.

6.1 Introduction

With nanotechnology becoming widely used, there is an increasing demand for rapid, parallel fabrication strategies for nanoholes and nanopillars. Some applications that require repetitive uniform nanoholes and nanopillars over large area are photonic crystals¹⁰⁰, memory devices¹⁰¹, nanofiltration¹⁰², solar cells¹⁰³, etc. Of all the applications, towards delaying the onset of thermal effect and superparamagnetic limit in the ultrahigh density recording media, patterned magnetic thin film nanostructures are intensively investigated^{70, 104}. For optimum performance of the patterned storage medium, understanding and controlling the magnetic properties of individual and interacting elements at reduced dimension are of key importance. Different patterned magnetic nanostructures like rectangles¹⁰⁵, dots⁴⁶ and rings¹⁰⁶ have been studied. Due to the requirement of the density increase in the developing recording technology, the magnetization reversal mechanisms in sub-100 nm sized patterns have recently attracted a lot of attention. In nanomagnets with size in this sub-100 nm range, the shape anisotropy becomes a key issue to the magnetization reversal. Using conventional lithography techniques, such as electron beam lithography¹⁰⁷, interference lithography¹⁰⁸, as well as x-ray lithography, magnetic reversal process in patterns of hundreds nm scale have been fabricated. However, the conventional lithography techniques are limited in resolution by the wavelength of light (λ) used and the system numerical aperture (NA), giving a minimum feature size of $\lambda/2NA$.

Fig. 6.1 schematic illustrations of numerical apparatus

Besides, in the application sense, finding lithographic processes which can pattern the thin films into nanoscale patterns at an economically acceptable cost is also a challenge. A variety of nanofabrication technologies have been developed to prepare large-area 2D or 3D colloidal patterned surfaces, such as self-assembly, spincoating, electric-field-induced electrokinetic flowing, and Langmuir-Blodgett deposition^{109,110,111,113,171}. In particular, self-assembly technique, which forming ordered periodic arrays of packed spherical configuration with uniformly sized microspheres, has been applied widely because of its unique features: it is inexpensive, inherently parallel, and enables high-throughput nanofabrication. Of all the appealing approaches to capitalize on the self-ordered structures found in nature, nanosphere lithography (NSL) is a well-known, low-cost, and quick preparation method, which makes use of self-assembly of nanospheres to fabricate nanostructure and nanostructure arrays. This technique is always used for production of periodic particle arrays and it strictly requires the nanospheres to form a perfect hexagonal closed monolayer. Original NSL has limited pattern design. Only triangular-shaped metal nanoparticles can be directly obtained from deposition through monolayer and double layer of close-packed nanospheres. However, by using shadow NSL as well as

reactive ion (plasma) etching (RIE), various shapes of nanostructures, such as triangular, hexagonal, rings, and chains, have been obtained by different ways through NSL. Shadow NSL is a combination of monolayer-preparation of PS spheres with tilted shadow evaporation¹⁷², while RIE is widely used to extend the close-packed spherical colloidal patterns based on self-assembly into nonclose-packed nonspherical patterns. Using NSL, Niu et al¹⁷³ demonstrated that the nanoscale magnetic dots with the size approaching the superparamagnetic limits could have an enhanced coercivity. Magnetic asymmetric rings are also fabricated via NSL assisted with oblique angle ion beam etching by Zhu et al¹⁴⁸ to study the switching process. In this work, we demonstrate a simple and inexpensive method to fabricate periodical nanotriangles masks based on original NSL. The fabricated masks were then used for the deposition of magnetic thin film patterns and further study of magnetic properties.

Monodispersed nanospheres with diameter of 465 nm, 365 nm and 200 nm in suspension of 2.6 wt% in water, surfactant-free, were diluted by an equal amount of ethanol. About 5 μ L of the prepared solution was pipetted onto the surface of a 3 cm 3 cm large clean silicon wafer. The Si substrates were pre-cleaned with acetone followed by ethanol cleansing. The silicon wafer was first kept in 10% dodecyl sodium sulfate solution for 12 hr, later the wafer was slowly immersed into a glass vessel which is filled with deionized water. The PS spheres then form a monolayer on the water surface. The monolayer was then lifted out from the water surface using another piece of clean Si substrate, and dried in air at room temperature.

The fabricated monolayer nanosphere arrays were then used as a template to create magnetic patterns. The Si/Ta (5 nm)/NiFe (8 nm)/Ta (5 nm) structure was deposited onto the template using electron beam evaporation technique. The deposition pressure was maintained at 2×10^{-6} torr, while the growth rate was kept at 2 Å/s. After the deposition, the nanospheres were lifted-off by immersing the template in a CHCl₃ solution, assisted by ultrasonic agitation. To confirm the formation of the triangular nanomagnets, the sample was imaged by scanning electron microscope

(SEM). Shown in Fig.6.1 are the SEM images of the triangular nanomagnets of different lateral dimensions.

Fig.6.2 SEM image of the fabricated triangular nanomagnets

The SEM image shows that the nanomagnets are equilateral and have uniform shape and size over a large area. Using PS nanospheres of different sizes (465 nm – 200 nm), equilateral nano-triangles of lateral sizes of 100 nm, 80 nm and 45 nm were obtained. The area comprised by uniformly distributed triangles with no connecting film is found to be of 20-30 μm , which is close the size of the beam spot of in our focused longitudinal MOKE measurement ($\sim 30 \mu\text{m}$). The size of each triangle is in the range of 80%-120% of the original design. All the measurements were carried out at room temperature.

6.2 Magnetic properties of the magnetic nanotriangles

Shown in Fig.6.3 are typical room temperature MOKE hysteresis loops (a, b and c) for the 15- nm-thick NiFe triangular nanostructures of different lateral sizes (L). During the measurements, an in-plane magnetic field was applied along the base direction of the nano-triangles.

Fig. 6.3 Typical room temperature MOKE (a, b and c) hysteresis loops for the 15nm thick NiFe triangular nanostructures of different lateral sizes (L) with an in-plane applied magnetic field.

The open symbols represent the magnetic signal from the triangular. The

magnetization loops of the triangular nanomagnets are markedly sensitive to the lateral dimensions. For $L = 100$ nm shown in Fig.6.3 (a), several features are observed in the loop. When the field is reduced from saturation condition, a two-step magnetization switching was observed. The magnetization switches at a low field of around +20 Oe, resulting a small plateau of ΔH_v with a field range of around 50 Oe. The presence of the plateau indicates the formation of an intermediate state during the switching process. Further increasing the field along the positive direction, a major jump in magnetization leads to reverse saturation. The coercivity of the 100 nm triangle is found to be 115 Oe. As the lateral dimension is reduced to 80 nm, the hysteresis loop shows a single- switched property as shown in Fig.6.3(b), which is a characteristic of coherent switching process that the magnetization reverses without any intermediate state. The measured coercivity is found to be larger, *i.e.* 120 Oe. When L is further reduced to 45nm, a near rectangular-shaped hysteresis loop is observed as shown in Fig.6.3(b), except that the coercivity value has increased to 200 Oe, as shown in Fig. 6.3(c). The signal-to-noise ratio is relatively low due to the small magnetic volume. The significant increase in the coercivity of the nano-triangle arrays as the dimensions are reduced is attributed to the change in the reversal mechanism mediating the reversal process in the nanomagnets. In order to obtain a better understanding of the reversal process in the different triangular nanomagnets, we used the OOMMF code¹³⁹ to carried out micromagnetic simulations . The parameters used in the simulations are saturation magnetization $M_s = 860 \times 10^5$ A/m, exchange stiffness constant $A = 1.3 \times 10^{-11}$ J/m, anisotropy constant $K = 0$ J/m³, and chosen cell size is 2 nm. The simulated hysteresis loops for dimensions corresponding to Fig. 6.3(a), 6.3(b), and 6.3(c) are shown in Fig. 6.4(a'), 6.4(b') and 6.4(c'), respectively.

Fig. 6.4 The simulated hysteresis loops for triangular nanomagnets with dimensions correspondingly

The shape of the measured MOKE hysteresis loop is well reproduced by the simulation. For $L = 100$ nm nano-triangles, a two-step hysteresis loop is obtained which confirms the intermediate state in the reversal process. The nanotriangles with lateral dimensions 80 nm and 45 nm are characterized by a single switching process, resulting in a rectangular loop which is also in accordance with our measurements. These single switching loops reveal that there are no metastable states during the switching process. Thus, the reversal mechanism of the 45 nm and 80 nm triangles is dominated by the coherent rotation process. The simulated coercivity and saturation field are relatively larger than the measured values, possibly because of the thermal

fluctuations factors are not taken into considerations in the micromagnetic simulations, resulting overestimated switching fields.

To aid our understanding of the spin reversal process in the nanotriangle, the simulated spin configuration corresponding to different conditions in the measured hysteresis loop are extracted. Shown in Fig.6.5 are the spin states for the nanotriangle with $L = 100$ nm, where the fields corresponding to the spin configurations are labeled on the simulated hysteresis loop.

Fig. 6.5 Captured spin states for the nanotriangle with $L = 100$ nm.

When the field is reduced from negative saturation the spins near the edge of the triangles begin to deviate from the field direction, with a tendency to align along the edges (I). On reversing the magnetic fields the positive direction, the spins located close to the bottom of the triangle remain unswitched. However, the spins located at the top corner rotate towards the perpendicular direction of the field. Consequently, a symmetric V-like spin configuration forms in the nano-triangle with the opening of the V opposite to the field direction (II). By increasing the field along the positive direction, the center of the V state sweeps towards the bottom (III). These processes

cause the first switch in the hysteresis loop. Further increasing the field in the positive direction, a plateau extending 100 Oe was observed in the simulated hysteresis loop. A relatively stable state was observed with top corner spins aligning perpendicular to the field direction, while those of bottom base aligning along the field direction, forming a V state with the opening at the bottom (IV). As the field is increased to positive saturation, the spins in the nano-triangle begins to rotate towards the field direction, resulting the second switch in the hysteresis loop. The spin configuration was a reversed V state (V). When the field is increased to a large positive value, the spin configuration reaches a positive saturation state (VI).

MFM imaging was carried out to observe the magnetic states of the triangular nanomagnets. MFM measurement on the 100 nm triangle was performed in the phase detection mode. A lift scan height of 50 nm was used for the scanning. Before scanning, the sample was magnetically saturated by applying a field of +1.5 kOe along the major axis, the field was then reduced to 0 Oe. Shown in Fig. 6.6 is the MFM image of the remanent state of the 100 nm triangular nanomagnets.

Fig. 6.6 MFM image of the triangular nanomagnets

From the MFM image, the magnetization configurations can be classified into three categories which are bright contrast combined with dark edge, dark contrast combined with bright edge and half dark and half bright triangles. These three states

are present in the enlarged MFM scanning image which shows a unit block of the triangles. Correlated the MFM images with the simulated spin states, the different contrast may indicate the presence of either a negative saturation or a positive saturation. While the half dark and half bright contrast stands for the V state. The small percentage of the V state observed in the MFM scanning are in agreement with the switching field distribution among the nanomagnets which corresponds to the gradual decrease in the magnetization before switch in the hysteresis loop of the 100 nm triangles

6.3 Conclusions

In summary, arrays of sub-100 nm NiFe nanoscale triangle were fabricated by using nanosphere lithography techniques and their properties were characterized by MOKE magnetometry. MOKE measurements show that with the increase of the lateral dimension of the triangle, the effect of the coercivity enhancement decreases. Micromagnetic simulation shows that the reversal mechanism of the 45 and 80 nm triangles is dominated by the coherent rotation process, while that in the 100 nm triangle is via the formation and rotation of an intermediate V-like state.

Chapter 7

Structural, Magnetic and electrical Properties of Reactive-sputtered Fe₃O₄ Thin Films

Thin films of magnetite (Fe₃O₄) are grown on a single-crystal Si/SiO₂ (100) substrate with native oxide using DC reactive sputtering technique at room temperature (RT) and 300°C. The x-ray diffraction (XRD) result shows the thermal energy during deposition enhances the crystallization of the Fe₃O₄ and x-ray photoelectron spectroscopy confirms the film deposited at 300°C is single-phase Fe₃O₄ while the film deposited at RT is mostly γ -Fe₂O₃. The electrical measurements show that the resistivity of the Fe₃O₄ film increases exponentially with decreasing temperature, and exhibits a sharp metal-insulator transition at around 100 K, indicating the Verwey transition feature. The saturation magnetization M_s of Fe₃O₄ film measured by vibrating sample measurement (VSM) at RT was found to be 445 emu/cm³.

7.1 Introduction

Electrical spin injection and detection has been demonstrated between ferromagnetic and normal metals¹⁷⁴ and in semiconductor/ magnetic semiconductor heterostructures¹⁷⁵. Due to the potential applications in various spin-based devices, the spin injection and detection between ferromagnetic metal and semiconductor has attracted great research effort recently. However, to achieve high efficiency spin injection from a ferromagnetic metal into a semiconductor is difficult. One particular obstacle is the conductivity mismatch between metals and semiconductors, which drastically limits the spin injection efficiency for ohmic contacts between metals and semiconductors¹⁷⁶. For nonohmic contacts, spin injection can be obtained by tunneling through a barrier at the semiconductor interface¹⁷⁷ or by using a magnetic tunnel transistor emitter.

An alternative route for spin injection is to use a half metallic ferromagnet, which has a completely spin-polarized conduction band, as the spin injector. As perfectly spin polarized sources, the conductivity mismatch between half metallic ferromagnetic material and semiconductor is circumvented even for ohmic contacts. In the case of tunneling, the high polarization of half metal gives a much larger spin accumulation in the semiconductor compared to 3d transition metal injectors. Among the possible half metallic compound such as Fe_3O_4 , $\text{La}_{0.7}\text{Sr}_{0.3}\text{MnO}_3$ and CrO_2 , Fe_3O_4 is candidate for future spintronics devices due to the high Curie temperature¹⁷⁸(TC) of 858K and exhibition of full negative spin polarization¹⁷⁹ at room temperature. Unfortunately the growth of Fe_3O_4 ferromagnets onto semiconductors tends to be more difficult than ferromagnetic transition metals, and very often the spin polarization is determined more by disordered interfacial layers than by the bulk properties of the ferromagnet itself. Furthermore, perfect spin polarization is a zero temperature property. At finite temperature, the two spin states are mixed to some extent. Nonetheless, Fe_3O_4 has a number of unusual properties (the 100% polarization

and high T_c etc) make it attractive as a spin polarized source. Consequently, the fabrication and properties of Fe_3O_4 films are of vital importance. In order to obtain optimal performance in the applications, Fe_3O_4 thin film growth with high quality is required. Different techniques have been used such as: molecular beam epitaxy¹⁸⁰, pulsed laser deposition, and sputtering¹⁸¹. Various substrates such as MgO, $MgAl_2O_4$, $SrTiO_3$, sapphire and Si have been used to study the influence of the lattice mismatch between the magnetite and the substrates. Results show that single crystalline thin films can be epitaxially grown on MgO(100) substrates, due to the good lattice match between the oxides^{182,183,184}. In the perspective of incorporating the half metallic ferromagnet and semiconductor, polycrystalline Fe_3O_4 thin films have also been deposited on Si substrates to study the properties¹⁸⁵. Besides Fe_3O_4 , other iron oxides, Fe_2O_3 , FeO, and some nonstoichiometric oxides, have been reported to be present in the deposited films, depending on the deposition conditions¹⁸⁶.

<i>Technology</i>	<i>Target</i>	<i>P, Pa</i>	<i>Ts, °C</i>	<i>Substrate</i>	<i>References</i>
PLD	$\alpha-Fe_2O_3$	3×10^{-1}	350	Si	JAP 83 (1998) 7049
	$\alpha-Fe_2O_3$	$<1 \times 10^{-1}$	350	MgO(100)	PRB 57 (1998)7823
	$\alpha-Fe_2O_3$	$<1 \times 10^{-1}$	350	$SrTiO_3$ (100)	
	$\alpha-Fe_2O_3$	$<1 \times 10^{-1}$	350	$\alpha-Al_2O_3$	
	$\alpha-Fe_2O_3$	$<1 \times 10^{-1}$	570	$SrTiO_3$	
	$\alpha-Fe_2O_3$	1×10^{-3}	350	MgO(100)	PRB 64 (2001)205413
	Fe_3O_4	10^{-4}	350	MgO(100)	
RF-MS	Fe_3O_4	10^{-2}	250	MgO	PRB 80 (2002)823
RF-MS	Fe_2O_3	Ar: 5×10^{-3}	400	MgO(001)	JAP104(2008) 013913
DC-MS	Fe	O2: 4×10^{-2} Ar + O2: 2	400	MgO	JAP106(2009) 033908
DC-MS	Fe	Ar: O2 27:6.8 1Pa	300	Si(100)	This work

Fig. 7.1 Preparation of Fe_3O_4 thin films in various methods

Fe_2O_3 is brown or reddish brown and hence can be easily distinguished from the other oxides. The FeO and Fe_3O_4 , which are black, are difficult to differentiate. However, the crystal structure of both of the later oxides is based on the fcc Bravias Lattice with lattice parameters a 0.8936 nm for Fe_3O_4 and 0.4307 nm for FeO . This makes XRD an important technique in the characterization of the Fe oxide. In this work, considering the compatibility with most of the devices, magnetite thin films were deposited on surface of SiO_2/Si using DC reactive magnetron sputtering. The Verwey transition¹⁸⁷ feature indicated in the electrical measurements shows that high quality Fe_3O_4 thin films have been produced.

7.2 Sample fabrication

Fe_3O_4 films were grown by DC magnetron sputtering on thermally oxidized Si(100) substrate at a process pressure of 3×10^{-3} Torr. Prior to deposition, the Si(100) substrates were first rinsed in acetone, 2-isopronal and de-ionized water. A buffer layer comprising of 5 nm thick Ta layer was then grown on the substrate. By referring to the existing work in the literatures, the Fe_3O_4 films were reactively sputtered from pure Fe (99.99%) target in an Ar- O_2 (27:6.8) mixture, with the substrate maintained at room temperature (sample A) and 300C (sample B), respectively. The base pressure was better than 5×10^{-7} Torr. The film thickness grown was kept at 60nm for both set of samples. Structural characterization of the deposited samples was carried out using conventional θ -2 θ X-ray diffraction (XRD) and X-ray photoelectron spectroscopy (XPS). The magnetic and transport measurements were conducted using vibrating sample magnetometer (VSM). For electrical measurements, contacts were made using standard optical lithography, metallization and lift off of 10 nm Cr/ 150 nm Au. The resistance was recorded using a two terminal method. Low temperature measurements were performed using a

physical measurement system (PPMS-Quantum Design).

7.3 Structural characterization of Fe_3O_4

Fig. 7.2(a) Typical θ - 2θ XRD pattern for Fe_3O_4 film grown onto substrate maintained at RT.

Shown in Fig. 7.2(a), is the typical θ - 2θ XRD pattern for sample A, Fe_3O_4 film grown onto substrate maintained at RT. The XRD pattern displays a single distinct Fe peaks in the (440) orientation, indicating that the polycrystalline Fe_3O_4 film has a preferred growth along the 110 orientation. We attribute this preferred orientation to the columnar growth structure of the film due the sputtering deposition technique¹⁸⁸.

Fig. 7.2(b) Typical θ - 2θ XRD pattern for Fe_3O_4 film grown onto substrate maintained at 300°C

The XRD pattern for sample B, on the other hand, displays four Fe oxide peaks along the (311), (511), (220) and (400) orientations, as seen in Fig. 7.2(b). Interestingly, the (440) peak, as seen in sample A, is totally suppressed. The additional peaks, as seen from the XRD patterns, indicate a structural change in the Fe_3O_4 film grown at 300°C . This may be due to the thermal energy from the substrate, promoting different film growth mechanisms.

The reactive sputtering of Fe with Ar and O_2 plasma may result in the formation of either or both of the two stable Fe oxides, namely $\gamma\text{-Fe}_2\text{O}_3$ and Fe_3O_4 . These two Fe oxides, share the same spinel structure, and as such cannot be distinguished in XRD measurement. To differentiate between the Fe_3O_4 and $\gamma\text{-Fe}_2\text{O}_3$ that may have formed during the reactive sputtering process, we conducted XPS scan on Fe2p using MgK radiation at 1256.3 eV. The Fe_{2p} spectrum of $\gamma\text{-Fe}_2\text{O}_3$ exhibits a satellite peak

at 719eV, while the Fe_{2p} spectrum of Fe_3O_4 is demarcated by a shoulder at 709 eV. The XPS scans for both samples, A and B, are shown in Fig. 7.3

Fig. 7.3(a) Fe XPS spectra for samples deposited on Si/SiO₂ at RT

From Fig. 7.3(a), we observed that the XPS of the sample A displays both a shoulder at 709eV and a peak at 717eV. This implies that the film grown on sample A may comprises both the formation of Fe_2O_3 and Fe_3O_4 state during growth. For Sample B, the peak at 717 eV is absent, as seen in Fig. 7.3(b), implying that the film grown on sample A consists of only one oxide of Fe, Fe_3O_4 . The shift of the peak from 719 eV may possibly due to the formation of other phase of iron oxide in sample

Fig. 7.3(b) Fe XPS spectra for samples deposited on Si/SiO₂ at 300°C

7.4 Temperature on the magnetic properties

To better understand the magnetic properties of the samples, we carried out magnetization loop measurement using VSM. The room temperature magnetization loops of the two sets of samples, measured with magnetic field applied parallel to the film plane, are presented in Fig.7.4. The insets show the normalized M-H loops for both sets of samples.

Fig. 7.4 Hysteresis loops measured at 300K for samples deposited at RT and 300°C

From Fig.7.4, we can see that sample B exhibits a much larger saturation magnetization of 445 emu/cm³, as compared to sample A, 134 emu/cm³. Fe₂O₃ is known to be diamagnetic and the contribution to the overall magnetization is negligible. As such, the total magnetic moment for both samples A and B are solely from the Fe₃O₄ in the film. The drastic increase in the saturation magnetization, for sample B, is due to the suppression of Fe₂O₃ phase formation, when the substrate is

maintained at 300°C. Interestingly, the saturation magnetization for sample B, is close to the value of a single crystal Fe_3O_4 , which is reported to be 471 emu/cm³,⁸². We attribute the high saturation magnetization of our film to the good structural quality. Another reason for the enhancement of the magnetization of Fe_3O_4 is possibly due to the thermal energy contributing to the crystallization of the Fe_3O_4 . This in turn reduces the thickness of amorphous Fe_3O_4 layer which is formed at the surface of the substrate due to the lattice mismatch between the substrate and the Fe_3O_4 . As seen from the inset in Fig. 7.3, sample A exhibits a much larger saturation field of 2000 Oe as compared to that of sample B, which is only 500 Oe. The high saturation field of the film A is possibly due to formation of thick amorphous layer at the substrate.

7.5 Effect of growing temperature on the electrical properties of the Fe_3O_4 film

We have further carried out transport measurement, on the samples. Shown in Fig. 7.5, is the resistivity of both sets of samples as function of temperature. For both sample A and B, there is an exponential increase of resistivity as the temperature is reduced.

Sample A display a room temperature resistivity of ~120K. When the temperature is reduced below 270 K, there is a gradual increase in resistance with decreasing temperature. For sample B, room temperature resistivity is 0.005292 Ω cm. As the temperature is reduced below 200K, we observed a gradual increase in resistivity with temperature. At 100K, a sharp increase in resistivity is observed, indicating a metal-insulator transition, a clear signature of the Verwey transition feature. The temperature discrepancy of the Verwey transition from that of bulk Fe_3O_4 , is due to cation vacancy¹⁸⁹ and the nonstoichiometric¹⁹⁰ deviation.

Fig. 7. 5. Electrical resistivity of a 60-nm-thick sputtered Fe_3O_4 film grown on Si/SiO₂ substrates plotted vs T. The Verwey transition can be clear seen in the film deposited at 300 °C but absent in the film deposited at RT.

While for the film A, the resistivity increases significantly and the Verwey transition is absent. It is possibly due to the formation of $\gamma\text{-Fe}_2\text{O}_3$ which makes resistivity decrease and the Verwey transition smeared out in the curve as compared to B. The Verwey transition and relatively high magnetization in the film B show we have successfully fabricated the Fe_3O_4 at RT on Si substrate using sputtering technique.

7.6 Conclusions

In summary, we have grown Fe_3O_4 thin film on thermally oxidized Si substrate maintained at different temperatures. The film was grown using DC reactive sputtering technique, where a Fe target was sputtered in Ar+O₂ plasma. We observed

that the sample with substrate maintained at 300°C, has a very large magnetic moment of 445 emu/ cm³. This was attributed to the suppression of the Fe₂O₃ phase, due to the substrate temperature promoting the crystallization of Fe₃O₄. Our transport measurement reveals a clear signature of Verwey transition at 100K, for sample grown at 300°C. The presence of the Verwey transition indicates that our Fe₃O₄ thin film is of very good structural quality. The abnormal electrical and magnetic properties of Fe₃O₄ sputtered at room temperature, in particular the non-saturation of magnetization even at high field, may be caused by the amorphous layer at the substrate.

Chapter 8

Summary and Future work

8.1 Summary

This thesis carried out a comprehensive investigation of the effect of various patterns and geometrical parameters on the magnetization reversal process of a soft magnetic thin film (NiFe). Ordered homogeneous nanomagnets were fabricated using electron beam lithography technique as well as nanolithography technique. The effect of geometrical parameters such as film thickness, lateral dimensions as well as asymmetric ratios was systematically studied. Magnetic information about the magnetic properties of the nanomagnet arrays was obtained using a home built longitudinal magneto optical kerr effect magnetometry. The magnetic pattern arrays are designed to be big due to the low resolution. The spin configurations of the nanomagnets were imaged using magnetic force microscope with in situ magnetic field. The major contributions made during the course of this study can be summarized as follows:

Firstly, a technique to trap a domain wall at the exact position in asymmetric magnetic ring is proposed. Detailed studies on the domain wall trapping behavior and the field effects on the trapped domain wall are performed both numerically and experimentally. Simple and reproducible switching was obtained by controlling the

geometry of the nonmagnetic ring. When the thickness is controlled to a range, a pair of head-to-head and tail-to-tail domain walls can be trapped at the narrow arm of the asymmetric ring. The phase diagram of the switching behavior through the asymmetric rings by different regimes and domain wall trapping is presented as a function of ring thickness, inner diameter and outer diameter. The trapping behavior is found to be dependent mainly on the thickness of the asymmetric ring. The sensitivity of the domain wall nucleated at the symmetric axis of the asymmetric ring is reducing with decreasing of the asymmetric ratio. For asymmetric ring with a larger asymmetry ratio, a smaller magnetic field is needed to annihilate the trapped domain walls. By controlling the geometry of the magnetic nanoring, a nucleation free switching process can be obtained. A 360° domain wall is trapped at the narrow arm when the ring adopts the vortex configuration. Moreover, the domain wall stability is strongly dependent on the vortex chirality of the asymmetric ring. This non-geometric defect domain wall trapping behavior in the asymmetric ring structure can be useful in the applications of domain wall devices. Such domain wall trapping feature has potential application for magnetic memory devices. Most importantly, it is possible to control the domain wall position without having to create defects or notches in the nanoscale ring.

Secondly, we revealed that the switching process is field orientation dependent through the study of MOKE hysteresis loops, micromagnetic simulations and MFM scanning on sub-100 nm asymmetric rings. A highly stable and chirality controllable flux-closure state is formed when the reversal field is applied along the asymmetric axis of asymmetric ring. The flux-closure state can be still formed when the diameter approaches 30 nm. The consistent presence of the flux-closure in this range in asymmetric ring is due to the asymmetry in the ring structure, which can cause the nucleation of the domain walls at the ring arm. Making use of the chirality sense of the flux-closure state, the asymmetric ring holds a promising application in memory devices.

Thirdly, the study of domain wall trapping in asymmetric magnetic rings was extended to sub-100 nm triangles NiFe triangle arrays, which were fabricated using NSL. The MOKE measurement and micromagnetic simulations were carried out to investigate the reversal mechanism of the arrays. Enhancement of coercivity compared to the thin film was observed in all the three arrays but in different degree from the MOKE measurement. With the increase of the lateral size of the triangle, the effect of the coercivity enhancing decreases. Micromagnetic simulation shows that instead of domain wall nucleation and annihilation in the thin film, the reversal mechanism of the 45 and 80 nm triangles is dominated by the coherent rotation. While in the 100 nm triangle, the magnetic reversal takes place via forming and reversing a V like state. The direct observation of the V state, positive and negative saturation in the 100 nm triangle array shows a good agreement between the experimental and micromagnetic simulations

Finally, we have grown Fe_3O_4 thin film on thermally oxidized Si substrate maintained at different temperatures. The film was grown using a Fe target in ArO_2 plasma, using DC reactive sputtering. The purpose of this study is to fabricate a kind of material with high spin polarization and can be possibly used in the spin injection devices. We observed that the sample with substrate maintained at 300°C , has a very large magnetic moment of 445 emu/cm^3 . This was attributed to the suppression of the Fe_2O_3 phase, due to the substrate temperature promoting the crystallization of Fe_3O_4 . Our transport measurement reveals a clear signature of Verwey transition at 100K, for sample grown at 300°C . The presence of the Verwey transition indicates that our Fe_3O_4 thin film is of very good structural quality.

8.2 Future work

In this thesis, a few novel findings in large area nanomagnets have been reported. There are still various promising avenues to explore the magnetic properties

of the patterned magnetic thin films. Since I am the first PhD student in the group, I have contributed significant part of my time in lab construction, equipment calibration and maintenance. Although in my work the experiments have been designed carefully and the study was carried out as systematically as possible, there are still a lot of further works to be carried out. Here, several key aspects are recommended for future research. The recommendations are divided into two parts.

For the domain wall trapping in the patterned asymmetric rings, an alternative approach such as MR which is already built up by me may be adopted to measure the field effect on the trapped DWs. A single asymmetric ring may be fabricated and aligned with electrodes. Fig.8.1 gives out a picture of metallic contacts aligned on an asymmetric ring.

Fig. 8.1 Illustration of MR measurements.

Through wire bonding, the device can be located in a chip carrier. Different kinds of domain walls may be injected into the structures by controlling the geometric parameters of the asymmetric ring. By applying magnetic field in a proper direction and then releasing, the domain wall will sit in desired location. Both the magnetic

field and DC current will be used to depin the magnetic domain wall. The transportation of the domain wall can be investigated using MR combined with the MOKE. The current can be applied programmable by choosing the pin of the chip carrier. The resistance may be calculated.

For the Fe_3O_4 ,

Different substrates such as GaAs (100) and (111) may be used as substrates for the thin film deposition. The electron transport and magnetic property measurement may be carried out to characterize the thin film quality. After successful deposition of Fe_3O_4 on the semiconductor substrates, spin injection devices using Fe_3O_4 as spin aligner may be fabricated. Nonlocal measurements such as lateral spin valve may be carried out using the home built MR system. The lateral spin injection device used for this study is going to be fabricated on a semiconductor channel substrate by means of the lithography and conventional lift-off techniques. Fig 8.2 gives out the schematic illustration of the nonlocal lateral spin valve.

Fig. 8.2 Schematic illustration of the nonlocal lateral spin valve.

The device consists of two magnetic wires bridged by a normal metal wire. The spin injection will be evaluated by nonlocal spin-valve measurement. The two magnetic wires are deliberately patterned into different width so as to facilitate the control of the magnetization configuration (parallel or antiparallel) by adjusting the magnetic

field. Different magnetic material and distance will be used to study the spin accumulation and spin diffusion.

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Appendix

MOKE control GPIB with lock with clusters in 0515.vi

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Connector Pane

MOKE control GPIB with lock with clusters in 0515.vi

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Front Panel

The front panel displays two large plots at the top. The left plot has a y-axis from 0.00000 to 1.50000 and an x-axis from -1.6 to 1.4. The right plot has a y-axis from 0.00000 to 1.50000 and an x-axis from -1.50 to 1.50. Below the plots is an 'update' button and a 'GPIB Address' field set to 12.

The main control area includes the following sections:

- Field Parameters:** +ve Field (kG): 2.00, -ve Field (kG): -2.00, Field Scale: -7.10000, No. points: 200, Averages: 1, Delay (ms): 0. Field ramp: linear. Range (0: 1 - Lowest): 1 - Lowest, 0. Overload button.
- Input Configuration:** Input Mode: A (Single-Ended Voltage). Input Connector Shells: Float, Ground. Coupling: DC, AC. Line Frequency Rejection Filter: OFF. Line Frequency: 50 Hz, 60 Hz. Input Device (Voltage Mode): Bipolar - Input Impedance 10 k, FET - Input Impedance 10 M.
- Output Offsets:** X Channel Offset (%): 0.00, Y Channel Offset (%): 0.00. Auto Offset button.
- Sensitivity:** Range: 500mV. Auto Sensitivity. Auto Measure. Auto AC Gain. AC Gain: 0dB. Phase Degrees: 0.030.
- Reference Channel - Single Reference:** Reference I/P: Internal. Reference Harmonic: 1. Oscillator Amplitude (Volts rms): 0.500. Oscillator Frequency (Hz): 1000.000. Reference Phase: -50.0 to 50.0, -100.0 to 100.0, -150.0 to 180.0. Auto Phase. +90 degrees. Degrees: 0.00.
- Output Channels:** Time Constant: 100ms. Slope: 12dB/octave. Synchronous TC: On, Off. Virtual Reference Mode.
- Save/Sequence:** Save? File name: T_072_010_10mA_15kOe#. Next sequence: 1. Ramp points: 50. Total Ramp Time (ms): 5000. STOP button.

At the bottom, there are two error status boxes:

- error in (no error):** status: no error, code: 0, source: [empty field].
- error out:** status: no error, code: 0, source: [empty field].

MOKE control GPIB with lockin with clusters in 0515.vi

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Block Diagram

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