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Materials for giant spin Hall effect devices

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MATERIALS FOR GIANT SPIN HALL EFFECT DEVICES

by

Avyaya Jayanthinarasimham

A Dissertation
Submitted to the University at Albany, State University of New York
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I dedicate this thesis to my loving parents, Shri. Shanti Swarup and late Smt. Roja Rani
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ABSTRACT

Studies presented in this thesis are an effort to control the growth of $\beta$ W and explore the in-plane current induced effects in a $\beta$ W and CoFeB bilayer. Physical vapor deposited W films beyond 5 nm transform from $\beta$ to the stable bulk $\alpha$ phase. $\beta$ W films with 5 nm thickness when integrated with the other films for large scale fabrication presents a small process window for etch and deposition errors. Also, CoFeB on W does not generate perpendicular magnetic anisotropy (PMA) even when it is capped with MgO-Ta(Capping) layers. The $\beta$ W with larger thickness process window and a CoFeB with PMA deposited on top of W is necessary for an ideal functioning spin Hall effect (SHE) device. This thesis will focus on overcoming the above mentioned challenges.

2 sccm of $O_2$ gas was introduced during the growth of $\beta$ W, this resulted in thicker films with $\beta$ W. If a large amount of $O_2$ was introduced, it resulted in complete oxidation and loss of crystallinity. Thus an optimum amount of oxygen is necessary. However, introducing $O_2$ during the deposition can effect other metals present on the wafer, which is not ideal. $N_2$ was utilized to achieve thicker $\beta$ W films. Upon introducing N with similar concentration of O, it lead to amorphization of W, thus revealing a kinetic control. A pulsed $N_2$ of 1 sccm at 2-second period was used to kinetically control the growth of $\beta$ W. Both the techniques were able to grow $\beta$ W from 5 nm up to 20 nm thick films. Films with N-assisted growth exhibited lower resistance and higher metallic character.

1 nm Ta, Mo and CoFe were used as insert layers between $\beta$ W and CoFeB to induce PMA. 1 nm Mo insert layer and 5 nm Mo under layer have largely different interfaces with CoFeB even when annealed in ultra high vacuum (UHV) environment. Thus, 1 nm Mo layer does not show any PMA. The CoFe insert layer adds to the bulk anisotropy and dominates the interface anisotropy, and does not lead to any PMA. The 1 nm Ta insert exhibits strong PMA in both as deposited and annealed condition. Hence, the stack
SUB|β-W|Ta|CoFeB|MgO|Ta was deposited and patterned into a Hall bar device. The competition between external magnetic field and internal effective fields is demonstrated. An empirical model is developed to estimate the spin Hall angle (SHA). The SHA is estimated $= 0.24 \pm 0.05$ for the entire stack. Current induced switching of single ferromagnetic metal (FM) is also shown and the SHE symmetry is preserved.

The results presented in the thesis demonstrate the full capacity to integrate SHE materials like $\beta$ W in a 300 mm integrated circuit fabrication facility. A prototypical device is fabricated and studied. Along with the challenges which are inherent to the effect and some insights to overcome them and a future outlook.
CHAPTER 1
INTRODUCTION AND LITERATURE REVIEW

1.1 Motivation

Discoveries in spin based electronics has revolutionized the world of digital electronics. Semiconductor industry’s ability to follow Moore’s law is at this point faced with a newer ideology of “More than Moore”, where devices are incorporated with functionalities which do not necessarily scale according to ”Moore’s Law” [2]. Spin-based electronics fits right into this category with it’s non-volatility, low power and low speed as it’s major functionalities [3].

Recently discovered spin Hall effect (SHE), in heavy metal (HM) like W, Ta and Pt, can be used to reorient the magnetic moment of an adjacent ferromagnetic metal (FM). A lateral current is passed through the spin Hall material (SHM) generates a transverse spin current. This spin current exerts a spin transfer torque on magnetic moment of an adjacent FM [4, 5, 6, 7, 8]. This effect can improve some of the existing device units, especially magnetic random access memory (MRAM) and proposed charge-coupled spin logic (CSL) devices.

The basic building block of a MRAM bit is a magnetic tunnel junction (MTJ) which consists of two FMs sandwiching a thin tunnel barrier. The magnetic moment of one of the FM is fixed and the other FM’s magnetic moment can be flipped. By passing appropriate amount of current through the fixed FM layer in the junction the resistance of the entire stack can be switched from a low resistance to high resistance state [9]. A traditional MTJ is a two terminal device which uses a common read and write path from a access transistor. This interlinks switching current density, read voltage and magnetoresistance. Thus, making it challenging to independently optimize them. A spin Hall material can be integrated under the free FM layer in the MTJ. Thus when a lateral current is passed through the spin Hall
material the resistance of the adjacent MTJ can be controlled, which makes the MTJ a three terminal device. Thus, switching current density, read voltage and magnetoresistance are no longer interdependent and can be independently optimized for better device performance.

CSL is a recently proposed device which takes advantage of SHE to manipulate magnetic moment of an adjacent FM [10]. It consists of read unit and write unit which are coupled through a dipolar interaction. The write unit consists of a FM grown on top a SHM. The read unit is a lateral-double MTJ structure. The magnetic moments of the fixed FM layers in the lateral-double MTJ structure are in opposite direction. Also there is a constant bias applied on these MTJs. An input current is applied on the write unit’s SHM and an output voltage is measured from the free layer’s in the read unit. As the current reaches a critical value the voltage at the output reverses creating an inverter type of characteristic.

The primary building unit in MRAM and CSL when integrated with SHM is HM|FM bilayer. The effect mainly takes place at the interface of HM and FM. The critical criteria for such devices is the spin current created at the interface of SHM and FM. The effect is quantified by spin Hall angle (SHA) which is the ratio of spin current to charge current. Larger SHA will yield stronger spin transfer torque (STT) which ultimately lowers the lateral charge current through the SHM. SHE is a direct consequence of spin orbit coupling (SOC) which is proportional to the fourth order of atomic number [11]. This makes paramagnetic metals with high atomic number, such as, W, ta and Pt good candidates for such applications. For large scale fabrication of such devices in-line integration is very critical. Fortunately, Ta and W are already available in the back end-of-the line (BEOL) which makes them very attractive candidates. Experimentally β phase of Ta and W are shown to exhibit larger SHA in comparison to their α phase. These multilayered structures inherently have structural inversion asymmetry (SIA) which creates an effective field due to Rashba effect at the interface [12, 11]. Also the bulk inversion asymmetry (BIA) present in the β phase can generate spin accumulation transverse to a longitudinal current [13]. The response of magnetic moment of a FM to the effective field at it’s interface is very similar to the presence
of spin accumulation at the same interface [14].

The $\beta$-phase W has a thickness limit 5 nm, beyond which it transforms into the $\alpha$ phase. This creates only a small process window to fabricate these devices successfully. Thus, the growth of thicker films but within the spin diffusion length (10 nm) is necessary for efficient large scale device fabrication [15, 16, 17]. Presence of impurities during the growth of W films promotes the $\beta$ phase. This property is exploited to growth thicker $\beta$ W films. The FM’s magnetic moment’s anisotropy is largely dependent on the HM it is deposited on. The FM’s magnetic moment needs to be perpendicular to the direction of the film plane, which is termed as perpendicular magnetic anisotropy (PMA). PMA in FM provides a promise for scalability and higher thermal stability [18]. The PMA of these FM films is largely dependent on the two interfaces of the FM film. CoFeB and MgO interface is known to exhibit strong PMA. The other interface, with W and CoFeB, can strongly influence the magnetic anisotropy. An insert layer of Ta is deposited to ensure PMA in the CoFeB layer. In plane current induced spin orbit effects are studied in Sub|$\beta$-W (5 nm)| Ta (1 nm)| MgO (1.6 nm)| CoFeB (1 nm)|Ta (Capping layer). Current induced magnetic moment reversal in CoFeB is achieved which unequivocally demonstrates the presence of a spin orbit torque (SOT) at the interface. The films are grown and patterned in a 300 mm integrated circuit fabrication facility. This is a strong promise for large scale integration of these materials. The next section of the thesis will focus on the efforts done by the peers in the community towards understanding growth $\beta$ W and metal based spin orbit effects.

1.2 Literature Review

1.2.1 Growth of $\beta$ phase tungsten

One of the earliest ways $\beta$ -W was obtained is by electrolysis of tungsten trioxide and alkali phosphates [19]. For a large amount of time the $\beta$ -W was considered as an allotrope of tungsten oxide. It was Hagg and Schonberg were the first to report $\beta$ -W as a metallic “Tungsten-oxide- W$_3$O” owing to it’s large concentration of O and metal like
conductivity in 1953 [19]. The lattice parameter was estimated to be 5.036 Å with a space group of pm3n was reported. An irreversible phase transformation to α phase was observed around 700°C. Not until 1968 was electron beam evaporation utilized to fabricate these films. Superconductivity for the β phase was observed around 3.2 K in these films [20]. During the growth of these films it was observed that a lower base pressure or presence of impurities during the deposition favoured the growth of β phase W. P. Petroff, et al. studied the RF-sputter growth deposited β-W. They reported a small grain size (50 Å) for a relatively thick film (100 Å). A thickness induced phase transformation to α is reported around 50-80 Å. A chemical formula of W$_3$W with a A15 crystal structure was reported [21]. Much later in 1992 M.S. Aouadi, et al. studied the DC magnetron sputter deposited tungsten. Various metrology techniques like x-ray diffraction (XRD), scanning tunneling microscopy (STM), x-ray reflectivity (XRR) were used to understand these films. Presence of WO$_x$ and β W was proposed to be the structure of the films [22]. Arita, et al. did a detailed study trying to relate the presence of oxygen atoms in the lattice and it’s impact on the XRD pattern of the β-W film. The A15 structure is a body centered cubic (BCC) with tungsten atoms occupying the tetrahedral void on the face [19]. It was concluded that the following structures have the similar XRD pattern (1) No oxygen atom in W$_3$W (2) Oxygen atoms in interstitial sites (3) Oxygen atoms occupies face sites and body sites with equal probability. Only distinguishable XRD pattern is when oxygen atoms occupy the lattice sites with unequal probability, which only happened under large concentrations of oxygen is present during the deposition [23]. This distribution of the non-symmetrical A15 and highly disordered crystal structure makes it difficult to study any structural transformations. Tungsten films evaporated on NaCl substrates were observed under high resolution electron microscopy which revealed defect stabilized A15-tungsten films [24].

The first report to display the long term stability and impurity concentration in the β W film was I.A. Weerasekara et al. They report a phase transformation after 6 months of depositing the films. They also report impurity concentration of 19 at. % of O atoms in a
40 nm film [25]. A much lower impurity concentration of 5 at. % was achieved by growing the films at slower rate of 2 Å per min [26]. Y.G. Shen, et al. presented a stress induced phase transformation of β-W. They observed the β phase W to be formed with stacking faults which were stabilized by the O atoms. Thus the presence of optimum concentration of O atoms was necessary for a particular thickness of film [27]. The first experimental evidence of O atom-removal induced phase transformation was presented by T. Karabacak, et al. They use a hot stage during transmission electron microscopy (TEM) to heat the sample and observe the removal of O atoms with the phase transformation of β-W to α around 950°C which is higher than maximum transformation temperature of 650°C due to absence of grain boundaries in the lamellae used for TEM [28]. Growth of β-W beyond a certain critical thickness is primarily kinetically controlled by the concentration of impurities available during the deposition which restrict the grain growth. Upon introducing large concentrations of inert impurities viz. N, amorphization of W is observed [17, 16, 29]. A N-atom adsorbed template assisted growth of β-W has also been proposed [30] The thermodynamic choice to crystallize in A15 symmetry under the critical thickness still needs to be explored.

Parallel to research on understanding growth of β-W, α-W was being explored as a potential candidate against Cu as an interconnect material in semiconductor industry [31, 32, 33]. Much work was also done to successfully transform the β-W fully to α-W either by heating during the deposition or utilizing a faster deposition rate. In both conditions a immense heat is generated during the deposition which onsets the rearrangement of atoms and eventually leads to a phase transformation [34, 35, 36].

1.2.2 Perpendicular magnetic anisotropy

The PMA is a result of a magnetic anisotropy at the interface which drastically differs from the magnetic anisotropy in the bulk. This kind of interface or surface anisotropy was first predicted by Ne’el as an response to lowered symmetry at the surface or interface [37]. The first experiments which had revealed such an interface anisotropy were performed in 1968
by Gradmann and Muller (1968) on ultrathin NiFe films on Cu(111) [38]. They demonstrated an easy axis perpendicular to the film plane for 1.8 monolayers of NiFe and furthermore that the magnetic anisotropy scaled with the reciprocal of film thickness. For multilayers, PMA was first observed in 1985 by Carcia, et al. in the Co/Pd and Co/Pt system [39]. Later on the much research was done on Pt/Co/AlO$_x$ interface to attain PMA [40, 41]. In a work done by Manchona, et al. on Pt/Co/AlO$_x$ trilayers the authors found that the PMA had a strong dependency on the content of oxygen present at the interface. The authors basically demonstrated that there is an optimum amount of oxygen, when present at the interface of Co and AlO$_x$ induces PMA. The oxygen from AlO$_x$ at the interface bonds with the p-orbital of Co atoms. With an optimum amount of O atoms are present at the interface makes the p-orbital out of the plane of the film. This induces an anisotropy in the magnetic moment of the Co film. An excess amount of O atoms will get diffused into the Co layer and the p-orbital no longer will have a magnetic moment out of the plane [42]. In 2010 Ikeda, et al. demonstrated PMA in a MTJ using CoFeB|MgO|CoFeB stack [43]. MTJs with FM top and bottom layer with PMA are of immense interest as they have a potential for realizing next-generation high-density non-volatile memory and logic device with their high thermal stability and low critical current for current-induced magnetization switching. In this paper, the authors use interfacial perpendicular anisotropy of CoFeBMgO. The perpendicular MTJs consisting of Ta|CoFeB|MgO|/|CoFeB|Ta show a high tunnel magnetoresistance ratio, over 120%, high thermal stability at dimension as low as 40 nm diameter and a low switching current of 49 $\mu$A. In 2011 Worledge, et al. from IBM published their work on spin torque switching of PMA Ta|CoFeB|MgO free layer with a synthetic antiferromagnetic reference layer [44]. A very low switching voltage of 290 mV was needed for a quasistatic switching. They were able to switch the FM free layer as quickly as 1 ns speed. This was the pivotal moment for MTJ to have obtain a potential to compete with existing memory technologies. The process and fabrication conditions of such stacks strongly influence the device properties. An optimum annealing temperature of 350°C was identified as an ideal temperature. Any
further annealing the or heat which increased the film/device temperature beyond 350°C usually resulted in deteriorating the PMA due to excessive boron diffusion and magnetic dead layer formation. The thickness of CoFeB plays a crucial role in competing with the interface anisotropy, a thicker CoFeB will generally lead to a in-plane magnetic moment, continuous films under 1.5 nm typically lead to PMA on an appropriate surface and capping layer [45].

Such a control of thickness on PMA was also observed in CoFe films grown on Ta and capped with MgO layers. A cross-over from in-plane to out-of-plane anisotropy was observed as the Co concentration in CoFe was varied. The results suggest that the degree of filling of valence bands in the CoFe adjacent to the interface, which determines the relative population of the anisotropic d-bands, controls the interfacial anisotropy brought on by CoFe-O hybridization at the metal/oxide interface [46]. Zhang, et al. presented a theoretical understanding behind the PMA. Their findings show that the magnetocrystalline anisotropy (MCA) is strongly dependent upon the CoFe composition and that the MCA decreases with increasing Co concentration. At a certain composition there is a transition at which the MCA changes sign from positive to negative. The origin of the MCA is related to the difference of density of states between d orbital around Fermi energy. One major challenge for CoFeB was the limited annealing temperature only up to 350°C which is low compared to the typical processing temperatures of semiconductor IC fabrication. Tantalum’s greater affinity to boron leads to the degradation of PMA in a Ta|CoFeB|MgO stack. Liu, et al. used a Mo underlayer which has a robust BCC crystal structure which does not allow B to diffuse through. This Mo|CoFeB|MgO stack allowed the annealing temperature up to 400°C in these films [47]. Worledge, et al. published an invited article on developing PMA. They use a fast-turn around technique to explore materials which are suitable for PMA. They measured the moment/area and anisotropy field of in-plane materials as a function of CoFeB thickness. Various seed layers were tried to explore their effect on PMA. This method led to the discovery of PMA in Ta|CoFeB|MgO at IBM with annealing up to 400°C with Fe insert
layer [48]. Utilizing the appropriate insert layer to change the interface is key to control interface anisotropy. Interfaces which are rough or which have a lattice mismatch with CoFeB (001) have difficulty supporting a PMA in CoFeB layer. For such systems utilizing an appropriate insert layer induces a template to growth the CoFeB with PMA. For instance tungsten’s surface is very rough and it does not support a PMA CoFeB. By utilizing Ta or Hf insert layer the interface anisotropy can be controlled to produce PMA in CoFeB [6, 49].

1.2.3 Spin Hall effect

Coupling between spin and charge currents was theoretically first predicted in semiconductors by D’yakonov and Perel’ in 1971 [50]. The very next year experimental evidence was collected for anomalous Hall effect in non-magnetic semiconductor InSb, which mainly was the evidence for spin dependent Hall effect [51, 52]. Much later Hirsch theoretically predicted and introduced the term “SHE” in paramagnetic metals in 1999 [53]. In 2003 SHE was theoretically predicted in p-doped semiconductors by Murakami, et al. [54]. A universal intrinsic SHE was give by Sinova, et al. in 2004 [55]. Systems with strong SOC tend to exhibit strong SHE.

From the first experimental evidence in 1972 little work was done in this area. It was Hirsch’s work in 1999 renewed people’s interest in the topic. The next experimental evidence for SHE was observed utilizing optical Kerr effect in InGaAs by Y.K Kato et al. in 2004 [56]. In this experiment a charge current was passed through a micron sized strip of InGaAs while a polarized laser was scanned on the sample. Due to transverse spin accumulation and optical Kerr effect the different reflectivity was observed around the edge of the sample. According to the universal intrinsic SHE mechanism such a transverse spin accumulations are possible. However, due inverse SHE any such spin accumulations should also generate transverse voltage, no such observation was made. In the light of previous statements, since this kind of transverse spin accumulation can occur due to defects in the crystal structure, one can attribute the defects present in the GaAs crystal to be generating
the optical response, in which case the intrinsic mechanism of SHE and the requirement for non-centrosymmetric crystal structures still requires an experimental evidence. The origin of the effect was very much in debate at that point. The theoretical platform was not yet available to completely understand the discovered physical phenomenon.

However, much later in 2010 Eric Garlid, et al. developed an all-electrical technique to detect SHE in Fe/InGaAs heterostructures [57]. In this work the authors utilize the spin transport phenomenon in ferromagnetic semiconductors previously developed. A Hall bar structure of InGaAs is studied with alternating magnetic and non-magnetic contacts. As the charge current travels through the longitudinal channel of the InGaAs spin currents starts to flow in the transverse direction. These spin currents are then collected by a FM-Fe contact. The difference in voltage of a Fe contact at different locations is utilized to estimate the average voltage drop, which is proportional to the transverse spin accumulation. At a constant longitudinal current magnetic field is swept to observe the spins precess in the applied magnetic field. The spin accumulation is then extracted from the Hanle spin precession measurement. In the paper, they successfully change the magnitude of SHE by changing by doping the channel with In. As the In doping increases it increased the SOC and it increase the SHE. They successfully related the SHE to the strength of SOC which reinforces the universal intrinsic mechanism which is dependent on the strength of SOC. The main advantage of the measurement is the long spin diffusion length of InGaAs system.

A similar experiment in a system with shorted spin diffusion length will not be possible. As the spin which are accumulated will relax completely until they reach the magnetic contact. To fabricate such a device will be extremely challenging as it will require the contacts to be a few nm apart. The first experimental evidence for SHE in metals was in Pt in 2007 by T. Kimura et al. [58, 59]. The work presents measurements of both the direct and inverse SHE in the same device. They used the direct SHE to inject a spin current from a Pt into a Cu, and measure the spin accumulation in the Cu using a permalloy contact. In measuring inverse SHE, they injected a nonlocal spin current from Py through Cu and
into Pt, and measured the transverse voltage generated across the Pt due to the inverse SHE. From both the experiments the SHA estimated is 0.0037. However, in estimating the SHA authors did not take into account the shunting of the voltage by Cu on Pt. Thus under estimating the SHA. This was a simple experiment and a pioneering effort which demonstrates the SHE in metals for the first time ever. The shunting effect was taken into account much later in 2011 and a SHA of 0.021 was estimated. However, this number is largely debated in comparison to the other groups [58].

The pivotal moment in this field was in 2010 when the charge current through Pt was used to switch the magnetic moment of adjacent Co dot by Miron et al. from ETH Zurich from Pietro’s group [12]. In this experiment a Co bit of about 500 nm was grown on the center of a Pt cross. The Co layer was capped with AlO$_x$ layer which provides strong interfacial anisotropy for the magnetic moment of the Co layer to be oriented out of the plane at the interface. Since there exists strong SOC all through the thickness of Co layer the magnetic moment through out the thickness are oriented in the out of the plane direction. In a multilayered structure like Pt$|$Co$|$AlO$_x$ there inherently there exists a SIA which generates a spin accumulation transverse to the charge current flowing through it. The Hall resistance of a FM is proportional to the Z component of the magnetic moment vector [60]. By measuring Hall resistance of the stack, the information about the magnetic moment can be extracted. In this paper the authors perform a anomalous Hall measurement while they sweep the magnetic field from a positive to a negative value, thus rotating the magnetic moment vector. They also perform a current sweep while applying a constant magnetic field parallel to the current direction to break the inversion symmetry. They observe an irreversible switching in the resistance value. This switch signifies the switching of magnetic moment from +Z to -Z direction. The constant magnetic field is applied all the three directions to ensure the reversal is symmetric. They also perform pulsed reconfigurable switching of the ferromagnetic layer’s magnetic moment [61].

At the time these two articles received much of the attention from the Spintronics
community. However, not much later Liu et al. from Dan Ralph group at Cornell published a similar work with an entirely different working model [4]. In this work, Liu and authors present a different technique to study the torque generated by the current induced effects. Here a ferromagnetic resonance (FMR) signal generated by the spin torque from the current induced SHE is estimated from the line-width of the resonance signal. The SHA is then extracted from the ratio of the charge current to the spin current. In the Cornell group’s work the spin torque generated was attributed to the SHE and in the ETH Zurich’s group it was attributed to Rashba spin orbit interaction. Much later in 2013 Paul et al. theoretically and experimentally proved the coexistence of SHE and Rashba effect in different experimental systems. However much later in 2015 Allen et al. from Intel unequivocally showed the coexistence of Rashba and SHE [62].

The major breakthrough came around in 2012 when the Cornell group Liu et al. and Pai et al. were able to utilize the SHE to switching the resistance of a MTJ [7, 5]. A new material was used to generate spin current through SHE. These were the $\beta$ phase of Ta and W they were estimated to have SHA of 0.15 and 0.3. 15% of the charge current flowing through a $\beta$ phase Ta is converted to spin current is accumulates transverse to the charge current. This transverse spin current is capable of exerting a spin torque on an adjacent FM’s magnetic moment. Up on exerting sufficient torque the magnetic moment of the FM will flip. In the works of Liu et al. and Pai et al. the authors show the direct application of SHE.

One major drawback in the theory of these systems is the spin diffusion lengths which are estimated for these materials. A very low value 1.4 nm spin diffusion length was estimated for $\beta$-Ta. Since, spin diffusion length in the spin-torque-FMR is used as a fit parameter rather than an estimated value. Some light towards the spin diffusion length was cast by Otani group. Niimi et al. published a work of verifying the spin diffusion lengths of strong SOC materials like Pt [15]. In this work they used the weak antilocalization effects in non-magnetic materials to estimate the SOC length and spin diffusion can be extracted from
these lengths were about 10 nm. An explanation for such a low value spin diffusion lengths observed from spin torque-FMR was attributed to the interference of magnetic moment from the adjacent FM. Niimi, et al. also measured a device of Pt wire with an adjacent FM and observed a reduction in the spin diffusion length. A more experimental way to analyse the SOT was developed by Kim et al. [63, 64]. In this work an alternating current is passed through FM/HM bilayer while applying the magnetic field in the place of the device but either transverse or parallel to the current. By which two the transverse and the longitudinal effective fields are extracted. A low frequency is utilized for the alternating current so that the magnetic moment is in phase with the longitudinal current. The second harmonic Hall voltage signal is collected while external magnetic field is swept either transverse or longitudinal direction. The slope of the second harmonic Hall voltage versus magnetic field give the effective field in that direction. The technique is very versatile and can be applied for FM with in-plane and out of-plane magnetic moments as well [65]. The technique was originally developed by Pi, et al. to estimate the tilt of the magnetic moment due to Rashba effect [66]. A major requirement of SHE induced switching in FM is the in plane external magnetic field required to break the inversion symmetry. A recent work by Lau et al. uses a antiferromagnet and a FM stack on top of the FM and HM bilayer. The FM under the antiferromagnet coupled and creates a bias field which is sufficient break the inversion symmetry thus deterministic switching is observed in the system [67]. In order for a comparable device performance a larger SHA is necessary. The highest reported SHA until 2016 is 0.5 by Demasius et al. from IBM-Spintronics group [68]. In this paper β-W is highly doped with oxygen atoms but still in metallic state. The reduced grain size and increased impurities add to the skew scattering and increase the spin current generated for a given charge current.
1.3 Thesis Plan

From the previous sections it is evident that fabricating the right stack of materials lies at the crux of SHE device performance. PMA in the FM deposited on a SHM with large SHA is the ideal stack. Thus, working with $\beta$-W becomes the obvious choice. As the thicker films start to transform to $\alpha$ a technique to grow thicker $\beta$-W would be necessary for a better process window, which is importance for large scale fabrication. Also CoFeB or Co FM on $\beta$ W is not a favorable stack for PMA even when capped with MgO. Thus a unique technique to grow the FM with PMA is needed. An insert layer of Ta between CoFeB and W will be presented. Current induced spin-orbit effects of a Si|SiO$_2$|$\beta$-W (5 nm)|Ta (1 nm)|CoFeB (1 nm)|MgO (1.6 nm)|Ta (Cap) will be studied and an empirical model to estimate the SHA will be derived.

The outline of this thesis will be as follows. Chapter 2 will present the theoretical background to understand spin transport. Chapter 3 will outline the experimental methods utilized to fabricate thick $\beta$-W and measure the current induced spin orbit effects in the proposed stack. The effect introducing O$_2$ and N$_2$ gas during the growth of $\beta$ will be detailed in chapter 4 and chapter 5 respectively. In chapter 6 will focus on the custom rotatable holder which was built to perform the electrical measurements. Chapter 7 will present the results of depositing an insert layer on PMA of CoFeB and in-plane current induced effects on devices with PMA. All the results will be discussed in chapter 8 and conclusions of this thesis will be given in chapter 9 with a future outlook.
CHAPTER 2
THEORETICAL BACKGROUND

2.1 Thin Film Deposition

Thin film deposition mainly involves three steps as follows.

1. Creating a vapor/flux of particles from a source.
2. Transport the vapor/flux to the substrate.
3. Condense/react the vapor/flux on the surface of the substrate.

Depending on the technique utilized to generate the vapor or flux the deposition technique can be broadly classified into a chemical method and physical method [69].

2.1.1 Physical method: Sputter Deposition

Sir W.R. Grove discovered surface coatings generated in the valve where he worked on glow discharge. Sir W. Thomson called the phenomenon "SPLUTTERING", in analogy with generation of drops of spilling out a liquid surface upon impingement of the primary drop. As a result of printer’s error the term "SPUTTERING" is now a widely accepted. The ejection of particles from a solid surface after exposure to impingement with heavy particles (mostly Ions) of sufficiently large energy is called sputtering. The initial understanding was that the solid surface got locally heated, which does happen in sputtering, and created a physical vapor. However, experimentally it was verified that the angular deposition of sputtered particles largely depended on the angle of the impinging particles. The sputter yields not only depend on the particle energy but also on their masses. Sputtered particles have higher kinetic energy than when they are evaporated thermally, without large heating of the sputter target. All these imply that it is the momentum transfer from the heavy particles to the target or solid surface which results in a physical vapor. In the case of single crystal targets
the sputter particles also exhibit a preferred direction. Since the momentum transfer is also directional and when a single crystal is being impinged there can be a unique direction where multiple impingements can occur due to the symmetry leading to a directional particle flux.

The easiest source of ions is the plasma of DC glow discharge which are explained hereafter. A diode system with gas pressure of about 100 mtorr when appropriate amount of voltage is applied creates glow and then arc. While ramping up the voltage initially generic I-V characteristic is observed. Until a critical voltage is reached where the primary ions are capable to ionize the neutral ions and create a self-sustainable glow-discharge and alternate charge carriers for current. In regions of abnormal glow a higher current is associated with a higher energy of ions and hence this can be controlled by voltage and is used for sputtering. Due to their higher inertia the ions generate a much wider double layer at the cathode than the electron layer at the anode. The highest voltage drop, also known as cathode fall, extends over this area, where ions obtain their kinetic energy and and are used for subsequent sputtering and for ejection of secondary electrons for sustaining the discharge. The electrons are accelerated towards the anode, for ionization, they ionize at the end of the cathode fall, generally characterized by a dark room (Krookes DR). There they completely lose their energy and thus accumulate in a somewhat higher density, manifested in an opposite field. In this region of the positive column, the field strength is low and charge carriers move by diffusion rather than by drift gradient.

The Krookes DR represents mainly the distance needed by an electron for an ionizing collision. Thus, a discharge cannot be ignited if the anode and cathode are closer than this distance. Thus, the distance between anode and cathode can be estimated from the Paschen’s Law, which is an extension to Krookes DR and given by the following relation.

\[
d = \frac{1}{P} \left( \alpha + \frac{\beta}{(V - \kappa)} \right)
\]  

(2.1)

where \(d\) is the distance between anode and cathode, \(P\) denotes the pressure, \(V\) voltage,
$\alpha, \beta, \kappa$ constants which depend.

### 2.1.1.1 Sputter deposition techniques

General DC Sputtering process parameters are as follows

- **DC typically 1-5 kV**
- **Current density = 1-10 mA/cm$^{-2}$**
- **Target area $\approx 10$ Sq. cm**
- **Separation distance**
- **Ar gas is used**

Substrate is biased negatively ($\approx 100$ V) the plasma and anode are positively charged. This mild bias improves the quality of films by ions precleaning the substrate and film surface (removing water vapor or trapper atoms). Ions can also be pumped out simultaneously during the growth to improve the quality. By ion impingement insulators will develop a charge with positive surface potential so that further impingement is restrained. Thus, DC sputtering can only be used for sputtering conductors. Another technique will be described shortly avoids such a charging and also creates a possibility to use lower pressure.

In a radio frequency sputtering system insulators can be deposited without any build up of charge. A capacitor is connected in series with the cathode and the RF source. The capacitor gets charged negatively from cathode as it draws from electrons than ions (heavier) from the anode. At a steady state condition there will be a charge build up on the capacitor, thus a DC bias on the target. Under an alternating bias, electrons and ions in the plasma both are responding but with different degree of response. The anode and the cathode i.e substrate and target both get sputtered depending on the frequency of source. Ions, as they are heavy than electrons cannot keep up with the quickly switching RF bias. Thus the electrons neutralize the charge build up on the target. The sputtering of targets when the
target is negatively charged. However, the target has a DC-self bias due to which the time compared for which the targets sputter is much longer than the time for which the substrate gets sputtered.

This process is not limited by substrate material. Thus dielectric materials like, MgO can be successfully deposited by such a technique. The discharge tube pressure is self-sustaining under as gas pressure down to slow is $2 \times 10^{-4}$ Torr. The effect is detectable above 50 kHz but typical frequencies are around 13.65 MHz. If a electron oscillates at the RF field while it collides with other particles using the RF energy, it will increase the total number of ionizing particles.

In a magnetron sputtering system a magnetic field is used to confine the plasma around the cathode in a DC sputtering system. Confining the plasma this way increases the efficiency of the sputtering system as the concentration of atoms increases which are available for impingement on the target material. The plasma mostly gets confined to the shape of magnetic flux line, which usually takes a shape of a ring. Thus the target preferentially sputters in this region. Deposition rate upto a few nm per sec can be estimated using this technique.

2.1.1.2 Sputtering yields

Unlike evaporation of thin films which is strongly dependent on the heat of sublimation ($\Delta H$) of the source, sputter yields do not have a strong dependence on the $\Delta H$. In evaporation of thin films the particle which is evaporated receives it’s energy from a heating element via heat conduction. In the case of sputtering the particle at the surface receives it’s energy to leave the surface of the target from an external atom or cluster of atoms through momentum transfer. Although certain heat is generated due to inelastic collision, at the surface the process is more controlled by an external source than an internal transfer of energy. The
sputter yield can mathematically expressed as

$$S = \frac{n_s}{n^+} = \left( \frac{m^+ m_s}{m^+ + m_s} \right) \times \frac{E_k}{\Delta H} \quad (2.2)$$

where $n_s$ and $n^+$ are the number of sputtered and impinging particles respectively, $m_s$ and $m^+$ are their respective masses, and $E_k$ is the kinetic energy of the impinging particles. The sputter yield is largely dependent on the mass of target atom and thus the atomic number of target atoms. They usually differ by order as the mass of the target atom changes a group in periodic table. When sputtering alloys, the lighter atom gets sputtered sooner than the heavier atom. This increases the concentration of refractory atoms on the surface. Higher concentration at surface increases the sputter yield of the refractory atoms, eventually this reaches a equilibrium. The equilibrium is reached when the distribution in sputter yields is compensated by the concentration distribution on the surface of target. In order to obtain highly pure alloys co-sputtering technique is sometime better. Advantages of sputter deposition system.

1. Widely applicable to different materials.
2. Good adhesion to the substrate is ensured.
3. Uniform thickness over large area is obtained. Very good for large scale IC fabrication.
4. Control of the thickness is possible as thickness proportional to the deposition time.
5. *In situ* substrate cleaning can be done by ion impingement.

Disadvantage of sputter deposition.

1. Source material must be malleable to form sheets
2. Deposition rate is slow only less than 40 Ås$^{-1}$.
3. Substrates cooling is necessary except for quick depositions.
2.1.2 Conditions for physical vapor transport

An ambient high vacuum base pressure is very necessary for an efficient sputter deposition. If any foreign particles are present, like water vapor, it might either react with the target atom flux or it can be adsorbed on the substrate or react with the newly formed film as well. All these aspects create a need of a very low base pressure, usually any vacuum below $10^{-7}$ is considered a good base pressure. Another reason why we want a good base pressure is due to the mean free path of the target particles, which in case of a higher pressure will be very short and thus the particles will never make it to the substrate, and even if they do it will be a very distorted and non-uniform flux of particles.

From the idea gas equation, the number of gas atoms per unit volume, $n$, at any given pressure $P$ is

$$n = \frac{P}{\kappa T}. \quad (2.3)$$

Which implies at atmospheric pressure and room temperature $n = 25 \times 10^{19}$ atoms are present in 1 CC of volume and at $P = 10^{-8}$ Torr here are still $3.2 \times 10^{8}$ atoms in 1 CC of volume. A rough estimation of mean free path (MFP) of particles as the average distance between two collisions through a unit length, with an atom diameter $d$ can strike $n d^2 \Pi$ gas atoms of similar size. The mean free path is thus given by

$$Mean \ free \ path = \frac{1}{\sqrt{2nd^2\Pi}}. \quad (2.4)$$

For instance, at $P = 10^{-3}$ Torr MFP = 5 cm (low pressure sputtering) and $P = 10^{-8}$ Torr the MFP = 5 km (Vacuum evaporation). The number particles without a collision incident after a distance of $x$ can be statistically estimated by

$$N(x) = N(x = 0)exp(-\frac{x}{MFP}). \quad (2.5)$$

In sputter system usual working pressure $P = 5 \times 10^{-2}$ Torr, at $x = 10$ cm, $N = N_0 exp(-100)$
≈ 0, none of the sputtered particles reach the substrate without a collision. 50 % of them already encounter the first collision in the first 0.7 mm. Thus sputtered particles reach the substrate from many different directions which improves the coverage of the substrate.

2.2 Nucleation and Growth of Thin Films

In this section the process of film growth from the first few atoms condensing on the surface of substrate to the final film will be discussed. The quantitative steps involved in the film growth are as follows:

1. Thermal loss of the heat from the ”hot” vapor atoms to the atoms on the surface of the substrate
2. Adhering of the target atoms on the substrate
3. Target atoms diffuse on surface and form clusters
4. Formation of supercritical clusters and conversion to islands
5. Island combing to form larger islands
6. Growth of the continuous film

2.2.1 Physical vapor-ambient equilibrium

The incoming vapor atoms get adsorbed on the substrate by dissipation of their kinetic energy to the substrate atoms within a few lattice vibrations (10^{14} Hz). The incoming vapor atoms have an average kinetic energy of \( \frac{3kT_s}{2} \), where \( T_s \) is temperature of source. The desorption kinetics are proportional to \( \exp(-\frac{E_d}{kT}) \), where \( E_d \) is desorption energy. Hence, \[
\frac{n_d}{n_a} = \gamma \exp\left(-\frac{E_d}{kT}\right). \tag{2.6}
\]
where \( n_d \) is number desorbing atoms, \( n_a \) is number adsorbing atoms, and \( \gamma \) is the attempt frequency for escaping. At equilibrium between absorption and desorption,

\[
R = \frac{dN}{dAt} = n_d. \tag{2.7}
\]

where \( R \) is the growth rate, \( N \) is the total number of atoms, \( A \) is the area of impact, \( t \) is time. Surface agglomeration of atoms or absorbed atoms:

\[
n_a = R \exp \left( \frac{E_d}{kT} \right) = R \tau. \tag{2.8}
\]

where \( \tau \) is mean residence time of adatoms at the substrate surface. For example, if \( R \) is about 10 \( \text{Ås}^{-1} \) with an atomic diameter of 1.5 \( \text{Å} \) gives \( R = 5 \times 10^{16} \text{atoms cm}^{-2} \text{s}^{-1} \). For Cu on sapphire substrate,

- \( E_d = 0.14 \text{ eV} \)
- \( \exp(\frac{E_d}{kT}) = 380 \)
- \( \tau = 10^{14} \text{ Hz} \)
- \( n_d = 2 \times 10^5 \text{ atoms cm}^{-2} \text{s}^{-1} \).

For Ag on sapphire substrate,

- \( E_d = 0.6 \text{ eV} \)
- \( \exp(\frac{E_d}{kT}) = 1.1 \times 10^{11} \)
- \( n_d = 5 \times 10^{13} \text{ atoms cm}^{-2} \text{s}^{-1} \).

During surface diffusion the adatoms can interact with other adatoms and form agglomerates or clusters. Under subcritical size of an agglomerate, adatoms can reversely combine with an agglomerate eventually reaching equilibrium. Number of clusters of radius \( r \) less than less
than the critical radius \((r^*)\) is given by

\[
\frac{n(r)}{n_a} = e^{\frac{-\Delta G(r)}{\kappa T}} \tag{2.9}
\]

where \(\Delta G(r)\) is the Gibbs free energy of formation of a cluster of radius \(r\). The clusters whose radius \(r \geq r^*\) grow at the expense of further adatoms irreversibly. Gibbs free energy decreases when an agglomerate reaches critical radius. \(\Delta G\) is given by the heat of condensation and the surface energy. When a small cluster starts forming it has a small size. With a small size it has a smaller surface area and lower surface energy given by \(\sigma K r^2\). The heat of condensation is given by the \(\Delta gv \dot{K} r^3\), where \(K\) and \(\dot{K}\) are constants, \(\sigma\) is the coefficient of surface tension and \(\Delta gv\) the heat of condensation per unit volume. Due to the small volume the Gibbs free energy of condensation does not compensate fully the surface energy thus it is reversible until it reaches a critical size. Beyond the critical size any addition of atoms reduces the Gibbs free energy in other words, the surface is expanded at the expense of heat of reaction or heat of condensation. There exists a threshold for nucleation beyond which film growth takes place. This is called as critical supersaturation. Experimentally it has been shown that the smallest critical nuclei can contain anywhere up to one to a few atoms. In accordance with an atomistic view Gibbs free energy of formation of cluster can be ideally replaced with the energy for decomposition of an aggregate into individual atoms. This gives a clearer picture in regard to assumption that nuclei and aggregate are made up for discrete atoms. Another atomistic theory with regard to decay and formation rates of super critical clusters was proposed by Frenkel and Zinsmeister.

### 2.2.2 Thin film growth

The decay of the super-critical cluster is much less probable to occur than the film growth. The maximum length within which the adatom can be located so that it can reach the nuclei in its lifetime is called the ”capture zone” of the nucleus. This was not considered before as the zone will be depleted of adatoms since they get incorporated irreversibly in
the nucleus. We already know the mean square distance travelled during N steps is $Na^2$. We assume this area to be a circle due to entropy reasons, circle has a equal probability from any direction. A cluster if present on this circle, it will most probably capture the adatom. Thus,

$$\langle l^2 \rangle = Na^2 = 2D_s \tau$$ \hspace{1cm} (2.10)

, where $D_s$ is the surface diffusion coefficient, the radius of the zone can be estimated as

$$r^2 \approx e^{\frac{E_d - E_{diff}}{\kappa T}} a^2$$ \hspace{1cm} (2.11)

For instance Cu on glass, $a^2 = 4 \text{Å}^2$, $e^{\frac{E_d - E_{diff}}{\kappa T}} = \exp(4.255)$ and $r = 16 \text{Å}$. The number of nuclei on a given unit area, surface coverage ($N(t)$) with capture zone is given by

$$N(t) = \frac{1}{D_s \tau} [1 - e^{-(ID_s \tau t)}]$$ \hspace{1cm} (2.12)

, where $I$ is a constant, $t$ is small time duration. A time past the time constant $\frac{1}{(ID_s \tau)}$ the time duration of nucleation is completed and the surface is mostly covered by capture zones. For complete condensation the deposition rate ($R$) should be high enough so that the reversible equilibrium between adatoms and clusters is not established. It can happen if

$$R \geq \frac{1}{a^2 \tau}.$$ 

Once the substrate is completely covered with capture zone further nucleation will not occur. Islands will start growing on the substrate either by adding more adatoms to them or by adatoms diffusion on the surface of the substrate. \textit{In situ} electron microscopy has shown that when two islands come close to each other they coalesce. Consider a situation where two cap shaped islands come close to each other. At the juncture of there will very strong outward forces due to large difference in surface tension. Which leads to quicker movement of atoms to this higher energy area. The islands will try to minimize the surface energy by changing their shape. Eventually they shrink into a shape with only a single-cap aggregate. Even if there existed a crystallographic orientation in islands before the coalescence it will
be dissolved after completion of coalescence process. The grain boundary of the islands prior
to coalescence will slowly disappear towards the end of the coalescence. The surface area
of the combined islands is actually lower than sum of individual islands. This creates new
uncovered surface on the substrate or film. These uncovered areas if are under the capture
zone of a nuclie, which has a high chance to happen, will trigger secondary nucleation. The
islands thus grow to give a uniform coverage to the substrate by adding more adatoms and
eventually form a continuous film.

2.2.3 Epitaxial growth

Any relation between structure of the film to the surface structure of the substrates
can be termed as epitaxy. However a more general understanding is when growth of single
crystal occurs on a single crystal substrate. Specifically, when a single crystal substrate of
same materials as the target film grows as a single crystal is called, iso, auto, or homoepitaxy.
A more rare complicated occurrence is when a single crystal film grows on a single crystal
substrate of a different material which is called heteroepitaxy. The features associated with
the heteroepitaxy are listed here after.

1. The geometrical alignment of the substrate surface structure with one lattice plane of
the film is a favorable but not necessary condition for epitaxial growth.

2. Above a certain substrate temperature ($T_e$) is required for the substrate for a epitaxial
growth of the target material. $T_e$ depends on the surface impurity of the substrate
and the deposition rate of the target material. An elevated temperature is required
due to lower surface contamination by desorption and higher surface mobility during
diffusion. It also favor larger nuclei, recrystallization and defect reduction.

3. Lower deposition rate is preferred to avoid large flux of atoms interfering in surface
mobility leading to over growth.

4. Contaminants forming amorphous layers can be a huge hindrance for epitaxial growth.
In some cases, epitaxially adsorbed contaminants may allow lattice mismatch and thus eventually allow epitaxial growth. A higher $T_e$ might be required for less clean substrates than freshly cleaved substrates in ultra high vacuum.

5. Electron or ion impingement prior to deposition on substrates can assist the removal of surface contaminants.

A widely used method to grow epitaxial films is molecular beam epitaxy (MBE). A recent technique called chemical beam epitaxy which combines MBE and metal organic chemical vapor deposition is being explored.

### 2.3 Spin Orbit Interaction

The spin orbit interaction (SOI) is a very crucial concept in spintronics physics. The strength of SOI changes the spin relaxation times. American physicist Kronig, from an incorrect idea of electron spin, led the formalism of SOI. Kronig thought an electron revolving around itself, i.e. electron spin, generates a magnetic moment which can interact with its orbital momentum. An electron orbiting the nucleus will experience an electric field from positively charged nucleus. Thus in rest frame of electron, there exists a magnetic field, nonexistent in the laboratory frame, through Lorentz transformation. The magnetic flux density given by Einstein’s relativity theory is as follows.

$$\vec{B} = \frac{\vec{\varepsilon} \times \vec{\nu}}{c^2 \sqrt{1 - \frac{\nu^2}{c^2}}}$$ \hspace{1cm} (2.13)

where $\varepsilon$ is electric field from nucleus, $\nu$ is orbital velocity and $c$ is speed of light in vacuum.

The equation 2.13 was later proved to be wrong by Thomas in his paper published in Nature (192). While using the Lorentz transformation to go from lab frame to electron’s frame there needs to be rotation added to align with the electron’s rotation as well. Since the electron is revolving and thus accelerating due to change in the direction of electron’s velocity. This when taken into account adds a factor of 2 in the denominator. The detailed
version of derivation can be found in Thomas’s paper [70] a simpler one can be found by Kroemer [71]. Although the idea of self rotating electron is not true, most of the derivations take the idea in developing the physics. But it must be remembered that electron’s spin state is a quantum mechanical concept, a solution to Schrodinger’s equation. This kind of semi-classical approach is prevalent in Spintronics. Thomas’ equation:

\[
\vec{B} = \frac{\vec{\varepsilon} \times \vec{\upsilon}}{2c^2\sqrt{1 - \frac{\upsilon^2}{c^2}}}
\]  

(2.14)

The interaction energy of a self rotating electron with spin angular moment \( \vec{\mu}_e \) in a magnetic field \( \vec{B} \) is given by

\[
E_{\text{Rel}} = -\vec{\mu}_e . \vec{B}
\]  

(2.15)

Landé proved that the ratio of magnetic moment (\( \vec{\mu}_e \) in units of Bohr magnetron \( \mu_B \)) of the electron to it’s angular momentum (in units of associated with self rotation) is a constant defined as gyromagnetic ratio or Landé g-factor:

\[
\frac{\text{magnetic moment}}{\text{angular momentum}} = -g_o \vec{\mu}_e = -g_o \mu_B \vec{s}
\]  

(2.16)

where \( \vec{s} \) is spin angular momentum.

From Equ. 2.16, Equ. 2.18 and Equ. 2.14

\[
E_{\text{Rel}} = -g_o \mu_B \vec{s} . \vec{B}
\]  

(2.17)

\[
E_{\text{Rel}} = g_o \mu_B \frac{\vec{\varepsilon} \times \vec{\upsilon}}{2c^2\sqrt{1 - \frac{\upsilon^2}{c^2}}} \vec{s} = g_o \frac{e\hbar}{2m} \frac{\vec{\varepsilon} \times \vec{\upsilon}}{2c^2\sqrt{1 - \frac{\upsilon^2}{c^2}}} \vec{\sigma}
\]  

(2.18)

where \( \vec{s} = \frac{\vec{\sigma}}{2} \) and \( \mu_B = \frac{e\hbar}{2m} \).

In a non-relativistic system, where the electron’s orbital velocity \( (\upsilon_{\text{orbital}}) \) is very less than the speed of light, in other words \( \frac{\upsilon_{\text{orbital}}}{c} \ll 1 \).
Magnetic field can be given from Bio Savart’s law

\[ \vec{B}_o = Ze \frac{\vec{r} \times u_{\text{orbital}}}{4\pi\epsilon_o C^2 r^3} \] (2.19)

The electric field from the Coulomb field is given by

\[ \varepsilon_o^* = \frac{Ze\vec{r}}{4\pi\epsilon_o r^3} \] (2.20)

Thus Equ. 2.19 can be rewritten as

\[ \vec{B}_o = \frac{\varepsilon_o^* \times u_{\text{orbital}}}{C^2} \] (2.21)

which is very similar to Equ. 2.18 barring the relativistic factor of \( \sqrt{1 - \frac{v^2}{c^2}} \) and Thomas’ factor of 2. By performing similar mathematical deductions as previously explained the non relativistic spin orbit interaction energy with, Thomas’ correction, can be given by

\[ \hat{E}_{\text{Rel}} = g_o \mu_B \hbar \frac{Ze}{8\pi\epsilon_o mc^2 r^3} \vec{l} \cdot \vec{s} \] (2.22)

. Which is a specific case of Equ. 2.18. Dirac has proven that \( g_o = 2 \) for a free electron.

The spin orbit interaction Hamiltonian (energy) can be written from Equ. 2.18 by converting the variables to operators.

\[ H_{\text{so}} = -\frac{e\hbar}{4m^2c^2 \sqrt{1 - \frac{v^2}{c^2}}} (\Delta \vec{V} \times \vec{p}).\vec{s} \approx -\frac{e\hbar}{4m^2c^2} (\Delta \vec{V} \times \vec{p}).\vec{s} \] (2.23)

where \( \vec{V} \) is potential and \( \vec{s} = -\vec{A} \vec{V} \) and \( \vec{p} \) is the momentum operator and velocity operator = \( \frac{\vec{p}}{m} \) and \( \vec{p} = -i\hbar \vec{\Delta} \). An electron in the conduction band does not experience the strong nuclear attraction compared to an electron in an atom. However, it may still experience a gradient in potential (\( \vec{s} = -\vec{A} \vec{V} \)) due to any other effects. For instance, an external electric field, due to conduction band discontinuity in a heterojunction or at interface of crystalline
metal and amorphous insulator. This electric field can lead to a spin-orbit interaction derived in Equ. 2.18. This kind of interaction was studied by E.I Rashba in 1960 and it is named after him. It is also called structural inversion asymmetry (SIA), since a structural facet induces inversion asymmetry in conduction band [72, 73, 74]. The electric field or potential gradient can originate from the crystallographic inversion asymmetry in a crystal. This kind of interaction was studied by Dresselhaus in 1954 and it is also termed as bulk inversion asymmetry (BIA) [75, 76].

2.3.1 Rashba interaction: Structural inversion asymmetry

The Hamiltonian of SOI in Equ. 2.23 shows a direct dependence of potential gradient of electrons. The origin of potential gradient can be from many physical origins. A 2-dimensional electron gas (2DEG) inherently has structural inversion asymmetry which causes a potential gradient in the system. Space charge regions and difference in conduction band edges at the interface of systems with different barrier heights can also cause a potential gradient. An electron travelling in such a system will experience a varying electric field which induces a effective magnetic field perpendicular to the electric field and the momentum of electron. This effective magnetic field induces spin orbit coupling (SOC) and creates a spin splitting in the spin-sub bands. The Rashba Hamiltonian and the corresponding spin-splitting energy dispersion are given by

\[ H_{\text{Rashba}} = \alpha (\vec{p} \times \Delta \vec{V} \cdot \vec{\sigma}) \]  

(2.24)

\[ E_{\vec{k}} = \frac{\hbar^2 k^2}{2m} + \alpha k \]  

(2.25)

where \( \alpha \) is Rashba coupling constant which is proportional to the average potential gradient in the 2DEG which represents the strength of Rashba spin orbit coupling (RSOC) and it can also be tuned by an external gate voltage and \( K \) is wave vector of the electron. When
a 2DEG is further confined to a 1D channel, the SOC induces energy dispersion in spin-up and spin-down electrons. In this K will be the wave vector component in the direction of current flow. The dispersion curves of opposite spins are shifted along K, rather than along energy as in Zeeman splitting. The difference between $K_↑$ and $K_↓$ at fermi level is the basis of spin field effect transistor (SpinFET) [77]. Despite many efforts to realize SpinFET, only one is reported until now [78].

### 2.3.2 Dresselhaus interaction: Bulk inversion asymmetry

Time inversion symmetry in solids establish a relation between energy dispersion and spin-sub bands as follows

\[
E_↑(k) = E_↓(-k)
\]  \hspace{1cm} (2.26)

If there exists bulk inversion symmetry which leads to

\[
E_↑(k) = E_↑(k-)
\]  \hspace{1cm} (2.27)

Thus in a system with both the symmetry existent, from Equ. 2.26 and Equ. 2.27 it gives

\[
E_↑(k) = E_↓(k)
\]  \hspace{1cm} (2.28)

an equal distribution of energy for up and down spins, which is true in Si.

However, systems with zinc blend kind of structure (InAs, GaAs et.c) there is BIA which breaks the degeneracy of spin sub bands and gives

\[
E_↑(k) \neq E_↓(k)
\]  \hspace{1cm} (2.29)

Thus creating a difference in spin sub band energies.
2.4 Ferromagnetic Metal film: Switching and Interface Anisotropy

2.4.1 Landau-Lifshitz Gilbert Equation

Landau-Lifshitz Gilbert equation (LLG) equation is at the heart of magnetization dynamics. It was initially proposed by Landau and Lifshitz [79] and much later a correction term was added by Gilbert [80, 81]. The equation attempts to relate magnetic moment of a ferromagnetic metal (FM) with the external field in a dynamical approach. The Landau-Lifshitz equation is given as

\[
\frac{d\vec{M}}{dt} = -\gamma_o \vec{M} \times \vec{H}_{\text{eff}} - \frac{\lambda}{M_s} (\vec{M} \times (\vec{M} \times \vec{H}_{\text{eff}}))
\]  
(2.30)

where \(\gamma_o\) is gyromagnetic ratio, \(\lambda\) is the Landau-Lifshitz damping constant and \(\vec{H}_{\text{eff}}\) is the total effective field. The Landau-Lifshitz Gilbert equation is given as

\[
\frac{d\vec{M}}{dt} = -\gamma_o \vec{M} \times \vec{H}_{\text{eff}} + \alpha \frac{\vec{M} \times d\vec{M}}{dt}
\]  
(2.31)

where \(\alpha\) is Gilbert damping constant and the constants are related as

\[
\dot{\gamma}_o = \frac{\gamma}{(1 + \alpha^2)}
\]  
(2.32)

\[
\lambda = \frac{\gamma_o \alpha}{(1 + \alpha^2)}
\]  
(2.33)

As it can be observed in both the Equ. 2.30 and 2.31 there are two parts in the right hand side (RHS). The first part \(\vec{M} \times \vec{H}\) is called as the Larmor precession term the second term \(\vec{M} \times (M \times \vec{H}_{\text{eff}})\) is called as the damping term.

The Larmor precession term creates a torque which is perpendicular to the magnetic moment and the \(\vec{H}_{\text{eff}}\). This is called as the field-like torque. The torque merely rotates the \(\vec{M}\) in a circular plane. The Z-component of the \(\vec{M}\) does not change by this torque. The damping term acts a torque which aligns the \(\vec{M}\) with the total effective field. The net work is done by the damping torque. Thus when a external magnetic field is applied on a FM
it aligns the magnetic moment of the FM with the external magnetic field. The internal effective anisotropy field can compete with the external magnetic field.

### 2.4.2 Interface Magnetic Anisotropy

Magnetic anisotropy in a FM film refers to the existence of preferred direction in space for the net magnetization of the film or device. If this effect arises from the interface of the FM and adjacent films then it is termed as interface anisotropy. This is much different than bulk anisotropy which orients the magnetic moments along a direction which derives from the crystallographic plane. The total effective magnetic anisotropy energy can be empirically separated in two parts, bulk and interface anisotropy. The relationship is given by

\[
K_{\text{eff}} = K_v + 2 \frac{K_s}{t},
\]

(2.34)

where \(K_{\text{eff}}\) is total effective magnetic anisotropy constant, \(K_v\) is volume anisotropy constant, \(K_s\) is the surface/ interface anisotropy constant, \(t\) is the thickness of the FM film. The layer is assumed to be bound by two identical surfaces, which in reality is never true. The relation also suggests that the anisotropy contribution from each surface is \( \frac{K_s}{t} \). This equation is used experimentally to predict the thickness range where a perpendicular magnetic anisotropy (PMA) in the FM can be found. A positive value of \(K_{\text{eff}}\) indicates a preferred direction for the magnetization vector. Experimentally \(K_{\text{eff}}\) can estimated from the following relation

\[
H_{\text{eff}} = - \frac{2K_{\text{eff}}}{\mu_0 M_s}
\]

(2.35)

where \(\mu_0\) is magnetic permittivity constant, \(M_s\) is saturation magnetization in emu and \(H_{\text{eff}}\) is the effective anisotropy field. This is the field needed to rotate the magnetic moment vector from easy axis to hard axis. It is experimentally determined by torque measurements or field sweep measurements using vibrating sample magnetometry (VSM). A graph of \(t \times K_{\text{eff}}\) is plotted. For all the negative values of \(K_{\text{eff}}\) in-plane magnetic moment is favored. For all the
values $K_{eff}$ is positive favours a out of plane magnetic moment. This limits the the thickness range within which PMA can be observed to $\leq t_\perp = \frac{2K_s}{K_v}$ which usually is around a 1.2 nm for CoFeB. The PMA arising from CoFeB and MgO interface is largely studied. However a generic mechanism for PMA via interface anisotropy is still under construction. A largely accepted mechanism says, the MgO and CoFeB interface have a lattice match so that MgO can crystalize in [100] direction so that CoFeB will as well crystallize in [100] direction. This added crystallographic symmetry is believed to align the p-orbital of CoFe at the interface of MgO. This alignment causes the magnetic moment to be out of the plane at the interface and due to strong SOC in CoFeB we observe a PMA throughout the thickness [82].

2.4.3 Switching of a FM|HM bilayer

When external magnetic field is applied to switch a FM|heavy metal (HM) bilayer with no strong in-plane currents, the switching is dominated by the applied magnetic field. A coherent switching occurs if there is no magnetic domain formation during the switching process. Under such a condition the net torque needs to be balanced for a switching event to occur. However under any magnetic domain formation the switching criteria would change to domain-wall nucleation and prorogation. However, under a condition when the net torque is zero it is the thermal fluctuations of magnetization which make the magnetic moment collinear with the field and thus makes the net torque non-zero. Similar switching behaviour is followed when a spin current exerts a spin torque on the magnetic moment.

When a spin current travels through the FM or spin accumulates at it’s interface, FM will start to experience a torque on it’s magnetic moment. When a sufficiently large density of current passes through the film the magnetization would switch. In such experiments current density is of more importance than current as the effect takes place at the interface and is dependent on the charges distributed over a given area. This phenomenon is termed as spin transfer torque, it was first predicted by J.C. Sloczewski at IBM in 1996. Sloczewski proposed an additional damping term to the classical LLG equation [83]. Independently,
Berger also proposed a similar term in the same year [84, 85]. These were two most pivotal papers for spintronics community.

When a unpolarized current passes through the FM the current collected on the other side is spin-polarized, this phenomenon is called spin filtering [86, 87, 88]. From simple conservation of angular momentum, there should be a torque acting on the FM which is opposite to the torque which spin-polarized the electrons in the first place. Now consider the above a system and we reverse the current direction i.e we flow a spin polarized current through a FM. From the previous conservation of momentum argument. There must be a torque acting on the FM by the incoming spin-current. This torque is proportional to the angle between the magnetic moment of FM and the spin polarization and the current density. The new torque term is given by

\[
\vec{T}_{\text{Stonsewski}} = \frac{J \hbar \epsilon}{2e \ell M_s^2} \vec{M} \times (\vec{M} \times \vec{m})
\]  

(2.36)

where J is charge current density through the junction, \(\epsilon\) is transfer efficiency, M is fixed layer’s magnetic moment, m is free layer’s magnetic moment or the moment associated with the incoming spin current.

In case of a oxide|FM|HM stack there will be spin accumulation at the interface of the FM and HM due to RSOC and spin Hall effect (SHE). In case of RSOC the inherent SIA creates a spin accumulation at the interface. In case of SHE the potential of gradient associated with it is different. The impurities in the metal can be a source of potential gradient and thus induce SOC or it can be due to strong L-S coupling associated with the crystal field splitting of the f-orbitals, or the BIA associated with the crystal lattice. These sources of potential gradient ultimately create a spin current transverse to charge current and spin polarization and is given by [89]

\[
\vec{J}_s \propto \vec{S} \times \vec{J}_c
\]

(2.37)
where $\vec{S}$ is spin polarization.

This spin accumulation at the interface of FM and HM exerts a torque and when sufficient current density is able to generate enough torque it will flip the magnetic moment. In the case of a FM with PMA there will be two kind of torques known as damping like torque and field like torque. The damping like torque is given by:

$$\vec{T}_{DampingLike} = \vec{M} \times (\vec{M} \times \vec{m})$$  \hfill (2.38)

The field like torque is given by:

$$\vec{T}_{FieldLike} = \vec{M} \times \vec{m}$$  \hfill (2.39)

The effect torque due to spin current can be understood by utilizing an effective magnetic field concept. The torques generated by spin current and effective fields are equal. The effective damping like field is

$$\vec{H}_{DampingLike} = \vec{M} \times \vec{m}$$  \hfill (2.40)

The effective field like field is

$$\vec{H}_{FieldLike} = H_{ST}\vec{m}$$  \hfill (2.41)

However, in order to observe a deterministic an external in-plane magnetic field is required. In-plane external field breaks the symmetry and current induced spin torque gives a deterministic switching through anomalous Hall resistance measurement. If charge current along $+X$ with applied field along $+X$ favor a magnetization direction (say $m_z > 0$), reversing the applied field, favors the reverse direction of magnetization ($m_z < 0$). This symmetry is unique for SHE-switching and needs to be evidenced experimentally. If there is no external magnetic field there will be two indistinguishable states for the same direction of current.
2.4.3.1 Empirical Model

Macrospin model was initially proposed to explain the switching observed in FM/HM. However in this model, it is assumed that there is no domain formation during the switching. A coherent switching with complete domain suppression is assumed. Domain suppression is attained by applying the in plane magnetic field at an angle from the plane about 4 $^\circ$ during the current sweep measurement. It will depend on the sample size and interface interactions like DMI, which might lead to domain formation prior to any switching. Under a macrospin model the switching occurs when torque balances the effective anisotropy field. However if there is some domain formation which is possible if the external field is very close to the plane of device. The switching criteria changes to the net effective perpendicular field equalizing to coercive field. An empirical model is developed to estimate the net effective field required to switch the FM. The net effective field will be a sum of external magnetic field’s out plane component and effective field from the SHE. The following equation is the effective from SHE

$$\vec{H}_{SHE} = -\tau^{SHE}(\vec{m} \times (\hat{Z} \times \vec{j}_c)) \quad (2.42)$$

Torque from spin current is given by:

$$\tau^{SHE} = \frac{\hbar \theta_{SHE}}{2|e| M_s t_F} \vec{j}_c \quad (2.43)$$

Spin Hall angle can be estimated by:

$$\theta_{SHE} = -\left(\frac{\Delta H_{in-sw}}{\sum H_{in-sw}}\right)\left(\frac{H_c 2|e| M_s t_F}{\hbar j_c \sin \phi_m}\right) \frac{1}{\sqrt{1 - (\frac{\Delta R_{||}}{\Delta R_{\perp}})^2}} \quad (2.44)$$

where $\theta_{SHE}$ is spin Hall angle (SHA), $\Delta H_{in-sw}$ is the difference in inplane switching fields for a particle current value, $\sum H_{in-sw}$ is the sum of inplane switching fields for a particle current value, $H_c$ is coercive field, $M_s$ is saturation magnetization, $t_f$ film thickness of FM, $e$ is charge of electron, $\vec{j}$ is the current density vector, $\phi_m$ is the tilt in magnetization due
to experimental misalignment, $\Delta R^\parallel$, $\Delta R^\perp$ difference and sum of Hall resistance values when magnetization was out of the plane and into the plane, when external magnetic field is swept in-plane ($\parallel$) and out of plane ($\perp$). The derivation of the above formula is detailed in appendix section A.

2.5 Hall effect family

2.5.1 Hall effect

Charge carriers flowing in X direction through a conducting material under an external magnetic field in Z direction will experience Lorentz force, in Y direction. This creates a transverse charge accumulation at the edge of the sample. This creates an electrical field in Y direction termed as Hall voltage. This was discovered by Edwin Hall in 1879 [90]. The Hall effect is mainly used for quantifying carrier type, density and mobility in materials. The ratio between current and transverse voltage is Hall resistance. Hall effect is directly proportional to the applied external magnetic field.

2.5.2 Anomalous Hall effect

A charge current in X-direction flowing in a ferromagnet will generate a charge accumulation in Y-direction without any external magnetic field. Edwin Hall found that nickel exhibits significantly high Hall voltage at low magnetic fields. The effect was termed as anomalous Hall effect, as the theory was developed much later [91]. Due to spin-dependent band structure or spin-dependent scattering, electrons with spin polarization in $+Z$ direction accumulate to one edge and whose spins are along $-Z$ direction accumulate at other edge. As the ferromagnet has majority and minority spins there will be a net electric field along Y direction.

2.5.3 Spin Hall effect

SHE was theoretically predicted by Dyakonov and Per’el in semiconductors in 1971 [92]. A charge current induces a transverse accumulation of up-spins and down-spins. This is
caused mainly due to spin-orbit interaction in non-magnetic semiconductors and paramagnetic metals. The first experimental evidence was collected in 1972, but was termed as spin-dependent anomalous Hall effect in InSb [51]. Much later about 30 years later the effect was observed in GaAs [56]. No external magnetic field is necessary to observe the effect. As there is no net magnetic moment the accumulated electrons on the edges have equal distribution, hence no net electric field. However, there will be a spin current flowing transverse to the charge current.

2.5.4 Quantum Hall effect

At large magnetic fields and low temperature (Liquid He) Quantum Hall effect (QHE) was discovered by Klitzing in Si/SiO$_2$ field effect transistor in 1980 [93]. A single electron in a strong magnetic fields can has quantized momentum due to Landau quantization. Thus, density of states in 2DEG under strong magnetic field split into equally spaced Landau levels. If the Fermi levels lies between two Landau levels the carriers occupy these levels. Due to which the Hall resistance plateaus and the longitudinal resistance becomes zero. The Hall resistance $= \frac{h}{\gamma e^2}$, where $\gamma$ is an integer depending on the number of filled levels.

2.5.5 Quantum spin Hall effect

In 2005, a year after the SHE was discovered, experimental evidence for Quantum spin Hall effect (QSHE) was also collected. QSHE was predicted by Kane et al., the model can be understood as a combination of two different QHE derived from Haldane’s model. The spin-up electrons exhibit a chiral QHE where as spin-down electrons exhibit achiral QHE. There will be a quantized spin-Hall conductance and zero charge conductance. QSHE was observed in the HgTe/CdTe quantum well structure by Bernevig et al. 2006 [94]

2.5.6 Quantum anomalous Hall effect

F. Duncan M. Haldane proposed a model which predicted QHE without any magnetic field in graphene. However, no experimental evidence was collected for this model. Experi-
mental evidence for Quantum anomalous Hall effect (QAHE) was collected by magnetically doping topological insulators, (Bi,Sb)$_2$Te$_3$, by Chang et al. in 2013 [95]. Topological insulators have strong spin-orbit coupling which enables to realize Anomalous Hall effect (AHE). A net magnetization in such structures, will suppress QSHE from one of the spin-channels (minority), thus realizing QAHE.

A summary of experimental prerequisites for each effect is given in table 2.1.

<table>
<thead>
<tr>
<th>Effect</th>
<th>Magnetic Field</th>
<th>Low Temperature</th>
<th>Net Magnetization</th>
<th>Spin Orbit Interaction</th>
</tr>
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<td>Not Required</td>
<td>Not Required</td>
<td>Not Required</td>
</tr>
<tr>
<td>Anomalous Hall effect</td>
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<tr>
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<tr>
<td>Quantum anomalous Hall effect</td>
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<td>Required</td>
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</tr>
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</table>

**Table 2.1:** Summary of Hall effects family
CHAPTER 3
EXPERIMENTAL METHODS AND TECHNIQUES

3.1 X-Ray Diffraction Pattern

The advent in which X-ray shine onto any crystalline material and these X-rays are scattered by the electron density on the material which lead to a diffraction pattern when collected by a detector is referred to as x-ray diffraction (XRD). The mathematical equation relating the X-ray scattering event to an atom is atomic form factor $f(Q)_a$ for a spherically symmetric density distribution

$$f(Q)_a = f = \int \rho(r)e^{iQr}d^3r,$$  \hspace{1cm} (3.1)

where $\rho(r)$ is the spatial electron charge density of the atom about it’s center of mass and Q is momentum transferred.

In a crystal with miller indices h,k,l when X-ray incident the scattering can be mathematically related to structure form factor, $F_{hkl}$ and is given by

$$F_{hkl} = \sum_j f_j e^{-2\pi i(hx_j+ky_j+lx_j)},$$  \hspace{1cm} (3.2)

where $x_j, y_j, z_j$ are the positions coordinates and $f_j$ is the atom form factor of the $j^{\text{th}}$ atom. The intensity of the X-ray scattered beam is $\propto |F_{hkl}|^2$. We know

$$e^{-2\pi i(hx_j+ky_j+lx_j)} = \cos(2\pi(hx_j + ky_j + lx_j)) - i \sin(2\pi(hx_j + ky_j + lx_j))$$  \hspace{1cm} (3.3)

. Mathematically the crystals symmetry gets transformed to the XRD by which we can study and estimate the lattice parameter, grain size and strain in the unit cell. For instance, if we consider a crystal with a 2-fold symmetry around Z axis. We can have three set of atoms

39
with \((o, o, z)\) atoms lying on \(Z\) axis itself. Also for each atom on \((x, y, z)\) there will be \((-x, -y, z)\). Now if this symmetry is preserved in the pattern \(F(h, k, l) = F(-h, -k, l) = F(\bar{h}, \bar{k}, \bar{l})\). Now,

\[
F_{hkl} = \sum_j f_j e^{-2\pi i (hx_j + ky_j + lz_j)}
\]  

(3.4)

\[
= \sum_j f_j e^{-2\pi i (lz_j)} + \sum_j f_j e^{-2\pi i (hx_j + ky_j + lz_j)} + \sum_j f_j e^{-2\pi i (-hx_j + -ky_j + lz_j)}
\]  

(3.5)

\[
= \sum_j f_j e^{-2\pi i (lz_j)} + \sum_j f_j e^{-2\pi i ((-h)x_j + (-k)y_j + (-l)z_j)} + \sum_j f_j e^{-2\pi i ((-h)x_j + (-k)y_j + (-l)z_j)}
\]  

(3.6)

\[
= F(\bar{h}, \bar{k}, \bar{l})
\]  

(3.7)

Thus any symmetry from the crystal structure is transferred to the pattern. When these incident X-ray strike the atomic planes and scatter. At far off distance (relative to wavelength of X-ray i.e. near detector) these beams from diffracting from different planes a certain criteria is necessary for these waves to have high intensity at the detector which is called Bragg’s law. Given by

\[
2d \sin \theta = n\lambda
\]

(3.8)

where \(d\) is lattice spacing, \(\lambda\) is wavelength of incident wave. Thus one can estimate lattice parameter, grain size, and strain from XRD. XRD was extensively used to study the phase of the W films. Rietveld and Le’Bail method was utilized to estimate the lattice parameter and phase of W films studied in this thesis. All the XRD patterns in this thesis were studied using a Bruker X-ray diffractometer (sealed Cu source) configured in a parallel-beam geometry with a fixed 0.5° incidence angle. Beam conditioning consisted of a graded parabolic mirror with mechanical slits on the incident beam and a parallel plate collimator for the diffracted beam. Detector \((2\theta)\) scans covered a range of 20° to 120° in 0.05° steps.
Instrument function parameters were determined using NIST Standard Reference Material 1976 ($\alpha$-Al$_2$O$_3$).

### 3.2 X-Ray Photoelectron Spectroscopy

X-ray photoelectron spectroscopy was performed using Thermoscientific Theta probe. The surface characterization technique x-ray photoelectron spectroscopy (XPS) studies the stoichiometry and chemical bonding at the surface and about 5 nm below it. Most of the W are films grown in $\beta$ phase by introducing either $O_2$ or $N_2$ gas during the deposition. This can lead to oxidation or nitride formation (much less probable). To know the stoichiometry of these films XPS was used. A monochromatized Al K$\alpha$ x-ray source producing photons at 1486 eV is used. As these X-ray pass through the material, their energy is absorbed by electrons in the material. If there is sufficient energy available to eject electron, they get ejected. The kinetic energy is the difference between energy of source and binding energy. The kinetic energy of electrons collected is relative to the Fermi level of the detector, which is electrically connected to the substrate, and thus in case W (metal) films it is the Fermi level of the sample. Energy conservation equation for the measurement is

$$ E_{kin} = \hbar \nu - E_B^{FL} - \Phi_s, $$

(3.9)

where $\hbar \nu$ is the energy of the incident photon, $E_B^{FL}$ is the binding energy of the electron relative to Fermi level, $\Phi_s$ is the work function of the detector, $E_{kin}$ is the measured kinetic energy of the collected electron. The binding energy spectra obtained from XPS are characteristic of the particular chemical state of elements and the chemical bonds in the material. As a metal gets oxidized it looses its metallic character and more energy is needed to eject an electron from its surface. W is known to form non-stoichiometric oxides and this is studied using XPS is W films.
3.3 Vibrating Sample Magnetometery

A vibrating sample magnetometry (VSM) measures the magnetic moments of ferromagnetic metal (FM) and heavy metal (HM) stacks under external magnetic field. The main magnet utilized in the Versalab is a superconducting magnet. This magnet can be operated up to 3 T. The film is mounted on a glass rod using GE varnish. The sample is lowered into a magnetic field using a sample rod. It is firmly connected to a sample rod which vibrates causing a changing magnetic field. A changing magnetic field induces a current in a pickup coil located around the sample due to Lenz law. The pick up current/ voltage is calibrated to the moment which is read out to the user.

\[ V_{coil} = 2\pi f C m A \sin(2\pi ft) \]  

(3.10)

where \( f \) is the vibration frequency, \( C \) is a tool specific coupling constant, \( m \) is the magnetic moment in the sample and \( A \) is the vibration amplitude. Thus the magnetic moment can be estimated while we sweep the magnetic field. This way we can know if the magnetic moment is out of the plane and sample has any perpendicular magnetic anisotropy (PMA). Thin films fabricated on \( SiO_2 \) substrate gives a diamagnetic signal. This signal can be subtracted by sweeping the field to larger magnetic field, where the FM is saturated and diamagnetic signal can be estimated.

3.4 Hall and Resistivity configuration

Electrical measurement setup consists of excitation signal probes and measurement parameter probes. Depending on the relative configuration of these two probes there can be two ways it can be done. A configuration when, say current, excitation signal is passed through a metal bar/rod device and measurement probes are connected so that they measure the electric field parallel to the current direction, the quantity \( \frac{V}{I} \) is called the longitudinal resistivity. A configuration where the measurements probes are connected to measure the
electric field perpendicular to the current path is termed as Hall configuration. The quantity $V_{Hall}$ is termed as the Hall/ transverse resistance. The reason behind observing a longitudinal resistance is a trivial voltage drop in that direction. However, the reasons for a Hall/ transverse voltage drop can be internal or external effects. Largely, these measurements were done by Edwin Hall and thus the name. In a broad sense, semiconductors under external magnetic field, we see a Hall signal due to Lorent’z force and in HM and FM we see a Hall signal due to internal spin orbit coupling (SOC) without any external magnetic field. Thus, Hall configuration is indicative of these effects in films studied in this thesis. The setup is simple but, it requires an involved theoretical understanding to interpret the results.

3.5 Sample preparation

All the samples presented in this thesis have been fabricated in a manufacturing scale intergraded circuit fabricating facility. The various tools used and their purpose is listed here.

1. **Canon Anelva**: physical vapor deposition (PVD) deposition of $\beta$-W.

2. **Singulus**: *In situ* deposition of FM and HM bilayers.

3. **193 nm Immersion Lithography**: Patterning the Hall bar and Cross bar structures.

4. **Jusung**: For methanol reactive ion etch of FM films

5. **Vistec VB300**: E-beam lithography tool, used for fabricating preliminary devices.

3.5.1 Growth of $\beta$ W, CoFeB, MgO, Ta thin films

All films presented in this thesis were deposited utilizing a PVD Ar Sputter technique described in 2.1.1. The base pressure was for all the deposition was at around E-9 Torr. The working pressure is around 2-5 mTorr. The following are the deposition parameters used for deposition of W using Canon Anelva PVD tool.
- Plasma power: 500 W
- Ar flow: 300 sccm
- Deposition rate: 5 Å/s

Extensive study of β W deposition was carried out on the Singulus deposition tool where, *in situ* deposition of FM and HM were possible. In case of Singulus the target is rectangular in shape and the substrate is linearly scanned under the target due to which it does not observe a constant flux throughout the deposition and a standard deposition rate does not apply. However, a standardized deposition rate can be estimate by back calibrating the thickness from the following equation.

\[
t = SDR \times Power \times \frac{P}{S}
\]  

(3.11)

where \( t \) is the thickness, SDR is standardized deposition rate, \( P \) represents the number of times the substrate was required to be scanned under the plasma to deposit the required thickness, and \( S \) is speed at which the substrate was moved. If the substrate is accelerated we deposit a film with thickness gradient. These thickness gradient films of CoFeB help in studying their PMA and a gradient of 135 %/mm was used for them. The parameters utilizing for deposition are listed in 3.1.

<table>
<thead>
<tr>
<th>Metal</th>
<th>Plasma Power (kW)</th>
<th>Ar flow (sccm)</th>
<th>SDR</th>
</tr>
</thead>
<tbody>
<tr>
<td>W</td>
<td>1.5</td>
<td>300</td>
<td>172</td>
</tr>
<tr>
<td>Ta</td>
<td>1.5</td>
<td>300</td>
<td>180</td>
</tr>
<tr>
<td>MgO (RF)</td>
<td>4</td>
<td>600</td>
<td>12.2</td>
</tr>
<tr>
<td>CoFeB</td>
<td>0.5</td>
<td>300</td>
<td>120</td>
</tr>
</tbody>
</table>

*Table 3.1: Deposition parameters in Singulus*

### 3.5.2 Device fabrication

Two Hall bar devices are studied and presented in this thesis. A 100 nm wide Hall and a 5 µm wide Hall bar. For all the structures the fabrication starts from bare Si wafer.
A thermally grown $SiO_2$ was deposited on Si. A buried M1 contact line was fabricated. A PVD $\beta$-W film of 5 nm and a Ta insert of 1 nm were deposited. A FM of CoFeB of 1 nm and 1.6 nm MgO and about 12 nm Ta capping layer was deposited in that order. The blanket films were then patterned into Hall bar. The wafer was then etched until the Hall bar pattern is left out. The top Ta layer acts as a hard mask protecting the interface from damage. The etch was a $CH_3OH$ reactive ion etch.
CHAPTER 4

$O_2$ assisted growth of $\beta - W$ from 5 nm to 20 nm

4.1 Introduction

Tungsten’s low resistive $\alpha$ phase is utilized as an interconnect material in integrated circuits and its high resistive $\beta$ phase has recently been shown to exhibit a giant spin Hall effect which can be utilized to switch the magnetically free layer in a TMR device [96, 5]. High Z metals have large spin orbit coupling which generates giant spin Hall effect and have been shown in Pt and the $\beta$ phases of Ta and W, which are potential materials for the non-volatile spin logic (NVSL) devices [10, 8, 7, 5, 92, 53]. The NVSL device places the giant spin Hall material as the first layer in the stack, at the bottom of the write unit [10]. In this location, the giant spin hall material must survive the etch process, which puts a lower limit on its thickness. For effective use, the giant spin Hall material’s thickness should be on the order of the spin diffusion length ($\lambda_s \approx 10$ nm) but also thick enough to be uniform [97, 98, 5].

Tungsten partially transforms from $\beta$ to $\alpha$ phase around 5 nm and the corresponding giant spin Hall angle correspondingly drops from 0.33 to 0.07 [5, 34]. It has been determined that during sputter deposition impurity concentration in the chamber, backside cooling of substrate and oxygen from sputter targets surface can inhibit the growth of $\alpha$ phase in favor of $\beta$ phase [27, 21, 34, 20, 25, 35, 26, 23]. Care must be taken when using oxygen to rule out the formation of a completely oxidized W film [22]. The challenges imposed by the etch process can be overcome by inverting the device and depositing the giant spin hall material on top of a the magnetic metal as the last layer. However, this growth condition would be different when compared to the non-inverted design, since silicon dioxide is commonly utilized as a starting substrate and W can reduce the silicon dioxide to form a thick $\beta$ phase material. Regardless the desired device geometry, knowledge of how to deposit $\beta$ W films on any substrate of any thickness is needed.
In this chapter our efforts to grow sputter deposited $\beta$-W films with thickness values from 5 nm to 20 nm on a Si substrate with and without a 5 nm thermal oxide ($\text{SiO}_2$) will be presented. A 2 sccm $\text{O}_2$ gas was introduced during the deposition process to facilitate the growth of $\beta$ W on Si and 5 nm SiO$_2$ substrates.

4.2 Experimental and Results

Tungsten films were deposited utilizing a 300 mm Anelva DC magnetron sputter system while varying the thickness, substrate surface, and $\text{O}_2$ in the chamber as discussed in 3.5.1. Two starting substrates of Si (non H-terminated) with and without a 5 nm SiO$_2$ layer grown using a standard thermal oxidation process were utilized. One 5 nm W film was deposited on a Si substrate. Three W films 5, 7.5, and 10 nm thick were deposited on a Si substrate with a 5 nm SiO$_2$ layer. Six films 5, 10 and 20 nm thick were deposited on a Si substrate with and without the 5 nm SiO$_2$ underlayer, while a 2 sccm of $\text{O}_2$ was introduced during the deposition. All sample details are summarized in Table 4.1. In addition, a fully oxidized tungsten sample was fabricated by maintaining a 20% $\text{O}_2$ partial pressure during the deposition on a 300 nm SiO$_2$ thermal oxide on a bare Si substrate.

The thickness of films was measured using a high resolution X-ray reflectivity with a Jordan Valley BEDE Metrix-L (BML) X-ray diffractometer. X-ray beam of a few millimeters (2 mm $\times$ 2 mm) from a 2.2 kW Cu sealed tube source (0.154 nm) was scanned over a range of 0 to 20000 arcseconds with a step size of 10 arcseconds at the center and edge of the wafer. The four point probe resistivity was measured with a KLA Tencor’s RS-100 instrument.
<table>
<thead>
<tr>
<th>Thickness (nm)</th>
<th>2 sccm O₂</th>
<th>5 nm SiO₂</th>
<th>Resistivity (µΩ cm)</th>
<th>Density (gm/c.c)</th>
<th>a (Å)</th>
<th>Dominant phase</th>
</tr>
</thead>
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<tr>
<td>4.9</td>
<td>Yes</td>
<td>Yes</td>
<td>188.92</td>
<td>19.98</td>
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<td>18.7</td>
<td>5.02</td>
<td>β</td>
</tr>
<tr>
<td>21.6</td>
<td>Yes</td>
<td>Yes</td>
<td>196.41</td>
<td>18.28</td>
<td>5.01</td>
<td>β</td>
</tr>
<tr>
<td>4.7</td>
<td>No</td>
<td>Yes</td>
<td>170.45</td>
<td>20.55</td>
<td>5.02</td>
<td>β</td>
</tr>
<tr>
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<td>5.03</td>
<td>β</td>
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<td>5.03</td>
<td>β</td>
</tr>
<tr>
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<td>No</td>
<td>29.07</td>
<td>17.9</td>
<td>3.17</td>
<td>α</td>
</tr>
<tr>
<td>7.1</td>
<td>No</td>
<td>Yes</td>
<td>85.19</td>
<td>21.71</td>
<td>3.06</td>
<td>α</td>
</tr>
<tr>
<td>9.7</td>
<td>No</td>
<td>Yes</td>
<td>23.57</td>
<td>19.28</td>
<td>3.16</td>
<td>α</td>
</tr>
</tbody>
</table>

Table 4.1: W films deposited under different substrates and process conditions [1]
4.2.1 X-ray diffraction analysis

The experiment was performed as described in section 3.1. The XRD patterns of all 11 W films are displayed in Fig. 4.1. Determinations of crystal structure and lattice parameter were performed using Rietveld refinement (Bruker TOPAS, version 4.2) with instrument function parameters being determined using fits to NIST Standard Reference Material 1976 ($\alpha$-$\text{Al}_2\text{O}_3$ plate). The 10 nm and 7.5 nm W films on 5 nm SiO$_2$ and 4.9 nm W on Si substrate without any oxygen being introduced form $\alpha$-W as the dominant phase with a lattice constants of 3.16 Å, 3.06 Å, and 3.17 Å, respectively. The other seven films either grown with a 2 sccm O$_2$ flow or with a 5 nm SiO$_2$ layer and all form a $\beta$-W dominant phase. The 5 nm W film grown on a 5 nm SiO$_2$/Si substrate under a 2 sccm of O$_2$ flow during the deposition forms $\beta$ phase with a lattice constant of 5.04 Å. Also a 5 nm W grown on same substrate but without 2 sccm of O$_2$ flow and 5 nm W grown on Si substrate with a 2 sccm O$_2$ flow both resulted in a $\beta$ phase W with a lattice constants of 5.02 Å and 5.03 Å. A 10 nm and 20 nm W films grown on 5 nm SiO$_2$/Si and Si substrates with a 2 sccm O$_2$ flow form a $\beta$ phase with lattice constants 5.02 Å, 5.02 Å and 5.01 Å, 5.03 Å respectively. Tungsten sample with 20 % O$_2$ partial pressure showed no significant crystalline diffraction pattern as displayed as a inset in Fig. 4.1.
Figure 4.1: X-ray diffraction pattern of 5 nm W films [1]
4.2.2 X-ray photoelectron spectroscopy

XPS was performed to study the stoichiometry of the films as described in section 3.2. Specifically the metallic and oxygen bonding states in these films. Three states; metallic-W$^0$, suboxide-W$^{+x}$ ($0<x<6$) and W$^{+6}$ are present in the films as shown in XPS plots in Fig. 4.2 and Fig. 4.3 [99]. The 4f$_{7/2}$ of W$^{+2}$ shows a peak at 32.5 eV which is very close to metallic W 4f$_{7/2}$ at 31.2 eV. An extra peak very close to W$^0$ was needed to obtain a better fit to the experimental data. However this peak was not sharp unlike other two peaks from metallic and oxide states. It is a rather broad peak and it varies from sample to sample.

A pair of scans of 5 nm W films one with a 5 nm SiO$_2$ growth substrate other on a Si substrate are shown in Fig. 4.2. Films with the SiO$_2$ substrate,(in $\beta$ phase), show a higher tungsten oxide peak than the film without SiO$_2$ layer. However, when compared with films grown on 5 nm SiO$_2$ substrate or samples with 2 sccm O$_2$ flown, they form a thicker oxide at the surface. Two scans of 10 nm $\alpha$ and $\beta$ films which show similar thicker native oxide in $\beta$ films are shown in Fig. 4.3. A W sample deposited with 20% O$_2$ partial pressure shows no metallic peak of W as shown inset in Fig 4.2.
Figure 4.2: X-ray photoelectron spectroscopy of a 5 nm $\beta$ and $\alpha$ phase W [1]
Figure 4.3: X-ray photoelectron spectroscopy of 10 nm $\beta$ and $\alpha$ phase W [1]
4.2.3 X-ray reflectivity

The data from XRR measurements is modeled as a four layered structure with Si as the substrate, SiO$_2$ as the pad oxide, W as the metal layer, and a native oxide layer on top. The surface oxide is included in the fits to improve the goodness of fit value. Periodic oscillations called Kiessig fringes are clearly visible in the reflectivity data shown in Fig. 4.4. The fringes are equally prominent in both the plots but the difference in periodicity of these oscillations for the two samples is clearly distinct with the 10 nm sample having double the periodicity than the 5 nm sample. These oscillations are due to the difference in refractive index between the layers and the thickness of the layers is calculated from the periodicity of the fringes. Thickness from XRR measurements have an error of 0.5 nm but are in good agreement with earlier reported values for their respective phases [23, 26].
Figure 4.4: X-ray reflectivity of 5 nm and 10 nm $\beta$ and $\alpha$ W [1]
CHAPTER 5
Pulsed $N_2$ gas assisted growth of $\beta - W$

5.1 Introduction

As discussed in the previous chapter 4 introducing $O_2$ during the deposition has been shown to stabilize $\beta$ tungsten enabling the growth of thicker films, but high concentrations of it forms an amorphous-like phase [16, 21, 25, 29, 28, 100, 27, 101]. In addition, it may be undesirable to introduce $O_2$ into deposition chamber which also grows ferromagnetic metals. Introducing $N_2$ gas during deposition is a natural alternative to $O_2$, however it forms tungsten-nitride crystals or an amorphous-like phase but not a $\beta$-phase [29, 101, 100]. It appears that $N_2$ drastically inhibits the grain growth (similar to high concentrations of $O_2$), which is undesirable [27]. This creates a need to form 10-20 nm thick $\beta$-W films without the assistance of $O_2$ gas flow and find the optimum conditions for $N_2$ flow during tungsten deposition.

This chapter will focus on our efforts which led to deposition of $\beta$ phase W films of thicknesses up to 20 nm using a 2 second periodic 1 sccm $N_2$ pulse. The growth kinetics are altered by changing the $N_2$ gas concentration in the chamber during the deposition which enables the deposition of up to 20 nm thick $\beta$-W. The crystal size of these films are $\sim$5 nm and reluctivities are $\sim$160 $\mu\Omega$ cm, which show promise to be introduced into a fabrication facility to grow $\beta$-W for large scale device fabrication.

5.2 Experimental and Results

Two substrates: single crystal Si(001), and 5 nm of SiN on Si(001) were chosen to deposit the W films. Four different thicknesses of W were deposited: 5, 7.5, 10, and 20 nm. Six 5 nm thick W films were grown, three on each substrate. Three films grown on the Si substrate with the three following different $N_2$ gas conditions: 1 sccm, 2 sccm, and a 2
second periodic-1 sccm pulse. Three 5 nm thick films were grown on the SiN(5 nm)/Si(001) substrate using the following three N\(_2\) gas conditions: 1 sccm, 2 sccm and no N\(_2\) during the deposition.

Six 7.5 nm thick W films were grown on both substrates under the following three different N\(_2\) gas conditions: 2 second periodic-1-sccm pulse, 1 sccm continuous flow and no N\(_2\) during the deposition.

Four 10 nm thick films were grown on the two different substrates with two different flow conditions: 2 second periodic-1-sccm pulse and without N\(_2\). Two 20 nm thick films on the two substrates were grown using a 2 second periodic-1-sccm pulse of N\(_2\) gas.

Lattice parameters and crystal sizes were calculated from X-ray diffraction patterns using the LeBail method (Bruker TOPAS, version 4.2) to characterize the phases and estimate the lattice parameter which are displayed in Table 5.1. x-ray reflectivity (XRR) and resistance measurements were done as described in section 4.2.
Thickness (nm) N₂ gas flow 5 SiN Resistivity (µΩ cm) Crystal size (nm) Lattice constant (°Å) Dominant phase
5(4.8) 1 sccm No 135.9 1.6 - Amorphous-like
5(4.5) 2 sccm No 168.9 1.1 - Amorphous-like
5(4.9) 2 sccm pulse No 135.9 2.3 5.04 β
5(4.9) 1 sccm Yes 185.8 1.8 - Amorphous-like
5(4.7) 2 sccm Yes 189.1 1.1 - Amorphous-like
5(4.7) No Yes 158.3 2.8 5.04 β
7.5(7.3) No No 24.9 7.9 3.15 α
7.5(7.1) 1 sccm Yes 172.7 2.3 4.9 β
7.5(7.7) 2 sccm pulse Yes 176.5 4.7 5.00 β
7.5(7.2) No No 24.3 8.3 3.15 α
7.5(7.1) 1 sccm No 166.55 2.2 5.04 β
7.5(7.7) 2 sccm pulse No 153.6 3.9 5.03 β
10(9.9) No Yes 23.7 10.2 3.14 α
10(10.5) 2 sccm pulse Yes 174.6 5 5.30 β
10(9.7) No No 20.9 10.4 3.14 α
10(10.4) 2 sccm pulse No 159.7 4.9 5.00 β
20(21) 2 sccm pulse Yes 163.3 7.8 5.00 β
20(21) 2 sccm pulse No 149.49 7.0 5.01 β

Table 5.1: W films deposited under different substrates and process conditions [1]

5.2.1 X-ray diffraction analysis

All diffraction experiments were performed as described in section 3.1. The X-ray diffraction patterns of 5 nm, 7.5 nm and 10 nm, 20 nm W films are displayed in Fig.5.1, Fig.5.2, Fig. 5.3, respectively. The four X-ray diffraction patterns of the 5 nm W films with 1 sccm and 2 sccm N₂ gas when grown on both, Si(001) and SiN(5 nm)/Si(001), substrates show amorphous-like diffraction patterns as displayed in Fig. 5.1. A strong crystalline diffraction is observed in the two 5 nm films grown on SiN(5 nm)/Si(001) without any N₂ gas and the 5 nm film deposited on the Si substrate with pulse N₂ gas. These have a lattice parameter of 5.04 Å when fit using LeBail method.

Two 7.5 nm W films, deposited on Si(001) and SiN(5 nm)/Si(001) substrates form an α dominated phase with lattice constant of 3.15 Å and crystallite size around 8 nm. The two 7.5 nm W films with continuous flow of 1 sccm N₂ gas on SiN(5 nm)/Si(001) and Si(001) substrates show a smaller crystallite size of 2.3 nm and 4.7 nm and a β dominant phase with lattice constants of 4.9 Å and 5.04 Å respectively. The smallest crystal size is seen in the two films grown with a pulsed N₂ gas condition on both substrates. Lattice constants and
crystal sizes are 5.00 Å, 4.7 nm and 5.03 Å, 3.9 nm for the SiN(5 nm)/Si(001) and Si(001) substrates respectively.

The X-ray diffraction patterns for 10 nm and 20 nm films show a smaller crystal size, as seen in Fig. 5.3, also the $\beta$-W(200), $\beta$-W(211) peaks appear in films with 2s-1-sccm N$_2$ pulse samples. These films show a reduction in crystallite size in comparison to their $\alpha$-W counter parts (similar thickness, Table 5.1). Lattice parameter and crystallite size are 5.00 Å, 5.01 Å and 7.0 nm, 7.8 nm for 20 nm $\beta$-dominated W films. The 10 nm $\alpha$ dominated W films lattice constants and crystallite size are 3.14 Å, and 10.4 nm, 10 nm. The 10 nm $\beta$ dominated films have a 5.00 Å and 5 nm lattice parameter and crystallite size, respectively.
Figure 5.1: X-ray diffraction patterns of the 5 nm W films deposited on the substrates as indicated [1].
Figure 5.2: X-ray diffraction patterns of the 7.5 nm W films deposited on the substrates as indicated [1].
Figure 5.3: X-ray diffraction patterns of the 10 nm and 20 nm W films deposited on the substrates as indicated [1].
5.2.2 X-ray photoelectron spectroscopy

XPS was performed to study the stoichiometry of the films as described in section 3.2. The XPS results for all the films show a WO$_3$ peak in addition to a metallic peak of W as displayed in the Fig. 5.4 and Fig. 5.5. The 5 nm W films with different N$_2$ gas flow conditions on two different substrates are displayed in Fig. 5.4. The 5 nm W films grown on a SiN(5 nm)/Si(001) substrate with a 2 sccm of N$_2$ gas during the deposition cycle are displayed in Fig. 5.4(a). This shows significant W$^{+0}$ peak at 30.7 eV and W$^{+6}$ peak around 35.06 eV [99, 22]. A broad suboxide peak W$^{+x}(0<x<6)$ was necessary to obtain a better fit at 34.1 eV. The 5 nm W film grown during a N$_2$ pulse was used during the deposition, as displayed in Fig. 5.4(b). This resulted in a increase in W$^{+0}$ peak at 30.6 eV and W$^{+6}$ peak around 35.3 eV with a suboxide of W$^{+x}(0<x<6)$ at 31.7 eV. A similar spectrum was observed for the rest of the 5 nm films with both metallic and oxide character of W in all the films.

The W film grown on a SiN(5 nm)/Si(001) substrate with W$^{+0}$ peak at 30.9 eV and W$^{+6}$ peak around 34.9 eV as displayed Fig. 5.5(a). No suboxide peak was necessary to obtain a better fit for this film. The 10 nm W film deposited during a N$_2$ pulse was used during the growth on a SiN(5 nm)/Si(001) substrate as displayed in Fig. 5.5(b). This resulted in W$^{+0}$ peak at 30.5 eV and W$^{+6}$ peak around 35.2 eV with a suboxide of W$^{+x}(0<x<6)$ at 31.9 eV was added to improve the fit. Similar spectrums were observed for the 7.5 nm and 20 nm films.
Figure 5.4: (a) X-ray photoelectron spectroscopy result of 5 nm amorphous-like W film. (b) X-ray photoelectron spectroscopy result of 5 nm βW film [1].
Figure 5.5: (a) X-ray photoelectron spectroscopy result of 10 nm $\alpha$ W film. (b) X-ray photoelectron spectroscopy result of 10 nm $\beta$W film [1].
CHAPTER 6
Custom Built Rotatable Holder for Magneto-transport measurements

6.1 Introduction

At this point the growth of $\beta$W upto 20 nm thickness has been well understood and explored. In order to further explore the spin Hall effect (SHE) in the $\beta$-W films magneto-transport measurements are necessary. As discussed in chapter 2.4.3, value of magnetoresistance is strongly dependent upon the angle between the external magnetic field and built-in effective fields in a ferromagnetic metal (FM) device structure. These built-in effective fields are either perpendicular or parallel to the device plane. The onsite Quantum design VersaLab is a versatile instrument that comes with magnetic field perpendicular to device plane. There are existing in-plane holders which provide a parallel configuration for the external magnetic field and device plane. However the devices need to be rewired to switch between parallel and perpendicular holders, which is challenging and time consuming [102]. This makes it difficult to measure the exact same device in both the directions. Commercially available rotatable holders exist but are expensive ($\approx$ $20,000).

This chapter will present our engineering efforts to build an economic holder out of aluminum which can be rotated without the need for rewiring. The estimated total cost of the entire setup is less than $1000. The designed holder is tested by performing anomalous Hall measurements in both orientations on multilayered stack of Si|SiO$_2$|Ta (5 nm)|CoFeB (1 nm)|MgO (1.6 nm)|Ta (Cap) which is patterned into a Hall bar. In addition, measurements to compare noise levels and estimate the offset in temperature of the rotatable holder to the standard holder are also presented.
6.2 Design and fabrication

The custom rotatable-holder (CRH) is built on the universal puck which comes with the Quantum design Versalab instrument. The CRH has two extreme modes of operation as shown in Fig. 6.1(a) & Fig. 6.1(c). An expanded view of the CRH assembly is shown in Fig. 6.1(d). It consists of a base, a stage, and a chip carrier assembly. The stage and base are made of aluminium which is chosen for it’s machinability over copper even though the thermal conductivity of aluminum is about half that of copper. The base has a cross shaped support with two straight rectangular poles on diagonally opposite fingers of the cross. The legs of the poles are curved instead of a sharp “L-shape” to give more rigidity to the poles, as these two poles will hold the stage. The fingers of the cross without poles have screw holes which fasten the base to universal puck. The uniform region of the magnetic field is centered at 40 mm above the top surface of a standard sample puck within ±0.1 % over a 25 mm axial length centered at that location [103]. Two screw holes are made on the pole which hold the stage at a height of 30 mm above universal puck and within region of uniform magnetic field. The stage is a cuboid of length (15.5 mm) equal to the separation between the two poles. The stage has two small forks sticking out on the same side of the cuboid (large). The two forks are used to support the chip carrier assembly. The forks have cuboid (small) shape with equal breadth and height. The heights of the large-cuboid and small-cuboid are different giving a step-edge shape from the profile view as seen in the Fig. 6.1(a). There are two pairs of screw holes on the stage. One pair is on the side-faces, one screw-hole on each face, which align to the screw-hole on the pole. The other pair of screw holes are on the top-face of the stage. These top-screw holes fasten the chip carrier assembly to stage.

The chip-carrier assembly has a male and female housing. The housing is a customized printed circuit board (PCB) obtained from OSH Park. The male housing is a square shaped PCB with six collinear gold-plated bond pads onto which the device can be wire bonded. Above each pad there is a pin-hole into which male-pins are soldered, providing an electrical contact between pad and pins. The female housing is a rectangular shaped board with the
same length as the male housing. The female housing consists of six through-holes which line-up with the centers of the pin-holes in the male housing. The width of the female-housing is so chosen to be much larger than diameter of these through-hole. The female-pins are push-fit type with a solder-cup termination. A non-magnetic cryogenic compatible phosphorous-bronze wire is soldered to the solder-cup at the end of each female-pin. The other end of this wire is soldered to a unique pad on the universal puck. By application of appropriate amount of pressure the female pins can be installed. Once installed into the female housing there is a lip-height of about 1 mm which doesn’t let the male and female housing touch each other. The step-height described earlier is equal to sum of the lip-height of the female-pin and the thickness of the PCB as seen in Fig. 6.1(a).

The pair of screws on the top face of the stage fasten the female-housing between the forks and male-housing, providing the packaging stability to the electrical-housings. Once the base, stage and chip-carrier are assembled on top of the universal-puck, device under test (DUT) is mounted onto the male housing using either N-grease for low-temperature or copper double-tape for room temperature measurements. A grounding strap should be worn by the user throughout all the experiments especially when handling the devices and rotating the holder to reduce static discharge from damaging the devices. The universal puck’s surface and metallic region of CRH are grounded. The male housing acts as an insulator between the sample leads and ground. Once the DUT is loaded the two screws on the rectangular poles fasten the stage to the base. These screws perform a dual function of fastening and also give a fulcrum for rotating the stage. Once the stage is rotated to desired angle the screws can be tightened to seize rotational motion as seen in the Fig. 6.1(a).
To demonstrate the working of the holder the sample plane is rotated to be perpendicular (90°) to the magnetic field as in Fig. 6.1(a), which is swept from positive to negative and back, while the Hall resistance is measured. This results in a strong perpendicular magnetic anisotropy (PMA) as displayed in Fig. 6.2(a) indicating this is the easy axis direction of the FM. The Hall resistance in a FM with PMA is proportional to the magnetic moment’s Z-component (perpendicular to device plane) [60]. As the magnetic field is swept perpendicular to device plane while measuring Hall resistance a discontinuous change is observed around 50 Oe as seen in Fig. 6.2(a). This discontinuous change in resistance signifies the reversal of magnetic moment in the Z direction. A symmetric reversal is observed around
−50 Oe. The difference between these two fields is the coercive field of the device. Films with PMA have their easy axis out of the device plane. The external magnetic field needed to rotate the magnetic moment from the out of the plane direction to the in-plane direction is signified as the effective anisotropy field. The effective anisotropy field and coercive field are intrinsic properties of a FM.

The stage is then rotated so the sample plane is parallel (0°) to the magnetic field as in Fig. 6.1(c). The same magnetic field sweep is performed and the Hall resistance is plotted in Fig. 6.2(b) indicating that this is the hard axis direction of the FM. When the magnetic field is around -10 KOe the magnetic moment of the FM is parallel to external field. When the magnetic field is zero the anisotropy field will point the magnetic moment in the $-z$ direction. As the field is further increased to around 1000 Oe the net torque due all the fields is sufficient to flip the magnetic moment in the $z$-direction, but slightly canted. As the external field is further increased, magnetic moment eventually points in the same direction as the external field. The magnetic moment at these large fields can be slightly canted if there is some experimental misalignment. The direction of the magnetic moment vector is indicated with the black arrows in Fig. 6.2.
Figure 6.2: Normalized Hall resistance plotted vs external magnetic field in two orthogonal directions with $I_{DC} = 0.25$ mA. The black arrows signify the magnetic moment vector in the ZY-plane. The blue (red) data points represent Hall resistance while magnetic field is swept in increasing (decreasing) direction. (a) The magnetic field is swept perpendicular to the device plane. (b) The magnetic field is swept in parallel to the device plane. Both the plots show strong perpendicular magnetic anisotropy.

As the DUT on the CRH is further away from the thermometer located under the Quantum Design sample puck (QDSP) the temperature at the DUT can vary from that of the puck. In addition, aluminum’s lower thermal conductivity than that of copper and
its distance from the thermometer may result in a temperature lag when heating or cooling the device. To estimate the offset in temperature, the longitudinal resistance of the Hall bar device is measured while sweeping the temperature on the QDSP and the CRH. N-grease was used to mount samples for low temperature measurements to ensure good thermal contact. The temperature coefficient of resistance \( \alpha \) is related to resistance via \( \Delta R_T/R_{T_o} = \alpha (T - T_o) \). A 2 K per minute ramp rate results in a 0.7 K temperature error in the QDSP [103]. Hence, a lower ramp rate of 0.5 K per minute and a wait time of 60 seconds at each temperature was incorporated in the measurements on the CRH to match the measurements performed on the QDSP, which is displayed in Fig. 6.3. The normalized relative change in resistance is plotted vs the relative change in temperature in Fig. 6.3. The resistance at 300 K is chosen as \( R_{T_o} \) and a linear fit to the data gives \( \alpha \) for both the CRH and QDSP which have a ratio \( f = \alpha_{CRH}/\alpha_{QDSP} \) equal to 0.988. The offset in temperature for the CRH is estimated as \( T_{Real} = f \times T_{Apparent} + (1 - f) \times 300 \), which gives an estimated offset \( \approx 3 \) K at 50 K. A longer wait time and slower ramp rate might yield smaller offset at lower temperature regions.

![Graph showing normalized relative change in resistance vs temperature](image)

**Figure 6.3:** The green (black) data points represent normalized relative change in resistance of the Hall bar device on a Quantum Design sample puck (Custom rotatable holder) while the temperature is swept. The slope from a linear fit to the data gives the temperature coefficient of resistance \( \alpha \) as indicated.
To investigate the noise levels in the CRH, Hall resistance vs out of plane magnetic field sweeps of similar devices on QDSP and CRH are performed and displayed in Fig. 6.4(a) and Fig. 6.4(b), respectively. The data from both the holders have very similar signal-to-noise where the total Hall voltage generated is influenced by the inherent noise from the setup at room temperature. The anomalous Hall resistance or voltage in a FM film is directly proportional to the longitudinal current in the Hall bar [60]. By increasing the longitudinal current in the Hall bar a larger signal-to-noise can be achieved. This is demonstrated using a larger DC current of 2.5 mA on a similar device mounted on the CRH with a much improved signal to noise as displayed in Fig. 6.5(a). However, using large currents can be detrimental to the device due to Joule heating. This can be overcome with an AC-Hall resistance measurement, which is performed on similar Hall bar device mounted on the CRH using a lock-in measurement technique. An AC signal amplitude of 0.1 V at 20 Hz was used to measure the Hall resistance which is plotted in Fig. 6.5(b) and displays larger signal-to-noise than the DC measurements.
Figure 6.4: Normalized Hall resistance of similar devices plotted vs external magnetic field with $I_{DC} = 0.5$ mA on different holders (a) Quantum design sample puck (b) Custom rotatable holder.
These various measurements demonstrate the performance of the custom built rotatable holder (CRH). It performed equivalently to the Quantum Design sample puck (QDSP) in all measurements except it requires a slower temperature ramp rate (0.5 K/min.) when heating or cooling the sample. This is attributed to the distance from the cold source and the choice of aluminum over copper. However, the measurements required to study SHE are at steady
state temperature either at 298 K or lower. The ease of sample rotation greatly benefits the measurements since it is extremely necessary to measure the same device at multiple angles.
CHAPTER 7
Magnetic anisotropy and Switching of
$\text{SUB}|\beta$-W$|\text{Ta}|\text{CoFeB}|\text{MgO}|\text{Ta(Cap)}$

7.1 Introduction

The growth of $\beta$-W and developing a custom holder to perform spin Hall effect (SHE) studies on devices has been the initial focus of this thesis. To further our understanding of SHE on these materials electrical transport measurements will be presented. In-plane current induced switching of ferromagnetic metal (FM) and heavy metal (HM) bilayers have been one of the pivotal discoveries in recent years. The advent of in-plane current induced switching of FM facilitates a 3-terminal magnetic tunnel junction (MTJ) which makes feasible independent optimization of magnetoresistance, switching current and read out voltage, otherwise not possible in a traditional 2-terminal design [3]. Longitudinal current in a HM like Ta, Pt and W creates a transverse spin current which has a spin angular momentum associated with it. The component of spin angular momentum perpendicular to the magnetic moment exerts a torque on it [4, 5, 7, 12, 61]. spin orbit coupling (SOC) is proportional to the fourth order of atomic number, thus metals like Pt, Ta and W become good candidates to explore this effect [11]. Crystal structures with bulk inversion asymmetry (BIA) and inherent structural inversion asymmetry (SIA) in multilayers like SUB$|\text{HM}|\text{FM}|\text{oxide}$ are fundamental requirements of the effect [13]. Until now $\beta$-W ahs the highest spin Hall angle (SHA) in crystalline metals of $0.30 \pm 0.02$. However, the tungsten and CoFeB interface does not typically yield a magnetic moment perpendicular to the film without magnetic anneal or insert layer [6, 49]. FM films with perpendicular magnetic anisotropy (PMA) are necessary for large scale fabrication due to their inherent scalability and thermal stability [18, 48]. In this chapter, efforts in finding the right insert layer to induce PMA in CoFeB on a $\beta$-W underlayer and also, studies of the effect of in-plane current’s direction on in-plane magnetic
field sweeps and estimate SHA will be presented.

7.2 Experimental and Results

All the films and devices presented in this chapter are deposited and fabricated utilizing the techniques and methods described in subsections 3.5.1 and 3.5.2. A blanket wafer with varying film thickness of CoFeB (0.8 nm to 1.2 nm) was deposited. The wafer was later cleaved at appropriate lengths, with unique thickness, and vibrating sample magnetometry (VSM) was measured to check for PMA. A total of three insert layers were studied, Ta, Mo and CoFe. Ta and Mo were chosen for their body centered cubic (BCC) crystal structure, Mo also exhibits higher thermal stability [47]. A CoFe dusting layer was also chosen to act as a boron sink thus MgO and CoFeB could crystallize in a (100). VSM measurements were performed prior to device fabrication with the three different insert layers the results are summarized in Fig. 7.1. An offset is added in the hysteresis loops in Fig. 7.1 to avoid overlap of the individual loops. Each sample was subjected to radiative annealing in ultra high vacuum (UHV) environment at 320°C. The estimated temperature loss due to radiation from substrate to sample is about 5 %. The films with Ta insert only revealed a PMA in CoFeB with a $M_s = 1180 \text{ emu/cm}^3$ and effective anisotropy field of 8000 Oe. As annealing temperature was increased for Ta samples to 365 °C and 410 °C the loss of PMA was observed. Films with Mo insert do not show any PMA throughout all of the annealing temperatures. Similarly films with CoFe dust layer did not result in a PMA CoFeB either.
Figure 7.1: VSM hysteresis loops of blanket films of Ta, Mo and CoFe insert layers under different annealing conditions. The CoFeB sample thickness is 1 nm in these samples.

Hall bar devices were fabricated with the stack SUB|β-W|Ta|CoFeB|MgO|Ta(Cap) with 1 nm Ta insert as it revealed PMA with 1 nm CoFeB. β phase of W was confirmed using x-ray diffraction (XRD) as shown in Fig. 7.2c. The stack was then patterned and etched to a 100 nm wide Hall bar, a top down scanning electron microscopy (SEM) is shown in Fig. 7.2(d). A magnetic field was applied out of the plane and swept as the Hall resistance is plotted in Fig. 7.2(a). An edge detection algorithm was used to estimate the discontinuous change in resistance. It is depicted in Fig. 7.2(b) using vertical red spikes and the coercive field of the device is estimated to be 163.8 Oe.
Figure 7.2: (a) Magnetic field is swept while Hall resistance is measured. The red (blue) circles indicate forward (reverse) sweep of magnetic field. (b) The result of edge detection algorithm is shown using a red line. The vertical spikes indicate the discontinuous changes in the data. (c) XRD pattern revealing $\beta$ phase W. (d) A top down SEM of patterned Hall bar device is shown. (e) A schematic of the stack studied.

The device was then rotated so that the magnetic field makes very small angle with the plane of the device. The effective fields and external magnetic field the FM experiences is vectorially shown in Fig. 7.3(a). As the sample is tilted so that magnetic field is close to the plane of device the magnetization vector tilts accordingly as in Fig. 7.3(b). Any misalignment in the magnetic field line and longitudinal current direction, which is possible given the small size of Hall bar, is depicted in Fig. 7.3(c). A top down view of possible misalignment is shown in Fig. 7.3(d). The magnetic field is swept in four different configurations. Positive magnetic field vector slightly away from the film-plane (180°) with positive and negative current direction shown in Fig. 7.3(g) and Fig. 7.3(h). Positive magnetic field vector slightly into the film-plane (178°) with positive and negative current direction shown in Fig. 7.3(e) and Fig. 7.3(f).
The in-plane magnetic field sweeps were also analyzed using edge detection algorithm for the discontinuous jumps. These point are termed as switching field (sw) and they are dependent upon current vector, external magnetic field vector and coercive field. The switching field for in-plane magnetic field at 182° is shown in Fig. 7.4. (a)-(d). To estimate the spin Hall angle the current flowing through the FM layer and through the HM layer needs to be estimated. A series of devices are measured which have varying thickness of CoFeB and W with Ta insert. The $\rho_{\text{CoFeB}} = 105.7 \ \mu \Omega \cdot cm$ and for $\rho_{\text{W-Ta}} = 224.0 \ \mu \Omega \cdot cm$ are estimated. To estimate SHA the following equation is derived by balancing the total fields in the Z-direction with the coercive field. The estimated effective field Z direction for various
100 µA and 50 µA at different angles is summarize in Table 7.1 and 7.2.

\[
\theta_{SHE} = -\left( \frac{\Delta H_{in-sw}}{\sum H_{in-sw}} \right) \frac{H_c 2|e|M_s t_F}{\hbar |\vec{j}| \sin \phi_m} \frac{1}{\sqrt{1 - (\frac{\Delta R}{\Delta R^2})^2}}
\]  

(7.1)

where \( \theta_{SHE} \) is SHA, \( \Delta H_{in-sw} \) is the difference in the in-plane switching fields for a particular current value, \( \sum H_{in-sw} \) is the sum of in-plane switching fields for a particle current value, \( H_c \) is the coercive field, \( M_s \) is the saturation magnetization, \( t_f \) is the film thickness of FM, \( e \) is the charge of electron, \( \vec{j} \) is the current density vector, \( \phi_m \) is the tilt in magnetization due to experimental misalignment, \( \Delta R^\parallel, \Delta R^\perp \) are the difference and sum of Hall resistance values when magnetization was out of the plane and into the plane, when external magnetic field is swept in-plane (\( \parallel \)) and out of plane (\( \perp \)). Using this formula and the measured data the SHA to be estimated = 0.24 ± 0.05.
Figure 7.4: (a)-(d) In-plane magnetic field swept while measuring Hall resistance for 100 µA. Edge detection algorithm used to estimate the discontinuous change in Hall resistance (e),(f) Longitudinal resistance versus the thickness of film for to estimate the resistivity of CoFeB and W-Ta films.

The in-plane current switching of FM is displayed in Fig. 7.5. As the in plane longitudinal current is swept and the Hall resistance is collected. A constant magnetic field is applied parallel to the current direction to break the inversion symmetry. A discontinuous jump in Hall resistance is observed around 1 mA and 1.25 mA. As the external magnetic field is reversed the switching polarity has reversed which is indicative of SHE switching. These measurements were performed on a 5 µm Hall bar device.
Figure 7.5: Current induced SHE switching under external magnetic field along longitudinal current direction. (a) Magnetic field 70 Oe (b) Magnetic field -70 Oe

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Table 7.1: Estimated effective field values for measurements with 100 µA current
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**Table 7.2:** Estimated effective field values for measurements with 100 $\mu$A current
CHAPTER 8
Discussion

8.1 O₂ assisted growth of β – W from 5 nm to 20 nm

The resistivity measurements are in agreement with earlier reported values for β and α phase W [34]. Physical vapor deposited tungsten forms a polycrystalline film either in low resistive α phase or high resistive β phase. The α phase is a body centered cubic (BCC) lattice of W atoms whereas β phase is A₃B type of structure with space group Pm̅3n where B occupies the body center and corner lattice sites, and A occupies two lattice sites in the face [19]. Peak positions from x-ray diffraction (XRD) patterns were utilized to indicate the β phase [23, 26]. No change in lattice parameter or resistivity was observed after six months [25].

A 5 nm thin W film grown on a 5 nm SiO₂/Si substrate was found to be in a β phase, whereas, films grown thicker than 5 nm, on a similar substrate, were pre-dominantly α. However, when 2 sccm of O₂ is introduced these thicker films form a β phase. Films as thin as 5 nm grown on a Si substrate form an α phase but when a 2 sccm O₂ is introduced into the chamber they form a β phase. Films thicker than 5 nm under similar substrate and deposition conditions form a β phase. When much higher amount of O₂ is present in the chamber (about 20% partial pressure) W was completely oxidized and no significant crystalline diffraction pattern is observed.

Introducing 2 sccm of O₂ did not completely oxidize W, rather oxygen assisted in stabilizing the β phase. Thicker oxide on β phase films can be attributed to larger oxygen gettering capacity over the α phase. In previous reports, peaks adjacent to metallic W peaks were attributed to WO₂ but a more realistic stoichiometry would be WₓOᵧ, where x and y depend on local oxygen concentration [22]. The farthest triplet peak belongs to W⁺⁶ from WO₃ formed either due to atmospheric oxidation after the deposition or the oxygen available
during the deposition. Collecting the angle resolved photoelectron signal shows surface (take off angle 81.1° with surface normal) is dominated with WO$_3$ and few angstroms deeper signal (lower take-off angles, at 24.8°) from W$_x$O$_y$ and W increase. This suggests a layered stack of WO$_3$/W$_x$O$_y$/W, as described by Aouadi, M.S. et al. [22]. All the β phase W films reveal a higher intensity peak from the W-O bonds when compared to the metallic bonds but clearly do not show a completely oxidized film. This technique can be easily extended to any deposition system to grow thicker β W films to survive the etch of the top ferromagnetic metal (FM) layer.

8.2 Pulsed N$_2$ gas assisted growth of β − W

The lattice constants of the β W films are in agreement with previous β W phase films grown with and without O$_2$ [22, 19, 23, 26, 104]. However, the films are less resistive than previous β-W films which are typically around ∼200 µΩ cm, which maybe beneficial for producing devices with lower resistances than films grown with O$_2$ [21, 16]. The α phase-W films have two peaks around W(110) these peaks were not commented for in earlier studies and fit well with a W$_5$Si$_3$. In addition, the β W(210) and α W(110) peaks are very close to each other which cannot be resolved using the Lebail method which does not take the site occupancy into account. However, the average grain size decreases whenever the β W(200) and (211) peaks appear around the β W(210)/α W(110). This characteristic smaller grain size is another indication of the formation of a β phase.

Depositing a 5 nm W film on SiN without any N$_2$ gas forms a β phase film. However depositing 5 nm on bare Si forms an α phase. This is similar to our previous findings where 5 nm of W deposited on SiO$_2$ forms a β phase, which was attributed to the uptake of O$_2$ causing the formation of β-W [16]. However the SiN substrate does not provide the oxygen and the roughness of the substrate and the thickness (5 nm) of W film constrain the average grain size to be around 2.8 nm, thus restricting the film to be in β phase. The Si substrate does not offer such a surface roughness, or source of oxygen, and the film readily forms an
A continuous $N_2$ gas flow renders the 5 nm thick W amorphous-like on both substrates similar to previous findings [29, 105, 100]. However, when a $N_2$ pulse is introduced, the concentration is sufficient to kinetically constrain the grain growth to form $\beta$ W. The XPS results of these films shows metallic character present in all the W films. The native oxide peak of $\beta$-W is higher than the $\alpha$-W of similar thickness. This can be interpreted as higher oxygen gettering capacity of $\beta$-W. The metallic peak of $\beta$-W is higher than amorphous-like W film of similar thickness and is attributed to the increased crystallinity in the $\beta$-W than the amorphous films.

As the thickness is increased to 7.5 nm and 10 nm there are more W atoms to nucleate and grain size increases to form thermodynamically stable $\alpha$ phase on Si and SiN substrates when no $N_2$ is introduced. When a continuous 1 sccm of $N_2$ gas is flown during deposition for the 7.5 nm W film on both substrates the film forms a $\beta$ phase W but with a reduced grain size. However, a $N_2$ pulse during the 7.5 nm growth yielded much sharper $\beta$-phase diffraction pattern with a larger grain size similar to the 5 nm thick film. For 10 nm and 20 nm thick W films on both Si(001) and SiN substrate only pulsing the $N_2$ resulted in $\beta$-W with larger average grain size and sharp diffraction patterns, indicating a high quality $\beta$-W film.

These findings demonstrate the significance of pulsing the $N_2$ to achieve $\beta$. When inert impurities such as $N_2$ are introduced into the deposition chamber, they interfere with the nucleation and grain growth of the deposited film. These impurities can either act as a roadblock to grain growth or act as a nucleation site resulting in smaller grain sizes [69, 106]. However if large concentrations of $N_2$ gas are introduced, about 1 or 2 sccm, it blocks W-metal from forming a crystalline phase and an amorphous-like film is formed. Upon lowering the concentration by pulsing, a kinetically stabalized phase which has much larger grain size is grown. The 7.5 nm film still crystallizes to form a $\beta$ phase in presence of 1 sccm continuous flow due to the availability of more W atoms but it has smaller grain size. The 5 nm thick
W lies on the boundary of kinetic and thermodynamic stability offered by the SiN substrate or continuous N$_2$ gas flow. For films 10 nm and above pulsing the N$_2$ is required to form β-W films with larger grains. Thus a thicker β W can be grown without the need of any O$_2$ during the deposition.

8.3 Magnetic anisotropy and Switching of

SUB|β-W|Ta|CoFeB|MgO|Ta(Cap)

8.3.1 Magnetic anisotropy due to insert layer

Although the CoFeB and MgO interface can induce strong interface anisotropy leading to perpendicular magnetic anisotropy (PMA), it is extremely necessary for the other interface of CoFeB to not induce a significant amount of roughness ruining the effect. Thus, no PMA was observed in SUB|βW(5 nm)|CoFeB(1 nm)|MgO(1 nm) films. The interface roughness of βW and CoFeB have large lattice mismatch and tungsten’s lower affinity to boron can lead to interference with the MgO and CoFeB interface. To induce PMA an insert layer of about 1 nm was deposited between CoFeB(1 nm)|βW(5 nm). The choice of insert layers was constrained by the available targets in a 300 mm tool (Ta, Mo, and CoFe).

Molybdenum was known to crystallize in BCC and was previously reported to show good thermal stability up to 400°C. However a 1 nm insert layer Mo does not behave similar to a 5 nm underlayer Mo, as no PMA was observed even after annealing the films. The CoFe dusting layer of 0.5 nm was also inserted to behave as a boron sink. However, the total FM thickness was contributing for the bulk anisotropy and dominating the interface anisotropy of MgO and CoFeB.

Ta insert resulted in a PMA stack. The sample was annealed under ultra high vacuum (UHV) environments at 320°C for one hour which improved the PMA and increased the effective anisotropy field to 8000 Oe from 4000 Oe in as deposited state. This is due the crystallization of MgO which allows the CoFeB to crystallize and reorient its moments out of the plane. As the annealing temperature is increased to 365°C and beyond the PMA
degraded due to boron diffusion and dead layer formation. This study sets the processing parameters for the inline fabrication of the Hall bar device.

### 8.4 Switching of SUB|β-W|Ta|CoFeB|MgO|Ta(Cap)

In light of previous results, a Hall bar structure with β-W and Ta insert layer was fabricated and patterned. The 100 nm Hall bar device were annealed for 10 min at 350°C. As a first measurement to test PMA an external magnetic field was swept perpendicular to the device plane while collecting the Hall resistance. A square hysteresis loop is seen which confirms the PMA in these films. Now the device is rotated to apply an external magnetic field parallel to current while the Hall resistance was measured. As the current direction was reversed the spin torque direction also reverses and results in a lower external field required for flipping. Thus, revealing a competition between external magnetic field and internal effective field. A discontinuous switching in the Z-direction is observed when the net field in the Z-direction is equal or greater than the coercive field 163.8 Oe. The internal effective field in the Z-direction is estimated and an analytical model is used to derive a relation to spin Hall angle (SHA). To estimate the longitudinal current distributed in the FM and heavy metal (HM) layers devices with different thickness were studied. A plot of thickness versus inverse of longitudinal resistivity is made. The slope of this plot is the resistivity of the metal whose thickness is varied.

The estimated SHA is about 0.24 ± 0.05, which is close to SHA observed by Hf insert layer study by Pai et al. but lower than one reported for βW underlayer and higher than Ta [7]. The lowering of SHA could arise from the Ta insert layer. Although, Ta induces PMA due to it’s strong spin orbit coupling (SOC) it can behave as a spin block and reduce the spin current passing through it. Since W and Ta both show spin Hall effect (SHE) we report the SHA for the entire stack of materials.
CHAPTER 9
Conclusions

The results presented in the thesis demonstrate the following unique scientific and engineering contributions.

- Kinetically controlling the growth of $\beta$ tungsten using $O_2$ or $N_2$.

- Developing perpendicular magnetic anisotropy (PMA) in CoFeB on $\beta$ W utilizing Ta insert layer.

- Demonstrate current induced spin Hall effect (SHE) switching of a single CoFeB film and estimated spin Hall angle (SHA) about $0.24 \pm 0.05$.

These results discussed in section 8.1 demonstrate that an oxygen controlled environment can be used to grow $\beta$ W films on silicon with and without a 5 nm thermal oxide for thicknesses up to 20 nm. The O atoms can come from reduction of the thermal oxide or $O_2$ flown in the chamber. $\beta$ W films thicker than 5 nm require 2 sccm supply of $O_2$ during deposition for both substrates. These results demonstrate how to deposit $\beta$ W on Si or SiO$_2$ which allows for growth at the bottom of device stack and maybe applicable to other $\beta$ phase materials such as Ta. However, flowing of $O_2$ can be detrimental to other materials in the deposition chamber or metals lines on the wafer. An inert gas like $N_2$ can be used to deposit thick $\beta$ phase films by kinetically controlling the growth by introducing a 2 second periodic pulse of 1 sccm $N_2$ gas from 5 to 20 nm thick films is demonstrated, as discussed in section 8.2. The inert nature of $N_2$ gas will not effect other metal targets in the deposition chamber and offers a much more versatile alternative than using $O_2$. These less resistive $\beta$ phase films should produce devices with lower resistances which maybe beneficial for improved efficiency and power consumption.
PMA in ferromagnetic metal (FM) films is very essential for their scalability and thermal stability. This was achieved utilizing a Ta insert between CoFeB and W layers. The boron affinity of Ta and its interface do not interfere with the CoFeB and MgO interface. Thus improving PMA upon annealing the films. The surface of a bulk film, 5 nm, largely differs with that of a, 1 nm, insert layer film in case of Mo. CoFe dust layer does not essentially act as a boron sink but increases the net contribution to bulk anisotropy, as presented in the subsection 8.3.1. The stack with Ta insert showed PMA both in blanket and patterned device. The difference in the inplane switching fields clearly exhibit the competition between spin orbit torque (SOT) and external field’s torque. A positive current with a positive field prefers a positive magnetic moment (value of Hall resistance). This symmetry of switching is observed in current sweeps and preserved in field sweeps with $\theta > 2\pi$. Sweeps with $\theta < 2\pi$ exhibit an opposite symmetry. This reversal in symmetry can be due to the field-alone induced switching in in-plane field sweeps. Further analysis would be needed to comment on the symmetry of the field sweeps.

9.1 Future outlook

With the major advantage of the 3-terminal device structure and still unknown formalism of SHE, makes this an interesting area both for academic and semiconductor industry research. The fundamental formalism for SHE is yet to be constructed for systems with spin orbit coupling (SOC), in metals with bulk inversion asymmetry (BIA). There exists well formulated theories for 2-dimensional electron gas (2DEG) and semiconductors with BIA. A significant spin-off from the metal BIA spintronics is the antiferromagnetic metal (AFMM)-spintronics. However, these AFMM are exotic materials which are difficult for large scale fabrication. A film with BIA, high Z, low resistivity and low spin mixing and high spin diffusion length will be the ideal for such applications. Films with high Z like Pt can change their properties with alloying. Pt forms an alloy with W, with proper annealing conditions it can be crystallized in a structure with BIA.
LITERATURE CITED


APPENDIX A

Derivation for Estimating Spin Hall Angle

Equation relating $\vec{M}, \vec{H}_{ext}, \vec{J}_c$ with the experimentally observed switching field. Magnetic moment vector is $\vec{M} = (|\vec{M}|, \alpha_m, \phi_m)$ in polar coordinates. Unit vector in cartesian coordinates:

$$\hat{m} = |\hat{m}| \cos \alpha \hat{Z} + |\hat{m}| \sin \alpha \cos \phi_m \hat{X} + |\hat{m}| \cos \alpha \hat{Z} \quad (A.1)$$

External magnetic field vector is $\vec{H}_{ext} = (|\vec{H}_{ext}|, \alpha_H, \phi_H)$ in polar coordinates. Unit vector in cartesian coordinates:

$$\hat{H}_{ext} = |\hat{H}| \cos \alpha_H \hat{Z} + |\hat{H}_{ext}| \sin \alpha \cos \phi_H \hat{X} + |\hat{H}_{ext}| \cos \alpha_H \hat{Z} \quad (A.2)$$

Effective field due to spin Hall effect (SHE) is $\vec{H}_{SHE}$, given by

$$\vec{H}_{SHE} = -\tau_{SHE} (\hat{m} \times (\hat{Z} \times \hat{J}_c)) \quad (A.3)$$

$$\vec{H}_{SHE}^z = -\tau_{SHE} (|\hat{m}| \sin \alpha_m \sin \phi_m) \quad (A.4)$$

where $Z$ component of SHE $\vec{H}_{SHE}^z$, and $\tau_{SHE}$ is torque due to SHE given by $\tau_{SHE} = \frac{\hbar \theta_{SHE} \vec{J}_c}{2 |e^{-1}| \mu_B t_f}$

Z component of applied in-plane magnetic field = $|\vec{H}_{ext}| \cos \alpha_H = H_{z}^{\text{in}}$

Total field in Z direction = $H_{SHE}^z + H_{z}^{\text{in}}$

Switching with $+\vec{J}_c$ occurs when Z field is equal to coercive field

$$\frac{\vec{H}_c}{2} = H_{SHE}^z + H_{z}^{\text{in}} = -\tau_{SHE} (|\hat{m}| \sin \alpha_m \sin \phi_m) + |\vec{H}_{sw+}| \cos \phi_H \quad (A.5)$$

Switching with $-\vec{J}_c$ occurs when Z field is equal to coercive field
\[
\frac{H_c}{2} = H_{SHE}^z + H_{in}^z = +\tau_{SHE} (|\hat{m}| \sin \alpha_m \sin \phi_m) + |H_{sw-}| \cos \phi_H
\] (A.6)

Spin Hall angle can be estimated as

\[\theta_{SHE} = -\left(\frac{\Delta H_{in-sw}}{\sum H_{in-sw}}\right) \left(\frac{H_c^2 |e| M_s t_F}{\hbar |\hat{j}| \sin \phi_m}\right) \frac{1}{\sqrt{1 - \left(\frac{\Delta R_{\|}}{\Delta R_{\perp}}\right)^2}}\] (A.7)

where \(\theta_{SHE}\) is spin Hall angle (SHA), \(\Delta H_{in-sw}\) is the difference in inplane switching fields for a particle current value, \(\sum H_{in-sw}\) is the sum of inplane switching fields for a particle current value, \(H_c\) is coercive field, \(M_s\) is saturation magnetization, \(t_f\) film thickness of ferromagnetic metal (FM), \(e\) is charge of electron, \(j\) is the current density vector, \(\phi_m\) is the tilt in magnetization due to experimental misalignment, \(\Delta R_{\|}, \Delta R_{\perp}\) difference and sum of Hall resistance values when magnetization was out of the plane and into the plane, when external magnetic field is swept in-plane (\(||\)) and out of plane (\(\perp\)).
I know that I know nothing.

–Socrates