Spin-dependent Electron Transport in Nanomagnetic Thin Film Devices

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Abstract: Spin-dependent Electron Transport in Nanomagnetic Thin Film Devices

Yun Zhou

Spin-dependent electron transport in submicron/nano sized magnetic thin film devices fabricated using the optical lithography, e-beam lithography and focused ion beam (FIB) was investigated with the primary aim to find the ballistic magnetoresistance (BMR) in thin film nanoconstrictions. All experimental results were analysed in combination with micromagnetic simulations. The magnetisation reversal processes were investigated in a submicron half-pinned NiFe stripe with a microconstriction. An asymmetric MR curve was observed, and micromagnetic simulations verified it was due to the exchange-bias on the left side, which changed the magnetic switching mechanism. The effects of different pinning sites on the magnetisation switching and domain wall displacement were studied in NiFe film and spin-valve based nanodevices. A sign of domain wall MR was seen on the transversal MR curve of the NiFe nanodevice due to the domain wall induced electron scattering. The size effect on the magnetisation switching and interlayer magnetostatic coupling was demonstrated and characterised in synthetic antiferromagnet (SAF)-pinned spin-valve nanorings. It has been clarified by micromagnetic simulations that these nanorings exhibit a double or single magnetisation switching process, which is determined by the magnetostatic coupling as a function of the ring diameter. The interlayer magnetostatic coupling was efficiently reduced in large SAF-pinned nanorings, resulting in a small shift of the
minor MR curve, which is beneficial to the magnetic memory applications. In-situ MR measurements and the investigation of domain wall properties have been carried out in FIB patterned NiFe film nanoconstrictions. Spin-valve like sharp transitions were observed on the MR curves in the 80 nm/130 nm wide nanoconstriction devices. However, our analysis of the results by micromagnetic simulations and domain observations with scanning electron microscopy with polarisation analysis (SEMPA) concluded that these sharp MR transitions originated from the anisotropic magnetoresistance (AMR) effect, due to the fast magnetisation rotation in the nanoconstriction, and not from BMR. The numerical investigation has proved that a further reduction of the constriction width/length is necessary for large MR values.
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Author’s Declaration

At no time during the registration for the degree of Doctor of Philosophy has the author been registered for any other University award without prior agreement of the Graduate Committee.

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Relevant scientific seminars and conferences were regularly attended at which work was often presented; external institutions were visited for the purpose of research cooperation and several papers prepared for publication.

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Chapter 1 Introduction and Motivation

1.1 Introduction

Basic research in the physical sciences can result in important developments in engineering, technology and commercial production.

In the past two decades, some exciting discoveries have revolutionised the magnetic recording industry. The most prominent examples are the giant magnetoresistance (GMR) and tunneling magnetoresistance (TMR) effects. These effects are related to the electron spin, which is the core of an emerging field known as spin electronics (or spintronics). The GMR device, which employs spin-dependent electronic transport in magnetic thin films, is the key technology for present computer hard disk drives (HDDs). While the TMR effect in magnetic tunnel junctions (MTJ), employing spin-dependent tunneling, is critical to the development of magnetic random access memory (MRAM).

The development of future high capacity mass-storage devices will need highly sensitive materials and devices with exceptional spin transport properties, which is characterised by a large MR ratio.

In this thesis we study issues of critical importance to the development of future spintronic data storage devices, such as large magnetoresistance (MR) effects at room temperature, including ballistic magnetoresistance (BMR) and domain wall MR.

1.2 Project Objectives

Since the discovery by García et al [1] of 200% magnetoresistance in mechanically formed wire nanocontacts, BMR in ferromagnetic point contacts has presented new
prospects of technological applications in information storage and spintronic devices. Further experiments with mechanically adjusted and electrodeposited magnetic nanocontacts improved the result up to 500-700% at room temperature [2-5]. Very large room temperature MR values of up to 500% were also confirmed in mechanically formed half-metallic oxide nanocontacts (Fe$_3$O$_4$ or CrO$_2$) [6-7] or oxide-metal hetero-nanocontacts (Ni-CrO$_2$) [7]. The large BMR effect is attributed to non-adiabatic spin scattering across nanometre sharp domain walls trapped at nanoconstrictions [2, 8-9]. Bruno has shown that atomically sharp domain walls can form in point contacts [10], giving rise to the non-adiabatic nature of spin scattering and a very large BMR at room temperature.

However, very recent experimental studies by Chopra et al. [11] of nano-sized Ni contacts, in which the reproducible BMR as large as 3000-4000% and even 100000% [12] was obtained, but cannot be explained by the existing theories, have raised new questions about the origin of such huge BMR values.

Thus more studies are certainly required to clarify the mechanism of the large BMR effect. The form and the width of the domain wall at the nanocontact (and the dependence of BMR on these factors) depend on the geometry of the nanocontact itself and remain to be investigated. Furthermore BMR results reported so far have been limited to mechanically formed or electroplated wire nanocontacts, so the exploration of BMR in thin film nanocontacts is very necessary. Since the beginning of this project, some results related to thin film nanocontacts have been reported by other researchers [13-17].

Aiming to integrate the complementary knowledge, infrastructure and expertise for the exploration of the spin dependent ballistic transport properties in thin film nanocontacts,
the consortium of our BMR project ("ballistic magnetoresistance in thin film nanocontacts") consists of five European leading institutes/universities: Consejo Superior de Investigaciones Científicas (CSIC), Spain, Centre for Research in Information Storage Technology (CRIST), University of Plymouth, Central Microstructure Facility (CMF) of Rutherford Appleton Laboratory (CCLRC), IAP of University of Hamburg (IAP) and Kazan State University (KSU). My PhD research is a main part of the BMR project.

The goals of our project are to employ state-of-the-art nanofabrication technology for the fabrication of thin film nanoconstrictions, and to carry out leading-edge research of spin dependent ballistic electronic transport in relation to physical sizes, micromagnetic structures, and domain wall motion and magnetoresistance in the vicinity of the nanocontacts. The aim is to achieve a better understanding of the observed spin-dependent transport mechanism.

1.3 Organisation of Thesis

The theme of this thesis is an experimental study of the spin-dependent electron transport properties in thin film nanocontacts, including both single-layer ferromagnetic and multilayer structures.

Chapter 2 gives a brief review of the general background, including the theory of spintronics and the basic principles of spintronic devices. A review is also given of the historical development and the state-of-the-art work carried out in this field.

Chapter 3 presents the experimental methods and micromagnetic simulation software used in this research. Section 3.1 describes the experimental methods, which includes thin-film deposition, microfabrication and nanofabrication techniques, and several
device characterisation methods. Section 3.2 briefly introduces two types of micromagnetic simulation software packages, which have been used for the micromagnetic modelling in the following chapters.

Chapter 4 covers all the experimental results along with micromagnetic simulations and relevant analysis. Section 4.1 describes the magnetisation reversal with the domain wall motion in both standard submicron NiFe stripe and half-pinned NiFe stripe with a microconstriction. Further related research in NiFe and spin-valve based nano-sized devices with different artificial pinning sites is presented in section 4.2. Section 4.3 gives a systematic description of the magnetoresistance, domain wall motion and the magnetic switching in NiFe nanorings, and the size effect on the magnetisation switching and interlayer magnetostatic coupling in spin-valve nanorings. Section 4.4 reveals that spin-valve like sharp transitions were observed on the MR curves in in-situ magnetotransport measurement of thin NiFe film nanocontacts using FIB nanofabrication. Further analysis was carried out by the micromagnetic simulation and SEMPA domain observations. Section 4.5 presents current-perpendicular-to-plane (CPP) magnetoresistance study of single-layer NiFe and spin-valve nanocontacts fabricated on SiN_x membranes.

Finally in Chapter 5, some important conclusions are drawn from this work.
Chapter 2 Background

2.1 The Development of Spintronics

Spintronics (spin electronics, or magnetoelectronics) exploits the intrinsic spin of electrons in addition to the fundamental electronic charge for device applications. This research field is the combination of two traditional branches of physics: magnetism and electronics. The aim is to find ways to manipulate the spin state of electrons in the magnetotransport process for spintronic applications.

2.1.1 History

The birth of spintronics was the discovery of the GMR effect in 1988 independently by A. Fert et al. [18] and P. Grünberg et al. [19]. The origins can be traced back further to the ferromagnet/superconductor spin-dependent tunneling research pioneered by P.M. Tedrow and R. Meservey [20], and initial experiments on magnetic tunnel junctions by M. Julliere in the 70’s [21].

2.1.1.1 Anisotropic Magnetoresistance (AMR)

Magnetoresistance (MR), defined as the change of the electrical resistance as a function of an external magnetic field, is a well-known phenomenon, and can be expressed as $\Delta R/R$, as shown in Fig. 2.1 (a).

In bulk ferromagnetic conductors, the leading contribution to the magnetoresistance is the anisotropic magnetoresistance (AMR) discovered in 1857 by W. Thomson (Lord Kelvin) [22].
AMR originates from the spin-orbit interaction, which tends to induce an anisotropic scattering of the conduction electrons in the exchange split spin $\uparrow$ and spin $\downarrow$ 3d-subbands [23], leading to a dependence of the electrical resistance on the relative orientations of the magnetisation and the electric current. The change in the resistance due to the AMR effect can be described by Eq. (1.1), where $\theta$ is the angle between the current and the magnetisation (Fig. 2.1 (b)).

$$R = R_{\text{min}} + \Delta R \cos^2 \theta$$  

(1.1)

![Graphs showing AMR effect](image)

Fig. 2.1. Anisotropic magnetoresistance change as a function of (a) the magnetic field, (b) the angle between the current and the magnetisation [24].

The AMR effect has caused great interest for industrial applications, such as magnetic sensors and read-out heads for magnetic disks. The AMR effect remained the most important contribution to the magnetoresistance of ferromagnets until 1988, when the GMR effect was found.
2.1.1.2 Giant Magnetoresistance (GMR)

It was a great sensation when, in 1988, A. Fert and P. Grünberg independently discovered that a much greater magnetoresistive effect (hence called "giant magnetoresistance" or GMR) can be obtained in Fe/Cr multilayers [18] and a Fe/Cr/Fe trilayer [19], respectively. These systems essentially consist of an alternate stack of ferromagnetic (e.g., Fe, Co, Ni, and their alloys) and non-ferromagnetic (e.g., Cr, Cu, Au, etc.) metallic layers. It has led to a whole series of important discoveries and opened the door for a new field - Spintronics.

A typical GMR device consists of at least two layers of ferromagnetic materials separated by a thin non-ferromagnetic spacer layer. When the magnetisations of the two ferromagnetic layers are aligned in parallel (less scattering), they show a low system resistance; whereas if the magnetisation vectors are antiparallel (more scattering), the system is in the high resistance state. Two types of GMR have been applied in devices: (1) the current-in-plane (CIP) structure, where the electric current flows parallel to the layers and (2) the current-perpendicular-to-the-plane (CPP) structure, where the electric current flows in a direction perpendicular to the layers.

GMR can also be observed in many other systems, such as pseudo spin valves (PSV), spin valves (SV) and granular thin films. In spin valves, one of the ferromagnetic layers is "pinned" by the antiferromagnetic (AF) layer, so its magnetisation direction remains fixed and the soft magnetic layer is "free" to rotate when an external magnetic field is applied.
The most important application of the GMR effect to date is in the spin-valve sensors used in hard disk read heads, which were first introduced in 1997 by IBM. The SV sensors have replaced the AMR-based heads.

So far, The GMR values exceeding 20% in symmetric spin valves [25] and spin valves with oxide specular layers [26] have been reported at room temperature.

2.1.1.3 Tunneling Magnetoresistance (TMR)

Giant magnetoresistance effects have also been found in systems comprising a thin insulating layer sandwiched between two ferromagnetic layers, that is, the TMR effect. The resistance of such a system changes with the relative orientation of the two magnetic layers. The TMR effect is a consequence of spin-polarised tunneling, which can usually be observed in structures with various tunnel barriers, such as AlO$_x$ and MgO.

Large magnetoresistance (over 10% TMR) in magnetic tunnel junctions (MTJ) was observed at room temperature in 1995 by J.S. Moodera [27] and T. Miyazaki [28]. To date ~70% TMR has been observed in AlO$_x$-based MTJs [29] and ~600% TMR in MgO-based pseudo-spin-valve MTJs [30].

At present the most ambitious project is the use of magnetic tunnel junctions as non-volatile magnetic random access memories (MRAM). An MRAM device can read/write the information by using the TMR effect. TMR can also be utilised in the read heads in HDDs.
2.1.1.4 Spin Torque Transfer (STT)

A key to the success of the MR-based applications is to achieve a simple and efficient control of the relative orientation of two ferromagnetic layers. An interesting realisation of such control was independently proposed by Berger [31] and Slonczewski [32] in 1996. Based on the fact that in GMR structures the relative orientation of magnetisations will affect the flow of spin-polarised current, they predicted the reverse effect, which is now known as the spin torque transfer effect: a spin-polarised current driven through a magnetic multilayer creates a torque on the magnetic layers, which can lead to a magnetisation reversal even without an applied field.

2.1.1.5 Domain Wall Magnetoresistance (DWMR)

For atomic-size constrictions, a very thin (sharp) domain wall predicted by Bruno in 1999 [10], which becomes smaller than the electron mean free path, may lead to a large contribution to the resistivity, or a large domain wall magnetoresistance.

If a DW is trapped in the nanocontact region, when the magnetisations at the two sides of the nanocontact are antiparallel, the polarised electrons will not be able to adiabatically traverse a sharp domain wall, leading to strong scattering (high resistance state); while in the parallel state, the electrons do not suffer domain wall scattering, corresponding to a low resistance state.

The large DWMR expected in magnetic nanocontacts have stimulated significant interest in the magnetotransport through domain walls due to possible applications of DWMR in spintronic devices.
2.1.1.6 Ballistic Magnetoresistance (BMR)

The first so-called ballistic magnetoresistance effect of over 200% was experimentally discovered in 1999 by Garcia et al. in Ni-Ni wires joined by a nanocontact, which was then increased to 700% and 1000% at room temperature [33]. In this case ideally the size of nanocontacts is smaller than the electron mean free path, so the electrons are believed to transverse the junction ballistically without loss of spin polarisation and no scattering.

Ideally the BMR effect is a promising candidate of future high capacity information-storage devices. But the huge BMR, as high as 3000-4000% or even higher, which cannot be explained by the existing theories, requires more studies to clarify the mechanism behind it.

2.1.2 Applications

Current efforts in designing and manufacturing spintronic devices involve two different approaches.

2.1.2.1 Metals-based Spintronic Devices

The storage density of hard drives is rapidly increasing exponentially, partially because metal-based spintronic devices like GMR and TMR sensors have increased the sensitivity of the read head and the areal density of information storage.

MRAM, based on arrays of TMR or spin torque transfer (STT) devices, is non-volatile, so information is stored even when the power is off. The first commercially available
MRAM product, Freescale's 4Mb Toggle MRAM, was released in 2006.

Based on STT writing technology, STT-MRAM has the potential to replace embedded SRAM and Flash as well as DRAM with the advantages of lower power-consumption, ultra-high speed and better scalability over conventional MRAM.

Another design in development, called RaceTrack memory, which was recently announced by S. S. P Parkin as another type of nonvolatile memory [33-35], encodes information into the magnetic domain walls along a ferromagnetic wire.

2.1.2.2 Semiconductor-based Spintronic Devices

Semiconductor based spintronic devices could be much easier integrated with traditional semiconductor technology.

Although electrical spin injection can be achieved in metallic systems by simply passing a current through a ferromagnetic metal, the large impedance mismatch between ferromagnetic metals and semiconductors prevents efficient injection across the metal-semiconductor interface. A solution to this problem is to use a ferromagnetic semiconductor source (e.g. Mn-doped Ga_{1-x}Mn_xAs) [36], which increases the interface resistance with a tunnel barrier [37], or using hot-electron injection [38].

Possible applications of semiconductor-based spintronics include magnetic sensors and non-volatile magnetic memory devices with more versatile design due to the ability to adjust potential variation and spin polarisation in the device channel by external voltages, device structures and doping profiles [39].
2.1.3 Basic Theories

2.1.3.1 Concept of Electron Spin

The concept of electron spin was first hypothesised in 1925 by S. A. Goudsmit and G. E. Uhlenbeck [40-41] on the basis of two types of experimental evidence. They were the closely spaced splitting of the hydrogen fine structure and the Stern-Gerlach experiment [42], in which a beam of silver atoms directed through an inhomogeneous magnetic field would be separated into two beams, indicating two possible magnetic moments for the electron. Both of these experimental results were consistent with the possession of an intrinsic angular momentum (with two possible states: ±1/2) and a magnetic moment for an individual electron. In a classical model, this could occur if the electron was a spinning sphere of charge, which generates the magnetic moment. This reasoning leads to the concept of electron spin.

Electron spin is an intrinsic property of the electron related to its intrinsic angular momentum characterised by the quantum number 1/2. In quantum mechanics, the angular momentum of the electron is quantised and its magnitude can only take values

\[ S = \sqrt{s(s+1)} \hbar = \sqrt{3/2} \hbar \quad (s = 1/2), \]

where \( \hbar \) is the reduced Planck's constant. The resulting fine structure corresponds to two possibilities for the z-component of the angular momentum,

\[ S_z = \pm \frac{1}{2} \hbar. \]

Each electron is found in one of two spin states, which can be described as spin up (↑) and spin down (↓). Generally there is a splitting of the spin-up and spin-down energy levels via the Zeeman effect, so electrons with their spins aligned with an external
magnetic field are less energetic than those with their spins anti-aligned. While for the traditional electric circuit, spins are randomly oriented, which have no effect on the current flow.

In order to make spintronic devices, the primary requirement is to have a system that can generate a current of spin polarised electrons. Ferromagnetic materials are particularly appropriate for such devices because of the spontaneous magnetisation, which creates an imbalance of spin population at the Fermi energy level in the electronic structure. The imbalance of energy states for spin-up and spin-down electrons leads to a difference in the resistance of the two types of electrons.

The spin direction of an electron in a solid-state environment is not conserved due to spin-flip scattering on magnetic impurities. The characteristic length scale is the spin-diffusion length $\lambda$, that is, how far an electron can travel in a diffusive conductor before it flips the spin. The polarisation relaxes to the equilibrium value:

$$P(x) = P_{\text{bulk}} + \Delta P \exp\left(-\frac{x}{\lambda}\right).$$

Here $\lambda$ depends on the material and its quality (e.g. purity), and the dimensions are typically between a few nm and a few tens of nm for the ferromagnetic devices. The spin polarisation of ferromagnetic metals is defined as:

$$\alpha = \frac{\sigma^{\uparrow} - \sigma^{\downarrow}}{\sigma^{\uparrow} + \sigma^{\downarrow}}$$

($\sigma^{\uparrow}$ and $\sigma^{\downarrow}$ are the spin-up and spin-down conductivity, respectively).

2.1.3.2 Two Current Model

Generally there are different theoretical spin-polarised electron transport mechanisms, including the free electron model and the ab-initio transport calculations etc. [43]. The appropriate formalism to describe the electron transport is determined by the transport...
regime applicable to a given system, which usually depends on the characteristic physical length scale versus the size of the given system.

One important length scale for the spin-dependent diffusive transport is the spin-diffusion length. As the spin-diffusion length in metals is usually much larger than the elastic mean free path, the transport can be described in terms of two independent spin channels.

Fig. 2.2. Sketch drawing of a spin valve with two ferromagnetic (FM1/FM2) layers separated by a nonmagnetic layer: electrons with spins parallel to the magnetisation (low resistance state) are less scattered than those with spins antiparallel to the magnetisation (high resistance state).

Mott's 'two current model' [44-46] has been utilised to describe the transport in ferromagnetic materials [23, 47-49] and the transport across the F/N interface [50], and to explain the CPP GMR effect [51].

This model assumes that the conduction electrons are divided into two spin channels: those with spins parallel to the magnetisation (majority) and those with spins antiparallel to the magnetisation (minority). Scattering rates for the two spin channels are very different due to different energy states at the Fermi level. As shown in Fig. 2.2, in the antiparallel magnetic state (high resistance state), conduction electrons are much more scattered than in the parallel magnetic state (low resistance state). Thus the
mechanism of the GMR effect is related to spin-dependent (scattering) electronic transport in magnetic multilayers or granular films or, more precisely, the change in the scattering rate as the magnetic configuration changes with the external magnetic field. Spin-dependent scattering makes a large contribution to the resistance of a GMR system.

The other critical element is the density of states. Due to the exchange interaction, the two channels are very different in the density of states, leading the difference in the mobility. One of the two channels shows high-mobility, while the other shows low-mobility. When an electrical current flows in a ferromagnetic metal, it is mediated primarily by the high-mobility channel, which creates the spin-polarised current. Thus ferromagnetic materials can be used as spin-polarised current sources in spintronic applications.

2.2 Magnetoresistance Effect

This section will give an introduction to some special magnetoresistance effects, including the spin-valve GMR effect with exchange-bias, current-induced switching of layers, domain wall magnetoresistance (DWMR), current-induced DWMR, ballistic magnetoresistance, and magnetoresistance in magnetic nanorings.

2.2.1 Spin-valve GMR Effect

The GMR effect has been found in the three different systems: multilayer systems, spin-valve systems and granular systems. In spite of the very high GMR measured in antiferromagnetically-coupled magnetic multilayers (Co/Cu and Fe/Cr etc.), they are not
the best materials for technological applications. This is due to the large magnetic fields, which are required to saturate the magnetisations of multilayers and to obtain a sizeable change in the resistance. As a consequence, the sensitivity, which is defined as $\Delta R/R$ per unit magnetic field, is very small.

A search of low field GMR structures in which an antiparallel configuration of the magnetisations could be achieved in different ways, as compared to the antiferromagnetic interlayer coupling, resulted in the invention of pseudo spin valves (PSV) and exchange-biased spin valves (SV).

Pseudo spin valves (shown in Fig. 2. 3 (a)) combine hard and soft magnetic layers with different coercivities, which are separated by a non-magnetic (NM) spacer layer. That the magnetisations of the soft and hard magnetic layers switch at different fields provides a field range in which they are antiparallel, showing a high resistance state [52-54]. Typical PSV structures are always trilayers: FM/NM/FM (FM=Fe, Co, CoFe or NiFe; NM=Cu, Au or Ag). PSV structures without the antiferromagnetic layer have been studied recently for applications in MRAM devices [55-60].

Exchange-biased spin-valve is the most successful type of spin-valve GMR structure (Fig. 2. 3 (b)), which is normally composed of four layers (FM1/NM/FM2/AF): a free layer (FM1), a non-magnetic spacer layer, a pinned layer (FM2), and an AF exchange pinning layer.

The behaviour of an exchange-biased spin-valve is illustrated in Fig. 2. 3 (c-d), which show, respectively, the magnetisation curve and magnetoresistance curve measured at
room temperature, with a structure of Ni$_{80}$Fe$_{20}$/Cu/ Ni$_{80}$Fe$_{20}$/Fe$_{50}$Mn$_{50}$ [61]. Exchange-biased spin valves with the GMR value exceeding 20% have been reported [62-63].

![Schematic illustrations of a pseudo-spin-valve and a spin-valve, a magnetisation curve and a magnetoresistance curve of Ni$_{80}$Fe$_{20}$/Cu/ Ni$_{80}$Fe$_{20}$/Fe$_{50}$Mn$_{50}$ spin-valve [61]. HB is the exchange-bias field.](image)

2.2.2 Exchange Bias

2.2.2.1 Introduction

The exchange bias effect comes from the exchange coupling between a ferromagnetic layer and an antiferromagnetic layer [64], which is widely utilised to control the magnetisation in the multilayer structures, such as spin valves and magnetic tunnel junctions. In such systems, the magnetisation of one FM layer is fixed by the exchange bias (EB) at the FM/AF interface, while the other FM layer rotates freely in an external magnetic field.
The exchange bias effect has two main characteristics. Firstly the MH hysteresis of an exchange-biased FM/AF bilayer is not symmetric about a zero magnetic field as shown in Fig. 2. 4 (b), and displays an obvious offset from a zero field, which is defined as the exchange coupling field and also described as the unidirectional anisotropy ($K_E$). This leads to the energy function, $W_E = -K_E \cos \theta$, where $\theta$ is the angle between the magnetisation direction of the FM layer and the preferred direction of the exchange anisotropy. Secondly, the coercivity enhancement in the pinned FM layer is usually observed.

The exchange biasing effect is key to utilising spin-valve and MTJs for memory devices. In recent years, AF materials such as FeMn, IrMn, NiO, NiMn, PdPtMn and PtMn have been exploited as an exchange-bias layer for SV and MTJ systems. Only an introduction to IrMn and PtMn will be given here, since they are two of the best candidates for the AF layer in spin valves and MTJs.

Fig. 2. 4. Easy-axis MH hysteresises of (a) a normal ferromagnetic system and (b) an exchange-biased FM/AF system.

(a) IrMn

So far, many researchers have reported properties of the exchange bias and thermal
stability of IrMn/FM structures.

IrMn has been identified as a promising antiferromagnetic material due to its high exchange bias energy ($J_k$), high blocking temperature ($T_B$), and low critical thickness (~7 nm). IrMn has a moderate pinning field (200 Oe - 400 Oe) in the as-deposited state or after a low temperature annealing. Thus IrMn appears to be a good candidate for the AF layer in spin valves and MTJs.

(b) PtMn

The best practical antiferromagnetic material for the exchange coupling used in the industry is PtMn with a high exchange coupling field, high blocking temperature of 380 °C, even though it needs a magnetic field annealing to obtain an ordered phase.

Compared with IrMn, PtMn has better pinning properties, but requires a high temperature annealing (> 270 °C), which is to facilitate a phase transition from the non-antiferromagnetic fcc (face-centred cubic) crystalline structure to the antiferromagnetic fct (face-centred tetragonal) structure. Such an annealing may degrade the magnetic properties of other layers of the GMR structure, which becomes worse with increasing the temperature.

2.2.2.2 Main Characteristics of Exchange-biased FM/AF Bilayer:

(1) Enhanced Coercivities

For an exchange-biased FM/AF bilayer, the coercivity $H_c$ of the pinned FM layer is usually enhanced in comparison with that of the corresponding free FM layer [64]. The
$H_C$ enhancement is linked to the anisotropy of the AF layer and the interfacial exchange coupling energy. The coercivity is also affected by the microstructure of the FM layer.

(2) **Blocking Temperature**

In most systems, the exchange bias decreases linearly with increasing the temperature. The exchange bias vanishes above a critical temperature, which is often denoted as the blocking temperature $T_B$.

(3) **EB Dependence on FM Thickness**

For the majority of the systems investigated, it has been observed that exchange bias is roughly inversely proportional to the FM layer thickness, $H_E \propto 1/t_{FM}$, indicating that exchange-bias is an interface effect. However this relation is not valid for ultrathin FM layers, for which considerable changes are expected in the interface structure.

(4) **EB Dependence on AF layer Thickness**

For many systems it has been found that the exchange-bias increases with the AF layer thickness. Above a certain critical thickness, which is material dependant, the shift of the exchange bias is independent of the AF layer thickness. This exchange bias vanishes if the AF layer thickness is below a critical value, that is, the pinning thickness.

This behaviour suggests that the intrinsic anisotropy of the AF has to be much stronger than the exchange interaction between FM and AF to induce the exchange bias. This can be expressed as: $K_A d_{AFM} \geq J_{int}$. The AF anisotropy is represented by the anisotropy constant $K_A$, and $J_{int}$ describes the exchange coupling between FM and AF.
Furthermore, in other systems, a peak in the shift of the exchange bias is shown, as the AF layer thickness is decreased, before the effect finally vanishes for a very thin layer. This behaviour was theoretically predicted for the case of changes in the domain structure as the AF layer thickness is decreased [65]. On the other hand, for very thick AF layers, the EB effect has been found to decrease in some special systems. This can be attributed to considerable changes in the microscopic structure of the AF with increasing thickness.

2.2.3 Current-induced Switching of Layers

The recent discovery of the spin-transfer torque effect [32, 66-77] has attracted much attention due to the novel physics and potential application in MRAM.

It has been seen from the GMR effect that the relative orientation of the magnetisations of FM layers affects the electric current, causing different resistances for different magnetisation configurations. The reverse effect that a spin-polarised current can affect the magnetic moment of a layer has also been theoretically predicted [32, 66, 78-79] and experimentally demonstrated [68-69, 76]. In the perpendicular transport geometry, the spin-polarised current may transfer angular momentum between layers, resulting in current-driven excitations in magnetic multilayers, which can be either the magnetisation reversal, or the generation of spin waves [76]. The reversal of the magnetisation here is due to the interaction between the magnetisation and the spin accumulation in a direction perpendicular to the magnetisation.

These effects have been experimentally studied by various groups on very narrow
multilayer pillars, typically between 150 nm and 60 nm in the lateral dimension [69-73, 80-81]. The structures investigated normally consist of simple sandwiches comprising a nonmagnetic space layer separating a thick magnetic layer and a thin one (typically Co 20 nm/Cu 4 nm/Co 4 nm). The thick Co layer serves as the “fixed layer,” whereas the thin Co layer is the “free layer.” The current through each layer is preferentially carried by the majority electrons leading to a spin-polarised current. The spin-polarised current exerts torques on the layers whenever the magnetisations of the FM layers are not collinear. Depending on the polarity of the current injected perpendicularly through the trilayer, the free layer can be switched between parallel and antiparallel alignments relative to the fixed layer, resulting in a hysteretic dependence of the resistance on the current via the MR effect in low fields. In high fields, the observation of a peak in the differential resistance for only one current polarity in Co/Cu/Co trilayers and Co/Cu multilayers is generally regarded as a sign of spin precession [68-70].

In a single ferromagnetic layer without the GMR effect, the STT effect was first observed by Myers [69]. Then Ji et al. [74] observed peaks in the differential resistance with a large field applied perpendicularly to a Co film. Guided by the prevailing interpretation of experiments [70-71], such signatures in both single-layer and multilayer systems were interpreted as spin wave excitations.

The great interest in the phenomenon of the current-driven excitations in magnetic multilayers lies both in trying to understand the underlying physics and in its potential applications: magnetisation reversal for the magnetic storage and spin-wave generation for the production of high frequency radiation. For present magnetic devices, the
magnetic moments are reversed via externally generated magnetic fields, which are complex and high energy-consuming. The reading/writing processes would be much simplified by applying a polarised current through the magnetic layer itself. Generally the magnetisation reversal by spin-transfer requires a high current density, $J_c \sim 10^7 \text{A/cm}^2$.

The Co/Cu/Co CPP-GMR nanopillars show $J_c$ in the range of $10^6$-$10^8 \text{A/cm}^2$ depending on the structure, and exhibit MR ratios of 0.5%-5%. Similar research has also been done using MTJ nanopillars. The AlO$_x$-based MTJs have $J_c$ similar to CPP-GMR pillars and exhibit MR ratios of 10%-30% [82-85]. The MgO-based MTJs have been shown to exhibit a high MR ratio of 130% with $J_c$ in a wide range of $8\times10^2$-$2\times10^7 \text{A/cm}^2$ [86-90].

The current density must be reduced by at least an order of magnitude before this effect could be considered for practical applications, such as STT-RAM [91-92]. To realise the production of Gbit-scale STT-RAM devices, $J_c$ needs to be reduced to about $5\times10^5 \text{A/cm}^2$.

2.2.4 Domain Wall Magnetoresistance

A domain wall (DW) is the interface between uniformly magnetised regions (domains) with different magnetisation directions. The magnetic configuration, which consists of oppositely magnetised ferromagnetic domains separated by domain walls, closely resembles that of a magnetic trilayer, i.e. two regions of oppositely pointing magnetisations separated by a thin non-magnetic spacer (shown in Fig. 2. 5). So it was predicted that a chemically homogeneous thin ferromagnetic film might also exhibit the
MR effect.

The interplay between the electron transport through domain walls and the magnetic properties of ferromagnetic nanowires and point contacts has become a major research area. This interest has been stimulated by the investigation of the fundamental physical properties behind this phenomenon and the promising applications. It is possible to find a giant magnetoresistance at room temperature, e.g. Ballistic MR, as discussed in the previous chapter, which has the potential for applications in high-density HDD read heads and magnetic sensors. Recently Dr. Stuart S. P. Parkin announced the next-generation nonvolatile memory "RaceTrack" utilising the control of magnetic domain walls along ferromagnetic nanowires [35].

![Diagram of magnetic geometry](image)

Fig. 2. 5. Schematic illustration of the similarity in the magnetic geometry between (a) an antiferromagnetic aligned GMR trilayer and (b) a ferromagnetic domain wall.

The domain wall width in bulk ferromagnetic materials is much larger than the electron mean free path, so scattering by the domain wall is weak. As the DW becomes as small as the size of the constriction, as theoretically proposed by Bruno [10], the mean free path becomes much larger than the DW width in ferromagnetic nanocontacts, which may lead to a large domain wall magnetoresistance (DWMR).
If a DW is trapped in the nanocontact region, when the magnetisations at the two sides of the nanocontact are antiparallel, the polarised electrons will not be able to adiabatically traverse the very sharp domain wall, and will thus suffer strong scattering resulting in a high resistance. In the parallel state, the electrons do not suffer domain wall scattering, corresponding to a low resistance.

The complex mechanism of DWs in nanoscale thin film elements is not yet well understood. The exact nature of the domain wall resistance is highly controversial, and there is hardly any agreement in the magnitude and sign of the effect. Here we briefly review the important theories that predict a positive or negative contribution of the DW to the electrical resistance if the DW is thin enough compared to the Fermi wavelength of the conduction electrons.

Three widely cited models that predict a positive DWMR are those of Cabrera and Falicov [93-94], Levy and Zhang [95] and Brataas et al. [96]. The Cabrera-Falicov model of DW scattering considers the reflection of incoming electrons by the effective potential created by the rotating magnetisation (and hence an internal exchange field) within the wall. The Levy-Zhang model of DW scattering considers spin-dependent potentials and scattering rates, namely potentials and electron relaxation times that are different for spin up and spin down (majority and minority) electrons in the ferromagnets. The third model, proposed by Brataas et al., considers both ballistic and diffusive transport through a DW.

There are also models that predict an intrinsic negative DW contribution to resistance.
Tatara and Fukuyama considered the effect of the DW on weak localisation [9]. Weak localisation is pronounced in low-dimension disordered systems and arises due to quantum interference, which enhances electron backscattering and the resistivity. The second model by Gorkom et al. found that the DW could be the region of enhanced conductivity, when the electronic structure of the DW is taken into account semi-classically [97]. Kent and Ruediger have proposed another mechanism by which the interplay between the surface scattering and the electron orbital motion within domains may reduce the resistivity [98].

2.2.5 Current-induced Domain Wall Magnetoresistance

The study of domain wall magnetoresistance by use of current-induced domain wall propagation has recently been focused on ferromagnetic nanowires and stripes with constrictions [99-105]. Single layer ferromagnetic wires patterned with nanoconstrictions can be used to trap the domain wall. By varying the measurement current, the domain wall is unpinned from the constriction by current-induced domain wall motion at a critical current depending on the material used. The difference in resistance between the two states when a domain wall is present at the constriction and when it is removed is used to obtain a direct measurement of the DWMR contribution.

This effect can also be used to switch the magnetic configurations of patterned spin-valve stripes with constrictions [101, 104, 106]. By trapping a domain wall at the constriction and subsequently removing it by increasing the measurement current, the spin-valve magnetic configuration is switched between the antiparallel and parallel
states, resulting in a sharp change of the magnetoresistance. Similar work has also been reported in patterned ring-shaped structures [107].

Switching by the domain wall motion [108] induced by the spin-polarised current rather than by an external field is a promising approach to the switching of magnetic nanostructures, since it brings simple fabrication processes, combined with the possibility of achieving fast and reproducible magnetic switching [102, 106-107, 109-110].

There are three origins of the interaction between a DW and an electrical current: the hydromagnetic drag force, which arises from the Hall effect and is not significant for very thin films [111-112], the current-induced field (Oersted field) and the spin transfer by s-d interaction if the current is spin polarised. The last effect (also called current-induced domain wall drag) has been predicted by Berger [113-114], and has an origin similar to the spin transfer mechanism found in magnetic multilayered structures: electrons transfer angular momentum to the domain wall when passing through it, pushing it in the direction of the electron flow [114]. It arises from the s-d exchange interaction between the spin-polarised electrons carrying the current and the local moments. The s-d interaction exerts a torque on the spins of the conduction electrons passing through a DW and rotates the polarisation direction of the current. Inversely, the spin-polarised current exerts an s-d exchange torque on the DW magnetisation, resulting in the domain wall motion. The DW drag by spin transfer can be significant if the DW is thin enough, in which the conduction electron spins cannot completely follow the local magnetisation direction. Berger [115] and Gan et al. [116-119] have demonstrated
this effect in thin magnetic films by injecting high dc current pulses and observing the displacement of domain walls by Kerr microscope and MFM, respectively. In these experiments the order of magnitude of the current pulses needed to move the DW is \( \sim 10^7-10^8 \text{A/cm}^2 \).

### 2.2.6 Ballistic Magnetoresistance

#### 2.2.6.1 Definition of Diffusive or Ballistic Regime

If the Fermi wavelength \( \lambda_F \) or the atomic diameter becomes comparable to sizes (diameters) \( d \) of the point contacts, they are called atomic-sized point contacts. When it comes to the mean free path, we have to differentiate between elastic and inelastic scattering. Generally the elastic mean free path (\( l_e \)) is smaller than the inelastic one (\( l_i \)). Here the inelastic mean free path is the path an electron travels between two inelastic scatterings. If the contact size \( d \) is much smaller than either of the mean free paths \( d \ll l_e, l_i \), the regime is called the ballistic regime, as shown in Fig. 2.6. In this case, the electron travels through the point contact without any scattering (except for the reflection on the domain wall). On the other hand, if \( d \gg l_e, l_i \), the electron travels in the diffusive mode in the contact, and accordingly the regime is called the diffusive regime.

![Fig. 2.6. Schematic diagrams of diffusive and ballistic regimes.](image-url)
2.2.6.2 Ballistic Magnetoresistance

In the ballistic regime, a particular MR effect arises when the electrons are made to flow through 1D-like channels formed in atomic size constrictions. Recently, Garcia reported MR in Ni and Co nanocontacts of over 200%, which they later extended to 700% and 1000% at room temperature [1]. In the nanocontacts the electron mean free path is assumed to be longer than the nanocontact size. The regime of electron transport is considered ballistic, thus the MR is called "Ballistic Magnetoresistance" (BMR). Practical applications require nanocontacts to be fabricated into a planar geometry, and two different planar geometries are always used in related research, as shown in Fig. 2.

Much work has recently been done in this area [4, 120-123].

A possible explanation for the BMR effect is non-adiabatic spin scattering across nano-sized sharp domain walls. More recently, small contacts have been obtained by electrodepositing Ni between two Ni tips. Several groups have reported very large MR [11-12, 124-129] with a record value of 100000% [12]. In these systems, the contact is larger than 10 nm, thus the domain wall scattering cannot account for the magnetoresistive effect. A possible explanation is that a dead layer about 1 nm thick forms just at the contact, due to the electrodeposition process, and acts as the

![Fig. 2.7. (a) Parallel geometry and (b) perpendicular geometry used in BMR research [1]].

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non-magnetic spacer of a CCP GMR trilayer [130]. Another possibility is that some oxygen ions are trapped at the contact. According to electronic structure calculations [131], Ni ions would induce a very large local spin polarisation to the oxygen, which would therefore be responsible for the very large BMR values. These two possible explanations are controversial. Alternative mechanisms involving magneto-mechanical effects that could lead to similar results have been overlooked, which throws doubt on the accuracy of BMR in planar structures [132].

2.2.7 Magnetoresistance in Magnetic Nanorings

2.2.7.1 The Best Candidate for MRAM Application

Understanding and controlling spin configurations and magnetisation processes of nanoscale magnetic elements have received considerable attention recently. This is not only due to the fact that these structures allow for the investigation of fundamental physical properties, such as the pinning and structure of domain walls at notches or constrictions, and the MR effect associated with domain walls, but also due to the potential applications, such as MRAM, magnetic logic and sensor devices [60, 133-136]. One key issue for these applications is to understand and precisely control the magnetic switching. To achieve this, one first needs to have well-defined magnetic states, and secondly the switching process needs to be simple, fast and reproducible, and also shows a narrow switching field distribution.

Any promising MRAM devices should have a high storage density. However, a high-density arrangement enhances the effect of the stray field between adjacent memory cells, which decreases the uniformity of the switching field of the free layer.
So far, magnetic switching mechanisms of various geometries, such as squares [138-139], wires [140-141], rectangles [142-144], needles, cylinders, truncated cones and disks [137, 145-146], have been studied, but it was found that their magnetic states are hard to be controlled with complicated domain structures and switching processes, due to edge roughness and shape fluctuations.

An alternative geometry is the circular element, which forms a vortex state at remanence, which minimises the role of the edge effect. However, it was found that the vortex state is only stable for disks with diameters larger than ~100 nm, depending on the material used and the film thickness [137]. Furthermore, the formation mechanism of the vortex state is complex and hard to control, and it has a broad switching field distribution [133].

In order to avoid such effects, a special MRAM memory design was proposed based on a ring-geometry magnetic multilayer [133]. There are at least two advantages of using a ring structure. The vortex state with closed magnetic flux in the ring structure can substantially reduce the exchange energy and minimise the stray field. Therefore, elements can be fabricated very close to each other, achieving an ultrahigh density. The ring structure also has less edge roughness, leading to a narrow switching field distribution and good switching reproducibility. The critical size above which the vortex state is stable is smaller for a ring structure than for a disk structure. Therefore, higher-density MRAM can be achieved in ring structures.
Additionally ring structures are well suited for the investigation of fundamental magnetic properties, including domain wall trapping, magnetisation reversal, and the influence of the geometry and the thickness on the switching field.

2.2.7.2 Magnetisation States and Switching Processes

(1) Magnetisation States

Ring structures can be controlled to display different stable magnetisation states. In narrow magnetic rings, MR and magnetisation measurements, imaging techniques and micromagnetic simulations suggest the existence of two magnetic states [147-148]: the flux-closure “vortex” state and the “onion” state. The vortex state occurs when domain walls disappear and the magnetisation runs continuously throughout the ring circumference, and almost no stray field is generated. The onion state is characterised by 180° head-to-head/tail-to-tail domain walls.

The two different states come from the competition between the magnetostatic energy and the exchange energy, and depend on the type of magnetic materials and are a function of geometric parameters, e.g. ring shape, ring thickness, ring width, and ring diameter. In the transition between different magnetic states, the anisotropy plays an important role.

For example, narrow rings have vortex states that are more stable [149-150]. In a wide ring, a stable vortex state and a metastable near single-domain state were found during magnetisation reversal. The narrow and wide rings have distinct characteristics: for instance, the wide ring undergoes a process of local vortex nucleation before evolving
into the vortex state, whereas the narrow one reaches the vortex state without a nucleation process.

For applications such as data storage devices, the diameter of the ring has to be scaled down to achieve a high density while maintaining the magnetic characteristics. However, it has been found in the micromagnetic simulation that as the lateral size of the rings is reduced, the vortex state becomes more and more unfavourable due to the increasing curvature of the ring, which results in an increasing exchange energy. Since the stray field-free vortex state has been suggested for the application in data storage, it is highly important to establish whether the vortex state can be maintained when the lateral dimension is reduced.

(2) Switching Processes

Generally, depending on the geometric parameters (ring thickness, shape, width and diameter), rings exhibit different switching behaviours, which might be a single switching process (the onion state to the reverse onion state), a double switching process (the onion state to the vortex state and then to the reverse onion state), or a triple switching process including a vortex core state [148, 151-152].

The stability of the vortex state depends on the geometric parameters of the ring structures [152-153]. In wide and thin rings, a direct transition from the onion state to the reverse onion state is favoured. In considerably wide rings, a complex multi-step switching process with the presence of the vortex core state occurs [154].

In addition, the switching process is dependent on the pinning of the head-to-head/
tail-to-tail domain walls due to edge roughness and other defects. In order to control the switching precisely, notches are introduced as artificial pinning sites along the ring circumference. Micromagnetic simulations show not only that two possible vortex states are attained during a complete hysteresis cycle, but also that by choosing the notch size and the position as well as the applied field direction, the circulation direction of the vortex state can be controlled [147], which is either clockwise or counterclockwise. The two chiralities of the vortex state have been proposed as the carriers for the information stored in high-density MRAM devices. With notches, one is also able to tune the switching of the rings from an onion state into the desired vortex state, as required for the MRAM application.

2.2.7.3 Research Tools and Measurement Methods

Several methods have been used to measure the magnetisation reversal collectively from an array of ring structures, including the magneto-optical Kerr effect (MOKE) [147-148, 152, 155], photoemission electron microscopy (PEEM), magnetic force microscopy (MFM) and superconducting quantum interference device (SQUID). However, because of the sample-to-sample variation, the collective result does not necessarily reflect the behaviour of an individual ring. Some techniques such as AMR measurements, scanning electron microscopy with polarisation analysis (SEMPA), and Lorentz microscopy, as well as MFM and PEEM, can be used to probe the magnetic reversal of an individual ring.

The AMR measurement is a very efficient method for research on a single ring structure.
Since the MR is dominated by the AMR effect in a ring structure, a maximum resistance should be found if the magnetisation is parallel (or antiparallel) to the current. The MR becomes lowered if the local magnetisation rotates away from the direction of the current, which corresponds to a domain wall present in rings, where some magnetisation components are not parallel to the current.

AMR measurements of ring elements can be taken by making contacts to them with nonmagnetic leads. NiFe single-layer rings with diameters in the micron range and widths of 100 nm and above have been studied by AMR measurements [156-160]. In these rings the AMR shows the maximum when the ring is in the vortex state, because the magnetisation is everywhere parallel to the current direction. Transitions between the onion and vortex states cause relative resistance changes around 0.1% at room temperature [149], and the domain walls existing at remanence in single-layer rings can be positioned and detected using external fields or current pulses [161].

### 2.2.7.4 Multilayer Nanoring Structures

To date, considerable studies of the magnetisation reversal in NiFe or Co single-layer rings with outer dimensions from ~100 nm to a few microns have been carried out.

Since Zhu et al. achieved robust magnetic switching in the multilayer GMR rings with enhanced MR response [162], there has been recent interest in the magnetisation reversal in ring-shaped multilayer structures, e.g. spin-valve NiFe/Cu/Co/ErMn rings, pseudo-spin-valve NiFe/Cu/Co rings [163-170], and MTJ rings [171-172]. In the spin-valve rings, the free layer shows a switching mechanism different from that of
single-layer rings, which is caused by the strong interlayer magnetostatic coupling. As a result, they present asymmetric minor MR curves with large shifts. Although the onion state still exists, normally a vortex state in the free layer can only be observed when the reference layer is in the vortex state [163-164, 167-169], which eliminates the interlayer magnetostatic coupling.

In the ring-shaped MTJ [171] Chen et al. found several common domain states, including the onion state and the vortex state, which are similar to what they observed in single-layer NiFe rings. The magnetostatic coupling also exists in MTJ rings, but does not affect the magnetic transition, which is different from the behaviour of the spin-valve rings. A size dependence of the magnetisation transition process was seen in MTJ rings.

Compared with single layer ring elements, ring-shaped multilayer structures, such as spin-valve and MTJ structures with higher MR ratios, provide a more sensitive way of investigating the magnetisation configurations in the free and pinned layers. Such structures with more domain states (the state combinations in two magnetic layers) are very attractive for spintronic applications such as memories or logic devices that require multiple stable resistance levels [172]. Generally for these multilayer structures, the magnetic layers may be coupled through the magnetostatic interactions, resulting in a change of the switching field of the free layer. This leads to a shift of the minor MR curve along the field axis.

Thus it is essential to investigate the effect of the magnetostatic interactions on the
magnetic switching in ring-shaped multilayer structures. The main magnetostatic interactions come from two mechanisms, the Néel coupling and the interlayer magnetostatic coupling. The Néel coupling due to the interface roughness is independent of the feature size [173-174]. The interlayer magnetostatic coupling is caused by the stray field from the domain walls and the edges of neighbouring magnetic layers [175] and it increases with a decrease in the feature size [176]. For a patterned device, the interlayer magnetostatic interaction field can be approximated by the demagnetisation field of the pinned layer acting on the free layer, which is estimated by the formula $H_D \sim C M_s t / L$, where $L$ is the length of the patterned device, and $M_s$, $t$ and $C$ are the magnetisation, thickness of the pinned layer and the proportional coefficient, respectively [177-179]. So the magnetostatic coupling field is proportional to the product of the thickness and the saturation magnetisation of the pinned layer [180]. As a result, this coupling field is much reduced in the SAF structure due to the decreased net magnetisation and a smaller effective CoFe thickness [181-182].
Chapter 3 Research Techniques

3.1 Experimental Techniques

Introduction

Our microfabrication is based on the combination of magnetron film deposition, photolithography and ion-beam milling. In terms of nanofabrication, there are two key techniques, e-beam lithography (EBL) and focused-ion beam (FIB), which are able to fabricate CIP and CPP devices with nanoconstrictions down to 20 nm in our research.

The magnetic and electrical transport properties of the structures, including MR curves as well as I-V characteristics, were detected by four-point probe measurement. In addition, a vibrating sample magnetometer (VSM) was used to measure M-H hysteresis loops. Atomic force microscopy (AFM) was often utilised to accurately measure the dimensions of the structures, thickness and surface roughness of thin films.

3.1.1 Thin Film Deposition

3.1.1.1 Magnetron Sputtering

Magnetron sputtering is a physical vapour deposition (PVD) technique, which is widely used for the deposition of magnetic as well as dielectric films.
For the magnetron sputtering, the deposition source takes the form of an electrode to which a high negative voltage is applied. The deposition chamber is filled with very low-pressure Ar gas, which is ionised by the voltage, forming the glow discharge plasma. Permanent magnets behind the electrode form a toroidal field (see Fig. 3.1), which confines the plasma in a ring close to the target surface. Positive Ar ions in the plasma are attracted towards the target by the applied negative voltage and strike it with a kinetic energy of several hundred eV. The powerful impact knocks atoms out of the target. The sputtered atoms are neutrally charged and so are unaffected by the magnetic trap.

When high-energy electrons collide with neutral Ar atoms, they can knock more electrons out of the atoms, resulting in the creation of more positive Ar ions. These positive ions are attracted to the target surface and the whole sputtering process keeps
going. As the electrons are trapped by the magnetic field, the path length and the degree of ionisation are increased. This increased ion density close to the target surface produces a high deposition rate.

Our sputtering facility, a Nordiko 9550 DC/RF sputtering system, is capable of processing sixteen 4-inch wafers per vacuum cycle, and contains 6 targets and a load lock, as shown in Fig. 3.2. The base pressure of the Nordiko 9550 is below $3 \times 10^{-8}$ Torr, which is very important to produce very smooth and dense films for spin transport studies. This fully computer-controlled system makes the deposition process very precise and reproducible.

3.1.2 Microfabrication Techniques

3.1.2.1. Mask Aligner

A mask aligner normally uses ultraviolet (UV) light as the exposure light source to lithographically transfer mask patterns onto the resist-coated wafer.
Our OAI 500 high-resolution mask aligner (shown in Fig. 3. 3) is a contact mode mask aligner. This is a high-performance mask aligner developed for ultra-precise, submicron level-to-level alignment lithography. In the contact mode, the mask is pressed against the resist-coated wafer during exposure and the radiation from a high intensity UV lamp is used to expose the photoresist on the wafer.

This tool is very essential for the mix and match between UV photolithography and e-beam lithography processes to achieve micron- and nano-sized devices in our research. The minimum feature size of this tool is \( \sim 0.8 \, \mu m \).

3.1.2.2. Lift-off

The basic procedure of lift-off/film deposition technique is shown in Fig. 3. 4. For the mask aligner system, UV light is shone through a mask to expose the pattern in the photoresist. After pre-baking, the exposed photoresist is developed in the 351 developer and gets the appropriate size of the undercut. Following post-baking, the thin film is deposited by using our sputtering deposition system. Lastly 1165 remover is utilised to remove the photoresist and thin films above it. If need be, ultrasonic agitation in heated 1165 remover can be applied. In addition, oxygen plasma treatment is good at removing residual resist ‘ears’ at the edge of the micro/nano-structures.

Fig. 3. 4. Schematic illustration of lift-off/film deposition technique: (1) coat and pattern photoresist,
(2) deposit thin films of desired materials, (3) swell photoresist with a solvent, (4) remove photoresist and thin films above it.

Fig. 3. Schematic illustration of film deposition/ion milling technique: (1) deposit thin films, (2) coat and pattern photoresist, (3) etch films using photoresist as the mask, (4) remove photoresist.

3.1.2.3. Ion Milling

In addition to lift-off/film deposition technique, the film deposition/ion milling technique (shown in Fig. 3. 5), is also very important for the fabrication of micron-scale devices. Our CVT Ion Miller (Ar⁺) is used to transfer the mask pattern into thin-film samples. After the resist is patterned, ion milling is used to physically etch away the unwanted areas of the film. Ar⁺ milling is a physical process, which involves no chemical reactions with samples.

3.1.3 Nanofabrication Techniques:

3.1.3.1 Focused Ion Beam

3.1.3.1.1 Introduction

Planar nanoconstrictions in ferromagnetic wires have been fabricated by several different methods including mechanically controllable break junction (MCBJ) [183],
self-terminating electrodeposition [184] and e-beam lithography. While the first method allows one to study very narrow wires, it usually leads to mechanically unstable structures. In electrodeposited nanocontacts, people have found a number of artefacts due to magnetostrictive, magnetostatic, and magneto-mechanical effects that can mimic BMR [132]. Although it is possible to produce 10-nm feature in resist with e-beam lithography, the transfer of the feature into a thin ferromagnetic film is not a well-established process yet.

A technique, which has recently become the focus of interest, is direct patterning by FIB milling [185]. FIB was mainly developed during the late 1970s and early 1980s, and the first commercial instrument was introduced over a decade ago [186]. Modern FIB systems are widely available in semiconductor research and processing environments. In a modern FIB system, the ion-beam ejected from a liquid Ga⁺ ion source, focused with a spot size ≤10 nm, is scanned across a sample in a manner analogous to the scanning electron microscope (SEM). The main applications arise from the use of Ga⁺ ions as the scanned species. These include composition imaging, direct etching of material, nanofabrication, and localised deposition and implantation of semiconductor or metal structures. What interests us in this technique is that Ga⁺ can be used to mill semiconductors or metals, and thereby pattern them into the desired shape [185, 187].

The unique combination of sub-10 nm resolution imaging with the ability to remove material in selected areas provides an ideal way of nanofabrication, which would otherwise be impossible, especially for sub-10 nm nanoscale structures. The directly etching ability of FIB removes extra patterning steps required in the e-beam lithography.
Additionally, Ga\(^+\) ions, which are many orders of magnitude heavier than electrons, are substantially less susceptible to the stray field, which is critical for the nanofabrication of magnetic samples.

![Schematic diagram of a FIB ion column](image)

**Fig. 3.6.** Schematic diagram of a FIB ion column [188].

FIB systems effectively combine together a scanning ion microscope and a precise milling system. By scanning the ion beam over a specimen and collecting ion beam-induced secondary electrons or ion signal, a surface image is formed. The position for a milled pattern can be selected from the image taken, and then the structure can be cut using Ga\(^+\) ions.

As shown in Fig. 3.6, the structure of the ion column is similar to that of SEM, and the major difference is that an ion beam is used instead of an electron beam. An ion beam is emitted from a liquid metal ion source by the application of a strong electric field. Normally people use a Ga\(^+\) ion source because of its stability, simplicity and the fact
that the $\text{Ga}^+$ ion beam gives good sputtering yields.

For related FIB nanofabrication in this thesis, we used an FIB system from Hamburg University (Fig. 3. 7). The beam current can be varied over a broad range, allowing for both a fine beam for the slow and precise milling, and a heavy beam for the fast and rough milling.

![Fig. 3. 7. (a) Schematic image and (b) real image of Hamburg FIB system [189].](image)

The Hamburg FIB system has the following features and characteristics:

- UHV $4 \times 10^{-10}$ mbar (ion getter-pumps)
- Cross beam-system, FIB on top
- CANION 31 Plus UHV-FIB by Orsay-physics
- Spin-detector for SIMPA (scanning ion microscopy with polarisation analysis)
- JEOL JAMP-30 SEM column, 58° tilt to normal
- High - precision JEOL - stage (motorised)
- Active vibration damping
• Lab with the magnetic-compensation system

• Sample size: 20 mm x 20 mm

3.1.3.1.2 Implantation Damage

FIB imaging inevitably induces some damage to the sample. Most Ga⁺ ions enter the sample after arriving at the surface, leading to the local implantation of Ga⁺ ions. The depth of the implanted area is related to the ion energy and the angle of incidence. The Ga⁺ implantation leads to a high level of surface impurities, and is detrimental to the magnetic properties of the magnetic films in our research.

To produce nanostructures without any modifications of the magnetic properties of the underlying material, it is necessary to minimise Ga⁺ implantation. It was found that a thin capping layer (such as Ta) can provide an efficient barrier to Ga⁺ implantation.

<table>
<thead>
<tr>
<th>Source</th>
<th>Liquid Ga source</th>
</tr>
</thead>
<tbody>
<tr>
<td>Acceleration</td>
<td>5 - 30 kV</td>
</tr>
<tr>
<td>Resolution</td>
<td>&lt; 20 nm</td>
</tr>
<tr>
<td>Beam current</td>
<td>1 pA — 50 nA</td>
</tr>
<tr>
<td>Writing field</td>
<td>500 μm x 500 μm</td>
</tr>
</tbody>
</table>

Table 3.1. Operating parameters of the Hamburg FIB system.

Also, we have tried varying the ion-beam parameters, including accelerating voltage, beam current etc. to minimise the effect of Ga⁺ implantation. The operating parameters of the Hamburg FIB system were given in Table 3.1.
3.1.3.2 E-beam lithography

3.1.3.2.1 Introduction

Electron beam lithography (EBL) is based on the principle that some macro-molecule polymers, e.g. PMMA, are sensitive to electrons and can be patterned by electron exposure. EBL is carried out by scanning an electron beam in a certain pattern across a resist-covered surface (resist exposure), and selectively removing either exposed or non-exposed regions of the resist (resist developing). The purpose is to create extremely fine structures in the resist, which can subsequently be transferred into another material.

The primary advantage of electron beam lithography is that it is one of the ways to avoid the diffraction limit of light and make submicron or nanoscale patterns. This has been realised due to the very small wavelength of the electron beam whereas the resolution in photolithography is limited by the wavelength of UV light.

This kind of maskless lithography has been widely used in mask-making for photolithography, small-quantity production of semiconductor components, and research in the nanometre regime.

On the other hand, the critical limitation of electron beam lithography is throughput, i.e., the very long time it takes to expose an entire wafer. A long exposure time leads to beam drift or instability during the resist exposure.

3.1.3.2.2 E-beam Lithography Nanofabrication

In our research, some nano-scale thin film devices were fabricated with electron beam lithography at the Rutherford Appleton Laboratory.

The fabrication started with a silicon wafer. A 70nm thick LOR layer was first coated
and pre-baked in an oven at 180°C for 20 minutes. A second resist layer, 70 nm thick PMMA (MW 350k), was then spin-coated and baked in the oven at 180°C for 1 hour. Electron beam direct writing was carried out with a high-resolution vector beam writer (Leica Cambridge VB6) at 100 kV beam energy and 500 pA beam current. The e-beam exposed resist was developed in a mixture of MIBK: IPA (1:3) for 60 seconds. The exposed PMMA layer defined the shape and dimension of the devices. The LOR resist in the opened area was then dissolved in CD26, an aqueous alkali solution, creating a clear undercut beneath the PMMA layer.

Thin films were then deposited by a sputtering deposition system. The lift-off process was carried out by soaking the sample in 1165 stripper. The second lift-off photoresist bilayer (LOR/1805) was then spin-coated. Using a mix-and-match technique, four contact electrodes made of 5nm Ta/200 nm Cu/5 nm Ta thin films were formed to connect the central part of the device by photolithography using the alignment marks patterned during the EBL exposure.

3.1.3.2.3 Optimisation of EBL Nanofabrication

Initially we started the first level lift-off with a single PMMA resist, which turned out to be unsuccessful. A LOR/PMMA bilayer was then introduced in the lift-off process. For the bilayer lift-off process, in the beginning we encountered difficulties in the complete removal of the resist along the edges of the nanoconstrictions, as shown in Fig. 3.8 (a). This problem was largely resolved by optimising the lift-off procedure. Nanoconstrictions with the minimum feature size of 30 nm have been successfully fabricated (see Fig. 3.8 (b)).
3.1.4 Device Characterisation

3.1.4.1 Vibrating Sample Magnetometer

A vibrating sample magnetometer (VSM) is an instrument that measures magnetic properties of magnetic materials as a function of magnetic field, temperature, and time. The VSM has become a widely used instrument since the invention of this experimental technique in 1956 by Simon Foner [190].

3.1.4.1.1. Principle of the VSM

If a magnetic sample is placed in a uniform magnetic field, created between the poles of an electromagnet, a dipole moment will be induced. The magnetic dipole moment of the sample will create a magnetic field around the sample, which is sometimes called the magnetic stray field. As the sample vibrates up and down with sinusoidal motion, this stray field changes as a function of time and a sinusoidal electrical signal can be detected by a set of pick-up coils according to Faraday's Law of Electromagnetic Induction. The signal has the same frequency of vibration, and its amplitude will be proportional to, the magnetisation of the sample, and relative position with respect to the pick-up coils system.
For a VSM system, a small sample (~0.5 cm by 0.5 cm) is fixed in a special sample holder located at the end of a sample rod mounted in an electromechanical transducer. The transducer is driven by a power amplifier which itself is driven by an oscillator at a fixed frequency, e.g. near 80 Hz. So, the sample vibrates along the Z-axis perpendicular to the external magnetic field. This vibration induces a signal in the pick-up coil system (shown in Fig. 3.9) that is sent to a differential amplifier or tuned amplifier and a lock-in amplifier that receives a reference signal supplied by the oscillator. The various components are connected to a computer interface. The output of this lock-in amplifier is a DC signal proportional to the magnetisation of the sample being studied.

Fig. 3.9. Schematic image of pick-up coils of a VSM system.

3.1.4.1.2. Our VSM System

Our VSM system (Fig. 3.10) was originally constructed by Mr. Khew Joong Harnn, a final year undergraduate project student. The sensitivity of this system has been improved recently, and can now provide precise magnetic measurements such as magnetisation hysteresis loops as a function of angle and field for very thin magnetic film devices.

The VSM system consists of six major parts, as shown in Fig. 3.11:
1. Water-cooled electromagnets and power supply

2. Vibration motor and sample holder

3. Pick-up coils

4. Hall sensor

5. Lock-in amplifier

6. LabView Computer Program

Fig. 3. 10. Our VSM system.

**Water-cooled Electromagnets and Power Supply**

The water-cooled electromagnets, along with the power supply, generate the external magnetic field.

**Vibration Motor and Sample Holder (with angle indicator)**

The sample holder rod is attached to the vibration motor, and the end of it is situated...
between the pole pieces. The motor moves the sample up and down at a set frequency. The sample rod can be rotated to achieve the desired orientation of the sample to the constant magnetic field. There are also different controls for the sample position.

**Pick-up Coils**

The sample produces an AC current in the pick-up coils at the same frequency as the vibration of the sample. The signal generated contains the information about the sample magnetisation.

**Hall Sensor**

A Hall sensor is used to measure the magnetic field generated by the electromagnets.

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![Block diagram of our VSM system.](image)

**Fig. 3.** Block diagram of our VSM system.

**Lock in Amplifier**

This amplifier is to recover the signal buried within the noise and to produce a DC
output that is proportional to the AC signal input. It is also used to provide the oscillation frequency to the motor, control the power supply voltage and the change-over relay.

**LabView Computer Program**

The LabView program installed in a computer automates the whole process by controlling the Lock-in Amplifier's output and acquiring the data from its input. The sample magnetisation and field strength collected are stored in a file, which is then used to plot the M-H loop. The data saved can be viewed using Microsoft Excel for further analysis.

**3.1.4.2 I-V and MR Measurement System**

**3.1.4.2.1 I-V Characteristics Measurement System**

For I-V characteristics measurements of samples with micro/nanoconstrictions, I developed a fully PC controlled measurement system using the combination of a Keithley Model 2520 meter and a 2182 nanovoltmeter as shown in Fig. 3. 12. This system provides DC current from 10μA to 1A and pulsed currents from 10 μA to 5A with pulse widths from 500ns to 5ms and pulse delays from 20μs to 500ms.

For this system, LabView programming is utilised to automate the process of the I-V characteristics measurement and plot the I-V loop. LabView programming uses a graphical programming method instead of writing down all command sentences in the traditional way. The programmer needs to add and arrange sub-VIs, codes and drivers according to the program requirement, which is like the flow diagram of the program where data flow determines the execution of the program.
A finished program in LabView is called the virtual instrument (VI), which includes two elements: the front panel and the block diagram. The function of the front panel (shown in Fig. 3. 13) is to provide the user with a graphical interface. The block diagram is where the code and drivers of the program are located. The graphical programming method is very useful in debugging the program on account of the visualisation of data flow.

In order to automate the I-V characteristics measurement, the following tasks are needed to be performed by the main program (please refer to Appendix 1 for the source code of the LabView program):

a) Initialise and set the settings of Model 2520 and Model 2182 meters.

b) Step the DC or pulse currents up or down if necessary.

c) Change the direction of the current.

d) Take readings of the signal from the 2520 and 2182 meters.

e) Save the data to a file for further analysis.

f) Plot the I-V loop as the readings are being taken.
But there is no voltage compliance in Model 2500 Current Source, so samples with micron- and nano-constrictions could easily break down by the unlimited high voltage during electrical measurements.

In addition, the measurement precision is much lower than we expected. To overcome these shortcomings, we have replaced the Model 2520 with a Model 6221 source meter. In contrast with the Model 2520, the Model 6221 can set required voltage compliance, which can effectively protect the micro/nanoconstriction samples. Besides, Model 6221 combined with Model 2182 has a much higher measuring resolution. Model 6221 can provide DC current from 10 pA to 105 mA and current pulses from 1 pA to 100 mA with pulse widths from 50 μs to 12 ms and pulse delays from 83.3 ms to 5 s. Thus this tool can carry out magneto-transport and I-V characteristics measurements for micro/nanoconstriction devices with substantially reduced overheating.
3.1.4.2.2 Four-point Probe Magnetic Measurement System

The four-point probe magnetic measurement system was improved by Mr. Kuan-Yi Yang. The front panel of this magnetic measurement system is shown in Fig. 3.14.

Using LabView programming with a GPIB interface card controlling the TTI PL 330TP Power Supply, a Keithley 2182 nanovoltmeter and a Keithley 6221 source meter as...
presented in Fig. 3. 15, this system provides precise measurements of MR curves as well as I-V characteristics for nano/micron-scale thin film devices.

3.1.4.3 Atomic Force Microscope

3.1.4.3.1 Introduction

The atomic force microscope (AFM), developed in the mid 1986, is a kind of scanning probe microscopy.

AFM is used to characterise the surface topography by measuring the interaction force acting between a fine probe and a sample. The probe is attached to the end of a cantilever and is brought very close to a surface. Attractive or repulsive forces resulting from interactions between the probe and the surface will cause a positive or negative bending of the cantilever. The bending is detected by a laser beam, which is reflected from the back side of the cantilever.

This section will only describe the Nano-R™ AFM in the close-contact mode, which was used in our research.

3.1.4.3.2 Components and Operation of AFM

Piezoelectric Ceramic Transducer

Precise mechanical motion in AFM is created from electrical energy using an electromechanical transducer. The electromechanical transducer commonly used in AFM is piezoelectric ceramic.

Force Sensors

The construction of AFM requires a force sensor to measure the forces between a small
probe and the surface being imaged. A common type of force sensor utilises the relationship between the motion of a cantilever and the applied force. The relationship is given by Hooke's law: $F = -K \times D$, where:

- $K$ is a constant, which depends on the material and dimensions of the cantilever
- $D$ is the motion of the cantilever.

The motion of the cantilever can be measured with the "light lever" method, which means that deflection of the cantilever causes the laser beam to move across the surface of the photo-detector. The motion of the cantilever is then directly proportional to the output of the photo-detector. Motions as small as 1 nm are routinely measured by AFM using this method.

**Feedback Control**

For AFM, feedback control is used to keep the probe in a "fixed" relationship with the surface while a scan is measured.

**AFM Theory & Instrumentation**

The theory and operation of AFM is similar to that of a stylus profiler. The primary difference is that probe forces on the surface are much smaller in AFM. Because of this, smaller probes can be used, and a much higher resolution can be achieved.

For AFM, a constant force is maintained between the probe and the sample while the probe is raster scanned across the surface. By monitoring the $Z$ motion of the probe as it is scanned, a 3D image of the surface is constructed.
The constant force is maintained by measuring the force on the cantilever with the light lever sensor and by using a feedback control electronic circuit to control the position of the Z piezoelectric ceramic. The motion of the probe over the surface is generated by piezoelectric ceramics that move the probe and force sensor across the surface in the X and Y directions, as shown in Fig. 3. 16.

Vibrating (close-contact) Mode

The cantilever of AFM can be vibrated using piezoelectric ceramics. When the vibrating cantilever comes close to the sample surface, the amplitude and phase of the vibrating cantilever may change. The feedback unit keeps either the vibration amplitude or phase constant. Changes in the vibration amplitude or phase are easily measured, and the
changes can be related to the force on the surface. This technique has many names, such as “close-contact” mode. It is important that the tip does not “tap” the surface (Fig. 3.17), as this may break the probe or damage the sample.

There are many useful analysis functions for the captured AFM images. Two main functions, topography and depth analysis, are mostly used in this thesis.

Nano-R AFM Instrument System

A Nano-R AFM instrument system includes Nano-R Stage, master computer, controller, video monitor, and track ball. A complete Nano-R™ AFM stage includes a video microscope, AFM scanner, sample puck, z motorised approach, and an x-y positioning stage, as shown in Fig. 3.18.

Fig. 3.18. Nano-R™ AFM stage.
3.2 Micromagnetic Simulation Software

Micromagnetic simulations were carried out to investigate the magnetisation reversal and the domain wall structures in patterned magnetic devices with constrictions. We have used two micromagnetic simulation software packages, LLC Micromagnetics Simulator™ and Object Oriented Micromagnetic Framework (OOMMF), and both of them are based on the Landau-Lifshitz-Gilbert (LLG) equation.

3.2.1 Landau-Lifshitz-Gilbert Equation

The equation that describes the dynamics of a magnetic structure in time is the Landau-Lifshitz-Gilbert equation of motion as shown below [192-193]:

\[ \frac{dM}{dt} = -\gamma \frac{M \times H_{\text{eff}}}{M_s} + \frac{\alpha}{M_s} M \times \frac{dM}{dt} \]  

The first term on the right-hand side of equation (3.1) describes the precession of the magnetisation \( M \) about the effective field and the second term accounts for the damping phenomenologically. The parameter \( \gamma \) is the gyromagnetic ratio, \( M_s \), the saturation magnetisation and \( H_{\text{eff}} \), the effective field which originates from the superposition of the external field and the anisotropy, exchange and demagnetisation fields. The dissipation (or magnetic damping) is described by the dimensionless Gilbert damping constant \( \alpha \) (\( \alpha = \lambda / \gamma M \), \( \lambda \) is the Landau-Lifshitz damping rate). Solving the LLG equation consists of an algebraic minimisation of the total energy \( E_{\text{total}} \) as follows:

\[ E_{\text{total}} = E_{\text{anisotropy}} + E_{\text{exchange}} + E_{\text{demag}} + E_{\text{seeman}} \]  

(3.2)
3.2.2 LLG Micromagnetics Simulator

Some of micromagnetic simulations in this project were carried out using a commercial micromagnetic software package, LLG Micromagnetics Simulator™. LLG Micromagnetics Simulator™, which was developed in 1997 by Michael R. Scheinfein [194], is a 3D simulation tool with integrated graphics that solves the LLG equation by relaxation and/or integration. With this software, one can characterise the micromagnetic structure and dynamics in either single layer or multilayer magnetic devices. Fig. 3.19 is a screen shot of the LLG working environment while a simulation is being run.

![Screen shot of the working environment of LLG Micromagnetics Simulator™.](image)

Fig. 3.19. Screen shot of the working environment of LLG Micromagnetics Simulator™.
Applications:

- LLG computes the equilibrium magnetisation distribution in small particles and in thin films, as well as computing fundamental properties, such as the coercive field, switching time, interlayer coupling strength, and domain wall length.

- LLG simulates the structure and response of magnetic devices, such as MRAM, spin valves, AMR and GMR heads, and magnetic sensors.

- LLG computes standard magnetic imaging contrast mechanisms realised in Lorentz microscopy, electron holography, SEMPA, and magnetic force microscopy (MFM).

In order to compare micromagnetic simulations with the experimental results, for example, a patterned NiFe device with artificial pinning sites (micron/nano-constrictions etc.); a device of special geometry with a constriction was input into the simulation with similar parameters as used in the experiments. The standard magnetic parameters for Ni$_{80}$Fe$_{20}$ were input into the model, including $A_{ex} = 1.05 \times 10^{-6}$ erg/cm and $M_s = 8 \times 10^5$ emu/cm$^3$. The cell size was $10 \times 10 \times 10$ nm$^3$, and the magnetisation in each cell has contributions from the anisotropy, exchange, magnetostatic and external fields.

3.2.3 Object Oriented MicroMagnetic Framework (OOMMF)

OOMMF is a public domain micromagnetic simulation software developed at the National Institute of Standards and Technology (NIST)/ITL. The program is designed to be portable and extensible, with a user-friendly graphical interface. The code is written in C++ and Tcl/Tk. Fig. 3. 20 is a screen shot of the OOMMF working environment.

The OOMMF simulation software, which was mainly developed by Mike Donahue, and
Don Porter [195], also uses the LLG equation. OOMMF employs the finite difference method, which requires the discretisation of a chosen geometry over a grid of cells.

Fig. 3. 20. Screen shot of OOMMF working environment.

Before using OOMMF, one needs to define the specific problem, which includes defining the magnetic properties (such as $A_{\text{ex}}$ and $M_s$ etc.), geometry of the sample, initial magnetic state and the external magnetic field to be applied. Simulation parameters such as the characteristics of the finite element mesh and the criteria for convergence are also very important parameters. The solution 'solves' the problem by integrating the LLG equation.

For the calculation, the micromagnetic problem is solved based on regular 2D meshes of squares with 3D magnetisation spins positioned at the centre of the square. Assuming a constant magnetisation within each cell, the anisotropy and applied field energy terms
are calculated. The exchange energy is calculated using the eight-neighbour bilinear interpolation with Neumann boundary conditions.

Calculating the magnetostatic field is the most time-consuming in the calculation process. In OOMMF, it is calculated as the convolution of the magnetisation for a given kernel, which represents different interpretations of the discrete magnetisation and the cell-to-cell magnetostatic interaction. Several kernels are supported within the software, but the simplest one is used for which the magnetisation is constant in each cell, and the average demagnetisation field through the cell is computed by using the formula described in [196]. This convolution is evaluated using fast Fourier transform techniques. The Landau-Lifshitz-Gilbert ODE, on the other hand is integrated using a second order predictor-corrector technique of the Adams type. Further details regarding to this process can be found in [195].

3.2.4 Why Use Both OOMMF and LLG Micromagnetics Simulator?

The OOMMF code is freely available, which makes it possible to compare our simulated results with established publications, which is a critical research criterion. In the beginning, we used OOMMF to simulate the domain wall states and magnetisation configurations of the single-layer magnetic structures, e.g. NiFe nanoring devices. Although OOMMF also has the capabilities of doing both 2D and 3D simulations, the original code cannot calculate and plot MR curves, which is however readily accessible with LLG Micromagnetics Simulator, so most of the simulation work was done with the latter afterwards. In addition, it is much easier to define a complex position-dependent mask with LLG Micromagnetics Simulator, which is quite important for the research on some complex structures, such as the half-pinned NiFe/IrMn device.

Several structures were simulated with both software, and the results are quantitatively
similar. The slight deviations had been expected, as the various software use different numerical discretisation schemes.
Chapter 4 Results and Analysis

4.1 Magnetisation Reversal with Domain Wall Motion in Submicron NiFe and Half-pinned (NiFe/IrMn) Devices with Constrictions

4.1.1 Introduction

In this chapter, we report on a study of the magnetisation reversal accompanied by domain wall motion in submicron thin magnetic film stripes with microconstrictions. In this research two different structures have been included: structure A is a submicron standard NiFe stripe with microconstrictions; structure B has the same geometry, but the NiFe layer on the left side of the stripe is pinned by an AF layer, and the other side is unpinned, which can be easily switched by a small field.

The idea is to realise the antiparallel alignment of the magnetisations on two sides of a constriction, which would bring in a 180° domain wall at the narrow constriction area, so the resistance difference would be observed once the domain wall annihilates in a high field.

4.1.2 Experiment

A Ta (5 nm)/ NiFe (5 nm) bilayer was deposited on a 3-inch thermally oxidised Si wafer by the Nordiko 9550 deposition system. The base pressure for deposition was below $3.0 \times 10^{-8}$ Torr. By photolithography and ion milling, a blank wafer was patterned into structure A: a 1.5 μm-wide stripe with two 0.8 μm-wide microconstrictions. As shown
in Fig. 4.1 (a), the whole stripe is divided into the left, middle and right parts by the constrictions. Based on structure A, structure B was further fabricated with the photolithography/film deposition technique, leaving the left part covered by an IrMn layer, as indicated in Fig. 4.1 (b). Here, we used a special photomask, which enabled the middle and right parts of the stripe to be covered by photoresist during photolithography process. In the following film deposition, the NiFe surface of the sample was given a sputter pre-clean to achieve enough exchange-bias in the NiFe/IrMn bilayer, followed by the deposition of a NiFe (3 nm)/IrMn (15 nm)/Ta (5 nm) stack and the resist-removal process afterwards. The top nonmagnetic electrodes processed by the lift-off technique are used to supply a dc current and perform the MR measurement with a standard four-point probe technique. As shown in Fig. 4.1 (c), both the left and middle parts of the stripe are directly connected to the nonmagnetic electrode, so the right part was excluded in the electrical measurement and also in the micromagnetic simulation afterwards.

Fig. 4.1. Schematic images of (a) structure A (NiFe only) with 0.8 μm-wide microconstrictions and (b) half-pined structure B with the left part of the stripe covered by an IrMn layer. (c) SEM image of half-pined structure B with 0.8 μm-wide constrictions.

The magnetotransport properties have been measured at room temperature by a Keithley 85
2182 Nano-Voltmeter and a Keithley 6221 AC and DC current source. For the longitudinal measurement, the magnetic field is applied along the direction of the current, parallel to the long stripe axis.

4.1.3 Results and Discussion

4.1.3.1. Structure A: a Normal NiFe Stripe with Microconstrictions

![Experimental and simulated MR curves of structure A.](image)

Fig. 4.2. (a) Experimental longitudinal MR curve and (b) simulated MR curve of structure A.

A negative longitudinal MR curve (MR = -0.09%) is shown in Fig. 4.2 (a). The MR measurement begins with saturating the device in a large positive field. Once the field decreases to zero, the resistance begins to drop at the remanent state. After applying a small magnetic field in the opposite direction, a sharp drop of the resistance occurs, and a minimum resistance value is seen. When the field reaches the coercive field of the stripe $H_c = -3$ Oe, an abrupt resistance increase appears, and the resistance quickly goes back to the high resistance state. There is a small decrease of the resistance in a higher field, which cannot be explained by the AMR effect, as the whole structure is saturated in a very high field with the magnetisation completely parallel to the external field, leading to the high resistance state. Thus this behaviour could be caused by a small
misalignment between the long axis of the stripe and the external field during the MR measurement [197].

In order to understand the experimental MR curve, we have done the micromagnetic simulation of the magnetisation reversal process of structure A in a longitudinal field using LLG Micromagnetics Simulator [194]. The parameters used for the simulations are the commonly used values for NiFe, the saturation magnetisation $M_s = 8 \times 10^3$ emu/cm$^3$, the exchange constant $A = 1.05 \times 10^{-6}$ erg/cm and the uniaxial anisotropy constant $K_u = 1.0 \times 10^3$ erg/cm$^3$. The cell size is 10 nm. The right part of the stripe was excluded in the simulation. As shown in Fig. 4.2 (b), the simulated MR curve matches the experimental curve except that there is no drop in the resistance in high fields. This indicates that the abnormal high-field MR behaviour might be due to a misalignment between the stripe axis and the external field during the MR measurement.

Fig. 4.3. Simulated magnetisation configurations of structure A (a) at the remanent state, (b) in a small reverse magnetic field and (c) in a field higher than the switching field.
As shown in Fig. 4.3 (a), at the remanent state, some transversal magnetic components emerge at the edge of the stripe, while most of the magnetic moments in the stripe are still aligned along the long stripe axis, which explains the resistance drop (Fig. 4.2 (a)) at remanence considering the AMR effect behind. When a small reverse field is applied, vortex domain walls are formed and more transversal magnetic components are introduced, as shown in Fig. 4.3 (b), which inevitably leads to a quick decrease of the resistance, corresponding to the resistance minimum of the experimental MR curve. Once the field reaches the switching field of the NiFe stripe, both the left and middle parts of the stripe almost finish the magnetisation reversal simultaneously, and get magnetised along the long axis as shown in Fig. 4.3 (c), which pushes the resistance back to the high state.

4.1.3.2. Structure B: a Half-pinned NiFe Stripe with Microconstrictions

The negative longitudinal MR curve (MR= -0.11%) of structure B, as shown in Fig. 4.4 (a), is not symmetric about the resistance axis unlike the corresponding curve for structure A. This can be justified by the pinning of the IrMn layer, which was deliberately deposited to pin the NiFe magnetisation on the left side of the submicron stripe for low field measurements.

For the MR measurement, after saturating the sample in a large positive field and then returning to the remanent state (point A), a resistance drop is observed, which is similar to the behaviour of structure A. After applying a small negative field, a sharp fall in the resistance occurs at point B. Based on the simulation data of structure A, it can be
inferred that some transversal magnetic components are introduced here. When reaching the coercive field of the stripe $H_c = -3$ Oe, an abrupt increase of the resistance is observed at point C. But instead of going up to the high resistance state, the resistance decreases slightly from point C to point D, which is totally different from the behaviour of structure A at the same stage. The quick saturation of the resistance does not occur until the magnetic field reaches about $-90$ Oe, higher than the pinning field of the IrMn layer.

Fig. 4. Longitudinal MR curves of structure B: (a) a half-cycle MR curve with the field sweeping from positive to negative, and (b) a complete MR curve.

Fig. 5. Simulated longitudinal MR curves of structure B: (a) a half-cycle MR curve with the field sweeping from positive to negative, and (b) a complete MR curve.
The micromagnetic simulation has been done to clarify the magnetisation reversal mechanism in the half-pinned structure B. As shown in Fig. 4. 5, the simulated MR curves agree very well with the experimental curves. Based on the simulation, the magnetisation switching behaviour of structure B, especially the unique part (point C to point D) of the MR curve, can be fully understood.

Between point B and point C (Fig. 4. 4 (a)), when the applied field reaches the switching field of the NiFe stripe, the middle part of the stripe without feeling the exchange bias switches the magnetisation first, which leads to the first jump up in the resistance, but there is no obvious magnetisation reversal in the left part of the stripe due to pinning from the IrMn layer, which can be seen in Fig. 4. 6 (a). Meanwhile, with the field going up, the left part shows a sign of the magnetic switching, and some transversal magnetisation components are introduced at the left edge and the left side of the constriction as well, as shown in Fig. 4. 6 (b). A $180^\circ$ domain wall (highlighted area in Fig. 4. 6 (b)) is seen at the interface between the pinned part and the unpinned part. This inevitably leads to a small drop in the resistance, corresponding to the abnormal resistance trace from point C to point D shown in Fig. 4. 4 (a). The left part of the stripe does not finish the magnetisation reversal until the external field is higher than the IrMn/NiFe pinning field, when the magnetisation in the NiFe layer is more parallel to the current direction (or the long stripe axis) with fewer transversal magnetisation components, as observed in Fig. 4. 6 (c). This results in a sharp increase in the resistance, leading to the final saturation state of the resistance in a high field.
Fig. 4. 6. Simulated magnetisation configurations of half-pinned structure B (a) at the switching field (H_s) of the NiFe stripe, (b) in a field higher than H_s but lower than the pinning field (H_{pin}), and (c) in a field higher than H_{pin}.

4.1.3.3. Half-pinned NiFe Stripe with a Nanoconstriction

With the help of the Hamburg group we carried out several trials of fabricating the half-pinned stripe with a nanoconstriction by FIB milling. Unfortunately no electrical measurements could be taken, because the sample surface was damaged by accident during FIB fabrication and no further samples have yet been produced.

4.1.4 Conclusion

The magnetisation reversal processes have been investigated in the submicron standard NiFe stripe and half-pinned NiFe stripe with a microconstriction. An asymmetric MR curve has been found in the half-pinned NiFe stripe with a microconstriction (structure B): with the field sweep from positive to negative, the left part does not quickly switch
the magnetisation, and the resistance stays almost constant when the negative magnetic field is lower than the pinning field of the IrMn layer. A $180^\circ$ domain wall can be seen at the interface between the pinned part and the unpinned part in the simulated magnetisation configuration. Micromagnetic simulations verify the special MR curve is caused by the exchange-bias on the left side of the NiFe stripe, which changes the magnetisation switching mechanism of the whole stripe.

Further FIB nanofabrication was not successful, although we had expected to see the effect of the motion of a single domain wall on the MR in the nanoconstriction area of such a half-pinned nano-device in low fields, which would further push our research on the domain wall MR.
4.2. Magnetisation Reversal with Domain Wall Motion in NiFe and Spin-valve Nano-devices with Different Pinning Sites

4.2.1 Magnetisation Reversal with Domain Wall Motion in NiFe Nano-devices with Different Pinning Sites

4.2.1.1. Introduction

The magnetisation reversal process and domain wall motion in submicron or nano-scaled ferromagnetic wires have been widely investigated by MR measurements [197-200]. In FM wires with constrictions [99, 102, 104, 106, 201-206] and ring structures with notches [107, 207], constrictions/notches can effectively act as pinning sites of the domain wall and have been used to study domain wall trapping, domain wall MR and current-induced domain wall motion.

In this section, we report a study on different magnetisation reversal processes in NiFe nanodevices with different pinning sites, which were fabricated from a 15 nm-thick NiFe micron stripe with a microconstriction. In our investigation, three structures have been studied: (a) pre-patterned structure - a NiFe Stripe with a microconstriction, (b) structure A - a NiFe stripe with a nanoconstriction, and (c) structure B - a NiFe stripe with a submicron triangular element and a nanoconstriction. The pinning sites are very effective in trapping domain walls and controlling the domain wall propagation. The motion of domain walls can be indirectly detected by MR measurements and verified in the micromagnetic simulations.
4.2.1.2. Experiment

A Ta (5nm)/ Ni$_{80}$Fe$_{20}$ (15nm)/ Ta (5nm) structure was deposited onto a thermally oxidised Si wafer by a Nordiko PVD system. The base pressure for deposition was below 2.0×10$^{-7}$ Torr. Samples were first photolithographically patterned into 10 μm x 2 μm stripes with a 0.8 μm-wide microconstriction (pre-patterned structure), as shown in Fig. 4. 7 (c). Two kinds of structures have been fabricated by FIB from this basic structure: structure A is a submicron stripe with a 150 nm-wide nanoconstriction, and structure B is a stripe with a submicron triangular element and a 150 nm-wide nanoconstriction, as shown in Fig. 4. 7 (a) and (b). The cuts were made with the Canion 31M Plus FIB, using 30 kV gallium ions (5 pA beam current). The whole submicron stripe is divided into two parts by the nanoconstriction, and each end of the stripe is connected to a big contact pad, which is covered by the nonmagnetic electrode. Four electrodes made of Ta (5 nm)/Cu (100 nm)/Ta (5 nm) were fabricated to connect the central device by the photolithography and lift-off technique, as shown in Fig. 4. 7 (d). These electrodes are used to supply a dc current (outer two electrodes are current probes) for the standard four-point probe MR measurement. The inner two electrodes are situated at the edge of the micron stripe, so the big pads are excluded in the MR measurement.

The magnetotransport properties were measured at room temperature by a Keithley 2182 Nano-Voltmeter and a 6221 AC and DC current source. For the longitudinal measurement, the magnetic field is applied along the direction of the current (parallel to the long stripe axis shown in Fig. 4. 7 (b)); while for the transversal measurement, the
field is applied perpendicular to the current in plane.

Fig. 4. 7. SEM images of (a) structure A with a 150nm-wide nanoconstriction, (b) structure B with a triangular element and a 150 nm-wide nanoconstriction, (c) the pre-patterned stripe with a 0.8 μm-wide microconstriction and (d) a structure connected with big pads covered by four nonmagnetic electrodes. Since the FIB pattern was done in the central area of the original micron stripe, the two ends of the stripe were left unpatterned, as highlighted with dashed boxes.

4.2.1.3. Results and Discussion

4.2.1.3.1. NiFe Stripe with a Microconstriction (Pre-patterned Structure)

The MR curve of the NiFe stripe with a microconstriction shows a normal transversal MR curve (MR = 0.56%) in a transversal field, as shown in Fig. 4. 8, where the low resistance state shows in the saturation field, and the high resistance state in the zero field.

In a longitudinal magnetic field, the longitudinal MR curve exhibits a negative MR of -0.08%) (Fig. 4. 9). The MR measurement begins with saturating the device in a large positive field. Then the field sweeps from positive to negative. Once the field decreases
to zero, the resistance begins to drop at the remanent state. After applying a small magnetic field in the opposite direction, a sharp drop of the resistance occurs, and a minimum resistance value is finally seen. When the field reaches the coercive field of the stripe ($H_c = -3 \text{ Oe}$), an abrupt jump up in the resistance appears, and the resistance quickly goes back to the high resistance state. There is a small decrease of the resistance at a higher magnetic field.

![Fig. 4. 8. The transversal MR curve of the pre-patterned structure with a 0.8 μm-wide microconstriction.](image1)

![Fig. 4. 9. The longitudinal MR curve of the pre-patterned structure with a 0.8 μm-wide microconstriction.](image2)
To provide an explanation of the longitudinal MR curve shown in Fig. 4.9, we have done a micromagnetic simulation of the magnetisation reversal process of the pre-patterned structure with a longitudinal field using the OOMMF (Object Oriented Micromagnetic Framework) package [195]. Parameters used in the simulations for Permalloy are the saturation magnetisation $M_s = 8 \times 10^5$ emu/cm$^3$, the exchange constant $A = 1.05 \times 10^6$ erg/cm and the uniaxial anisotropy constant $K_{\alpha} = 1.0 \times 10^3$ erg/cm$^3$. The cell size is 10 nm x 10 nm. The above conditions are the same for all micromagnetic simulations of different structures in this section.

![Simulated magnetisation configurations of the pre-patterned structure](image)

Fig. 4.10. Simulated magnetisation configurations of the pre-patterned structure (a) at the remanent state, (b) in a small reverse field, and (c) in a reverse field higher than the switching field.

In the simulation, the structure is first saturated to the left, and then the magnetic field switches from left to right. As shown in Fig. 4.10 (a), at the remanent state, a small amount of transversal magnetic components emerge at the edge of the stripe, although most of the magnetisations are still aligned along the long stripe axis, which explains...
the resistance drop at remanence in Fig. 4. 9 considering the AMR effect. When a small reverse field is applied, more transversal magnetic components are introduced in the stripe, which inevitably leads to a sharp decrease of the resistance, corresponding to the resistance minimum of the MR curve. When the reverse field is close to the coercivity of the micron stripe, vortex domains are formed in the stripe, as shown in Fig. 4. 10 (b). When the field is higher than the switching field of the NiFe stripe, both the left and right parts of the stripe almost finish the magnetisation reversal simultaneously, and get magnetised along the long axis as shown in Fig. 4. 10 (c), which pushes the resistance back to the high resistance state.

In the simulation no decrease in the resistance is observed at a very high magnetic field, so what we have seen in the experiment might be due to the misalignment between the stripe axis and the external field during the MR measurement [197].

4.2.1.3.2. Structure A: NiFe Stripe with a Nanoconstriction

![Graph](image.png)

**Fig. 4. 11.** The transversal MR curve of structure A with a 150 nm-wide nanoconstriction.
For FIB-patterned structure A with a nanoconstriction, the transversal MR curve (MR = 0.68%) is shown in Fig. 4.11. Here we observe the so-called bell-shaped behaviour, that is, an MR enhancement around a zero magnetic field [99, 208]. It is believed that this enhancement is due to the domain wall pinning at the nanoconstriction near a zero field by the reversal from the saturation state, which can be explained by the domain wall scattering mechanism [95, 208]. This phenomenon is first observed when measured at a dc current of 50 μA, but it disappears at a current of 500 μA, corresponding to a critical current density of $2.2 \times 10^7$ A/cm$^2$, which is consistent with the reported behaviour of a NiFe wire with a nanoconstriction [208]. Thus this bell-shaped behaviour of the MR curve is in line with the domain wall scattering mechanism.

It might be suspected that the geometry of structure A may contribute to the special shape of its MR curve. But actually this can be excluded, because there is no MR enhancement found in the pre-patterned structure with a microconstriction, which has almost the same geometry as structure A except for the constriction area. Thus it is the nanoconstriction (with the domain wall scattering mechanism behind) that makes such a big difference, leading to the bell-shaped behaviour of structure A, although the AMR effect may have a small contribution. To clarify the uncertainty, we need to locate nonmagnetic electrodes as close to the nanoconstriction as possible to minimise the AMR contribution.

Fig. 4.12 exhibits the longitudinal MR curve of structure A at a longitudinal magnetic field, which shows a negative MR of 0.17%. During the MR measurement, the structure is first saturated at a high negative field, and then the magnetic field sweeps from
negative to positive. Once the field direction changes to positive, the resistance decreases sharply before reaching the minimum state. With the further increase of the field, the resistance shows the first sharp increase at a field of $H_{c1}=6$ Oe. Afterwards between $H_{c1}$ and $H_{c2}=20$ Oe, there is no obvious resistance change. Finally when the field reaches $H_{c3}=23$ Oe, the resistance returns to the original high state in the second abrupt increase.

![Graph showing MR curve](image)

**Fig. 4. 12.** The longitudinal MR curve of structure A with a 150 nm-wide nanoconstriction (arrows show the trace of the MR curve when the field sweeps from negative to positive).

For an insight into the experimental longitudinal MR curve in Fig. 4. 12, the magnetisation reversal process of structure A at a longitudinal field has been simulated using the OOMMF simulation package. The unpatterned stripe area (shown in Fig. 4. 7 (a)) was included in the simulation, but is not shown below for the reason of space.

With the field sweeping from negative to positive, at a small positive field, the unpatterned micro-stripe area initiates the magnetisation switching, followed by the switching of the left and right parts of the patterned nano stripe, but the magnetisation reversal of the latter is not completed yet due to several vortex domains existing close to...
the edges). Then the domain walls move towards to the nanoconstriction area until they are pinned there, as shown in Fig. 4. 13 (b)), corresponding to the first sharp resistance increase (Fig. 4. 12). The field keeps increasing, but within a narrow field range, the amount of the transversal magnetic components at the stripe edge and the constriction area remaining nearly the same, which is in agreement with the resistance plateau of Fig. 4. 12. When the external field reaches the depinning field of the nanoconstriction, domain walls finally annihilate at the nanoconstriction and the stripe edge completely switches the magnetisation, leading to the magnetisation reversal completed in the whole stripe as shown in Fig. 4. 13 (c), which corresponds to the second sharp rise of the resistance of Fig. 4. 12.

Fig. 4. 13. Simulated magnetisation configurations of structure A (a) at the remanent state, (b) at the switching field of the NiFe stripe, and (c) in a field higher than the depinning field of the nanoconstriction.
4.2.1.3.3. Structure B: NiFe Stripe with a Submicron Triangular Element and a Nanoconstriction

For structure B, the transversal MR curve (MR=0.58%) is shown in Fig. 4. 14 (a). Similar to the behaviour of structure A, a small MR enhancement can be observed around a zero magnetic field.

![Transversal MR curve and longitudinal MR curve of structure B](image)

Likewise under a longitudinal field, structure B shows a negative longitudinal MR effect (MR = -0.16%) in Fig. 4. 14 (b). As for the pre-patterned structure, structure B shows a sharp resistance decrease at a small reverse field, resulting in the minimum resistance state.

As shown in Fig. 4. 14 (b), on reaching the field $H_{c1}=4.3$ Oe, the resistance rises sharply. From $H_{c1} = 4.3$ Oe to $H_{c2} = 13$ Oe, there is a small increase in the resistance. Once the field is higher than $H_{c2} = 13$ Oe, the resistance shows the second abrupt jump up. Between $H_{c2} = 13$ Oe and $H_{c3} = 21.5$ Oe, the resistance again only changes slightly. On reaching $H_{c4}=23$ Oe (the same as the depinning field of the nanoconstriction of structure A), the resistance returns to the original high state in the third jump up.

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A similar micromagnetic simulation was also performed on structure B by using OOMMF and its magnetisation reversal process can then be explained as follows. The unpatterned stripe area (Fig. 4.7 (b)) was included in the simulation, but is not shown below.

Fig. 4.15. Simulated magnetisation configurations of structure B (a) at the remanent state, (b) at the switching field of the micron-stripe, (c) at the switching field of the left part of the patterned stripe, and (d) in a field higher than the pinning field of the triangular element and the nanoconstriction.

With the field sweeping from negative to positive, at a small positive field, the magnetisation reversal is initiated in the unpatterned micro-stripe area. Being wider than
the left part of the patterned stripe, the right part has a lower coercivity, so it follows the magnetisation switching of the unpatterned micro-stripe, and is magnetised along its long axis, corresponding to the first abrupt resistance increase shown in Fig. 4. 14 (b), and the domain wall moves towards the nanoconstriction before it is trapped there, as shown in Fig. 4. 15 (b). As the field increases, the left part of the patterned stripe begins to reverse its magnetisation, leading to the second resistance jump-up in Fig. 4. 14 (b), followed by the movement of the reverse domain wall towards the right until it reaches the base of the triangular element, where it feels the pinning, as illustrated in Fig. 4. 15 (c). When the external field is high enough to overcome the pinning energy induced by the sharp expansion in width, the domain wall is depinned at the triangular element. Almost simultaneously the pinned domain wall annihilates at the nanoconstriction, as shown in Fig. 4. 15 (d). The final domain wall annihilation at two pinning sites results in the third sharp increase of the resistance in Fig. 4. 14 (b).

Fig. 4. 16. Simulated magnetisation configurations of (a) the pre-patterned structure, (b)
Comparing the pre-patterned structure (with a microconstriction) with two types of FIB-patterned structure A and B (with nano-sized pinning sites), we notice that the longitudinal MR value of the pre-patterned structure is -0.08%, which is half as large as those of two FIB-patterned structures, -0.17% and -0.16%, respectively.

A comparison of the above longitudinal MR values may easily lead to a misleading interpretation that the domain wall MR is negative in the longitudinal measurement.

However, when taking a close look at the simulated magnetisation configurations of the above structures (as shown in Fig. 4.16), at a very small reverse field (lower than the switching field of the micron stripe), where the dip of the MR curve appears, some difference is seen between the pre-patterned and FIB-patterned structures. In addition to the transversal magnetic components emerging at the ends of the micron stripe, both FIB-patterned structures show some extra transversal magnetic components at the joint part between the micron stripe and the nano stripe. Furthermore, for structure B, a small amount of transversal magnetisation components appear at the two sharp corners of the triangular base. The extra transversal magnetisation components in the two FIB-patterned structures result in their larger negative MR compared with the pre-patterned structure, as AMR evolves as an angular variation of the resistance with 
\[ R \propto \cos^2\theta, \]
where \( \theta \) is the angle between the local magnetisation and the current direction (the AMR effect causes a decrease in resistance, when the magnetisation components are perpendicular to the current in the sample). Thus the large negative longitudinal MR of structure A and structure B can be explained by the AMR contribution, although the
Influence of DWMR may play a minor role.

In addition, due to the bell-shaped behaviour, the transversal MR magnitudes of structure A and B are 0.68% and 0.58%, respectively, slightly higher than that of the pre-patterned structure (MR~0.56%). There might be a contribution from DWMR, which is more pronounced in structure A, since the enhanced MR of structure B is reduced by its high resistance.

As to structure A, a series of samples with different nanoconstriction widths have been further studied for the dependence of the transversal MR on the constriction width. The transversal MR increases from 0.57% to 0.80% when the nanoconstriction width decreases from 200 nm to 50 nm, which can be interpreted as due to both AMR and DWMR. It can therefore be inferred that DWMR plays a more important role in the transversal MR for devices with narrower constrictions and there is a positive contribution to the resistance from DWMR. This can be justified by the fact that the amplitude of the DW scattering depends on the width of the domain wall [95, 209].

To clarify the existence of DWMR, we need to put contact electrodes as close to the nanoconstriction as possible (at a distance smaller than 20 nm from the constriction) to minimise the contribution of the long stripe (AMR contribution) to the magnetoresistance, which is then dominated by the contribution of the nanoconstriction, so ideally we can separate the normal AMR from DWMR. In reality we could only shorten the distance down to 2-3 μm, so this task is very tough considering the resolution of our photolithography and the unavoidable error in the mix
and match process for the electrode fabrication.

4.2.1.4. Conclusion

In summary, domain wall motion induced magnetisation reversal processes in constricted NiFe nanodevices have been studied by MR measurements and the micromagnetic simulations. By using different pinning sites in the patterned nanodevices, different magnetisation reversal processes, as indicated by different experimental magnetoresistance curves, have been observed in the simulations through the control of the domain wall pinning and displacement. The transversal MR curves of FIB-patterned structures cannot be fully explained in terms of the AMR effect, indicating the possible contributions from DWMR. We have shown that it is possible to design submicron integrated ferromagnetic stripes/wires to selectively achieve the position control of domain walls, which have the potential application in nanowire memory devices, e.g. the RaceTrack memory.
4.2.2 Magnetisation Reversal with Domain Wall Motion in Spin-valve Nano Devices with Two Different Pinning Sites

4.2.2.1. Introduction

In section 4.2.1, we reported a study of different magnetisation reversal processes in NiFe nanodevices with different pinning sites.

This section is focused on the magnetisation switching process in the spin-valve nano-device with the same geometry, as the spin-valve structure has a higher MR ratio, providing a more sensitive way of investigating the magnetisation configurations. In the experiments, the blanket spin-valve film was first photolithographically patterned into a micron stripe. Then, using FIB nanofabrication, a submicron triangular element and a 150 nm-wide nanoconstriction were cut in the middle of the stripe. A combination of the above two different pinning sites is effective in trapping the domain walls and controlling their propagation directions. The motion of domain walls can be indirectly detected by MR measurements and reproduced in the micromagnetic simulations.

4.2.2.2. Experiment

The synthetic exchange-biased spin valves of Ta (2 nm)/NiFe (FL) (4 nm)/Cu (2.5 nm)/CoFe (PL₁) (2 nm)/Ru (0.7 nm)/CoFe (PL₂) (2.5 nm)/IrMn (8 nm)/Ta (2 nm) were deposited by the Nordiko PVD system on thermally oxidised Si wafers (Dr Zhengqi Lu fabricated such spin-valve films years ago, which were also used in my study of spin-valve nanorings in section 4.3). The base pressure for deposition was below $3.0 \times 10^{-8}$ Torr. The process conditions were optimised separately for each of the target
A uniform magnetic field of 50 Oe was applied in the substrate plane during the film deposition to induce a uniaxial magnetic anisotropy for the ferromagnetic layers. The annealing process was undertaken in an argon atmosphere with a magnetic field of 6 kOe applied along the easy axis of the free layer.

The spin-valve film on the blanket wafer was first photolithographically patterned into 10 µm wide micron stripes with a 0.8 µm-wide microconstriction in the middle. Then spin-valve nanodevices with two different pinning sites were further fabricated by FIB from this basic structure: a submicron stripe with a nano-sized triangular element and a 150 nm-wide nanoconstriction in the centre, as shown in Fig. 4.17 (a). The cuts were made with the Orsay Physics Canion 31 Plus UHV FIB using 30 kV gallium ions (5 pA beam current).

![Fig. 4. 17. (a) SEM image of a spin-valve nano-device (highlighted area) with a nano-sized triangular element and a 150 nm-wide nanoconstriction.](image-url)
Fig. 4.17. (b) SEM image of the nano-device (highlighted area) connected with big pads covered by four nonmagnetic electrodes.

The whole nano-device is split into two parts by the nanoconstriction and the triangular element, and each end of the submicron stripe is connected to a big contact pad (Fig. 4.17 (b)), which is part of the structure and covered by four nonmagnetic electrodes. These nonmagnetic electrodes composed of Ta (5 nm)/Cu (100 nm)/Ta (5 nm) layers, which were processed by photolithography and lift-off process based on a mix-and-match technique, are used to supply a dc current and perform MR measurements with a standard four-point probe technique (the two inner electrodes are used for the voltage measurement, and the outer two for the current input). The magneto-transport properties have been measured at room temperature by a Keithley 2182 Nano-Voltmeter and a Keithley 6221 AC and DC current source. All MR curves mentioned in this section were measured longitudinally with the external magnetic field applied along the long axis of the stripe (the easy-axis of the free layer).

4.2.2.3. Results and Discussion

The MR measurement of the unpatterned spin-valve film was taken before patterning, showing MR $\sim 7.8\%$. In the high-field MR measurement (Fig. 4.18 (a)), the magnetisations of PL$_1$ and PL$_2$ of SAF are kept at the antiparallel alignment in a magnetic field up to 1 kOe. An effective exchange field ($H_{ex}$) of 1.3 kOe (defined as the field at which the MR amplitude is half of the maximum in a small field) is obtained. The two strongly antiferromagnetically coupled CoFe layers of the SAF are finally brought into a parallel orientation in a field of 5 kOe.
Fig. 4. 18. (a) High-field MR curve and (b) low-field MR curve of the unpatterned spin-valve film, and (c) low-field MR curve of a submicron spin-valve stripe with a microconstriction.

The low-field (minor) MR curve of the unpatterned spin-valve film, as shown in Fig. 4. 18 (b), gives an MR of 7.8%, which reflects the magnetisation reversal of the NiFe free layer only. A -14 Oe shift of the minor MR curve can be attributed to the Néel coupling.

After patterning by photolithography and ion-beam milling, the submicron spin-valve stripe with a microconstriction shows an MR of 7.4% during the low-field measurement, as presented in Fig. 4. 18 (c).

By using FIB milling, two different pinning sites were patterned in the submicron spin-valve stripe. The low-field MR curve of such a spin-valve nano-device exhibits
several sharp resistance transitions, as shown in Fig. 4.19.

![Low-field MR curve of the FIB-patterned spin-valve nano-device with a triangular element and a 150 nm-wide nanoconstriction.](image)

Fig. 4.19. Low-field MR curve of the FIB-patterned spin-valve nano-device with a triangular element and a 150 nm-wide nanoconstriction.

The magnetoresistance of the patterned nano-device is dominated by the GMR effect and therefore controlled by the relative magnetisation alignment of the free and pinned layers. Thus the resistance transitions in Fig. 4.19 only correspond to different magnetisation configurations in the NiFe layer, since there is no magnetisation reversal in the pinned CoFe layers of the SAF structure at low field measurements.

The GMR ratio of the FIB-patterned nano-device, which is about 0.72%, is much lower than that of the unpatterned spin-valve film (~7.8%) and patterned spin-valve stripe (~7.4%). The GMR degradation can be attributed to the Ga⁺ ion induced intermixing. As has been found, the high-energy Ga⁺ ions (30 keV) are responsible for interface intermixing and the reduction of interface effects like GMR [210-211]. Halo ions of the outer beam profile hit the device and reduce the GMR effect by low-dose intermixing. Most of the halo ions are blocked by the 15.2 nm thick materials above the Cu spacer.
layer responsible for the GMR. The remaining GMR is still enough to monitor the
different domain states inside the nano-device.

The shift of the minor MR curve is -15 Oe for the spin-valve nano-device, similar to
that of the unpatterned films (-14 Oe), so there is no obvious change in the interlayer
coupling after FIB patterning.

As shown in Fig. 4.19, the low-field MR curve of the nano-device displays three abrupt
transitions when the field sweeps from positive to negative. These distinct resistance
transitions correspond to three different magnetisation states in the NiFe layer
respectively, which are labelled as $L_1$, $L_2$, and $L_3$ in Fig. 4.19. A positive field of 100 Oe
is applied to saturate the magnetisation of the NiFe layer prior to the MR measurement.
When the field changes to negative, the first sharp increase of the resistance ($L_1$)
appears in a small field of -20 Oe, followed by the second resistance transition ($L_2$)
when the field decreases to -24 Oe. Then in a field of -29 Oe, the resistance quickly
jumps up to the high resistance state, which is the third sharp transition of the resistance
($L_3$).

Similarly when the field sweeps from negative to positive, the low-field MR curve also
shows three sharp resistance changes ($L_1'$, $L_2'$ and $L_3'$). Obviously these resistance
transitions are shifted to the left (similar to the minor MR curve), which can be
attributed to the interlayer coupling between the magnetic layers.

The magnetisation reversal process and MR curve of the nano-device with two different
pinning sites have been simulated using LLG Micromagnetics Simulator [194]. 3D
micromagnetic simulations have been done based on a NiFe/Cu/CoFe nanoring stack. A cell size of 10 nm x 10 nm x thickness was used for each layer. Parameters used in the simulations for Permalloy were the saturation magnetisation $M_s=8\times10^2$ emu/cm$^3$, the exchange constant $A=1.05\times10^{-6}$ erg/cm and the uniaxial anisotropy constant $K_{u2}=1.0\times10^3$ erg/cm$^3$; parameters for CoFe were $M_s=1.5\times10^3$ emu/cm$^3$, $A=2.05\times10^{-6}$ erg/cm, $K_{u2}=1.0\times10^4$ erg/cm$^3$.

![Simulated MR curve of the FIB-patterned spin-valve nano-device with a triangular element and a 150 nm-wide nanoconstriction.](image)

Fig. 4. 20. Simulated MR curve of the FIB-patterned spin-valve nano-device with a triangular element and a 150 nm-wide nanoconstriction.

Although the whole SAF was not included in the simulation volume to save the computation time, a pinning field of 3 kOe was applied to the CoFe layer, and an interlayer coupling field of -15 Oe between the NiFe and CoFe layers was also added to the input. The contact pads were included in the simulations, but not shown here due to limited space. The micromagnetic simulation results provide an insight into our experimental results, particularly the MR curve, as shown in Fig. 4. 20.

As found in the simulations, when the field sweeps from positive from negative, the two big pads and the right two-step stripe first begin the magnetisation switching of the
NiFe layer, corresponding to the first abrupt resistance transition in Fig. 4. 19, which is followed by the pinning of the domain wall at the nanoconstriction, as shown in Fig. 4. 21 (b). With the increase of the negative field, the left nano stripe starts the NiFe magnetisation reversal (Fig. 4. 21 (c)), which results in the second sharp increase of the resistance in Fig. 4. 19. As a result, the domain wall moves towards the base of the triangular element, where it feels the pinning due to the sharp expansion in width. Finally a higher field helps the domain walls annihilate at the triangular element and the nanoconstriction (Fig. 4. 21 (d)), which results in the last resistance jump-up in Fig. 4. 19. Meanwhile, the NiFe layer reaches the resistance saturation state.
Fig. 4. 21. Simulated magnetisation configurations of the NiFe layer of the FIB-patterned spin-valve nano-device (a) at the remanent state, (b) at the switching field of the micron-stripe, (c) at the switching field of the left part of the patterned stripe, and (d) in a field higher than the pinning fields of the triangular element and the nanoconstriction.

From Fig. 4. 19 and Fig. 4. 20, we see that the simulated MR curve agrees well with the experimental MR curve, which shows three sharp resistance transitions when the external field switches from positive to negative or vice versa.

Overall, the magnetisation switching behaviour of the NiFe layer of the spin-valve nano-device is very similar to that of the single-layer NiFe counterpart, as discussed in section 4.2.1. The only difference shown in this section is that the switching fields of three different parts (micro stripe, nano stripe and the pinning sites) of the spin-valve device are lower than those of the counterpart, which can be explained by the very thin NiFe layer (~4 nm) of the spin-valve structure, compared with the 15 nm thick single-layer counterpart.

4.2.2.4. Conclusion

In summary, domain wall motion induced magnetisation reversal processes in spin-valve nanodevices with two artificial pinning sites have been studied by low-field MR measurements and the micromagnetic simulations. It has been shown that in low fields, by trapping domain-walls at different pinning sites, the control of magnetisation switching procedure can be realised in the NiFe layer of submicron spin-valve nanodevices, as found in NiFe nanodevices with the same geometry.
4.3 Magnetoresistance, Domain Wall Motion and Magnetic Switching in NiFe and Spin-valve Nanorings

The main content of section 4.3.1 is motivated by Dr Zhengqi Lu’s research on NiFe nanorings. The author has conducted micromagnetic simulations to clarify and demonstrate the experimental data of Dr Lu, and then extended this research to spin-valve based nanorings.

4.3.1 Magnetoresistance, Domain Wall Motion and Magnetic Switching in NiFe Nanorings

4.3.1.1. Introduction

Nanoscale magnetic elements have received considerable attention recently due to the potential applications in high-density magnetic memory devices [212]. One key issue in these applications is to achieve controlled magnetic switching that is both fast and reproducible.

The magnetic switching mechanisms of different geometries, such as wires, rectangles, needles, cylinders, and disks, have been studied, but it was found that their magnetic states are hard to control. To achieve the controllable magnetic switching, one needs to have well-defined and reproducible magnetic states. A promising candidate that fulfils these criteria is the ring structure [107, 148, 213]. There exist two distinct stable states in a narrow ring at remanence, the “vortex” state with a flux-closure domain and the “onion” state with two head-to-head/tail-to-tail domains. Rings under a combination of an applied field and a current exhibit a range of switching behaviours from the forward
onion state to the reverse onion state, via a single switching process (forward onion to reverse onion), or a double switching process (onion to vortex, and then vortex to reverse onion).

In this work, we have constructed a ring structure connected to the nano stripe with nanoconstrictions. We present a systematic magnetoresistance study of the ring with various applied currents at different field angles. Evidence for the current-induced domain wall motion is also presented based on MR measurements.

4.3.1.2. Experiment (All the experimental work discussed below was conducted by Dr Lu.)

The NiFe nanoring connected to the nano stripe with nanoconstrictions was fabricated by e-beam lithography using LOR/PMMA bilayer resists. A 70 nm LOR was first coated and pre-baked in an oven at 180°C for 20 minutes. A second layer of resist, 70 nm PMMA (MW 350k), was then spin-coated and baked in an oven at 180°C for 1 hour. E-beam direct writing was carried out with a high-resolution vector beam writer (VB6) from Leica at 100 kV beam energy and 500 pA beam current at the Rutherford Appleton Laboratory. The exposed resist was developed in a mixture of MIBK: IPA (1:3) for 60 seconds. The PMMA layer defined the geometry of the devices. The LOR resist in the open area was then dissolved in CD26 developer, creating a clear undercut beneath the PMMA layer. A SEM image of a patterned structure is shown in Fig. 4. The 125nm-wide stripes have two 30 nm-wide constrictions. The ring has an outer diameter of 750 nm and an inner diameter of 500 nm. A film stack of Ta 2nm/NiFe 5nm/Ta 2nm
was then deposited by the magnetron sputtering system. The lift-off process was carried out by soaking the sample in 1165 stripper. Using a mix-and-match technique, four nonmagnetic electrodes made of 2nm Ta/200 nm Cu/2 nm Ta films were formed to connect the central part of the device by the optical lithography. The magneto-transport properties were measured at room temperature by a Keithley 2182 nanovoltage meter and a 6221 AC/DC current source. The magnetic field was applied in the plane and the field angle was measured with respect to the long axis of the stripe. The dc current was up to ±1 mA, which corresponds to a current density up to $10^8$ A/cm$^2$.

Fig. 4. SEM images of (top) a patterned NiFe nanoring connected to the nano stripe with 30nm wide nanoconstrictions and (bottom) a nanoring device connected to four nonmagnetic electrodes.

4.3.1.3. Results and Discussion

4.3.1.3.1. Field-induced Magnetisation Switching

MR measurements were taken with varied currents in magnetic fields along different angles. Positive current corresponds to the electrons flowing from right to left, and vice
versa. A typical MR curve with a current of 0.15 mA in a magnetic field along the 30° direction is shown in Fig. 4.23 (a).

![MR curves of a patterned NiFe nanoring device for a variety of currents at different field angles](image)

Fig. 4.23. MR curves of a patterned NiFe nanoring device for a variety of currents at different field angles: (a) current $I=0.15$ mA, field angle $\theta=30^\circ$ (critical switching fields $H_{d1}$, $H_{d2}$, $H_{d3}$ and $H_{d4}$ are labeled with dotted lines); (b) $I=0.02$ mA, $\theta=90^\circ$; (c) $I=0.05$ mA, $\theta=90^\circ$; (d) $I=0.15$ mA, $\theta=90^\circ$.

Two distinct jumps are observed with a sweeping field, which indicates that there are two different configurations for the magnetisation in the ring. Starting with the field saturated along 30°, the magnetisation in the ring changes from configuration $a$ towards configuration $b$ after relaxing the field, indicated by the gradual increase in resistance. When it reaches configuration $b$, the maximum resistance state is reached. When the field arrives at the first critical switching field $H_{c1}$, there is a sharp drop in resistance, corresponding to the magnetisation in the ring switching from configuration $b$ to configuration $c$. As the field is decreased to the second critical switching field $H_{c2}$, a
clear jump up in resistance occurs, which may indicate that the magnetisation of the ring switches from configuration \(c\) to configuration \(e\). Configuration \(d\) is finally reached at the negative saturation field. When the applied field sweeps back from the negative field, the magnetisation in the ring first switches from configuration \(e\) to configuration \(f\) at the third critical switching field \(H_{c3}\), and then to configuration \(b\) at the fourth critical switching field \(H_{c4}\). Configuration \(a\) is reached at the positive saturation field in the end.

The schematic magnetisation configurations (a-e) are shown in the insets of Fig. 4. 23 (a) based on micromagnetic simulations below.

It can be seen in Fig. 4. 23 (a-b) that the direction of the external magnetic field can affect the magnetisation reversal of the ring element. For the magnetic field tilted away from the long axis direction of the stripe (e.g. the 30° field, Fig. 4. 23 (a)), when the current is low, the MR curve shows two resistance drops at the critical switching fields \(H_{c1}/H_{c3}\), followed by two sharp resistance rises at the critical switching fields \(H_{c2}/H_{c4}\).

For a field perpendicular to the long axis of the stripe, an enhancement of MR is observed near zero fields at a low current as shown in Fig. 4. 23 (b). When the current density approaches a critical current density (\(\sim 2 \times 10^7\) A/cm\(^2\) in the constriction, corresponding to a current of \(\sim 150\) µA), the MR enhancement is still observed during the magnetic field sweep from negative to positive. However, there is no MR enhancement when the magnetic field sweeps from positive to negative as shown in Fig. 4. 23 (c). This asymmetry may come from the defects in the ring. With a further increase of the current, no enhancement in MR is observed for either sweep direction of the field as shown in Fig. 4. 23 (d). The enhancement in MR might be attributed to the
domain wall trapped at the constrictions [214]. When a domain wall is present at the constriction, there is an additional magnetoresistance, termed domain wall magnetoresistance. The domain wall is depinned when the applied current density is above the critical density due to the current-induced domain wall motion. In our experiments, it is estimated that the enhanced MR is ~0.12% by comparing Fig. 4. 23 (b) with (d). Here it needs to be noted that domain wall MR may also exist when a 30° field is applied, but any small MR enhancement at zero fields can be easily masked by the near-zero-field dips on the MR curve (as showed in Fig. 4. 23 (a), there are two sharp drops in the resistance near zero fields, corresponding to the magnetisation in the ring switching from the onion state to the vortex state).

![Simulated MR curve of a patterned nanoring device in a 30° magnetic field](image)

Fig. 4. 24. Simulated MR curve of a patterned nanoring device in a 30° magnetic field (critical switching fields $H_{c1}$, $H_{c2}$, $H_{c3}$ and $H_{c4}$ are labeled with dotted lines).

To investigate the magnetisation configurations of the NiFe nanoring, the micromagnetic equilibrium equation was solved for each applied field on a square mesh using LLG Micromagnetics Simulator [194]. The intrinsic parameters of NiFe used here
are the saturation magnetisation $M_s = 8 \times 10^2$ emu/cm$^3$, the exchange constant $A = 1.05 \times 10^{-6}$ erg/cm and the uniaxial anisotropy constant $K_{u2} = 1.0 \times 10^3$ erg/cm$^3$. A cell size of 10 nm x 10 nm was used.

It can be seen in Fig. 4. 24 that the simulated MR curve in a 30° field, as shown in Fig. 4. 24, agrees qualitatively with the experimental curve in Fig. 4. 23 (a). Because only the AMR effect was considered in the simulation, the experimental MR curve in a 30° field can be explained by the AMR effect. The distinct jumps in the MR curve are due to changes in the domain state of the ring element (onion to vortex, or vice versa) and the magnetisation configurations of the nano stripes.

![Fig. 4. 25. (a - e) Simulated magnetisation configurations of the nanoring device when the external field sweeps from right to left along the 30° direction: (a) shifted forward onion state, (b) forward onion state, (c) incomplete vortex state, (d) reverse onion state, (e) shifted reverse onion state.](image_url)
It is shown in the micromagnetic simulation in a 30° field that, when the field decreases from the positive saturation to zero, the magnetisation of the ring switches from configuration \(a\) (shifted forward onion state, Fig. 4. 25 (a)) to configuration \(b\) (forward onion state, Fig. 4. 25 (b)). Due to the 30° field, the head-to-head domain wall is away from the notch (shifted forward onion state) in a high positive field, but then it moves to the notch (forward onion state) after relaxing the field. Meanwhile, the device resistance rises as the magnetisation in the ring is gradually aligned with the current direction. At remanence the maximum resistance is seen, as the magnetisation and the current are both parallel to the perimeter of the ring. When the field reaches \(H_{c1}\), configuration \(c\) (incomplete vortex state) is formed in the ring. Fig. 4. 25 (c) shows that there is a domain wall trapped at the right ring notch, and the spins are tilted away from the current direction in both the ring and stripe, which results in a drop in the resistance in Fig. 4. 23 (a). Here it needs to be noticed that this vortex state is incomplete due to the trapping of the domain wall at the notch, which agrees with related research on the magnetic switching in NiFe ring structures with notches by M. Kläui et al. [215]. As the field arrives at \(H_{c2}\), the reverse onion state (configuration \(d\), Fig. 4. 25 (d)) is created in the ring, followed by the magnetisation switching of the right stripe, when the domain walls depin from the notch and the nanoconstriction, leading to a jump up in resistance. Configuration \(e\) (shifted reverse onion state, Fig. 4. 25 (e)) is finally reached in a higher negative field.

Based on the simulations, we know that when the external field is along the 30° direction, for a low current, both transitions from the forward onion state to the reverse
onion state and from the reverse onion state to the forward onion state go through a double switching process (onion to vortex and vortex to reverse onion and backwards). In addition, the main MR contribution is from the AMR effect in a 30° field.

For a magnetic field perpendicular to the stripe long axis, both transitions (onion to reverse onion and vice versa) go through a single switching process without the vortex state at all values of the current, as shown in Fig. 4. 23 (b-d). It was found in the simulations that the magnetisation is pointing upwards at the forward onion state, and downwards at the reverse onion state, as shown in Fig. 4. 26 (a, c). In addition, there is a metastable intermediate/left onion state (Fig. 4. 26 (b)) with the magnetisation pointing to the left when the domain wall is pinned at the left notch of the ring. There is no MR enhancement seen in the simulation that is based on the AMR effect, so the enhanced MR might be due to the domain wall scattering at the constrictions.

Fig. 4. 26. (a - c) Simulated magnetisation configurations of the nanoring device when the external field sweeps from upwards to downwards in the 90° direction: (a) forward onion state, (b) intermediate/left onion state, (c) reverse onion state.
4.3.1.3.2. Current-assisted Magnetisation Switching

To fully investigate the influence of the current on the domain wall, MR curves were taken with various currents for the applied field along 45° in the plane. The positive current corresponds to the electrons flowing from right to left. A typical MR curve at a current of +20μA is shown in Fig. 4. 27 (a), which is similar to what is observed in a 30° field in Fig. 4. 23 (a). There are two distinct jumps observable when sweeping the field, which indicates different magnetisation configurations in the ring structure. On the basis of the simulations, the MR curve in Fig. 4. 27 (a) can be explained as follows. Starting with the applied field saturated along 45° direction, the magnetisation in the ring changes from the shift forward onion state (configuration a) towards the forward onion state (configuration b) after relaxing the field, when the resistance gradually increases as the magnetisation in the ring is more aligned in the direction of the current. When it reaches configuration b, where the head-to-head domain wall moves to the notch, a maximum MR is seen, as the magnetisation and the current are parallel to the perimeter of the ring. When the field arrives at the first critical switching field $H_{c1}$, the magnetisation in the ring switches from the forward onion state (configuration b) to the incomplete vortex state (configuration c), which corresponds to a clear drop in MR. Similar to the switching behaviour with a 30° field, there is a domain wall trapped at the right ring notch, where the magnetisation tilts away from the current direction, hence the resistance is lowered. As the field decreases to the second critical switching field $H_{c2}$, the magnetisation in the ring switches from configuration c to the reverse onion state (configuration e), which is reflected by a jump up in MR as the trapped domain walls
are annihilated. The shifted reverse onion state (configuration d) is reached at the negative saturated field. When the applied field sweeps back from the negative field, the magnetisation in the ring switches from the reverse onion state (configuration e) to the incomplete vortex state (configuration f) at the third critical switching field $H_{c3}$, and then to the forward onion state (configuration b) at the fourth critical switching field $H_{c4}$, followed by the formation of the shifted forward onion state (configuration a) at the positive saturated field.

![Image of MR curves](image)

**Fig. 4.** The MR curves of a patterned nanoring device in a 45° magnetic field for different currents: (a) $+20\ \mu\text{A}$, (b) $+500\ \mu\text{A}$, (c) $+1000\ \mu\text{A}$ and (d) $-700\ \mu\text{A}$. The circles represent the MR curve sweeping from positive to negative and the triangles represent the MR curve sweeping from negative to positive.

MR curves at different currents are presented in Fig. 4. Clearly the applied current affects the switching behaviour of the ring elements. When the current density is lower than the critical current density, both transitions (from the forward onion state to the
reverse onion state and vice versa) go through the double switching process (onion to vortex and vortex to reverse onion, and backwards). For the positive current, when the applied current is higher than 500 μA, which corresponds to a current density of $4 \times 10^7$ A/cm$^2$ in the ring, the transition from the forward onion state to the reverse onion state becomes a single switching process (onion to reverse onion directly), while the transition from the reverse onion state to the forward onion state is still through the double switching process. Both transitions go through the single switching process with an applied current of higher than 900 μA. For the negative current, when the applied current is higher than 700 μA, corresponding to a current density of $5.6 \times 10^7$ A/cm$^2$ in the ring, the transition from the forward onion state to the reverse onion state goes through the double switching process. But the transition from the reverse onion state to the forward onion state goes through the single switching process. The asymmetry for the negative and positive currents may come from the defects in the ring. Both transitions go through the single switching process with an applied current of higher than 1000 μA.

![Fig. 4. Switching fields ($-H_{c2}$ and $H_{c4}$) from the vortex state to the onion state as a function of the current.](image)

The switching fields ($-H_{c2}$ and $H_{c4}$) from the vortex state to the onion state for different
currents are shown in Fig. 4. 28. The switching field $-H_{c2}$ decreases monotonously with increasing the positive current, so does the switching field $H_{c4}$ with increasing the negative current. The transition from the vortex state to the onion state is a nucleation process, followed by the spread of the reverse domain. According to the current induced domain wall drag due to spin transfer predicated by Hung and Berger [116], the electron flow in the same direction as the domain wall motion exerts an exchange torque on the constituent electrons of the domain wall, effectively promoting the spread of the reverse domain, which reduces the switching field ($-H_{c2}$ for the positive current and $H_{c4}$ for the negative current). While the electron flow in the opposite direction resists the spread of domain, leading to the increase of the switching field ($-H_{c2}$ for the negative current and $H_{c4}$ for the positive current). However, in our experiments, it is noticed that the increase in the switching field for an electron flow opposite to the propagation direction of the domain wall is smaller than the decrease in the switching field for an electron flow in the direction of the domain wall propagation for both $-H_{c2}$ and $H_{c4}$.

Furthermore, it is found that the switching fields for both high positive and negative currents are lower than those with weak currents. This suggests that in addition to the directional spin-torque effect, there is a symmetric effect that lowers the switching field for the applied current regardless of the direction. As the dc current is increased, the symmetric effect starts to dominate and the switching field decreases. There are three possible mechanisms, which could explain the symmetric effect. The first mechanism is the hydromagnetic domain wall drag force, which is associated with the Hall effect but negligible in films thinner than 100 nm, so this effect was ruled out in our research.
The second mechanism is the Oersted field, which is not enough to force the domain wall transition as it is rotational. The third mechanism is the Joule heat effect, which reduces the local pining force on the domain wall independent of the current direction. This effect may dominate in the high current situation. Thus the Joule heat makes a contribution to the reduction of the switching field at a high current.

It is known that the equilibrium magnetic states and magnetisation reversal processes are determined by the energy minimum. Two states (onion and vortex) in the ring mainly come from competition between the magnetostatic (stray field) energy and exchange energy, which depend on the geometric parameters such as the film thickness, ring width and ring diameter when there is no magnetic field or current. The onion state shows high stray-field energy, while the vortex state shows low stray-field energy but high exchange energy due to the strongly twisted spins. Although the vortex is more stable at remanence in our experiment, the vortex state is not observed at a current higher the critical current. In this situation, the nucleation of a reverse domain is more favourable than the depinning of a domain wall due to the spin torque transfer effect: the magnetisation in the ring directly switches from the forward onion to the reverse onion without going through the vortex state for a positive current as shown in Fig. 4. 27 (b) and so does the magnetisation in the ring switch from the reverse onion to the forward onion for a negative current shown in Fig. 4. 27 (d). With a further increase of the applied current, the thermal excitation can overcome energy barriers and force the ring to switch directly from the forward onion state to the reverse onion state and backwards without passing the vortex state.
4.3.1.4. Conclusion

MR measurements have been taken on the NiFe nanoring connected to the nano stripe with nanoconstrictions to investigate the magnetic switching process as a function of the field direction and the applied current.

For the transversal magnetic field, an enhancement in MR is observed around zero fields at a low current, which is due to the domain wall trapped at the nanoconstriction. No enhancement is observed with the current density above $3 \times 10^7$ A/cm$^2$. But the magnetisation in the ring goes via the single onion-to-onion switching process regardless of the current value.

For the applied field tilted away from the perpendicular direction, it is shown that the current has an effect on the switching fields of the magnetisation configurations of the ring and therefore on the transition process. The magnetisation in the ring goes via a double switching process (onion - vortex - reverse onion) at a low current, but through a single switching process (onion - reverse onion) when the current density is higher than the critical current density.

The switching field from the vortex state to the onion state decreases with increasing the current when the electron flow is in the direction of the domain wall propagation, while the opposite electron flow increases it.

Based on Dr. Lu’s experimental data, I have conducted micromagnetic simulations and found the detailed magnetic switching process: a double switching process for a 30° field, but a single switching process for a 90° field. It has also been confirmed that the
MR signal can be explained by the AMR effect when the magnetic field is in the 30° direction, but the enhanced MR in a 90° field may come from the domain wall scattering at the constrictions rather than the AMR effect.
4.3.2 Size Effect on Magnetic Switching and Interlayer Magnetostatic Coupling in Spin-valve Nanorings Exchange-biased by Synthetic Antiferromagnets

4.3.2.1. Introduction

Considerable studies of magnetic ring structures have been carried out in recent years due to their potential application in high-density MRAM [133, 147-148, 152-153, 155-157, 160-161, 216-217]. To date most of the work has focused on single-layer NiFe or Co rings.

Compared with single layer ring elements, ring-shaped multilayer structures, such as spin-valve and MTJ structures with higher MR ratios, provide a more sensitive way of investigating the magnetisation configurations in the free and pinned layers. Since Zhu et al. achieved robust magnetic switching in the multilayer GMR rings with enhanced MR response [162], there has been recent interest in the magnetisation reversal in ring-shaped multilayer structures, e.g. spin-valve NiFe/Cu/Co/IrMn rings, pseudo-spin-valve NiFe/Cu/Co rings [163-170], and MTJ rings [171-172, 218]. In the spin-valve rings, the free layer shows a switching mechanism different from that of single-layer rings, which is caused by the strong interlayer magnetostatic coupling. As a result, they present asymmetric minor MR curves with large shifts.

Adding a SAF structure to spin valves or MTJs is a good way to decrease the shift of the minor MR curve by reducing the interlayer magnetostatic coupling, which is desirable for better device performance in many applications, such as MRAM, read
heads or magnetic sensors. So far there are no reported data on the behaviour of SAF-pinned spin-valve rings, and in particular there are no investigations of the dependence of the magnetisation switching process on the magnetostatic coupling in such rings, in spite of their importance.

Here we present a study by a combination of MR measurements and micromagnetic simulations of the size effect on the magnetic switching behaviour and the interlayer magnetostatic coupling in SAF-pinned spin-valve rings. In our research, SAF-pinned spin valves were adopted to reduce the magnetostatic coupling between the free and pinned layers, aiming for minor MR curves with small shifts.

4.3.2.2. Experiment

Spin valves with a SAF structure of (substrate)/Ta (2 nm)/NiFe (4 nm)/Cu (2.5 nm)/CoFe (2 nm)/Ru (0.7 nm)/CoFe (2.5 nm)/IrMn (8 nm)/Ta (2 nm) were deposited by a Nordiko PVD system on a thermally oxidised Si wafer. The base pressure for the deposition was below $3 \times 10^{-8}$ Torr. The process conditions were optimized separately for each of the target materials. A uniform magnetic field of 50 Oe was applied in the substrate plane during the deposition to induce an in-plane uniaxial magnetic anisotropy for the ferromagnetic layers. The annealing process was undertaken at 250°C in an argon atmosphere in a magnetic field of 6 kOe applied along the easy axis of the free layer.

Samples were first photolithographically patterned into the shape of a submicron disk connected with stripes and injection pads in both ends, as shown in Fig. 4. 29 (a). The
injection pads are connected to four large nonmagnetic electrodes (Fig. 4.29 (b)) of Ta (5 nm)/Cu (100 nm)/Ta (5 nm), which serve as electrical connection pads to perform MR measurements with a standard four-point probe. Nanorings were then fabricated by focused-ion beam (FIB) from this basic structure, as shown in Fig. 4.30. A 100 nm wide nanoconstriction on the right stripe was also cut by FIB. The cuts were made with the Orsay Physics Canion 31 Plus UHV FIB using 30 kV gallium ions (5 pA beam current) [189]. It needs to be pointed out that our ring structure is different from those used in other work, due to the special sample fabrication procedure (photolithography plus FIB milling) and the requirement of our MR measurement system. Such a geometry (injection pads, stripes and a nanoconstriction) was initially adopted for research into the domain wall MR in the single-layer NiFe nanorings [207, 219] and also used in our previous investigation of the BMR effect [220].

Fig. 4.29. SEM images of (a) a submicron spin-valve disk connected to the injection pads before FIB patterning and (b) a device connected to four nonmagnetic electrodes.

As shown in Fig. 4.30, nanoring A has an outer diameter of 1600 nm and an inner diameter of 1200 nm; nanoring B has the outer and inner diameters of 600 nm and 200 nm, respectively. The ring width is 200 nm for both types of rings. The nano stripes are
200 nm-wide for both nanorings, and 1000 nm and 600 nm-long for nanoring A and nanoring B, respectively.

The magneto-transport properties of three types of samples, unpatterned spin-valve films, nanoring A and nanoring B, have been measured at room temperature using a 4-point probe system equipped with a Keithley 2182 Nano-Voltmeter and a Keithley 6221 AC and DC current source. The magnetic field is applied in the plane of the substrate along the easy axis of the films.

![SEM images of (a) nanoring A and (b) nanoring B connected with nano stripes (with a 100 nm-wide nanoconstriction in the right stripe).](image)

Fig. 4. 30. SEM images of (a) nanoring A and (b) nanoring B connected with nano stripes (with a 100 nm-wide nanoconstriction in the right stripe).

4.3.2.3. Results and Discussion

The minor (low field) MR curves of the unpatterned synthetic spin valve films, nanoring A, and nanoring B are shown in Fig. 4. 31 (a), (b) and (c), respectively. The unpatterned spin valve sample has a GMR of 7.8%. A shift of -10 Oe was observed in the minor MR curve, which was due to the Neel coupling [173-174], also referred to as the "orange peel" coupling. The GMR for both nanorings is about 1.3%, which is much lower than that of the unpatterned spin-valve films. And the shifts of the MR curves are -12 Oe and -25 Oe for nanoring A and nanoring B, respectively, higher than that of the
unpatterned films.

Fig. 4. Minor (low-field) MR curves of (a) unpatterned films, (b) nanoring A and (c) nanoring B.
and the upper right inset shows the simulated MR curve of the corresponding nanoring, respectively. The lower left inset of (b) (courtesy of T. J. Hayward et al. [221]), is the minor MR curve showing a shift of 40 Oe of the Co (8 nm)/Cu 6 nm/NiFe (6 nm) pseudo-spin-valve ring with the outer diameter of 2 μm and the width of 180 nm.

As shown in Fig. 4. 31 (b), the minor MR curve of nanoring A exhibits four distinct resistance levels labelled as L₁, L₂, L₃ and L₄. Starting from a positive field of 200 Oe, as the field sweeps from positive to negative, the first big transition occurs at a small negative field, leading to the first resistance plateau (L₁) followed by another two intermediate resistance states: the second (L₂) and the third plateaus (L₃). The high resistance state (the fourth transition, L₄) is observed at a field of -120 Oe. While for nanoring B, its minor MR curve shows only two distinct resistance levels, which are labeled as L₁' and L₂' in Fig. 4. 31 (c), respectively. These resistance levels only correspond to different magnetisation configurations in the NiFe free layer, since there is no magnetisation reversal in the CoFe pinned layers at low field measurements.

The large GMR ratio drop in the patterned nanoring device can be attributed to the Ga⁺ ion induced intermixing during the FIB fabrication process. As has been found, the high-energy Ga⁺ ions (30 keV) are responsible for the interface intermixing and the reduction of interface effects like GMR [210-211]. Halo ions of the outer beam profile hit the device and reduce the GMR effect by low-dose intermixing. Most of the halo ions are blocked by the 15.2 nm thick materials above the Cu spacer layer responsible for the GMR. The remaining GMR is still enough to monitor the different domain states inside the ring structure.
Nanorings A and B show larger shifts in the minor MR curves than the unpatterned films. As we know, this can be ascribed to the increase of the magnetostatic interactions (the vector sum of the interlayer magnetostatic coupling field and the Néel coupling field) between the free and pinned layers of the SAF structure. Because the Néel coupling is independent of the feature size [173-174], the shift difference between the patterned ring elements and the unpatterned films is due to an increase in the interlayer magnetostatic coupling as the feature size reduces after patterning [176].

It is important to examine the contribution of each CoFe layer to the total interlayer magnetostatic interaction. Considering the thickness difference of the two CoFe layers, the negative magnetostatic coupling from the thin lower CoFe layer only partially cancels the positive coupling from the thick upper CoFe layer, so the net coupling field is positive. This net coupling field favours the parallel alignment of the free layer and the lower CoFe layer, the same as the Neel coupling acting on the free layer.

Based on the above analysis, the interlayer magnetostatic coupling in our SAF pinned spin-valve rings, especially those with an outer diameter of 1.6 μm, is much lower than that of spin-valve nanorings without the SAF [163-164, 167-169]. This can be further verified by comparing the shift of the minor MR curve between SAF-pinned nanoring A (-12 Oe shift) and a pseudo-spin-valve ring (with an outer diameter of 2 μm, -40 Oe shift), as shown in the lower left inset of Fig. 4.31 (b), and the much larger shift of the latter indicates a much higher magnetostatic coupling [221].

But it needs to be noted that the magnetostatic coupling rises with the decrease of the
ring diameter. As shown in Fig. 4. 31 (c), the shift of the minor MR curve of the small nanoring B is doubled compared with nanoring A, which may hint at a different magnetisation reversal process being responsible.

To clarify the magnetisation reversal mechanism in nanorings of different sizes, we have done a 3D micromagnetic simulation of the NiFe (4 nm)/Cu (2.5 nm)/CoFe (0.5 nm)/trilayer nanoring device using LLG Micromagnetics Simulator [194]. A cell size of 10 nm x 10 nm x thickness was used for each layer. Parameters used in the simulations for Permalloy were the saturation magnetisation $M_s=8\times10^2$ emu/cm$^3$, the exchange constant $A=1.05\times10^{-6}$ erg/cm and the uniaxial anisotropy constant $K_{u2}=1.0 \times10^3$ erg/cm$^3$; parameters for CoFe were $M_s =1.5\times10^3$ emu/cm$^3$, $A=2.05\times10^{-6}$ erg/cm, $K_{u2}=1.0 \times10^4$ erg/cm$^3$. A damping constant $\alpha$ of 0.5 and a GMR value of 1.3% were used in the computations.

Although the whole SAF was not included in the simulation volume to save the computation time, a pinning field of 3000 Oe was applied to the pinned layer, and an interlayer coupling field (different values for different ring sizes, the same as that obtained in the MR measurements) between the free and pinned layers was also added to the input. The injection pads were included in the simulations, but only part of them is shown below due to limited space.

For nanoring A, its simulated MR curve is given in the upper right inset of Fig. 4. 31 (b), which agrees well with the experimental MR curve. It is seen in the low-field simulation that the CoFe pinned layer stays in the single domain state, which can be explained by the large antiferromagnetic coupling from the SAF structure.
Fig. 4. Simulated magnetisation configurations in the NiFe free layer of nanoring A at (a) $H = 115$ Oe, (b) $H = -15$ Oe, (c) $H = -60$ Oe, (d) $H = -102$ Oe, (e) $H = -112$ Oe, respectively, with the external field sweeping from positive to negative (right to left).

The simulation also shows that the NiFe free layer of the ring stack exhibits a
single-domain state at the positive saturation field. When the field decreases from the saturation field, the magnetisation of the NiFe free layer gradually follows the circumference of the ring because of the shape anisotropy. The single-domain state then switches to the forward onion state, where there are two head-to-head/tail-to-tail domain walls formed at the joint between the ring and the nano stripes, as illustrated in Fig. 4. 32 (a). When the magnetic field decreases and sweeps from zero to negative, the first sharp transition occurs at a low negative field, where the magnetisation of the NiFe free layer in the injection pads is reversed as shown in Fig. 4. 32 (b), leading to the first sharp step increase of the resistance (due to the antiparallel configuration with respect to the pinned layer), followed by the first plateau (L₁). Further increase of the negative field promotes the magnetisation switching in the left stripe and in the right part of the right stripe (between the nanoconstriction and the right injection pad), as seen in Fig. 4. 32 (c), which corresponds to the second jump-up in resistance (L₂). There is no apparent magnetisation switching thereafter until the negative field reaches a point where the domain wall depins from the nanoconstriction, leading to a complete magnetisation reversal of the right stripe as shown in Fig. 4. 32 (d). In addition, the vortex state is formed in the ring area after a reverse domain wall sweeps across the lower half-ring and switches the magnetisation, which begins by the expansion of the reverse domain from the stripe, where the MR curve shows the third jump-up in the resistance followed by the third plateau (L₃). Eventually, a higher negative field leads to the fourth step increase in the resistance and the final resistance saturation state (L₄), when a reverse domain nucleates and grows in the upper half-ring to flip the magnetisation direction, leading to the formation of a reverse onion state in the ring (Fig. 4. 32 (e)). As presented 142
above, a vortex state is seen in the free layer of the SAF-biased spin-valve nanorings during the magnetisation reversal when the CoFe pinned layer is in the single-domain state, which is very similar to what we found in single-layer NiFe nanorings [207, 219], but different from the behaviour of the conventional spin-valve rings without the SAF, where normally no vortex state is shown in the free layer unless the pinned layer is in the vortex state [163-164, 167-169]. We also noticed that a vortex state is still achievable in the free layer of the conventional spin-valve nanorings by using asymmetric injection pads, and applying off-axis external fields or using asymmetric ring geometries [165-166, 170].

To summarise, nanoring A shows a normal double switching process (a forward onion state to a vortex state, and finally to a reverse onion state). From the shift of its minor MR curve, we know there is a magnetostatic coupling from the CoFe pinned layers, and the normal double switching process with the vortex state shows that such a coupling is small in this large nanoring.
For nanoring B, the simulated MR curve is also consistent with the experimental one, as shown in the inset of Fig. 4. 31 (c). Its simulated magnetisation switching process (see Fig. 4. 33) of the NiFe free layer is different from that of nanoring A, as expected from their different experimental MR curves. Starting from the positive saturation, at the beginning of the magnetisation switching, nanoring B behaves similarly to nanoring A. When the field decreases from the positive saturation to zero, nanoring B switches from the single-domain state to the forward onion state in Fig. 4. 33 (a), showing two head-to-head/tail-to-tail domain walls. The first sharp increase of the resistance is also observed at a low negative field, corresponding to the magnetisation reversal in the NiFe free layer of the two injection pads (Fig. 4. 33 (b)). However, due to the strong magnetostatic interaction between the free and pinned layers caused by the small diameter of nanoring B, it is too hard for the reverse domains in the NiFe free layer to expand quickly at a low field. Consequently, over a wide field range, there is no abrupt resistance change (or intermediate vortex state as seen in nanoring A). Instead, it shows a slow and continuous increase in the resistance (a gradual, upward slope L1, which is highlighted with a circle in Fig. 4. 31 (c)). Once the field is high enough to overcome
the magnetostatic interaction, the reverse domains in the NiFe free layer quickly initiate
the expansion into the nano stripes. The strong negative field also promotes the
annihilation of domain walls at the constriction, followed by the formation of a reverse
onion state in the ring: reverse domains quickly nucleate and sweep across the ring, and
the magnetisation direction is switched to negative. As a result, the previous
head-to-head/tail-to-tail domain walls annihilate and the reverse tail-to-tail/head-to-head
domain walls form at the joint between the nano stripes and the ring, which is shown in
Fig. 4.33 (c). At this point, the resistance quickly jumps up to the saturation state (L2),
showing the second sharp increase in the resistance.

In summary, nanoring B exhibits a single onion-to-reverse onion switching process
without the vortex state, which is different from the behaviour of nanoring A, but
comparable to that of the conventional spin-valve nanorings without the SAF [163-164,
167-169]. Such a similarity is obviously caused by the increased interlayer
magnetostatic interactions in a small nanoring.

As described above, there is a dependence of the magnetic switching behaviour on the
ring diameter. For a large diameter (nanoring A), the double transition process (onion -
vortex - reverse onion) occurs, due to a weak interlayer magnetostatic coupling; but for
a small diameter (nanoring B), the single transition process (onion - reverse onion)
appears, and no vortex state exists because of a much stronger magnetostatic coupling.

It can be seen that the interlayer magnetostatic coupling increases with a reduction of
the ring diameter. The reason lies in two mechanisms. Firstly as the ring diameter
decreases, magnetic moments cannot exactly follow the perimeter of the ring due to the
decrease of the circumferential shape anisotropy, leading to an enhancement of the
magnetostatic coupling from the ring edge [176]. Secondly when the ring becomes
smaller, the stray field from the domain walls in the pinned layers becomes more
important.

It needs to be noted that the interlayer magnetostatic field is very non-uniform across
the area of the ring. This magnetostatic field is minimised in the flux-closure vortex
state. But in the onion state, the magnetisation is not parallel to the edge and there are
two head-to-head/tail-to-tail domain walls, leading to surface charges. This causes a
large magnetostatic field near the ring edge/domain walls, and this field decreases
rapidly with increasing distance from the ring edge/domain walls, as found by M.
Laufenberg et al. [222].

As we know, it is the interlayer interaction between the free and pinned layers of a
spin-valve structure which causes a shift in the minor MR curve, so that nanoring A
shows a small shift in the minor curve can be explained by a weak interlayer
magnetostatic coupling, due to its large ring diameter, as discussed above.

As small nanorings are preferred for high-storage density MRAM, increasing the ring
diameter is not the ideal way to drop the interlayer magnetostatic coupling for smaller
shifts of the minor MR curves. It is well understood [175] that there are two other
options when adding the SAF structure to the spin-valve rings to reduce the
magnetostatic coupling: one is to minimise the thickness difference in the two CoFe
layers for a lower net magnetisation and a smaller effective CoFe thickness, resulting in
the lower magnetostatic interaction; the other is to make the upper CoFe layer a little
thinner than the lower layer, so that the polarity of the net magnetostatic coupling from
the CoFe layers is flipped to favour the antiparallel alignment of the free and pinned
layers. Thus the negative magnetostatic coupling can be used to compensate the Neel
coupling (favouring the parallel alignment), aiming for a near-zero net coupling field.

To summarise, we have investigated the effect of the ring size on the magnetic
switching process of the SAF-pinned spin-valve nanorings, and found a strong
dependence of the interlayer magnetostatic coupling on the ring diameter.

In addition, we noticed in our experiments that the injection pads also contribute to the
magnetoresistance, resulting in the first sharp resistance transition on the MR curve. To
check whether the unique switching behaviour of SAF-pinned nanorings is caused by
the injection pads, we carried out further simulations of isolated SAF-pinned rings
without injection pads. As shown in Fig. 4.34 (a-c) and (d-e), respectively, the large
isolated ring shows a double switching process with a vortex state, and the small one
shows a single switching process with no vortex state. The simulation results confirm
that the isolated rings behave in the same way as the rings with injection pads. It can
therefore be inferred that although the symmetric injection pads may have a small effect
on the magnetic switching of the nanoring, the switching behaviour of the ring is more
determined by the interlayer magnetostatic coupling, which depends on the ring
diameter.
Fig. 4. Simulated magnetisation configurations in the NiFe free layer of (a-c) isolated nanoring A and (d-e) isolated nanoring B without the injection pads with the external field sweeping from positive to negative (right to left).

4.3.2.4. Conclusion

In this work, a detailed picture of the magnetic switching behaviour of the synthetic spin-valve nanorings connected to nano stripes with a nanoconstriction has been given by a combination of MR measurements and 3-D micromagnetic simulations. The SAF-pinned nanoring devices exhibit asymmetric minor MR curves with different resistance levels, corresponding to different magnetisation configurations in the NiFe free layer, as clarified by the micromagnetic simulations. The magnetic reversal behaviour is influenced by the magnetostatic coupling as a function of the ring diameter. Large nanorings exhibit a double (onion - vortex - reverse onion) switching process, while small nanorings show a single (onion - reverse onion) switching process due to the strong magnetostatic coupling between the free and pinned layers. The interlayer magnetostatic coupling, which leads to a shift of the minor MR curve, is ring size
dependent. The large SAF-pinned spin-valve nanoring presents a smaller shift in the minor MR curve than the small nanoring.
4.4 FIB Nanofabrication and In-situ Magneto-transport Measurement of Thin NiFe Film Nanoconstrictions

This section presents the collaborative work with Hamburg University on the FIB fabrication and in-situ magneto-transport measurement results of elements containing nanoconstrictions of controlled dimensions. The experimental results were analysed in combination with the micromagnetic simulations. The author would like to thank the Hamburg group, especially Mr. Daniel Stickler, for the FIB nanofabrication, in-situ MR measurements, SEMPA observations and some valuable discussions.

4.4.1 Introduction

A reduction of domain wall (DW) width has been predicted in nano-scaled constrictions [10]. Controlling and manipulating domain walls are of considerable interest because of the relevant physics phenomena and the potential for applications such as magnetic logic and memory devices. From this point of view, it is critical to understand the magnetic structure of the domain wall and its contribution to the resistivity of a magnetic device.

Experiments on domain wall MR showing a positive effect have been studied on different ferromagnetic structures, e.g. thin films [223], constricted wires [214, 224] nanowires and micro-sized elements with a nanoconstriction [225].

Also of interest in this field is the possibility of pinning domain walls at constrictions and depinning them using a magnetic field or a current. We introduced a nanoconstriction to a submicron NiFe stripe by FIB milling, which then allowed us to
control the domain wall motion at the constriction, so that the resistance difference could be detected when the domain wall pins and annihilates at the constriction with the magnetic field sweep.

Based on photolithography pre-patterned submicron NiFe stripes, the fabrication of nanoconstrictions in the CIP geometry was carried out with the Hamburg FIB system. Such a nanoconstriction made from the pre-patterned NiFe stripe is shown in Fig. 4. 35.

In the following magnetoresistance measurements only the conventional AMR effect was observed, as shown in Fig. 4. 36 (a). This reveals that the domain wall is hardly pinned at such a structure due to the very short length and the large angle (θ~90°, as shown in Fig. 4. 35 (a)) of the nanoconstriction. Our results are consistent with micromagnetic simulations performed by G. D. Li et al. [226], which shows that no domain wall is pinned in similar devices with a large angle when the applied field is along the main axis of the NiFe stripe. In addition, the AMR contribution from the micro-sized NiFe stripe could mask any small MR contribution from the domain wall.

Fig. 4. 35. (a) SEM image of an FIB fabricated nanoconstriction, which was made by milling a line from left and right of a pre-patterned NiFe stripe. (b) A magnified SEM image reveals the different milling cycles. At first the broad structure was milled to remove the NiFe material. After that the
sharp lines were milled to prevent any by-pass resistance.

The structures were changed accordingly. The pre-patterned structure, as well as the geometry of the constriction, was modified. The new nanoconstriction is longer and has a smaller constriction angle, as shown in the inset of Fig. 4.36 (b). With such new structures we found possible evidence for the magnetoresistance due to the domain wall pinned at a nanoconstriction, which is shown in Fig. 4.36 (b). For more details, please refer to section 4.2.1.

![Fig. 4.36. MR curves of submicron NiFe stripes with (a) a short nanoconstriction with a large angle (90°) and (b) a constriction with a small angle of 45° (modified geometry).](image)

During the first investigations a major problem with the small constriction was the instability due to contamination and oxidation. It was believed that the heat and static discharge caused the destruction of the constriction when current was driven through the thin film structure during the MR measurements. Also the smaller the constriction was, the more unstable due to oxidation it became. This issue was discussed with our partners, and we decided to change our strategy.

We expected a significant improvement would be made by employing a UHV in-situ experiment, in which both the structure fabrication and the MR measurements are
performed in-situ. This measure was expected to prevent contamination and oxidation of the nanoconstriction. Additionally it allows for a parallel production of a large set of different nanoconstrictions on one thin film sample. The idea was implemented into the Hamburg FIB/SEM dual beam system with a micromanipulator, which allows in-situ resistance measurement of the nanoconstriction device.

A side view of this dual beam system is shown in Fig. 4. 37. The FIB column is mounted in the vertical position pointing downwards, while the SEM column is arranged at ~58° to the vertical direction. FIB is used to create the submicron ferromagnetic structure with a nanoconstriction by milling out the blanket film. The fabrication process and the micromanipulator steering are controlled via SEM. The micromanipulator is attached to the frame of a goniometer stage.

For the in-situ MR measurement, the thin film sample is set between two pole pieces of a self-made electromagnet, as shown in the inset of Fig. 4. 37. A very thin tungsten wire (~diameter of 25 μm) with a sharp tip (~diameter of 300 nm) is welded to the end of the jib of the micromanipulator, which makes the contact to the sample (the inset of Fig. 4. 37).
The size dependent resistance and magnetoresistance of FIB fabricated nanoconstrictions have been successfully investigated by utilising this new tool.

4.4.2 Experiment

A 30 nm thick NiFe film with a 3 nm Pt capping layer is first deposited on an insulating substrate. The following experiments, including the FIB fabrication and in-situ MR measurements (one measurement is shown in Fig. 4. 38 (a)) were performed in the UHV dual beam system. The investigated structure is a nanoconstriction between two elliptical NiFe electrodes. The two elliptical shaped elements each have a major axis of 7 μm and minor axes of 3 μm and 4 μm, respectively (see Fig. 4. 38 (b)). The two ellipses have an overlap of 200 nm, and the connection has been cut into a nanoconstriction with the width ranging from 80 nm to 200 nm. There is a small overlapped area between the big ellipse and the blanket film.

Fig. 4. 38. (a) Sketch image of the in-situ MR measurement system, where the current flows from the micromanipulator tip to the blanket film (which is grounded during the measurement setup stage
to prevent charging and high discharge currents that could damage the nanoconstriction, but the grounding is removed afterwards) through a nanoconstriction; (b) SEM image of the FIB patterned elliptical electrodes connected by a nanoconstriction (Courtesy of D. Stickler).

The ellipse geometry of the magnetic electrodes was designed so that their magnetisations can be reliably controlled between the antiparallel and parallel configurations [227]. In the micromagnetic simulation of this geometry, it was found that the local magnetisations on opposite sides of the constriction region are in the antiparallel state with a domain wall trapped in the constriction region at a small reverse field, although the vortex states are shown in the elliptical electrodes. For sufficiently high external fields, both electrodes are aligned parallel to the field.

This was double checked and verified by scanning electron microscopy with polarisation analysis (SEMPA) and MFM. SEMPA images were taken at remanence after the Pt capping layer removed by ion-milling, as SEMPA cannot image surfaces when the magnetic field is applied to the sample. Our SEMPA is able to measure both perpendicular and in-plane components of the magnetisation with a high resolution of 10 nm. The domain patterns in such ellipses are shown in the Fig. 4. 39 (a-c).

Fig. 4. 39. Domain images of the ellipses: (a-b) two SEMPA images display the magnetisation distribution in two perpendicular magnetisation components. The arrows indicate the magnetisation sensitive axes. (c) An MFM image of the same structure (Courtesy of D. Stickler).
NiFe/Pt resistivity values have been measured in-situ on a 2 x 4.2 \( \mu \text{m}^2 \) stripe structure. The resistivity of the 30 nm NiFe/3 nm Pt bi-layer is \( \rho = 52.8 \pm 1.8 \ \mu \Omega \cdot \text{cm} \), and the resistance change is \( \Delta \rho = 0.58 \pm 0.03 \ \mu \Omega \cdot \text{cm} \), so the magnetoresistance is \( MR = 1.1 \pm 0.1 \% \).

For each nanoconstriction device, two different in-situ MR measurements need to be taken. For the first MR measurement, the centre of the small ellipse is contacted via a micromanipulator tip [189]. The current is driven through the patterned constriction to the blanket film, which is grounded and serves as the second electrode (Fig. 4.38). A magnetic field of up to 300 Oe can be applied within the film plane. To extract the resistance and MR of the nanoconstriction, the tip is then placed on the big ellipse for the second measurement, so that the difference between the two measurements almost only indicates the resistance \( R_{nc} \) and the resistance change \( dR_{nc} \) of the nanoconstriction, although there is a small contribution from part of the two ellipses.

### 4.4.3 Results and Discussion

Fig. 4.40 shows the magnetoresistance measurements of one sample with a 1:1 aspect ratio (AR = width/length=80 nm/80 nm) nanoconstriction. The external field is applied parallel to the major axis of the elliptical elements (along the x axis shown in Fig. 4.40 (d)), so the current (along the y axis) is flowing perpendicular to the magnetisation at high magnetic fields. The MR curve (Fig. 4.40 (c)) is the difference between the first measured MR curve (Fig. 4.40 (a)) with the tip on point 1 (the current through the nanoconstriction) and the second measured MR curve (Fig. 4.40 (b)) with the tip on point 2 (no current through the nanoconstriction). These MR curves show that
approaching from the positive field, the MR curve (Fig. 4. 40 (a)) shows a sharp increase of the resistance at a field of -80 Oe, and a steep decrease of the resistance at a reverse field of -125 Oe. The extracted resistance difference, or the extracted MR curve (Fig. 4. 40 (c)), which almost only reflects the resistance of the constriction, shows two similar sharp resistance changes, but there are no sharp MR peaks around the zero fields.

The resistance change of 0.15 Ω is very close to the expected value of 1.1% of the nanoconstriction resistance (~16 Ω) (the estimated resistance is 16 Ω for an 80nm x 80nm constriction if using the resistivity of 52.8 μΩ.cm) considering the AMR effect.

The resistance difference extracted from the two measurements is 47.6 Ω, so that the resistance of part of the two ellipses (~31.6 Ω), which contribute to the extracted MR curve (from the end of the constriction to the tip contact position in the ellipse), is almost twice the resistance of the constriction (~16 Ω).
Fig. 4.40. The two MR curves (a) and (b) were obtained when the micromanipulator was positioned on the small and the large ellipses, respectively; (c) is the extracted MR curve from the first two curves (a-b). The blue and red arrows indicate the sweep sequence of the external field. (d) SEM image of two ellipses connected with an 80 nm wide nanoconstriction (Courtesy of D. Stickler).

Because the sharp resistance rise in Fig. 4.40 occurs half way between the saturation field and the zero field, when there is no obvious magnetisation reversal (e.g. no vortex state) in the ellipses, it is expected that the sharp resistance rise is due to the magnetisation rotation of the nanoconstriction area from perpendicular to parallel to the current. Similarly, the magnetisation jumps back to the direction perpendicular to the current when the reverse field is high enough, corresponding to the sharp drop of the resistance.

Fig. 4.41. SEMPA images of two ellipses connected with an 80 nm wide nanoconstriction (Courtesy of D. Stickler).

To clarify the magnetic switching mechanism behind, the magnetic domain structure of an 80 nm wide nanoconstriction connected with two ellipses was imaged at remanence with SEMPA. For the SEMPA observations, a positive saturation field was first applied along the x-axis, and then switched to a small reverse field of -30 Oe (to introduce the
vortex states into the ellipses without switching the magnetisation of the nanoconstriction) before it was switched off. SEMPA images in Fig. 4. 41 show that the vortex states exist in the elliptical electrodes at remanence. Because the vortex states of two ellipses have the same rotation sense, a 180° domain wall is formed in the constriction, which is seen as a small yellow area between the red and green domains of the ellipses, indicating that the magnetisation of the constriction is aligned to the y-axis. This verifies the magnetisation rotation from the x-axis to the y-axis in the nanoconstriction area when the positive field is lowered.

(1) Micromagnetic Simulations

To clarify the magnetisation switching mechanism in the above structures, we’ve done some micromagnetic simulations with LLG Micromagnetics Simulator. The cell size is 10 nm x10 nm. The intrinsic parameters of NiFe used here are the saturation magnetisation $M_s=8\times10^2$ emu/cm$^3$, the exchange constant $A=1.05\times10^{-6}$ erg/cm and the uniaxial anisotropy constant $K_{u2}=1.0 \times 10^3$ erg/cm$^3$. This yields an exchange length $L_{ex} = \sqrt{A/M_s} \approx 13$ nm, so the above cell size is sufficiently small. Part of the blanket film was included in the simulations.

![Graph showing R vs H](image)
Fig. 4.42 (a) Simulated MR curve of the 80 nm x 80 nm nanoconstriction area between the two elliptical electrodes, and (b-g) are the simulated magnetisation configurations (corresponding to different resistance states on the simulated MR curve, as indicated by the arrows) of the whole device when the applied field sweeps from right to left.

As shown in Fig. 4.42 (a), the simulated MR curve of the 80 nm x 80 nm nanoconstriction area qualitatively agrees with the extracted MR curve in Fig. 4.40 (c). Fig. 4.42 (b-g) shows the evolution of the magnetisation configurations in the two elliptical electrodes connected by an 80 nm x 80 nm nanoconstriction when the external magnetic field sweeps from positive to negative (from right to left) along the x axis.

During the simulation, the device is first saturated at the positive saturation field (~250 Oe), when the magnetisation of the constriction is aligned to the external field, as shown in Fig. 4.42 (b). When the field decreases to a lower value (~100 Oe), the magnetisation of the constriction quickly tilts to the y axis (corresponding to the resistance jump-up in Fig. 4.40 (c)), leading to two 90° domain walls inserted into the constriction as shown
in Fig. 4.42 (c). When the field reverses, the vortex states gradually evolve in the ellipses: first the vortex state appears in the big ellipse (Fig. 4.42 (d)), as the connected blanket film facilitates the magnetisation reversal of the big ellipse, followed by the formation of the vortex state in the small ellipse, leading to a 180° domain wall formed in the constriction (Fig. 4.42 (e)), which separates the two elliptical electrodes with the local magnetisations on the opposite sides of the constriction region in the antiparallel alignment. When the reverse field rises more, the two ellipses finish the magnetisation switching, resulting in the formation of the reverse single-domain state for both (Fig. 4.42 (f)); meanwhile the 180° domain wall disappears, and two 90° domain walls appear again in the constriction area. When a higher field is reached (~ 130 Oe), the magnetisation of the nanoconstriction quickly rotates back to parallel to the external field (Fig. 4.42 (g)), leading to a sharp drop of the resistance. As stated above, the very sharp resistance rise/drop in Fig. 4.40 (c) is caused by the fast magnetisation rotation in the nanoconstriction area.

Because the nanoconstriction size is much larger than the electron mean free path and part of the elliptical electrode is included in the resistance measurement, it was expected that the AMR effect is the dominating source of the magnetoresistance. As we know, AMR originates from the spin-orbit coupling [23] and is observed as an angular variation of the resistance with $R \propto \cos^2 \theta$, where $\theta$ is the angle between the local magnetisation and the current direction. The AMR effect causes a decrease in resistance, when the magnetisation components are perpendicular to the current in the sample. Conversely it results in a resistance rise when the magnetisation components are parallel.
to the current. Because of the AMR effect, the fast 90° rotation of the constriction magnetisation can lead to the sharp rise/drop in the resistance, considering all the current flows through the nanoconstriction, as shown in the extracted MR curve of Fig. 4. 40 (c).

In a series of experiments we have investigated the magnetoresistance as a function of the constriction width, both 130 nm and 180 nm, and their MR curves are shown in Fig. 4. 43 (a-d). The resistance values of the wider constrictions scale with their widths, which are ~9.2 Ω and ~7.5 Ω for 130 nm and 180 nm wide constrictions respectively, as the resistance contribution from the ellipses stays almost the same (~31 Ω).

![Image](image_url)

Fig. 4. 43. The MR curves of two ellipses connected with (a) a 130 nm wide nanoconstriction and (c)
a 180 nm wide nanoconstriction, respectively, when the micromanipulator was positioned on the small ellipse. Curves (b) and (d) are extracted MR curves after excluding the contributions from the ellipses (Courtesy of D. Stickler).

As shown in Fig. 4. 43 (b), the resistance jumps appear at near zero fields, which is reasonable considering the higher aspect ratio (1.6: 1) of the 130 nm x 80 nm constriction. As the aspect ratio (AR) increases, the shape anisotropy rises with the preferred direction along the x-axis, so the magnetisation of the nanoconstriction does not show a fast rotation to the y-axis until near zero fields. It can also be found that the resistance rise is less obvious than the drop for the 130 nm x 80 nm constriction.

But there are no sharp resistance jumps for the 180 nm x 80 nm constriction device, as shown in Fig. 4. 43 (d), due to its much higher aspect ratio ~2.2:1, resulting in a slow magnetisation switching to the y direction after relaxing the external field from the saturation state.

Fig. 4. 44. Resistance and maximum resistance change of nanoconstrictions against the nominal constriction width. The blue rectangles indicate the resistance, and the red dots show ΔR of the
constriction when sweeping the magnetic field (Courtesy of D. Stickler).

The whole set of experimental data is put together in Fig. 4.44 which shows the constriction resistance as function of the constriction width (blue rectangles). As one would expect, we find the resistance of the constriction scales inversely with its width, which is consistent with the domain-wall scattering theory [95]. The maximum resistance change $\Delta R$ (red dots) increases when decreasing the nominal constriction width from 200 nm to 100 nm (due to the Gaussian distribution of the ions in the beam, the constriction is actually about 20 nm smaller than the number given in Fig. 4.44). The increase in $\Delta R$ is probably due to the change of the domain wall structure as the latter is dependent on the constriction dimension. Further investigation of domain wall properties will be presented later.

Further decrease of the nominal constriction width leads to a drop in $\Delta R$, especially when it is below 50 nm. The same trend is also found for the MR of the constrictions. As shown in Fig. 4.45, the experimental MR peaks at the constriction width of 180 nm (MR \(\sim\)0.4%, corresponding to a resistance of \(\sim\)40 $\Omega$) and decreases as the constriction width drops, especially for the sub-30 nm one (MR \(<\)0.05%, corresponding to a resistance \(>\)100 $\Omega$).

This is in contradiction to other recent experiments [13, 214], Levy-Zhang domain-wall scattering theory [95] and BMR theories [209, 228-229], which predicted a positive contribution of the domain wall to the resistance change in a nanoconstriction structure, which increases rapidly when approaching the ballistic transport regime, where the constriction dimension is smaller than the electron mean-free path, typically around 5
nm for NiFe films.

![Graph showing resistance change and MR of constrictions against constriction resistance](image)

**Fig. 4.** Resistance change and MR of constrictions against constriction resistance (Courtesy of D. Stickler).

In particular the big drop (Fig. 4.44) of ΔR in the smallest (sub-30 nm) constrictions needs to be noticed. The magnetoresistance curves of the tiny constrictions (Fig. 4.46) do not show any feature that is related to the effect of the decrease of the constriction width. The MR measurement exhibits only the normal AMR characteristics of a high-resistive constriction device.

One possible explanation is that in the smallest constriction the remaining material is no longer purely ferromagnetic due to the Ga⁺ ions induced interface intermixing, which has damaged the magnetic properties of the tiny nanoconstrictions. The second possibility is that the domain wall structure has changed because the aspect ratio of the constriction has reversed in the smallest constriction. But it was found in simulations that the smallest constriction shows almost the same MR as the wide constrictions. This confirms that it is the Ga⁺ ions implantation rather than the aspect ratio that has caused
the MR deterioration in very narrow constrictions.

![Fig. 4. 46. MR curves of a sub-30 nm nanoconstriction device measured (a) across the constriction, (b) without crossing the constriction, which show the same characteristics (Courtesy of D. Stickler).](image)

**2. Domain Wall Width and Profile**

Bruno [10] has provided the following definition of the domain wall width $w$:

$$w = 4 \left\{ \int_{-\infty}^{\infty} \left[ \dot{\theta}(y) \right]^2 dy \right\}^{-1} \tag{4.1}$$

where $\theta(y)$ is the rotation angle of the magnetisation and $\dot{\theta}(y) = d\theta / dy$ for a domain wall in the $y$ direction. The prefactor is chosen so that this definition results in $w_0 = 2\sqrt{A/K}$ for a domain wall in an unconstrained geometry, where $A$ is the exchange stiffness, and $K$ is the uniaxial anisotropy constant. On reducing the lateral dimensions, the domain wall width is more determined by the constriction geometry rather than the material parameters [230-232].

We have used definition (4.1) to compute the domain wall width in the nanoconstriction...
between the elliptical electrodes based on the simulation data we have collected. The magnetisation distributions of the nanoconstriction at fields, where a domain wall is pinned in the constriction (the vortex states are shown in the two electrodes), were used to calculate the width of the domain wall. The domain wall profiles and their respective widths are shown in Fig. 4.47.

![Simulated domain wall profiles in constrictions as a function of the y position in the constriction area.](image)

**Fig. 4.47.** Simulated domain wall profiles in constrictions as a function of the y position in the constriction area. Each DW profile was achieved by plotting $M_x/M_s$ along the central line of constriction (along the y-axis direction, as indicated by the dashed line in Fig. 4.48 (c)).

All the constrictions we have studied have the same constriction length of 80 nm, but have widths varying from 20 nm to 180 nm. A reduction of the domain wall width is seen with decreasing the constriction width, and the smallest domain wall width is 94 nm for the 20 nm x 80 nm constriction.

For a constriction with the width not greater than the length (80 nm), it seems more energetically favourable for the magnetisation of the constriction to split into two 90° domain walls when the vortex states are observed in the electrodes, as indicated by the...
domain wall profiles in Fig. 4. 47, which is in agreement with [225, 233]. In addition, as illustrated in Fig. 4. 48 (a-d), there are two 90° domain walls and an intermediate domain in the constriction area, and the latter shrinks with increasing the constriction width. When the constriction width increases to 100 nm, a single 180° domain wall is formed in the constriction as indicated by the domain wall profile in Fig. 4. 47, and there is no obvious intermediate domain seen in Fig. 4. 48 (e). But once the constriction width is 130 nm or greater, a complex vortex domain wall is formed in the constriction.

The domain wall deforms inwards in the constriction centre (small domain wall width) but expands at the constriction edges (large domain wall width) to minimise the total energy in the constriction, as shown in Fig. 4. 48 (f-g). Vortex domain walls have been experimentally observed in the NiFe and Co systems [231, 234].

As presented above, significant changes in the domain wall profile/width have been found in the simulations. Surprisingly, there are almost no obvious changes in the simulated MR, which varies between 0.25% and 0.26%, when reducing the constriction width from 180 nm to 20 nm.

The above domain wall width calculations were carried out using not only the dimensions of the fabricated nanoconstrictions but also smaller dimensions (especially smaller lengths), which is achievable with the FIB nanofabrication. It was found that the domain wall width can be reduced to ~28 nm for a 20 nm x 20 nm constriction, so not only the width but also the length of the constriction affects the domain wall width.
So far, we have not found any evidence of large MR values. There could be some reasons for this. Firstly, it is certain that the domain wall structure is determined by both the width and the length of the constriction. Secondly, the magnetisation configuration with a sharp domain wall (when the DW width is much smaller than the wavelength of electrons at the Fermi surface) pinned at the nanoconstriction turns out to be the exceptional case [15, 235]. Thirdly, reaching the ballistic regime is prevented by the
reduction in the electron mean free path due to the enhanced surface scattering at the nanoconstriction [236]. In addition, we have to take into account the effect of the Ga$^+$ ion implantation that is detrimental to the magnetisation of magnetic devices, which is much worse with the decrease of the constriction width.

4.4.4 Conclusion

In summary, in-situ MR and domain wall properties have been investigated in the nanoconstriction devices. We were able to measure the MR of the domain wall in the constriction as a function of the constriction width.

Spin-valve like sharp transitions were observed on the MR curves in the 80 nm/130 nm wide nanoconstriction devices superimposed on the normal AMR curves, which may indicate the existence of BMR or DWMR. However, our analysis of the results by micromagnetic simulations and SEMPA domain observations concluded that these sharp MR transitions originated from the AMR effect, due to the fast magnetisation rotation in the nanoconstriction. This switching behaviour vanishes for constrictions wider than ~130 nm due to changes in the shape anisotropy, and does not exist in sub-30 nm constrictions, probably due to the Ga$^+$ ions implantation.

Experimental MR peaks at the constriction width of 180 nm and decreases as the constriction width drops, especially for the sub-30 nm one. This is in contradiction to other recent experiments, domain-wall scattering theory and BMR theories. Conversely, the simulated MR magnitude is almost constant in spite of the significant changes in the domain wall profile/width when varying the constriction width from 20 nm to 180 nm.
Thus the reduction of MR in the smallest constrictions could be caused by the Ga$^+$ ion implantation damage, which is much worse with the decrease of the constriction width.

To summarise, it is essential to further decrease both the width and length of the constriction and at the same time lower the Ga$^+$ ion implantation to realise large MR values in the ballistic regime. However, this is beyond the capability of the current FIB technology.
4.5 CPP Nanoconstriction Devices on SiN$_x$ Membranes

4.5.1 Introduction

So far, most work, searching for the BMR effect, has been focused on CIP (current-in-plane) type nanocontacts fabricated by electrodeposition, e-beam lithography, and focused-ion-beam (FIB) etc. Only very limited work has been done on CPP (current-perpendicular-to-plane) structures [237-238], especially spin-valve based multilayer CPP nanocontacts.

![Fig. 4. 49. Si wafers with a window coated with SiN$_x$ membranes.](image)

In our research, FIB was utilised to dig a nano-sized pinhole (diameter d = 20-50 nm) in a SiN$_x$ membrane (as shown Fig. 4. 49) for the fabrication of CPP spin valve nanocontacts. Then multilayer film stacks were deposited on both the front and back side of the SiN$_x$ membrane to fill the pinhole and form a spin-valve structure, e.g. FM$_1$/SiN$_x$/FM$_2$/AF/electrode or FM$_1$/SiN$_x$/Cu/FM$_2$/AF/electrode, which creates a multilayered magnetic nanoconstriction. When a current flows through such a device, the current is concentrated in the constriction area due to its high resistance. As a result, if the nanoconstriction is small enough, only two domains may contribute to the
magnetoresistance of the device, one from each FM layer. Since one metallic electrode is deposited on each side of the membrane, the CPP geometry is formed with the current flowing perpendicularly through the layers. The new idea of our CPP nanocontacts is to fix the magnetisation of one FM layer (pinned layer), and apply a small field to flip the other FM layer (free layer), so a 180° domain wall is trapped in the nanocontact when the antiparallel magnetisations formed at its two sides. This type of nanocontact structures is very suitable for the research of the BMR effect and domain wall MR effect.

This section will present some current-perpendicular-to-plane (CPP) MR measurements on single-layer NiFe and multilayer spin-valve nanocontacts.

4.5.2 Experiment

Commercial Si wafers (3x3 mm² and 5x5 mm² in size) with a window of 10 x10 μm² in the centre, which was coated with a SiNₓ membrane (Fig. 4.49), were used as substrates for the fabrication of CPP nanoconstrictions. FIB milling was utilised to mill a nano-sized pinhole through the SiNₓ membrane. A NiFe/Ta film stack was first deposited through the pinhole on one side of the wafer using a Nordiko 9550 deposition system, and then a CoFe/IrMn/Ta/Cu/Ta stack deposited on the other side after pre-sputter cleaning the surface of the first NiFe layer, as indicated in Fig. 4.50. In order to achieve the deposition onto both sides, the wafers have to be taken out of the vacuum chamber and are reloaded after one-side deposition. A special mask was used to shield the edges of the wafer from film deposition so that there is no shunting current
between the top and bottom film stacks and a current flows perpendicular through the layers during the MR measurement.

![Schematic images of CPP nanoconstriction fabrication in SiN$_x$ membranes](image)

Fig. 4. 50. Schematic images of the CPP nanoconstriction fabrication in SiN$_x$ membranes: (a) FIB fabrication of a nano-sized pinhole in the membrane, (b) (FM$_1$) film deposition on one side of the membrane, (c) (FM$_2$+AF) film deposition on the other side of the membrane.

![Schematic diagram of four-point probe CPP MR measurement of nanoconstrictions](image)

Fig. 4. 51. Schematic diagram of the four-point probe CPP MR measurement of nanoconstrictions.

The MR measurements were carried out with the four-point probe method using silver paint to make the necessary electrical connections, as shown in Fig. 4. 51. The magnetotransport properties were measured at room temperature using a Keithley 2182 Nano-Voltmeter and a Keithley 6221 AC and DC current source.

**4.5.3 Results and Discussion**

The above experiments have been carried out on wafers with two different SiN$_x$ membrane thicknesses, 100 nm and 30 nm, respectively. For samples fabricated on the...
100 nm-thick SiN$_x$ membranes, it was found that they were non-conducting during the MR measurement. The most likely reason is that the aspect ratio of this kind of pinholes is so high that the deposited film stack cannot fully fill the pinhole, where voids may exist in the pinhole. It is well known that it is hard for traditional techniques such as the standard PVD deposition to void-free fill a high aspect ratio nano-sized via or pinhole. However, due to the very small size of the pinholes, it is not possible to visually check the quality of the pinhole filling unless high-resolution TEM analysis can be utilised.

![MR curve](image)

Fig. 4. 52. MR curve of a CPP nanoconstriction (front side: NiFe 30nm/Ta 4nm, back side: CoFe 6nm/IrMn 15nm/Ta 4nm/Cu 30nm/Ta 5nm) in the SiN$_x$ membrane.

While using the 30 nm-thick membranes, all nanoconstriction devices were conducting. Fig. 4. 52 shows one MR curve (MR $\sim$ 0.1%) measured on a nanoconstriction with a stack of NiFe 30nm/Ta 4nm on one side and a stack of CoFe 6nm/IrMn 15nm/Ta 4nm/Cu 30nm/Ta 5nm on the other side. A number of wafers with different FIB milling conditions were tested. Multilayer stacks were deposited using different target/substrate spacings to optimise the pinhole filling. Unfortunately there is a distance limit of the
target to the substrate and the substrate bias is not available in our deposition system, which is able to improve the film coverage of the pinhole. We also once tried e-beam evaporation to fill the pinholes. Therefore no significant difference was made to the magneto-transport properties of the CPP devices after all the above process adjustments, and all measured MR curves indicated an AMR origin.

In order to clarify the ambiguity on the interface quality of the multilayer stack in the pinhole, we carried out the following two experiments:

1. A conventional spin-valve stack was deposited on a blanket Si wafer following the identical deposition and wafer reloading sequence, i.e., after the deposition of the first Ta/NiFe bilayer, the wafer was taken out of the chamber and reloaded, and then the second stack in the sequence of Cu/CoFe/IrMn/Ta was deposited on the same side of the wafer after the surface of the first NiFe layer was pre-sputter cleaned. The CIP MR curve of such a spin-valve sample is presented in Fig. 4. 53 (a), showing ~5% GMR.

2. The same multilayer stack was repeated on patterned wafers with the SiNx membrane,

![Fig. 4. 53. (a) CIP MR curve of the blanket spin valve films and (b) CPP MR curve of a similar film stack deposited through a pinhole in the SiN_x membrane.](image)

2. The same multilayer stack was repeated on patterned wafers with the SiN_x membrane,
but deposited through the pinholes, i.e., the NiFe/Ta bilayer was first deposited on one side of the wafer, and then the wafer was taken out of the chamber, turned upside down and reloaded, followed by the deposition of the Cu/CoFe/IrMn/Ta stack on the other side after the pre-sputter cleaning. A typical measured CPP MR curve of such a sample is shown in Fig. 4. 53 (b), where the MR value is only ~0.2% and there is no sign of the exchange-bias between the CoFe and IrMn layers.

We would expect a CPP GMR curve from this device if the interface quality of the film stack deposited through the pinhole was similar to that of the blanket spin-valve sample shown in Fig. 4. 53 (a). However this was not achieved. Obviously the MR curve shown in Fig. 4. 53 (b) has the AMR origin only.

4.5.4 Conclusion

Our experimental results present the difficulties in realising spin-valve typed CPP nanocontacts in SiN_x membranes.

Firstly, some small voids might be created when the film stack deposited into the high aspect-ratio pinholes. Secondly, it is not an easy thing to form a continuous film stack with a smooth interface inside the 20-50 nm wide pinholes, as we had hoped for. As we know, the interface smoothness is critical to achieve the GMR effect in spin valves.
Chapter 5 Conclusions

Spin-dependent electron transport in submicron/nano sized ferromagnetic thin film devices fabricated using the optical lithography, e-beam lithography and FIB has been systematically explored with the main objective to find BMR in the nanoconstriction devices. The important results are given below:

The magnetisation reversal processes have been investigated in the submicron standard NiFe stripe and half-pinned NiFe stripe with a microconstriction by MR measurements and micromagnetic simulations. An asymmetric MR curve is observed in the half-pinned device, because the left part does not quickly switch the magnetisation, and the resistance stays almost constant when the reverse field is lower than the pining field of the IrMn layer. A 180° domain wall is seen at the interface between the pinned part and the unpinned part in the simulated magnetisation configuration. Micromagnetic simulations verify the special MR curve is caused by the exchange-bias on the left side of the NiFe stripe, which changes the magnetisation switching mechanism of the whole stripe.

Domain wall motion induced magnetisation reversal processes in NiFe nano devices with different pinning sites have been studied by MR measurements and micromagnetic simulations. A sign of domain wall MR is shown in the transversal MR curves of two FIB-patterned structures with nanoconstrictions, which can be attributed to the domain wall scattering mechanism. We realised the control of the domain wall pinning and displacement with two types of pinning sites (a triangular element and a
nanoconstriction), which was verified in the micromagnetic simulations. Similar domain wall motion induced magnetisation switching processes were discovered in the NiFe layer of the spin-valve based nano devices with the same dimensions. Thus it is possible to design complex submicron single-layer NiFe film or spin-valve based magnetic wires to selectively achieve position control of domain walls for potential applications in nanowire-based magnetic memory and logic devices.

Magnetisation switching and reversal process have been investigated in the e-beam patterned NiFe nanoring with nanoconstrictions in the connected wire by MR measurements as a function of the magnetic field direction and the applied current. For the applied field direction perpendicular to the wire, an MR enhancement is seen around zero fields at a low current, which is due to the domain wall scattering at the nanoconstriction (similar to what we observed in NiFe nano devices with a nanoconstriction). The magnetisation in the ring goes via the single onion-to-onion switching process regardless of the current value. For the applied field tilted from the perpendicular direction, it is shown that the current has an effect on the switching fields and therefore on the transition process. The magnetisation in the ring goes via a double switching process at a low applied current, but through a single switching process when the current density is higher than the critical current density. The switching field from the vortex state to the onion state decreases with increasing the current when the electron flow is in the direction of domain propagation, while the opposite electron flow increases it.

To extend the nanoring research, a study of the size effect on the magnetisation
switching and interlayer magnetostatic coupling has been carried out in synthetic antiferromagnet (SAF)-pinned spin-valve nanorings by MR measurements and the micromagnetic simulations. It has been demonstrated in simulations that SAF-pinned spin-valve nanorings exhibit a double (onion - vortex - reverse onion) or single (onion - reverse onion) magnetisation switching process depending on the ring diameter. It is also revealed that the magnetostatic coupling between the CoFe pinned layers and the NiFe free layer is dependent on the ring size, and it plays a more important role in the magnetisation switching of small nanorings. The interlayer magnetostatic coupling was efficiently reduced in the large SAF-pinned nanorings, resulting in a small shift of the minor MR curve, which is beneficial to the magnetic memory applications.

In-situ MR measurements and the investigation of domain wall properties have been conducted in FIB-milled nanoconstrictions. The MR of the nanoconstriction device was measured as a function of the constriction width. Spin-valve like sharp transitions were observed on the MR(H) curves in the 80 nm/130 nm wide nanoconstriction devices superimposed on the normal AMR curves, which may indicate the existence of BMR or DWMR. However, our analysis of the results by micromagnetic simulations and SEMPA domain observations concluded that these sharp MR transitions originated from the AMR effect, due to the fast magnetisation rotation in the nanoconstriction. This switching behaviour vanishes for constrictions wider than ~130 nm due to a change in the shape anisotropy, and does not exist in sub-30 nm constrictions either.

Experimental MR peaks at the constriction width of 180 nm and decreases as the constriction width drops, especially for the sub-30 nm one. This is in contradiction to
other recent experiments [13, 214], BMR theories [209, 228-229] and domain-wall scattering theory [95].

Conversely, the simulated MR magnitude is almost constant in spite of the significant changes in the domain wall profile/width when varying the constriction width from 20 nm to 180 nm. Thus the reduction of MR in the smallest constrictions could be caused by the Ga⁺ ion implantation damage, which is much worse with the decrease of the constriction width.

Through my thesis work, several challenging issues have been found for the realisation of the spin-dependent electron transport in thin film nanoconstrictions, including the nanofabrication of sub-30 nm constrictions, the design and control of domain wall structures in the nanoconstrictions, the physical quality of the nanoconstrictions (less Ga⁺ ion implantation), and the protection of the nanoconstrictions from oxidation/electrostatic discharge (ESD) damage after the sample fabrication and during the MR measurement. From all the nanoconstriction devices studied in this project, no experimental evidence for BMR was observed. This conclusion is in contradiction to the original publications by one of the partners in the consortium [1, 121-122], however, is in line with the concerns raised by Egelhoff et al. [132]. Although the existence of BMR cannot be absolutely ruled out due to the limitations of the current nanofabrication techniques, we are convinced that the reported BMR originated from the experimental artefacts.
List of References


[38]. X. Jiang, R. Wang, S. van Dijken, R. Shelby, R. Macfarlane, G. S. Solomon, J.


2871 (2003).


   (1999).
   (2000).
[100]. A. Hirohata, Y.B. Xu, C.C. Yao, H.T. Leung, W.Y. Lee, and S.M. Gardiner, J.
[101]. J. Grollier, D. Lacour, V. Cros, A. Hamzic, A. Vaurès, A. Fert, D. Adam, and G.
[104]. C. K. Lim, T. Devolder, C. Chappert, J. Grollier, V. Cros, A. Vaurès, A. Fert,
[107]. M. Kläui, C. A. F. Vaz, J. A. C. Bland, W. Wernsdorfer, G. Faini, E. Cambril,


[129]. N. Garcia, M. Muñoz, G. G. Qian, H. Rohrer, I. G. Savelief and Y-W. Zhao,
[132]. W. F. Egelhoff, Jr., L. Gan, H. Ettedgui, Y. Kadmon, C. J. Powell, P. J. Chen,
   A. J. Shapiro, R. D. McMichael, J. J. Mallett, T. P. Moffat, and M. D. Stiles,
[135]. D. A. Allwood, G. Xiong, M. D. Cooke, C. C. Faulkner, D. Atkinson, N.
[137]. R.P. Cowburn, D.K. Koltsov, A.O. Adeyeye, M.E. Welland, D.M. Tricker,
[138]. E. Gu, E. Ahmad, J. A. C. Bland, L. M. Brown, M. Ruhrig, A. J. McGibbon,


[194]. http://llgmicro.home.mindspring.com/


APPENDIX 1: Source Code of LabView Program

The main block diagram:

a) Initialise and set the settings of the current source meter.

Fig. appendix 1.

Fig. appendix 2.
b) Step the DC or pulse currents up or down and change the direction of the current.

Fig. appendix 3.

Fig. appendix 4.
c) Initialise Model 2182 meter and take readings of the signal (4 Sub VIs are included).

Fig. appendix 5. Scan.vi.

Fig. appendix 6. buffer_clear.vi.
Fig. appendix 7. buffer_config.vi.

Fig. appendix 8. buffer_read.vi.
Fig. appendix 9. buffer_read.vi (1).

Fig. appendix 10. buffer_read.vi (2).
d) Calculate relevant physical quantities and save the data.

Fig. appendix 11.

Fig. appendix 12.
Fig. appendix 13.

Fig. appendix 14.

205
e) Save the data to a file and plot the I-V loop instantly.

Fig. appendix 15.

Fig. appendix 16.
## APPENDIX 2: List of Abbreviations

<table>
<thead>
<tr>
<th>Abbreviation</th>
<th>Description</th>
</tr>
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<tbody>
<tr>
<td>AF</td>
<td>Antiferromagnetic</td>
</tr>
<tr>
<td>AFM</td>
<td>Atomic force microscopy</td>
</tr>
<tr>
<td>AMR</td>
<td>Anisotropic magnetoresistance</td>
</tr>
<tr>
<td>BMR</td>
<td>Ballistic magnetoresistance</td>
</tr>
<tr>
<td>CIP</td>
<td>Current-in-plane</td>
</tr>
<tr>
<td>CPP</td>
<td>Current-perpendicular-to-plane</td>
</tr>
<tr>
<td>DW</td>
<td>Domain wall</td>
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<tr>
<td>DWMR</td>
<td>Domain wall magnetoresistance</td>
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<tr>
<td>EB</td>
<td>Exchange bias</td>
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<tr>
<td>EBL</td>
<td>E-beam lithography</td>
</tr>
<tr>
<td>FIB</td>
<td>Focused ion beam</td>
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<tr>
<td>FM</td>
<td>Ferromagnetic</td>
</tr>
<tr>
<td>GMR</td>
<td>Giant magnetoresistance</td>
</tr>
<tr>
<td>HDDs</td>
<td>Hard disk drives</td>
</tr>
<tr>
<td>LLG</td>
<td>Landau-Lifshitz-Gilbert</td>
</tr>
<tr>
<td>MCBJ</td>
<td>Mechanically controllable break junction</td>
</tr>
<tr>
<td>MFM</td>
<td>Magnetic force microscopy</td>
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<tr>
<td>MOKE</td>
<td>Magneto-optical Kerr effect</td>
</tr>
<tr>
<td>MR</td>
<td>Magnetoresistance</td>
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<tr>
<td>MRAM</td>
<td>Magnetic random access memory</td>
</tr>
<tr>
<td><strong>MTJ</strong></td>
<td>Magnetic tunnel junction</td>
</tr>
<tr>
<td>----------</td>
<td>--------------------------</td>
</tr>
<tr>
<td><strong>NM</strong></td>
<td>Non-magnetic</td>
</tr>
<tr>
<td><strong>OOMMF</strong></td>
<td>Object Oriented MicroMagnetic Framework</td>
</tr>
<tr>
<td><strong>PEEM</strong></td>
<td>Photoemission electron microscopy</td>
</tr>
<tr>
<td><strong>PSV</strong></td>
<td>Pseudo spin valve</td>
</tr>
<tr>
<td><strong>PVD</strong></td>
<td>Physical vapour deposition</td>
</tr>
<tr>
<td><strong>SAF</strong></td>
<td>Synthetic antiferromagnetic</td>
</tr>
<tr>
<td><strong>SDT</strong></td>
<td>Spin dependent tunnelling</td>
</tr>
<tr>
<td><strong>SEM</strong></td>
<td>Scanning electron microscope</td>
</tr>
<tr>
<td><strong>SEMPA</strong></td>
<td>Scanning electron microscopy with polarisation analysis</td>
</tr>
<tr>
<td><strong>SQUID</strong></td>
<td>Superconducting quantum interference device</td>
</tr>
<tr>
<td><strong>STT</strong></td>
<td>Spin torque transfer</td>
</tr>
<tr>
<td><strong>SV</strong></td>
<td>Spin valve</td>
</tr>
<tr>
<td><strong>TMR</strong></td>
<td>Tunneling magnetoresistance</td>
</tr>
<tr>
<td><strong>UV</strong></td>
<td>Ultraviolet</td>
</tr>
<tr>
<td><strong>VI</strong></td>
<td>Virtual instrument</td>
</tr>
<tr>
<td><strong>VSM</strong></td>
<td>Vibrating sample magnetometer</td>
</tr>
</tbody>
</table>
APPENDIX 3: Publications

Articles:


**Conference Posters/Presentations:**


Size Effect on Magnetic Switching and Interlayer Magnetostatic Coupling in Spin-Valve Nanorings Exchange-Biased by Synthetic Antiferromagnets


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2Institut für Angewandte Physik, Universität Hamburg, Hamburg 20355, Germany

This paper presents studies of the size effect on magnetic switching and interlayer magnetostatic coupling in synthetic antiferromagnet (SAF)-pinned spin-valve nanorings connected to nano stripes with a nanoconstriction. Micromagnetic simulation has been successfully used to reveal the detailed magnetization reversal process of these nanorings. It was observed that SAF-pinned spin-valve nanorings exhibit a double (onion—vortex—reverse onion) or single (onion—reverse onion) magnetization switching process depending on the ring diameter, which is contrary to narrow single-layer NiFe rings that show a double switching process only. Micromagnetic simulations of the SAF-pinned spin-valve nanorings suggest that the magnetostatic coupling between the pinned layers of the SAF and the free layer is dependent on the ring size: it plays a very important role in the magnetization switching of small nanorings (680 nm outer diameter), but only a minor role in the switching of big nanorings (1600 nm outer diameter). An efficient reduction of the magnetostatic interaction results in a small shift of the minor magnetoresistance curve, which is beneficial to the magnetic memory applications.

Index Terms—Domain wall motion, magnetization reversal, magnetostatic coupling, nanoring, spin-valve.

I. INTRODUCTION

CONSIDERABLE studies of magnetic ring structures have been carried out in recent years due to their potential application in high-density magnetic random access memory (MRAM) [1]–[12]. To date, most of the work has focused on single-layer NiFe or Co rings [2]–[12], where there exist two distinct magnetic states: the vortex state with a flux-closure domain and the onion state with 180° head-to-head/tail-to-tail domain walls, due to the competition between the magnetostatic energy and the exchange energy.

Compared with single-layer ring elements, ring-shaped multilayer structures, such as spin-valve and magnetic tunnel junction (MTJ) structures with higher magnetoresistance (MR) ratios, provide a more sensitive way of investigating the magnetization configurations in the free and pinned layers. Such structures with more domain states (the state combinations in two magnetic layers) are very attractive for spintronic applications such as memories or logic devices, that require multiple stable resistance levels [13].

Generally for these multilayer structures, the magnetic layers may be coupled through the magnetostatic interactions, resulting in a change of the switching field of the free layer. This leads to a shift of the minor MR curve along the field axis.

Thus, it is essential to investigate the effect of the magnetostatic interactions on the magnetic switching in ring-shaped multilayer structures. The main magnetostatic interactions come from two mechanisms, the Néel coupling and the interlayer magnetostatic coupling. The Néel coupling due to the interface roughness is independent of the feature size [14], [42], [15]. The interlayer magnetostatic coupling is caused by the stray field from the domain walls and the edges of neighboring magnetic layers [16] and it increases with a decrease in the feature size [17]. For a patterned device, the interlayer magnetostatic interaction field can be approximated by the demagnetization field of the pinned layer acting on the free layer, which is estimated by the formula

$$H_D \sim C_M s / L,$$

where \( L \) is the length of the patterned device, and \( M_S, t, \) and \( C \) are the magnetization, thickness of the pinned layer, and the proportional coefficient, respectively [18]–[20]. So the magnetostatic coupling field is proportional to the product of the thickness and the saturation magnetization of the pinned layer [21]. As a result, this coupling field is much reduced in the SAF structure due to the decreased net magnetization and a smaller effective CoFe thickness.

Since Zhu et al. achieved robust magnetic switching in the multilayer GMR rings with enhanced MR response [22], there has been recent interest in the magnetization reversal in ring-shaped multilayer structures, e.g., spin-valve NiFe/Cu/Co/FeMn rings, pseudo-spin-valve NiFe/Cu/Co rings [23]–[30], and MTJ rings [31]–[33]. In the spin-valve rings, the free layer shows a switching mechanism different from that of single-layer rings, which is caused by the strong interlayer magnetostatic coupling. As a result, they present asymmetric minor MR curves with large shifts. Although the onion state still exists, normally a vortex state in the free layer can only be observed when the reference layer is in the vortex state [23], [24], [27]–[29], which eliminates the interlayer magnetostatic coupling.

In the ring-shaped MTJ [31], [32] Chen et al. found several common domain states, including the onion state and the vortex state, which are similar to what they observed in single-layer NiFe rings. The magnetostatic coupling also exists in MTJ rings, but does not affect the magnetic transition, which is different from the behavior of the spin-valve rings. A size dependence of the magnetization transition process was seen in the MTJ rings [32].

Adding a SAF structure to spin valves or MTJs is a good way to decrease the shift of the minor MR curve by reducing the interlayer magnetostatic coupling, which is desirable for better device performance in many applications, such as MRAM, read heads or magnetic sensors. Chen's group introduced the SAF to the MTJ rings [33], and found an incomplete vortex state. They attributed this to the pinning of the surface roughness rather than the magnetostatic coupling, which was neglected in the micromagnetic simulations and their analysis.

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Digital Object Identifier 10.1109/TMAG.2010.2089466
So far there are no reported data on the behavior of SAF-pinned spin-valve rings, and in particular there are no investigations of the dependence of the magnetization switching process on the magnetostatic coupling in such rings, in spite of their importance.

Here, we present a study by a combination of MR measurements and micromagnetic simulations of the size effect on the magnetic switching behavior and the interlayer magnetostatic coupling in SAF-pinned spin-valve rings. The intention is to produce minor MR curves with small shifts through the reduction in the magnetostatic coupling between the free and pinned layers of SAF pinned spin valves.

II. EXPERIMENTAL TECHNIQUES

Spin valves with a SAF structure of (substrate)/Ta (2 nm)/NiFe (4 nm)/Cu (2.5 nm)/CoFe (2 nm)/Ru (0.7 nm)/CoFe (2.5 nm)/IrMn (8 nm)/Ta (2 nm) were deposited by a Nordik PVD system on a thermally oxidized Si wafer. The base pressure for the deposition was below $3 \times 10^{-8}$ Torr. The process conditions were optimized separately for each of the target materials. A uniform magnetic field of 50 Oe was applied in the substrate plane during the deposition to induce an in-plane uniaxial magnetic anisotropy for the ferromagnetic layers. The annealing process was undertaken at 250°C in an argon atmosphere in a magnetic field of 6 kOe applied along the easy axis of the free layer.

Samples were first photolithographically patterned into the shape of a submicron disk connected with stripes and injection pads in both ends, as shown in Fig. 1(a). The injection pads are connected to four large nonmagnetic electrodes [Fig. 1(b)] of Ta (5 nm)/Cu (100 nm)/Ta (5 nm), which serve as electrical connection pads to perform MR measurements with a standard four-point probe. Nanorings were then fabricated by focused-ion beam (FIB) from this basic structure, as shown in Fig. 2. A 100 nm wide nanoconstriction on the right stripe was also cut by FIB. The cuts were made with the Orsay Physics Canion 31 Plus UHV FIB using 30 kV gallium ions (5 pA beam current) [34]. It needs to be pointed out that our ring structure is different from those used in other work [21–13], [23]–[33], due to the special sample fabrication procedure (photolithography plus FIB milling) and the requirement of our MR measurement system. Such a geometry (injection pads, stripes and a nanoconstriction) was initially adopted for research into the domain wall MR in the single-layer NiFe nanoring [35], [43] and also used in our previous investigation of the ballistic magnetoresistance (BMR) effect [36].

As shown in Fig. 2, nanoring A has an outer diameter of 1600 nm and an inner diameter of 1200 nm; nanoring B has the outer and inner diameters of 600 nm and 200 nm, respectively. The ring width is 200 nm for both types of rings. The nano stripes are 200 nm wide for both nanorings, and 1000 nm and 600 nm long for nanoring A and nanoring B, respectively.

The magneto-transport properties of three types of samples, unpatterned spin-valve films, nanoring A, and nanoring B are shown in Fig. 3(a), (b) and (c), respectively. The unpatterned spin valve sample has a GMR of 7.8%. A shift of $-10$ Oe was observed in the minor MR curve, which was due to the Neel coupling [14], also referred to as the "orange peel" coupling. The GMR for both nanorings is about 1.3%, which is much lower than that of the unpatterned spin-valve films. And the shifts of the MR curves are $-12$ Oe and $-26$ Oe for nanoring A and nanoring B, respectively, higher than that of the unpatterned films.

As shown in Fig. 3(b), the minor MR curve of nanoring A exhibits four distinct resistance levels labeled as $L_1$, $L_2$, $L_3$, and $L_4$. Starting from a positive field of 200 Oe, as the field sweeps from positive to negative, the first big transition occurs at a small negative field, leading to the first resistance plateau ($L_1$) followed by another two intermediate resistance states: the second
The high resistance state (the fourth transition, $L_4$) is observed at a field of $-120$ Oe. While for nanoring B, its minor MR curve shows only two distinct resistance levels, which are labeled as $L'_1$ and $L'_2$ in Fig. 3(c), respectively. These resistance levels only correspond to different magnetization configurations in the NiFe free layer, since there is no magnetization reversal in the CoFe pinned layers of the SAF structure at low field measurements.

The large GMR ratio drop in the patterned nanoring device can be attributed to the Ga$^+$ ion induced intermixing during the FIB fabrication process. As has been found, the high-energy Ga$^+$ ions (30 keV) are responsible for the interface intermixing and the reduction of interface effects like GMR [37], [38]. Halo ions of the outer beam profile hit the device and reduce the GMR effect by low-dose intermixing. Most of the halo ions are blocked by the 15.2 nm thick materials above the Cu spacer layer responsible for the GMR. The remaining GMR is still enough to monitor the different domain states inside the ring structure.

Nanorings A and B show larger shifts in the minor MR curves than the unpatterned films. As we know, this can be ascribed to the increase of the magnetostatic interactions (the vector sum of the interlayer magnetostatic coupling field and the Néel coupling field) between the free and pinned layers of the SAF structure. Because the Néel coupling is independent of the feature size [14], [42], [15], the shift difference between the patterned ring elements and the unpatterned films is due to an increase in the interlayer magnetostatic coupling as the feature size reduces after patterning [17].

It is important to examine the contribution of each CoFe layer to the total interlayer magnetostatic interaction. Considering the thickness difference of the two CoFe layers, the negative magnetostatic coupling from the thin lower CoFe layer only partially cancels the positive coupling from the thick upper CoFe layer, so the net coupling field is positive. This net coupling field favors the parallel alignment of the free layer and the lower CoFe layer, the same as the Néel coupling acting on the free layer.

Based on the above analysis, the interlayer magnetostatic coupling in our SAF pinned spin-valve rings, especially those with an outer diameter of 1.6 $\mu$m, is much lower than that of
This can be further verified by comparing the shift of the minor MR curve between SAF-pinned nanoring A (-12 Oe shift) and a pseudo-spin-valve ring (with an outer diameter of 2 µm, -40 Oe shift), as shown in the lower left inset of Fig. 3(b), and the much larger shift of the latter indicates a much higher magnetostatic coupling [39].

But it needs to be noted that the magnetostatic coupling rises with the decrease of the ring diameter. As shown in Fig. 3(c), the shift of the minor MR curve of the small nanoring B is doubled compared with nanoring A, which may hint at a different magnetization reversal process being responsible.

To clarify the magnetization reversal mechanism in nanorings of different sizes, we have done a 3-D micromagnetic simulation of the NiFe (4 nm)/Cu (2.5 nm)/CoFe (0.5 nm) trilayer nanoring device using LLG Micromagnetics Simulator [40]. A cell size of 10 nm x 10 nm x thickness was used for each layer. Parameters used in the simulations for Permalloy were the saturation magnetization $M_s = 8 \times 10^5$ emu/cm$^3$, the exchange constant $A = 1.05 \times 10^{-6}$ erg/cm and the uniaxial anisotropy constant $K_{u2} = 1.0 \times 10^6$ erg/cm$^3$; parameters for CoFe were $M_s = 1.5 \times 10^6$ emu/cm$^3$, $A = 2.05 \times 10^{-6}$ erg/cm, and $K_{u2} = 1.0 \times 10^6$ erg/cm$^3$. A damping constant $\alpha$ of 0.5 was used in the computations.

Although the whole SAF was not included in the simulation volume to save the computation time, a pinning field of 3000 Oe was applied to the pinned layer, and an interlayer coupling field (different values for different ring sizes, the same as that obtained in the MR measurements) between the free and pinned layers was also added to the input. The injection pads were included in the simulations, but only part of them is shown below due to limited space.

For nanoring A, its simulated MR curve is given in the upper right inset of Fig. 3(b), which agrees well with the experimental MR curve. It is seen in the low-field simulation that the CoFe pinned layer stays in the single domain state, which can be explained by the large antiferromagnetic coupling from the SAF structure. The simulation also shows that the NiFe free layer of the ring stack exhibits a single-domain state at the positive saturation field. When the field decreases from the saturation field, the magnetization of the NiFe free layer gradually follows the circumference of the ring because of the shape anisotropy. The single-domain state then switches to the forward onion state, where there are two head-to-head/tail-to-tail domain walls formed at the joint between the ring and the nano stripes, as illustrated in Fig. 4(a). When the magnetic field decreases and sweeps from zero to negative, the first sharp transition occurs at a low negative field, where the magnetization of the NiFe free layer in the injection pads is reversed as shown in Fig. 4(b), leading to the first sharp step increase of the resistance (due to the antiparallel configuration with respect to the pinned layer), followed by the first plateau ($L_1$). Further increase of the negative field promotes the magnetization switching in the left stripe and in the right part of the right stripe (between the nanoconstriction and the right injection pad), as seen in Fig. 4(c), which corresponds to the second jump-up in the resistance ($L_2$). There is no apparent magnetization switching thereafter until the negative field reaches a point where the domain wall depins from the nanoconstriction, leading to a complete magnetization reversal of the right stripe as shown in Fig. 4(d). In addition, the vortex state is formed in the ring area after a reverse domain wall sweeps across the lower half-ring and switches the magnetization, which begins by the expansion of the reverse domain from the stripe, where the MR curve shows the third jump-up in the resistance followed by the third plateau ($L_3$). Eventually, a higher negative field leads to the
process (a forward onion state to a vortex state, and finally to an asymmetric ring geometry [25, 26, 27]).

Injection pads, and applying off-axis external fields or using the conventional spin-valve nanorings by using asymmetric injection pads, and applying off-axis external fields or using asymmetric ring geometries [25, 26, 27].

It has been noticed that a vortex state is still achievable in the free layer when the pinned layer is in the vortex state [23, 24, 27–29]. We also noticed that a vortex state is still achievable in the free layer unless the pinned layer is in the vortex state [23, 24, 27–29]. We also noticed that a vortex state is still achievable in the free layer unless the pinned layer is in the vortex state [23, 24, 27–29].

To summarize, nanoring A shows a normal double switching process (a forward onion state to a vortex state, and finally to a reverse onion state). From the shift of its minor MR curve, we know there is a magnetostatic coupling from the CoFe pinned layers, and the normal double switching process with the vortex state shows that such a coupling is small in this large nanoring.

For nanoring B, the simulated MR curve is also consistent with the experimental one, as shown in the inset of Fig. 3(c). Its simulated magnetization switching process (see Fig. 5) of the NiFe free layer is different from that of nanoring A, as expected from their different experimental MR curves. Starting from the positive saturation, at the beginning of the magnetization switching, nanoring B behaves similarly to nanoring A. When the field decreases from the positive saturation to zero, nanoring B switches from the single-domain state to the forward onion state in Fig. 5(a), showing two head-to-head/tail-to-tail domain walls. The first sharp increase of the resistance is also observed at a low negative field, corresponding to the magnetization reversal in the NiFe free layer of the two injection pads [Fig. 5(b)]. However, due to the strong magnetostatic interaction between the free and pinned layers caused by the small diameter of nanoring B, it is too hard for the reverse domains in the NiFe free layer to expand quickly at a low field. Consequently, over a wide field range, there is no abrupt resistance change (or intermediate vortex state as seen in nanoring A). Instead, it shows a slow and continuous increase in the resistance (a gradual, upward slope L2, which is highlighted with a circle in Fig. 3(c)). Once the field is high enough to overcome the magnetostatic interaction, the reverse domains in the NiFe free layer quickly initiate the expansion into the nano stripes. The strong negative field also promotes the annihilation of domain walls at the constriction, followed by the formation of a reverse onion state in the ring: reverse domains quickly nucleate and sweep across the ring, and the magnetization direction is switched to negative. As a result, the previous head-to-head/tail-to-tail domain walls annihilate and the reverse tail-to-tail/head-to-head domain walls form at the joint between the nano stripes and the ring, which is shown in Fig. 5(c). At this point, the resistance quickly jumps up to the saturation state (L2), showing the second sharp increase in the resistance. In summary, nanoring B exhibits a single onion-to-reverse onion switching process without the vortex state, which is different from the behavior of nanoring A, but comparable to that of the conventional spin-valve nanorings without the SAF [23, 24, 27–29]. Such a similarity is obviously caused by the increased interlayer magnetostatic interactions in a small nanoring.

As described above, there is a dependence of the magnetic switching behavior on the ring diameter. For a large diameter (nanoring A), the double transition process (onion—vortex—reverse onion) occurs, due to a weak interlayer magnetostatic coupling; but for a small diameter (nanoring B), the single transition process (onion—reverse onion) appears, and no vortex state exists because of a much stronger magnetostatic coupling.

It can be seen that the interlayer magnetostatic coupling increases with a reduction of the ring diameter. The reason lies in two mechanisms. First, as the ring diameter decreases, magnetic moments cannot exactly follow the perimeter of the ring due to the decrease of the circumferential shape anisotropy, leading to an enhancement of the magnetostatic coupling from the ring
edge [17]. Second, when the ring becomes smaller, the stray field from the domain walls in the pinned layers becomes more important.

It needs to be noted that the interlayer magnetostatic field is very nonuniform across the area of the ring. This magnetostatic field is minimized in the flux-closure vortex state. But in the onion state, the magnetization is not parallel to the edge and there are two head-to-head/tail-to-tail domain walls, leading to surface charges. This causes a large magnetostatic field near the ring edge/domain walls, and this field decreases rapidly with increasing distance from the ring edge/domain walls, which was found by M. Lauenberg et al. [41].

As we know, it is the interlayer interaction between the free and pinned layers of a spin-valve structure which causes a shift in the minor MR curve, so that nanoring A shows a small shift in the minor curve can be explained by a weak interlayer magnetostatic coupling, due to its large ring diameter, as discussed above.

As small nanorings are preferred for high-storage density MRAM, increasing the ring diameter is not the ideal way to drop the interlayer magnetostatic coupling for smaller shifts of the minor MR curves. It is well understood [16] that there are two other options when adding the SAF structure to the spin-valve rings to reduce the magnetostatic coupling: one is to minimize the thickness difference in the two CoFe layers for a lower net magnetization and a smaller effective CoFe thickness, resulting in the lower magnetostatic interaction; the other is to make the upper CoFe layer a little thinner than the lower layer, so that the polarity of the net magnetostatic coupling from the CoFe layers is flipped to favor the antiparallel alignment of the free and pinned layers. Thus, the negative magnetostatic coupling can be used to compensate the Neel coupling (favoring the parallel alignment), aiming for a near-zero net coupling field.

To summarize, we have investigated the effect of the ring size on the magnetic switching process of the SAF-pinned spin-valve nanorings, and found a strong dependence of the interlayer magnetostatic coupling on the ring diameter.

In addition, we noticed in our experiments that the injection pads also contribute to the magnetoresistance, resulting in the first sharp resistance transition on the MR curve. To check whether the unique switching behavior of SAF-pinned nanorings is caused by the injection pads, we carried out further simulations of isolated SAF-pinned rings without injection pads. As shown in Fig. 6(a)–(c) and (d)–(e), respectively, the isolated ring shows a double switching process with a vortex state, and the small one shows a single switching process with no vortex state. The simulation results confirm that the isolated rings behave in the same way as the rings with injection pads. It can therefore be inferred that although the symmetric injection pads may have a small effect on the magnetic switching of the nanoring, the switching behavior of the ring is more determined by the interlayer magnetostatic coupling, which depends on the ring diameter.

IV. CONCLUSION

In this work, a detailed picture of the magnetic switching behavior of the synthetic spin-valve nanorings connected to nanocontacts with a nanoshunt has been given by a combination of MR measurements and 3-D micromagnetic simulations. The SAF-pinned nanoring devices exhibit asymmetric minor MR curves with different resistance levels, corresponding to different magnetization configurations in the NiFe free layer, as clarified by the micromagnetic simulations. The magnetic reversal behavior is influenced by the magnetostatic coupling as a function of the ring diameter. Large nanorings show a normal double switching process with a vortex state, while small nanorings show a single switching process due to the strong magnetostatic coupling between the free and pinned layers. The interlayer magnetostatic coupling, which leads to a shift of the minor MR curve, is ring size dependent. The large SAF-pinned spin-valve nanoring presents a smaller shift in the minor MR curve than the small nanoring.
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REFERENCES


