

Topological Insulator Heterostructures for Next Generation of Magnetic Devices

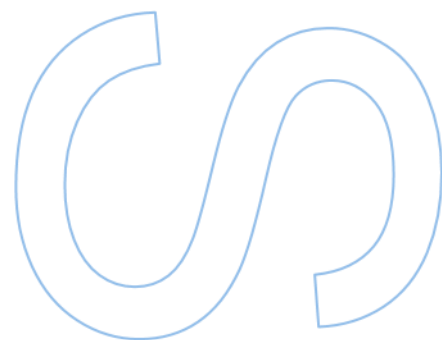
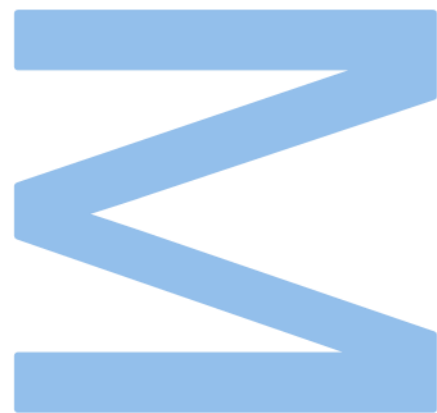
Bruno Miguel Martins Fernandes

Master in Engineering Physics

Department of Physics and Astronomy

Faculty of Sciences of University of Porto and Faculty of Engineering of
the University of Porto

2024



Topological Insulator Heterostructures for Next Generation of Magnetic Devices

Bruno Miguel Martins Fernandes

Master in Engineering Physics
Department of Physics and Astronomy
2024

Supervisor

Prof. Dr. André Pereira, Auxiliar Professor, Faculty of Sciences of
University of Porto

Co-supervisor

Dr. Ana Lúcia Pires, Junior Researcher, Faculty of Sciences of
University of Porto

"...that's the biggest lie I was ever told: "It's not that simple."

So, what's the truth?

That it is. That if you just do the thing that they tell you, you can't, then it's done. And you realize it is that simple... And that it always was. "

Miles Teller as Vinny Pazienza, in *Bleed for This*

Acknowledgements

If you want to go fast, go alone. If you want to go far, go together. I know it is a really cliché sentence, heard and spoken a million times, but during this thesis I saw the power of it very often. The most valuable lesson I've taken from this work isn't just about physics but about the importance of collaboration—working with people who can guide and trade knowledge with you, no one know can do it all alone. First and foremost, my deepest gratitude goes to my supervisor, Professor André Pereira. Thank you for always pushing me to think bigger and for embracing the innovative ideas that shaped this thesis. Your quick, five-to-ten-minute “micro-meetings” often cleared up days of confusion in an instant. There were times when an hour of discussing physics with you felt like I was getting half a semester's worth of insights.

I also owe a special thanks to Dr. Ana Pires, whose support whether in Lab or how to write a thesis was indispensable. And to Mafalda Moreira, who was the key to the experimental side of things: without your help, I'd have only half a thesis to show.

To Catarina Dias, a special thank you for teaching me about optical lithography, and for your endless patience in helping me. I definitely owe you a piece of cake for all the hours you spent guiding me through it. To Ana Sofia Queiróz, thank you for remembering me how to work in a cleanroom. A big thank you to Professor Sergiy Bunkaev and Gleb Kikazei for their assistance with FMR measurements. Your help was invaluable.

To my master's companions, João Benídio and André Miranda, I couldn't have asked for better partners on this journey. You made it all more enjoyable, and your company was as important as the work itself. I'd also like to thank my friends for the Barchelor's, specially Marta Silva, that without you I would not be the same. To my girlfriend for always believing in me, and to whom I shared the most about the bad and good days of this journey.

Finally, a big thanks to my mother who never completely understood what I was doing during my dissertation, but always supported me during this, and all phases of my life. You have given me everything for me to go and conquer the world. To my adventurous sister, whose energy and excitement never failed to lift my spirits during tough times. To my father that instigate in me, since I was a child, the love for physics and thought me to never quit. To all of you I dedicate my work.

Resumo

Os isoladores topológicos são uma classe de materiais quânticos com propriedades eletrónicas únicas, que têm despertado grande interesse pelo seu potencial na criação de dispositivos de baixo consumo energético. A sua estrutura de bandas permite a criação de estados de superfície robustos, com acoplamento entre spin e momento. Estas características possibilitam a criação de correntes de spin polarizadas — uma propriedade fundamental para aplicações em spintrónica.

Um dos principais objetivos desta dissertação é a exploração das propriedades de Bi_2Te_3 , um material conhecido como um forte isolador topológico, e o seu impacto em heteroestruturas magnéticas. Para este propósito, amostras com espessuras nanométricas de FeCo (11 nm) e Bi_2Te_3 (7 nm), foram fabricadas recorrendo a uma técnica de deposição por *DC Magnetron Sputtering*. A análise estrutural dos filmes finos de FeCo permitiu observar a presença de nanocristais de diâmetro $D = (10.8 \pm 0.4)$ nm, embutidos numa matriz de material amorfo. Relativamente às propriedades magnéticas, verificou-se uma magnetização de saturação, no plano, de $M_s = 558.3 \text{ emu/cm}^3$ e um campo coercivo de $H_c = 110$ Oe. Por outro lado, com a adição de uma camada de Bi_2Te_3 observou-se uma redução significativa nos valores de M_s e H_c .

No que diz respeito à medição de fenómenos de transporte eléctrico, foram produzidas estruturas com uma geometria em barra de Hall com dimensões inferiores a 100 μm , utilizando máscaras de poliéster flexíveis e de baixo custo económico. A produção destes dispositivos foi realizada em dois locais distintos: em ambiente sala limpa e em ambiente tradicional. O procedimento de litografia foi otimizado para ambos, sendo as condições providenciadas pela sala limpa preferíveis para a realização de padrões complexos. De salientar, no entanto, que o sistema experimental desenvolvido para a realização de litografia em ambiente tradicional forneceu uma abordagem rápida e económica para a fabricação de dispositivos. Os resultados das medições de transporte eléctrico demonstraram a presença do Efeito Anómalo de Hall em todas as amostras. Para estas medições, obteve-se um campo de anisotropia, perpendicular ao plano das amostras, de $H_s=16$ kOe e uma resistividade de saturação média de $\rho_s=10 \mu\Omega\cdot\text{cm}$. O baixo coeficiente de temperatura, $\text{TC} = 0.006\%/K$, e o desempenho estável numa gama de temperaturas de $[3-300]$ K demonstram a adequação destas heteroestruturas para aplicações em sensores de Hall. A permanência destes dispositivos em regime linear para campos próximos de 16 kOe, revelam a sua possível utilidade em aplicações onde elevados campos magnéticos são considerados, como em ressonância magnética, por

exemplo. Em termos de eficiência energética para dispositivos lógicos, foi possível alcançar a inversão da magnetização a uma densidade de corrente tão reduzida quanto $J_c = 3.3 \times 10^5 \text{ A/cm}^2$, superando sistemas homólogos utilizando metais pesados, onde a densidade de corrente aplicada é uma ordem de grandeza superior. Estes resultados destacam o potencial das heteroestruturas criadas de FeCo/Bi₂Te₃ para aplicações em spintrônica, assim como para tecnologias avançadas de sensores magnéticos.

Palavras-chave: Isoladores Topológicos, Filmes Finos, Heteroestruturas Ferromagnéticas, Sensor de Hall, Spin-Orbit Torque

Abstract

Topological insulators are a class of quantum materials with unique electronic properties, attracting attention for their potential in building low-consumption energy devices. Their distinctive band gap allows robust, non-trivial metallic surface states with spin-momentum locking, enabling the generation of spin-polarized currents—crucial for spintronics applications. This thesis presents a detailed investigation of the incorporation of Bi_2Te_3 , a material that holds signatures of a topological regime, in ferromagnetic heterostructures, focusing on its potential for next generation magnetic devices. The samples, with nanometric thicknesses of FeCo (11 nm) and Bi_2Te_3 (7 nm), were produced by DC Magnetron Sputtering technique and micro-patterned using optical lithography. Structural and magnetic analysis of FeCo thin films revealed an amorphous phase with nanocrystals of $D = (10.8 \pm 0.4)$ nm, possessing an in-plane saturation magnetization of $M_s = 558.3 \text{ emu/cm}^3$ and coercive field $H_c = 110 \text{ Oe}$. A significant reduction in M_s and H_c values was observed with the addition of a Bi_2Te_3 layer.

Regarding micro-fabrication, sub-100 μm Hall bar structures were produced using low-cost, flexible, polyester masks. The micro-structures production was done in two distinct environments: in a clean room and in a benchtop setup. The lithography procedure was optimized for both environments, with cleanroom conditions proving to be essential for intricate patterns. Despite this, the benchtop lithography setup provided a low-cost, rapid approach for device fabrication.

Magnetotransport measurements, employing the Hall Bar devices, demonstrated the presence of the Anomalous Hall Effect (AHE) across all samples. Concerning these measurements, an out-of-plane anisotropy field of $H_s = 16 \text{ kOe}$, and an average saturation resistivity of $\rho_s = 10 \mu\Omega\cdot\text{cm}$ were obtained. The low temperature coefficient of sensitivity, $\text{TC} = 0.006\%/K$, and stable performance across a temperature range of $[3\text{--}300] K$ demonstrate the suitability of these heterostructures for Hall sensor applications, especially in high magnetic field environments such as MRI. In terms of spin-orbit torque (SOT) efficiency, magnetization switching was achieved with a current density as low as $J_c = 3.3 \times 10^5 \text{ A/cm}^2$, surpassing existing heavy metal systems, where the applied current density is one order of magnitude superior. These results highlight the potential of $\text{FeCo/Bi}_2\text{Te}_3$ heterostructures for spintronic applications and offer practical insights into their fabrication and functionality in advanced sensor technologies.

Keywords: Topological Insulators, Thin films, Ferromagnetic Heterostructures, Hall Sensor, Spin-Orbit Torque

Table of Contents

Acknowledgements	ii
Resumo	iii
Abstract	v
Table of Contents	vii
List of Tables	ix
List of Figures	x
List of Abbreviations	xiii
Preface	1
1. State-of-the-art	5
1.1. Spin-Transfer-Torque	5
1.2. Spin-Orbit Torque	6
1.2.1. Spin Hall Effect	6
1.2.2. Edelstein Effect	7
1.2.3. SOT Geometries	8
1.3. Magnetoresistive Random Access Memory	9
1.3.1. History and Evolution	9
1.3.2. SOT-MRAM	11
1.4. Topological Insulators	13
1.4.1. Topological Band Theory	14
1.4.2. Electronic Transport Behavior	15
1.4.3. SOT Topological Devices	17
2. Fabrication of Magnetic Thin Films	21
2.1. Starting point	21
2.2. Magnetron Sputtering	22
2.3. Morphological Characterization	23
2.3.1. X-Ray Reflectometry	23
2.3.2. X-Ray Diffraction	24
2.4. Magnetic Characterization	26
2.4.1. SQUID Magnetometry	26
2.4.2. VNA Ferromagnetic Resonance	28
2.5. Conclusions	30
3. Micro-patterning of SOT heterostructures	31
3.1. Optical Lithography Methodology	31

3.2. First Generation Devices	33
3.2.1. Mask Design and Production	33
3.2.2. Benchtop Lithography Set-up	35
3.2.2.1. Errors and Adaptions	37
3.2.2.2. Alignment	38
3.2.3. Clean Room Fabrication	39
3.2.4. Exposure Time Optimization	40
3.3. Second Generation Devices	42
3.3.1. Mask Design and Production	42
3.3.2. Adaptions	42
3.3.3. Clean Room Fabrication	44
3.4. Conclusions	45
4. Magnetotransport	46
4.1. Anomalous Hall Effect	46
4.1.1. In-plane X Field	47
4.1.2. In-plane Y Field	48
4.1.3. Out-of plane Z Field	49
4.1.4. Temperature dependence	52
4.2. Second Harmonic Measurement Set-up	54
4.3. Magnetic devices	55
4.3.1. High Field Hall Sensor	55
4.3.2. Room-Temperature Current Driven Magnetization Switching	56
4.4. Conclusions	58
5. Final Remarks	59
5.1. Conclusions	59
5.2. Future Work	60
5.2.1. Material Engineering	61
5.2.2. Magnetic Devices Testing	61
5.2.3. New Concept of Magnetic Device	61
Bibliography	63
A. Thermal annealing	75
B. VNA-FMR Experimental Set-up	76
C. Lithography	77
C.1. Clean Room Parameters and Classification	77
C.2. Alignment Errors	79

List of Tables

2.1. Magnetic properties of sputtered thin films	27
3.1. Lithography process parameters for Benchtop set-up.....	39
3.2. Lithography process parameters for cleanroom	41
4.1. Parameters from the Anomalous Hall Effect analysis for clean room device and bench- top setup device, at 300 K.....	51

List of Figures

1.	Evolution of Moore's Law across decades. Adapted from [2].	1
1.1.	Illustration of a magnetic nano-pillar composed by two ferromagnetic layers (FM1 and FM2) and one non-magnetic layer. Electrons become spin polarized at FM1 and injected into FM2. In FM2, both s-electrons and d-electrons start to precess due to exchange interaction. As a result of a large local magnetic moment, the angle of precession of d-electrons is smaller than s-electrons. The lost spin components by s-electrons are transferred to the local spin moment, giving origin to a torque actuating on $\vec{M}_2$. Adapted from [18].	5
1.2.	(a) SHE: When entering a material with large SOC, a charge current produces a transverse spin current. (b) ISHE: a spin current leads to a charge accumulation on the same side of the conductor. Adapted from [22].	7
1.3.	a) Energy dispersion of the 2D Rashba interface. b) Fermi contour exhibiting orthogonality between spin and momentum for Rashba states. c) Energy dispersion relation in TI surface. d) Fermi contour exhibiting orthogonality between spin and momentum for TI surface. From [27].	8
1.4.	Different types of SOT magnetization switching in NM/FM heterostructures. From [30].	9
1.5.	History of development of MRAM technology.	10
1.6.	Different MRAM devices: STT-MRAM (a), three-terminal SOT-MRAM (b) and two terminal SOT-MRAM (c). Adapted from [43].	11
1.7.	SOT-MRAM device illustration produced by Wang <i>et al.</i> [46].	12
1.8.	Topologically inequivalent surfaces represented mathematically by different genus...	13
1.9.	From trivial insulator, characterized by a Chern number of $n_c = 0$, to a non-trivial insulator ($n_c=1$), there must exist gapless states at the surface between the two for a continuous transitions between topologically inequivalent phases. From [52].	14
1.10.	The origin for the peculiar properties of strong 3D TI's lies in hybridization of the band structure, due to strong spin orbit coupling, that gives rise to surface states protected by TRS. Diagram of topological insulator effects: Spin-momentum locking and Anti-localization effect; and key parameters: Spin Hall Angle and Spin diffusion Length.	16
1.11.	a) MR results for positive applied bias current and b) negative bias current b). c), f) scheme of low and high (d), e) resistance states and correspondence to relative current to magnetization orientation c),d),e),f).	18
1.12.	Word performed by Khang <i>et al.</i> [57]. a) 25- μm Hall Bar device. b) Anomalous Hall resistance measurement by sweeping a perpendicular magnetic field.c) Anomalous Hall resistance vs DC current sweep.d) Measurement of second harmonic.	19
1.13.	Comparison of SOT efficiency of several materials and respective power consumption. Reproduced from [43].	20
2.1.	Resistivity as a function of temperature for two 97 nm samples: a)without thermal annealing and b) annealed at 300°C; c) Magneto-conductivity for various thicknesses of sputtered Bi_2Te_3 thin films, with and without annealing [64, 65].	21
2.2.	Magnetron Sputtering chamber a), FeCo target b) and deposited FeCo/ Bi_2Te_3 thin films in glass substrate c).	23
2.3.	XRR spectrum and fitting of 5 min magnetron sputtering deposition of FeCo.	24

2.4. a) XRD spectrum of original FeCo target and b) XRD spectrum of sputtered 110 nm FeCo thin film.	25
2.5. In-plane M(H) loops for FeCo(110nm) and FeCo(11nm)/ Bi ₂ Te ₃ (7nm), with and without annealing. On the inset graph it is represented the full range of magnetic field (-5T to 5T), in which the data were collected.....	26
2.6. FMR spectra for FeCo(110) nm at different external magnetic fields H_{ext}	28
2.7. Counterplots of Resonance frequency, f_{res} for each applied magnetic field H_{ext} for a) FeCo(110), b) FeCo(110)/Bi ₂ Te ₃ (14), c) FeCo(11)/Bi ₂ Te ₃ (7) and d) annealed FeCo(11)/Bi ₂ Te ₃ (7) (nm).	29
3.1. Scheme of optical lithography procedure.	31
3.2. Left: Mask design elaborated in AutoCAD; in orange and green virtual symmetry lines. Right: Example of Printed mask.	34
3.3. a) AutoCAD drawing of test device. b) Microscope observation of printed mask. c) Region of the contact pads of the printed mask, at a higher magnification.....	34
3.4. a) 20- μ m width arm HB mask and b) 100- μ m width arm HB mask.....	35
3.5. Lithography process encountered for Benchtop set-up.....	36
3.6. Benchtop Lithography set-up: Spinner Station. a) 1-Spinner, 2- Connection to vacuum pump, 3- Arduino, 4- Spinner motor. b) 1- Sample holder, 2- Mask holder. c) Micropositioners. d) 1- UV lamp, 2- Diffusive Glass, 3- External power source.	36
3.7. Microscope images: a) Test device Photoresist pattern after development. b) Test device after Bi ₂ Te ₃ deposition and lift-off. c) Hall Bar photoresist pattern after development. d) Hall Bar after Bi ₂ Te ₃ deposition and lift-off.	37
3.8. Microscope images: a) Photoresist pattern after development at 200 μ m scale and b) Photoresist pattern after development at 100 μ m scale.	38
3.9. a) AutoCAD mask of contacts. b) Microscope image of patterned contacts in photoresist misaligned with deposited material from previous lithography process.	39
3.10. a) Spinner PWM50 in Lithography room I and b) Mask aligner in Lithography room II.	40
3.11. Microscope images of photoresist after development for exposure times of: a) 20s, b) 30s, c) 35s and d) 40s.	41
3.12. Double-cross Hall bar geometry: The structure consists of a channel with length L and width w , featuring four arms for voltage measurements. The distance between consecutive arms is denoted by a , while the perpendicular distance from the arms to the channel is p . The voltage contacts width is represented by c . From [78].	42
3.13. a) AutoCAD drawing of elaborated double cross Hall Bar of parameters $L = 734 \mu\text{m}$, $W = 160 \mu\text{m}$, $c = 50 \mu\text{m}$ and $p = 73 \mu\text{m}$; and b) Produced flexible polyester photomask.	43
3.14. Microscope images of a) Second Alignment mark of 1 mm width and b) Second alignment mark of 250 μ m width.....	43
3.15. Double Cross Hall Bar through different stages :a) Photoresist pattern after development and b) Deposited material (14 nm Bi ₂ Te ₃) after lift-off, and c) second lithography step with the photoresist pattern of the contacts after development.	44
4.1. Scheme of employed geometry for magnetotransport measurement.	46
4.2. a) Ordinary Hall Effect and b) Anomalous Hall Effect. From [80].	47
4.3. Horizontal Rotator and respective dockable sample board for ETO resistivity module from <i>Quantum Design</i>	47
4.4. a) Geometry used: applied magnetic field parallel to the current direction and b) Transverse resistivity as function of applied in-plane field. The Hall Bar illustrations along the curve represent the increase of the magnetization vector in the X-axis, according to the applied field.	48

4.5. Transverse resistivity as a function of in-plane Y field for a) clean room device and c) benchtop set-up device; b) geometry used for this measurement. In c) the HB scheme shows the rotation of the magnetization from the easy to the hard axis.	49
4.6. Anomalous Hall Curves for a) Clean room device and b) Benchtop setup. The inset graph shows the saturation resistivity as a function of temperature.	50
4.7. Deconstruction of the measured signal into OHE and AHE components for the clean room device.	51
4.8. Temperature dependence of transverse voltage, under absence of an applied magnetic field. On the inset is the clean room device used for the measurement.	52
4.9. Work from Chen <i>et al.</i> [84]: a) Hysteresis loops at 8 K for anomalous hall resistance and magnetization, for $\text{Bi}_2\text{Te}_3/\text{MnTe}$ heterostructure; b) and c) individual contributions of AHE for MnTe and Bi_2Te_3 , respectively.	53
4.10. Experimental set-up created for the measurement of the spin Hall angle. It consists of a lock-amplifier that acts both as the source and detector for the second harmonic voltage; a voltage to current converter module composed by two OP-77 and five resistors, and finally a test sample.	54
4.11. Sensitivity of the out-of plane AHE curve dependence with temperature, for clean room device.	56
4.12. a) Geometry used for type X SOT switching and b) Hall resistance results for a current sweep between -5mA, 5mA, under $H_z = 50\text{Oe}$ applied field.	57
5.1. Representation of Lateral Spin Valve. A spin polarized current is created at the topological insulator channel. By passing through the ferromagnetic stripes it is intended to revert one of the stripes magnetization.	62
5.2. a) Contacts view; b) active region of the device containing the topological insulator channel and the ferromagnetic stripes; c) Deposited Bi_2Te_3 on silicon wafer.	63
A.1. Annealing furnace	75
B.1. a) VNA ANRITSU 37247D; b) Helmholtz coils to produce static external magnetic field and c) Coplanar Waveguide.	76
C.1. Test device B. a) AutoCAD drawing. b) Microscope image of printed mask. c) Patterned photoresist. d) Deposited Bi_2Te_3	77
C.2. Scheme of clean room strategies to prevent contamination.	77
C.3. ISO clean room standards and FS209E equivalent. From [91].s.	79
C.4. Microscope Image of photoresist after development in second lithography process, for electrical contacts: a) Good Alignment, b) Vertical Misalignment and c) Vertical and Horizontal Misalignment.	80

List of Abbreviations

FINFETS Fin field-effect transistors

DG-MOSFET Double Gate Metal Oxide Semiconductor Field-Effect Transistors

TI Topological Insulator

AHE Anomalous Hall Effect

STT Spin-Transfer-Torque

SOT Spin-Orbit-Torque

SOC Spin-Orbit Coupling

FM Ferromagnetic

NM Non-Magnetic

SHE Spin Hall Effect

ISHE Inverse Spin Hall Effect

EE Edelstein Effect

IEE Inverse Edelstein Effect

MRAM Magnetoresistive Random Access Memory

MR Magnetoresistance

AMR Anisotropic Magnetoresistance

GMR Giant Magnetoresistance

TMR Tunnel Magnetoresistance

MTJ Magnetic Tunnel Junction

PMA perpendicular Magnetic Anisotropy

HB Hall Bar

OHE Ordinary Hall Effect

AHE Anomalous Hall Effect

XRR X-Ray Reflectometry

XRD X-Ray Diffraction

SQUID Superconducting Quantum Interference Device

VNA Vector Network Analyzer

FMR Ferromagnetic Resonance

Preface

Motivation

The ever-evolving nature of the microelectronics industry is guided by an empirical observation made in 1965, known as Moore's Law [1]. According to it, the number of transistors in an integrated circuit will double every two years, accompanied by a reciprocal reduction in the size of the device components. We are now entering the Angstrom era, where transistor dimensions are shrinking to the atomic scale. By 2030, this technological leap is projected to enable the integration of up to 1 trillion transistors in a single device, as shown in fig.1:

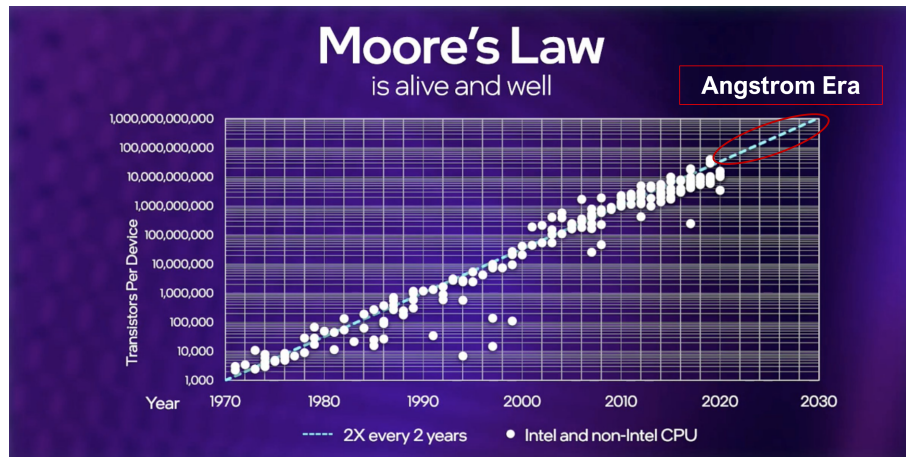


FIGURE 1: Evolution of Moore's Law across decades. Adapted from [2].

One major limitation of evolving towards smaller node processes is the promotion of a large current leakage for the entire chip. To solve this constrain, semiconductor manufacturers around the globe have presented alternative chip architectures such as the Fin field-effect transistors (FINFETs) [3], the double gate metal oxide semiconductor field-effect transistors (DG-MOSFET) [4] and carbon nanotube FETS [5]. Nevertheless, the majority of these structures do not yet comply in one crucial parameter: volatility, i.e., the incapability of retaining a logical state.

To continue the trajectory of exponential growth in computational power, researchers are now shifting their focus to a different approach: one that uses the electron's spin as an intermediary for logical state switching, instead of its charge. In this realm of expertise,

named spintronics, new ways of minimizing the very large-scale integration (VLSI) cost functions [6] are explored. Specifically, the power consumption problem in semiconductors is surpassed in a significant manner: by encoding information in magnetic structures non-volatility is assured, since there are not magnetic modifications of the system with respect to time, nor electrical power is necessary to sustain a logical state.

The groundbreaking discovery that definitely promoted a significant interest in this field was made by two independent research groups, led by Albert Fert and Peter Grünberg, respectively, with the finding of the Giant Magneto-Resistance in 1988 [7, 8]. Since then, the improvements in the spintronics field has widen up the range of applications, having its part on the development of non-volatile memory disks, neuromorphic hardware and quantum computing [9–11].

One approach to advancing current technology, aside from seeking new kinds of electronics, or adaptations to new architectures, is through the implementation of distinct materials. In that sense, **topological insulators** (TI's) are a category of quantum materials distinguished by their unique electronic characteristics, receiving interest for their potential in building dissipationless devices [12]. This class of materials required an extension of Landau's theory of phase transitions, since they could not be described by local order parameters, rather by topological invariants. The distinctive band gap structure of TI allows the existence of non-trivial metallic surface states, robust against back-scattering, with spin-moment locking. This last characteristic translates into the generation of electrical currents exhibiting well-defined spin polarization, crucial for spintronics devices.

Considerable effort is being employed in harnessing the unique characteristics of topological insulators to enhance the performance of existing devices. One evident application lies in its use in field-effect transistors, where the topological insulator material can serve as the channel [13]. Other not so common approaches include the topological p-n junction, local electrical topological interconnections and magneto-electric topological devices [14–16]. These materials might hold the key to unlocking unprecedented levels of performance and efficiency, ensuring that the pace of technological advancement continues unabated in the post-silicon era.

Research Goals

The purpose of this thesis is to explore the potential of 3D topological insulators, in specific Bi_2Te_3 , for the development of magnetic devices. The focus will be on exploring how Bi_2Te_3 can enhance the efficiency of magnetization switching in $\text{FeCo}/\text{Bi}_2\text{Te}_3$ thin film heterostructures. For that, we divide our main objective in three specific aims:

- **Fabricate ferromagnetic/topological insulator heterostructures using optical lithography.**
- **Understand how Bi_2Te_3 affects the magnetic properties of FeCo** and contributes to the performance of the heterostructure.
- **Determine the lowest magnetization switching current density:** A critical goal is to assess the minimal current required for magnetization switching in these heterostructures, aiming for reduced power consumption.

These goals aim to bridge fundamental research and practical applications, advancing the development of energy-efficient SOT devices.

Thesis Structure

The first chapter introduces fundamental theoretical concepts that provide the foundation for understanding the physics behind magnetic memory devices. It then traces the history and evolution of MRAM technology, leading into a discussion of the current state of the art. The chapter concludes with a brief exploration of topological band theory and highlights fundamental properties of topological insulators relevant to energy-efficient spintronics applications.

The second chapter focuses on the fabrication and characterization of thin films, covering the morphological and magnetic properties of the produced samples.

The third chapter is dedicated to optical lithography techniques, conducted in two distinct settings: a clean room environment and a benchtop setup in a standard controlled laboratory. This section describes the step-by-step process of creating micro-patterns, starting from mask design and production, addressing the challenges encountered during the lithography process, and concluding with the development of functional devices.

The final chapters, focusing on magnetotransport measurements such as the Anomalous Hall Effect (AHE), room-temperature magnetization switching, and second harmonic

measurements, provide the ultimate evaluation of the fabricated samples' suitability for magnetic devices.

1 State-of-the-art

In spintronics, two primary approaches narrow the gap between fast computing units and slow memory units, effectively tackling the issues of energy inefficiency and time delay. One approach involves manipulating magnetization via spin-transfer-torque (STT), while the other achieves the same purpose through spin-orbit-torque (SOT). Both methods are employed in effective nonvolatile memory devices, diverging in the created architectures, switching speed and expected endurance.

1.1 Spin-Transfer-Torque

The STT effect considers the transport of spin angular momentum between two magnets, by means of a spin-polarized current. A typical device is depicted in fig.1.1, where two ferromagnetic (FM) layers (FM1 and FM2) are separated by a non-magnetic spacer layer (that can be a metal or an insulator). When a bias voltage is applied, the electrons flowing through the device are first polarized in FM1 and then injected into FM2. Hence, the spin-polarized current created in FM1 can interact with the second magnetic layer, by exchange interaction, and generate a torque. In 1996, Slonczewski [17] predicted that depending on the device design and on the magnitude of the applied magnetic field, the FM2 can either switch from one static magnetic orientation to the other, or, be induced in steady-state precession, upon the actuation of a direct current.

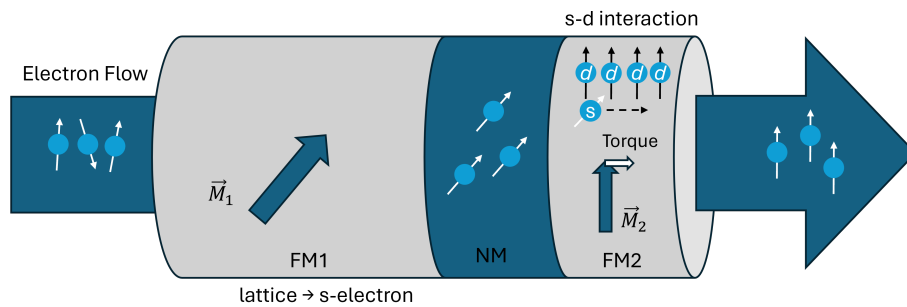


FIGURE 1.1: Illustration of a magnetic nano-pillar composed by two ferromagnetic layers (FM1 and FM2) and one non-magnetic layer. Electrons become spin polarized at FM1 and injected into FM2. In FM2, both s-electrons and d-electrons start to precess due to exchange interaction. As a result of a large local magnetic moment, the angle of precession of d-electrons is smaller than s-electrons. The lost spin components by s-electrons are transferred to the local spin moment, giving origin to a torque actuating on $\vec{M}_2$. Adapted from [18].

In a simplified view, this phenomenon can be understood as an exchange interaction between conduction electrons (s-electrons) and electrons that possess local magnetic moment (d-electrons) [18]. Considering that this s–d exchange interaction conserves the total spin angular momentum, any reduction in the angular momentum of conduction electrons is matched by an equivalent increase in the angular momentum of the d-electron system. As a result, within the magnetic pillar, if the spin angular momentum of a conduction electron is altered due to the s–d interaction at FM2, this change in angular momentum must be transferred to the d-electrons within that layer. This type of torque is known as "spin-transfer-torque" .

1.2 Spin-Orbit Torque

Contrarily to the STT, the spin-orbit torque does not consider the transfer of angular momentum between two magnets. Instead, it is generated from a single material heterostructure, typically using a material with high spin-orbit coupling (SOC) (ex: Ta, β -W, Bi_2Se_2), which can "naturally" produce a spin-polarized current capable of exerting a torque on a ferromagnetic layer. It can be described in terms of two distinct effects: the Spin Hall Effect (SHE) and the Edelstein Effect (EE), and their respective counterparts Inverse Spin Hall Effect (ISHE) and Inverse Edelstein Effect (IEE), which will be explored in the following sections.

1.2.1 Spin Hall Effect

The SHE was first theorized in 1971, by Mikhail Dyakonov and Vladimir Perel [19, 20], although it took more than 30 years to be directly observed in semiconductors [21]. It can be summarized as an electrical transport phenomenon where an applied charge current gives rise to a transverse spin-current. It occurs in bulk materials that present high spin-orbit coupling, such as Ta and Pt, for example.

For the description of SOT following the SHE, a bilayer structure of a ferromagnetic layer (FM) and a non-magnetic one (NM) (usually a heavy metal) is employed. When considering a charge current entering the NM layer, the spin-orbit scattering produces an accumulation of opposite spins in opposite faces of the conductor, as illustrated in fig.1.2.a):

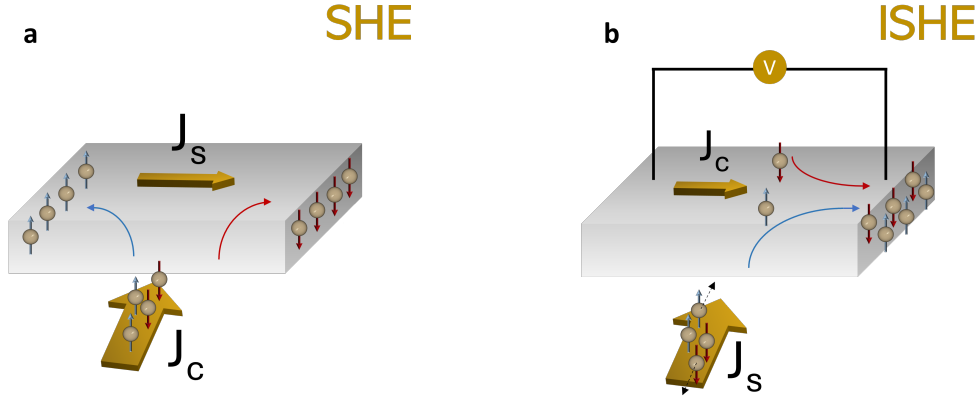


FIGURE 1.2: (a) SHE: When entering a material with large SOC, a charge current produces a transverse spin current. (b) ISHE: a spin current leads to a charge accumulation on the same side of the conductor. Adapted from [22].

This process leads to the appearance of a transverse spin current, J_s . The interaction of a spin current with an adjacent ferromagnetic layer occurs via transfer of angular momentum, known in this specific case as spin-orbit-torque. If large enough, the exerted torque is capable of switching the magnetization of the magnetic layer [23]. To quantify the efficiency of charge to spin inter-conversion in this process, the spin-Hall angle θ_{SH} is used, according to the equation [24]:

$$\mathbf{J}_c = \theta_{SH} \mathbf{J}_s \times \sigma \quad (1.1)$$

Where $\mathbf{J}_c$ and $\mathbf{J}_s$ are the charge and spin currents, respectively, and σ is the spin polarization unit vector. Conversely, by manipulating the magnetization of the FM material a spin current can be generated. Through ferromagnetic resonance excitation, for example, the damping of the precessional spins allows a flow of angular momentum to the NM layer, by means of a spin current. The entering of spin polarized electrons in the NM layer induces a voltage across the heavy metal, a phenomenon recognized as the accumulation of opposite spins on the same side of the conductor, known as the Inverse Spin Hall Effect - fig.1.2.b).

1.2.2 Edelstein Effect

As the SHE, the Edelstein effect (EE) also accounts for the creation of SOT, when combining a layer with high spin-orbit-coupling material with one with magnetic exchange. However, contrarily to the SHE/ISHE where the spin/charge accumulation occurs at the

bulk, in the EE/IEE the spin-to-charge inter-conversion is bi-dimensional [24].

The EE occurs when an electrical current is constricted at an interface or surface, alongside with the creation of spin-polarized electrons. In certain 2D materials, such as transition metal dichalcogenides like MoS₂ or WSe₂ [25], structural inversion symmetry breaking leads to the creation of states that are described by a Rashba Hamiltonian [26]. This symmetry breaking causes spin degeneracy to be lifted, resulting in the electron's spin being locked to its momentum within each energy branch, as shown in fig.1.3.a).

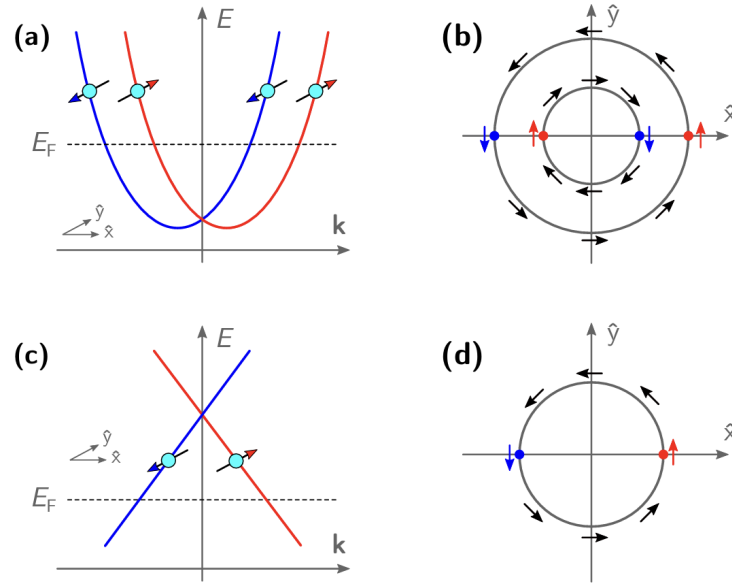


FIGURE 1.3: a) Energy dispersion of the 2D Rashba interface. b) Fermi contour exhibiting orthogonality between spin and momentum for Rashba states. c) Energy dispersion relation in TI surface. d) Fermi contour exhibiting orthogonality between spin and momentum for TI surface. From [27].

Topological Insulators also display bi-dimensional charge transport, spin-moment locking and surface states. However, these are topologically protected states, where the Hamiltonian describes a spin-split linear dispersion- fig.1.3.c). The spin accumulation produced in these materials, with either one of these type states, is capable of generating a torque on a ferromagnetic layer and induce magnetization switching.

The discovery of the EE was made by V.M. Edelstein [28] in 1990 and experimentally observed in 2013 by Sánchez *et al.* [29], when employing a spin current in a Bi/Ag bilayer and detecting a charge current.

1.2.3 SOT Geometries

The switching of the magnetization using SOT, in ferromagnetic heterostructures, is generally accomplished by one of three fundamental geometries: type Z, type Y and type X.

They are distinguished by the orientation of their magnetic easy axis relative to the accumulated spin current, as illustrated in fig.1.4. If we define the x-axis as the direction of the current, and the z-axis as the out-of-plane direction, the easy axis in these structures can be along the z-axis (fig.1.4.a) , the y-axis (fig.1.4.b)) and the x-axis (fig.1.4.c)).

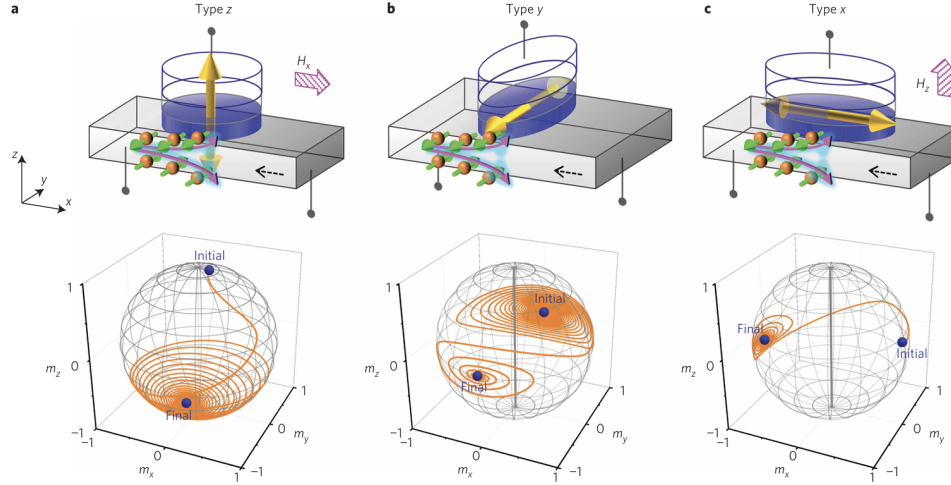


FIGURE 1.4: Different types of SOT magnetization switching in NM/FM heterostructures. From [30].

For the type Y, the accumulated spin at the non-magnetic layer aligns with the magnetization, and its switching mechanism is essentially similar to spin-transfer torque (STT) switching. In contrast, in type Z and type X, the electron's spin are orthogonal to the direction of the magnetization. Thus, for deterministic magnetization switching, these geometries require the application of a static external magnetic field. Nevertheless, types X and Z are capable of achieving faster switching compared to type Y [30]. This is because, in type Y, switching is only completed after multiple precessions of the magnetization (fig. 1.4.b)), whereas in types X and Z, the magnetization switches immediately after the current is applied, as shown by macrospin simulations (fig.1.4.a) and b)). Additionally, in type Y, the probability of successful switching depends on the duration of the applied current pulse, while in type X, switching is almost independent of pulse duration [31].

1.3 Magnetoresistive Random Access Memory

1.3.1 History and Evolution

With respect to technological implementation, the discussed phenomenon of STT and SOT, although distinct from each other, are both employed in Magnetoresistive Random Access Memory (MRAM) devices.

The MRAM is characterized by using the magnetoresistance (MR) effect as the reading principle, regardless of the writing method. In fig.1.5, the evolution of MRAM technology is shown, where the three different types of MR effects are situated according to their chronological implementation. Initially, the Anisotropic Magnetoresistance (AMR) effect was used. This effect, discovered in the mid-19th century, describes how the electrical resistance of a material changes with the angle between the direction of electric current and magnetization [32]. A significant advancement came with the discovery of the Giant Magnetoresistance (GMR) effect, in 1988, that propelled the improvement of magnetic sensors [7], specially hard disk drives. In 1994, the reading principle evolved to Tunnel Magnetoresistance (TMR), thanks to the work of T.Muyazaki *et al.* and Moodera *et al.* [33, 34] on Magnetic Tunnel Junctions (MTJs). These MTJs * achieved an 18% MR ratio, the highest value obtained until then, at room temperature.

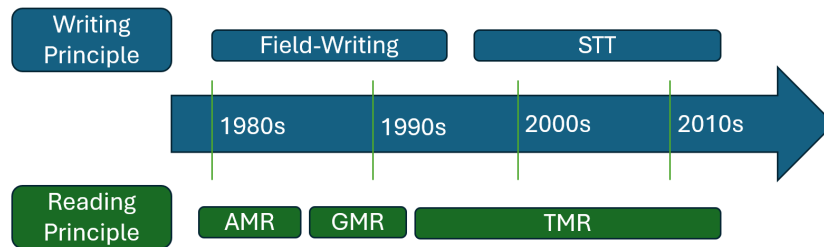


FIGURE 1.5: History of development of MRAM technology.

In these MTJs, amorphous Al_2O_3 was initially used as the tunnel barrier, enabling MRAM to reach megabit capacities. However, the writing mechanism still relied on field-writing, which implied currents in the order of the mA, making it incompatible with the thickness of the wires used. This factor prevented MRAM to achieve even further memory density. In the 2000s, the writing principle changed to STT-writing, leading to a higher device efficiency. This method was theoretically proposed by J. Slonczewski [17], in 1996, and experimentally demonstrated by Myers *et al.* [35] using GMR, and by Huai *et al.* [36] using Al_2O_3 -MTJ.

Despite all these improvements, the event that propelled MRAM technology was the introduction of a crystalline MgO barrier, in 2004 [37, 38]. As a consequence, the MR ratio increased from 70% to 200%, for MTJs with in-plane magnetization that presented a stable reference layer. At that time, higher MR ratios could be obtained at the cost of an unstable reference layer. In such conditions, the diffusion of elements to the barrier degrades the entire device, making it unsuitable for memory applications. In 2007, the path to gigabit

*Name given to elements that use the TMR effect as reading mechanism.

density MRAM was traced with the fabrication of perpendicular MTJs. In fact, perpendicular MTJs were thought to be almost impossible to produce, nevertheless, H.Yoda *et al.* [39] demonstrated the possibility of STT-writing in perpendicular CoFeB/MgO/CoFeB devices.

1.3.2 SOT-MRAM

In the current STT-MRAM technology, a two terminal geometry, shown in fig.1.6.a), with an oxide layer sandwiched by two FM layers is used. In this case, while one of the magnetic layers is pinned, the other is free to rotate and therefore alter its state. Here, the read and write events share the same electrical path. The logical state is defined by the relative display of the two magnetic layers, i.e, parallel ("0") or antiparallel ("1"), that present low or high TMR values, respectively.

However, one drawback of this technology is its difficulty in reducing the writing time below 10 nanoseconds, mainly due to the incubation time [40]. Also, for a fast switching a large current needs to be employed through the oxide layer, resulting in reliability problems and accelerated degradation of the barrier [41].

A solution to these issues is found in SOT-MRAM, which adopts a three-terminal architecture. This design allows the read and write operations to follow separate paths, as shown in Fig. fig.1.6.b), offering several advantages. First, it provides a wider write margin, which increases the operating frequency, since the critical current is inversely proportional to the pulse width [42]. Additionally, the three-terminal MTJ design improves endurance because the write current does not pass through the tunnel barrier. However, this design is less suitable for high-density applications, as it requires a larger area and twice the number of transistors per cell compared to STT-MRAM.

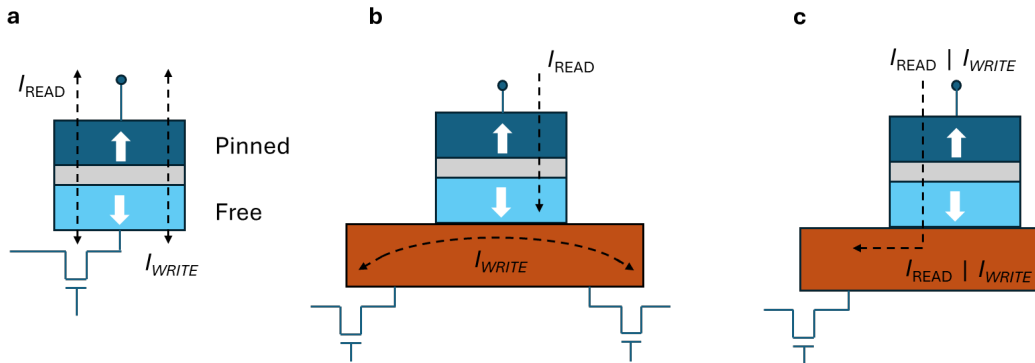


FIGURE 1.6: Different MRAM devices: STT-MRAM (a), three-terminal SOT-MRAM (b) and two terminal SOT-MRAM (c). Adapted from [43]

As a conciliatory alternative, two-terminal devices where STT and SOT mechanisms are both employed, as illustrated in fig.1.6.c), have been develop to achieve both marks, i.e, high density storage and good operational speed [43].

Additionally, since today's memory storage technologies (such as DRAM and flash memories) use two-terminal devices, there is a greater likelihood of integrating SOT-MRAM into the industry without having to redesign the entire memory chip. Through simulations of perpendicular anisotropic MTJs [44], it was concluded that combining the STT with the SOT effect completely eliminate the incubation delay, reducing the write energy more than 90% compared to a typical system. On the other hand, Sato *et al.* [45] came to the conclusion that in a two-terminal device, there was still a contribution of the SOT mechanism even when the current was applied vertically. By considering this hybrid architecture (fig.1.7), Wang *et al.* [46] demonstrated that the need of an assistant field for switching in SOT-MRAM disappears, due to the presence of the STT. The study involved fabricating p-MTJ (perpendicular magnetic tunnel junction) stacks on thermally oxidized silicon substrates. The stacks, consisting of layers like Ta, CoFeB were deposited by direct current magnetron sputtering, while MgO was deposited on radiofrequency mode. These layers were patterned into three-terminal devices with nanopillars of varying radii using electron beam lithography and ion milling. These devices included Hall probes to detect magnetization direction via the anomalous Hall effect.

In sum, while one mechanism provides a lower incubation time and therefore a higher speed (SOT), the other dictates the final magnetic orientation (STT), allowing a combination of low current density and field free switching.

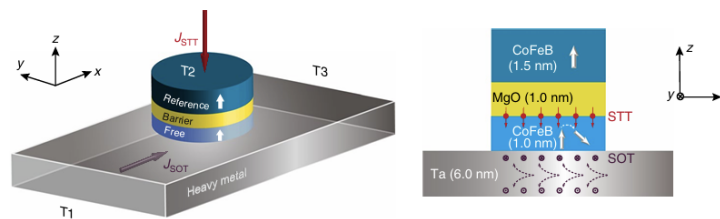


FIGURE 1.7: SOT-MRAM device illustration produced by Wang *et al.* [46].

Besides architectural development, other ways of improving the efficiency of magnetic logical devices is through the implementation of unique materials. In the case of two terminal SOT-MRAM, the search for materials with enhanced SOT is critical. Topological insulators, semimetals, 2D materials and heavy metals have been found as leaders groups for the purpose [47]. The first direct consequence of employing such distinctive materials is

a significant reduction in the switching current density. As a result, the likelihood of device degradation will be lower since the writing current is further from the breakdown voltage. In this work, we pretend to explore the potential of topological insulators as distinctive materials to improve SOT-MRAM efficiency.

1.4 Topological Insulators

Long before the introduction of topological order, phase transitions in condensed matter physics were thought to be governed solely by symmetry breaking, according to Landau's Theory. However, in 1980, a work developed by von Klitzing *et al.* [48] demonstrated that by confining a 2D electron gas in high magnetic fields, at helium temperature, the Hall resistance showed quantized plateaus $R_{Hall} = h/ve^2$ *, without any apparent symmetry breaking. Consequently, this newfound phenomenon, known as Integer Quantum Hall Effect (IQHE), required a broadening of Landau's theory to be accurately portrayed, which was accomplished with the help of topology.

Topology, in mathematics, refers to the study of geometric objects that are invariant under continuous deformations. The characterization of such objects is usually done by means of the Gauss-Bonnet theorem [49]. As a result, two surfaces are said to be topologically equivalent if they share the same genus g , which is fundamentally the number of holes of a given object. Conversely, surfaces with different genus can't be smoothly deformed into one another, i.e., without breaking, closing or opening holes. An illustrative example of topological inequivalent surfaces is given in fig.1.8, where a sphere ($g=0$) cannot be deformed into a torus ($g=1$).

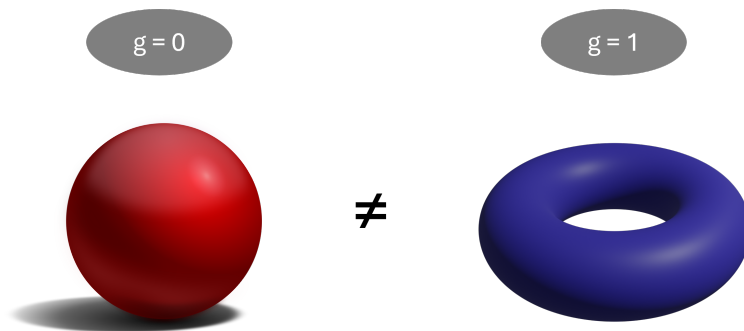


FIGURE 1.8: Topologically inequivalent surfaces represented mathematically by different genus.

* h - Planck's constant; v - Filling factor; e - Electron charge

1.4.1 Topological Band Theory

The remaining question is how can topology be used to classify phases of matter. In this regard, the adiabatic continuity principle offers the appropriate solution. According to it, if two quantum states, separated by a finite energy, can be deformed into one another upon a slow perturbation of the Hamiltonian of the system, such that it remains in the ground state, then they are topological equivalent states [50]. In other words, the equivalency between states can be done by establishing an adiabatic path in which the energy gap between them remains finite. As a result, to connect topological inequivalent states implies a phase transition where the energy gap disappears [49].

To classify the different phases of matter according to these new principles, topological invariants are used in the same manner that the genus is employed to classify distinct geometric objects. Consequently, instead of using the Gauss-Bonnet theorem a similar form is introduced to define the TKNN topological invariant. It is defined as the integral of the Berry curvature of states over the Brillouin Zone [49]. In a 1982 paper [51], Thouless, Kohmoto, Nightingale, and den Nijs (TKNN) demonstrated that the filling factor ν in the IQHE is actually a topological invariant correspondent to the Chern Number, n_c . In terms of band structure, topological insulators distinguish themselves from ordinary insulators due to the presence of gapless states and band inversion. Indeed, due to high spin-orbit coupling an energy-order inversion of conduction and valence bands occurs at the band-gap (fig.1.9).

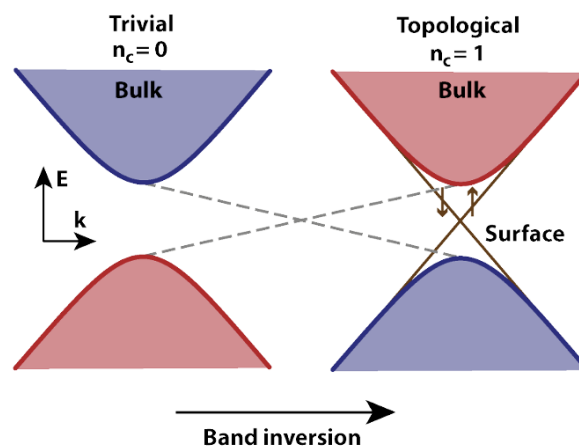


FIGURE 1.9: From trivial insulator, characterized by a Chern number of $n_c = 0$, to a non-trivial insulator ($n_c=1$), there must exist gapless states at the surface between the two for a continuous transitions between topologically inequivalent phases. From [52].

Conversely, for ordinary insulators their energy band are topologically equivalent to those

of vacuum or air, being denoted as topologically trivial ($n_c = 0$). However, for topologically non-trivial insulators ($n_c \neq 0$) the hybridization of energy bands occurs at the bulk due to high spin-orbit coupling. In this manner, to continuously deform one phase to another, there must exist, at the surface between ordinary and non trivial insulators, gapless states that connect both phases by closing the band-gap.

In strong*, 3D topological insulators, the gapless states appear as Dirac Cones and are topological surface states protected by time-reversal symmetry (TRS). Given this, TRS also dictates that electrons with opposite momentum must have opposite spins, being this way the spin locked to the momentum.

1.4.2 Electronic Transport Behavior

Due to time-reversal symmetry and spin-momentum locking, backscattering (which would require a spin flip) is highly suppressed in these materials, as long as time-reversal symmetry is preserved (i.e., in the absence of magnetic impurities), resulting in large spin diffusion lengths[†]. All these factors, lead to the creation of large spin currents, and enhanced θ_{SH} , which is a crucial parameter for spintronics applications. In fig.1.10, some of the important attributes of 3D topological insulators for spintronics are presented.

While the enhanced values of spin Hall angle[‡] and spin diffusion length for TI's are both a consequence of spin-moment locking and TRS, the Weak Antilocalization Effect holds a distinct signature of a topological insulator behavior.

*i.e. that display edge states protected by time reversal symmetry.

[†]Spin diffusion length is defined as the average distance between two spin-flip events under zero electric field corresponds to the spin diffusion length.

[‡]In this case, as we assume the presence of surface states and spin-moment locking, the charge to spin conversion is bi-dimensional and the correct designation should be attributed to the Edelstein Effect. However in the literature for magnetic memory devices the standard value used for characterizing charge to spin conversion is the θ_{SH} .

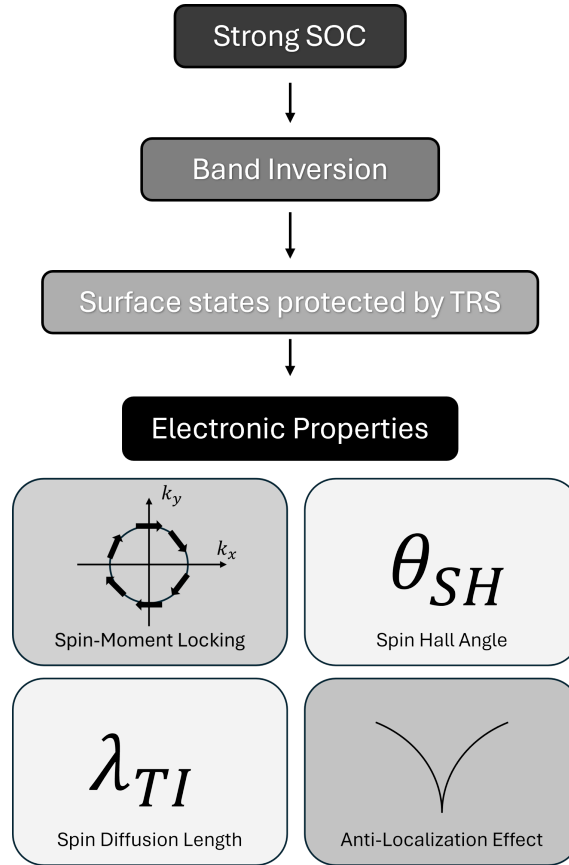


FIGURE 1.10: The origin for the peculiar properties of strong 3D TI's lies in hybridization of the band structure, due to strong spin orbit coupling, that gives rise to surface states protected by TRS. Diagram of topological insulator effects: Spin-momentum locking and Anti-localization effect; and key parameters: Spin Hall Angle and Spin diffusion Length.

As the system's temperature decreases, the phase coherence length, L_Φ , increases *, what in a weakly disordered system leads to a quantum diffusive regime. In this regime, the quantum interference of electron paths affects conductivity.

In systems with strong spin-orbit coupling (SOC), WAL occurs when backscattering causes the electron's spin to rotate, introducing a π Berry phase. This phase shift results in destructive interference between time-reversed scattering loops, leading to a positive correction in conductivity. The overall effect is a reduction in the material's resistivity, distinguishing WAL from Weak Localization (WL), where constructive interference increases resistivity.

When large magnetic fields are applied, WAL is suppressed as the magnetic flux introduces phase shifts between electron paths, breaking TRS and returning the system to a classical regime. This results in a V-shaped MR cusp, which can be described by the

*It represents the average distance an electron can travel before its phase becomes randomized.

Hikami-Larkin-Nagaoka (HLN) model [53], as a change in 2D magneto-conductivity ($\delta\sigma_{2D}$):

$$\Delta\sigma_{2D} = -\alpha \frac{e^2}{\pi h} \left(\Psi \left(\frac{1}{2} + \frac{B_\Phi}{H} \right) - \ln \left(\frac{B_\Phi}{H} \right) \right) \quad (1.2)$$

where the α coefficient indicates the type of localization (WL with $\alpha = -1$ and WAL with $\alpha = \frac{1}{2}$ per spin channel), Ψ is the digamma function, H is the transverse magnetic field, and $B_\Phi = \frac{h}{8e(L)^2}$ is the characteristic magnetic field which depends on the phase coherence length, which evaluates the amount of phase-shifting scattering centers in the system, working as a probe of film quality. More information about the origins of WAL can be found in [54].

1.4.3 SOT Topological Devices

The integration of TIs into magnetic memory devices is highly anticipated due to their previously mentioned electronic properties. Experimental evidence of one of the most remarkable features, spin-moment locking, came in a work developed by Tian *et. al* [55], by fabricating a lateral spin valve. The device consisted of a 12 nm exfoliated Bi_2Se_3 flake, contacted by 40 nm ferromagnetic Ni electrodes, defined by e-beam lithography and deposited by e-beam evaporation. The separation between the contacts and their widths range from 200-800 nm, ensuring that the polarized spin currents generated by spin-momentum locking are preserved over the expected spin diffusion length of the TI material (nanometer range [56]).

The intention of the authors was to apply a DC bias current through the TI flake and measure the voltage at the two FM electrodes, while sweeping an in-plane magnetic field. Their results show an asymmetry in MR profile as a function of the field, as show in fig.1.11. These findings are consistent with the existence of a spin polarization in the channel that is maintained with the reversion of the applied magnetic field. Given a certain current direction and respective spin polarization, if the magnetic orientation is parallel to the spin current we have a low resistance state (fig.1.11.c) and f). Otherwise, if either one of this parameters is inverted, (i.e the magnetic field or the applied current) we have a high resistance state (fig.1.11.d) and e)).

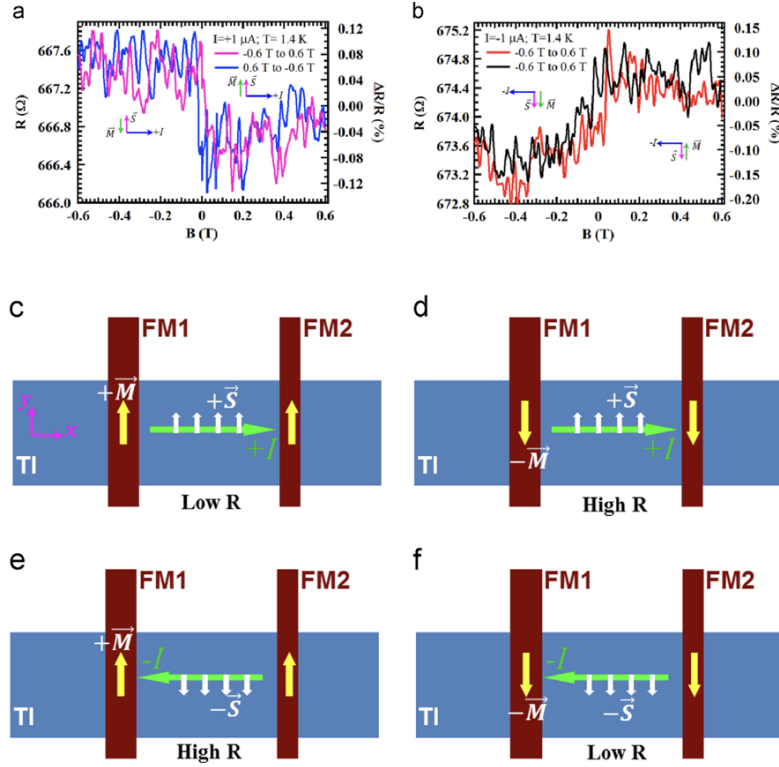


FIGURE 1.11: a) MR results for positive applied bias current and b) negative bias current. c), f) scheme of low and high (d), e) resistance states and correspondence to relative current to magnetization orientation c),d),e),f).

These results highlight the potential of spin-polarized currents in spintronics, as they enable the development of devices with tunable resistance states based on the relative alignment of spin and magnetic orientations. Consequently, in STT-MRAM, for example, the need for a magnetic layer that serves as spin polarized disappears, since TIs can produce spin-polarized currents directly. This leads to more compact designs and increasing data storage density, making them highly promising for future memory technologies.

On the other hand, in SOT-MRAM, two of the most important parameters are the **spin-orbit efficiency** $\theta_{SOT} > 1^*$ and the **critical density of current** J_w , which represents the current density required to reverse the magnetization orientation. A straightforward approach to study spin-orbit torques (SOTs) involves using a Hall bar geometry, as shown in fig. 1.12. Typically, the characterization of structures aimed at investigating SOTs follows a consistent pattern. This analysis is usually conducted through: measurements of the anomalous Hall resistance (fig.1.12.b)); SOT-driven magnetization switching by sweeping a DC current (fig.1.12.c)); and second harmonic Hall measurements (fig.1.12.d)).

*Also known as the Spin Hall angle θ_{SH} .

Once the critical current density (J_c) is determined, magnetization switching can potentially be achieved by applying current pulses of specific durations (τ). An example of such characterization is presented through the work of Khang *et al.* [57], illustrated in fig.1.12. The authors used a type Z geometry for SOT switching, where they employed BiSb, the first discovered 3D TI, on top of a ferrimagnetic CoTb layer with perpendicular magnetic anisotropy (PMA), with a Pt interface layer. Their aim was to demonstrate that non-epitaxial sputtered BiSb, even with bad crystal quality and low electrical conductivity, could provide ultralow power magnetization switching, proving its value towards realistic spintronic devices. They reported $J_c^{BiSb} = 3.5 \times 10^5 \text{ A/cm}^2$, at $H_x = +500 \text{ Oe}$, which is significantly lower than $J_c^{Ta} = 8 \times 10^6 \text{ A/cm}^2$ for the same heterostructures using Ta as the SOT material. Second harmonic measurements showed an effective spin Hall angle of $\theta_{SOT} = 3.2$, outperforming several heavy metals and topological insulators with similar CoTb ferrimagnetic layers [58].

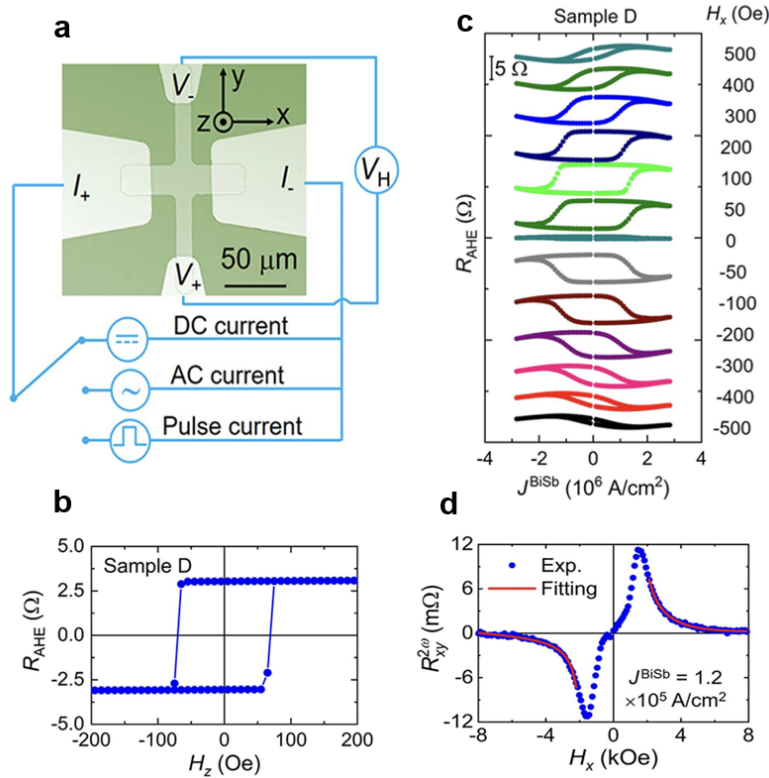


FIGURE 1.12: Work performed by Khang *et al.* [57]. a) 25-μm Hall Bar device. b) Anomalous Hall resistance measurement by sweeping a perpendicular magnetic field. c) Anomalous Hall resistance vs DC current sweep. d) Measurement of second harmonic.

Concerning other materials, Shao *et al.* demonstrated the efficiency of $(\text{BiSb})_2\text{Te}_3$ in ferromagnetic heterostructures of CoFeB, with perpendicular magnetic anisotropy (PMA), at room temperature. They employed a switching current of 4.5 mA, in a 20-μm width Hall

Bar device, what resulted in a density current as low as $J_{sw} = 3.0 \times 10^5 \text{ A/cm}^2$ [59]. Also, second harmonic measurements detailed a spin Hall angle of $\theta_{SOT} = 2.66$.

Despite their high resistivity, topological insulators display large values of θ_{SOT} , comparatively to other materials, as illustrated in fig.1.13. This enhancement is, of course, justified by their band structure that imposes electrons with spin-momentum locking, improving the charge to spin conversion.

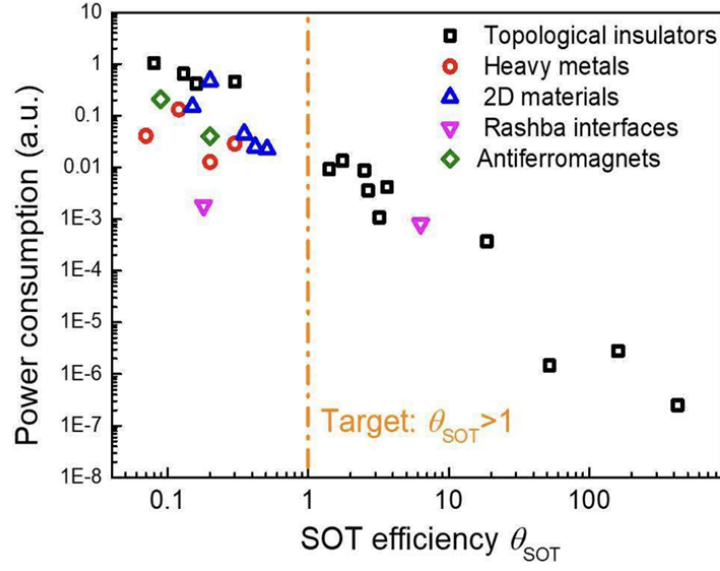


FIGURE 1.13: Comparison of SOT efficiency of several materials and respective power consumption. Reproduced from [43].

Another important research direction is respecting the implementation of field free switching of MRAM cells with PMA. As mentioned previously this task can be accomplished by combining SOT and STT mechanisms in a single device. Nonetheless, other forms include the use of van der Waals magnetic materials [60], employing an antiferromagnetic layer [61], tilted anisotropy [62], and incorporating an additional in-plane magnetic layer in the MTJ structure [63].

2 Fabrication of Magnetic Thin Films

2.1 Starting point

As previously mentioned, one of the primary objectives of this work is to fabricate logic devices using simple bilayer structures. These structures require one layer to be a highly efficient spin-orbit torque (SOT) material and the other to be a ferromagnetic material. For this purpose, we have selected Bi_2Te_3 , a topological insulator previously studied by our research group, as a potential efficient SOT generator. Additionally, we chose FeCo as the ferromagnetic material, which, contrarily to Bi_2Te_3 , has not been studied before in its thin film form by our research group.

In fig.2.1, some of the important results obtained during a fellow researcher master thesis [64] are illustrated. These data essentially demonstrate the effect of thermal annealing on DC Magnetron Sputtered Bi_2Te_3 thin films, concerning the electrical and magnetotransport properties.

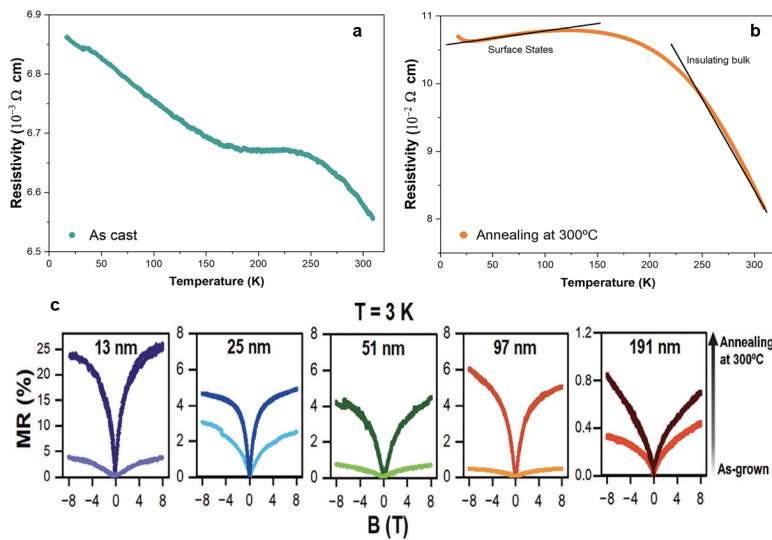


FIGURE 2.1: Resistivity as a function of temperature for two 97 nm samples: a) without thermal annealing and b) annealed at 300°C; c) Magneto-conductivity for various thicknesses of sputtered Bi_2Te_3 thin films, with and without annealing [64, 65].

Upon analysis of the data, there is evidence of the possible topological regime*, tuned through thermal annealing:

*To establish the confirmation of a topological regime, characterized by the presence of surface states, a more specialized technique as ARPES would have to be conducted.

- **Electrical Transport Transition** : The temperature-dependent conductivity measurements of two 97 nm samples—representative of the as-cast and optimized thin films—reveal a significant evolution in the intrinsic transport behavior. In fig.2.1.a), the as-cast sample exhibits characteristics similar to an n-type semiconductor. However, after annealing (fig. 2.1.b), the sample shows a markedly different response. Upon lowering the temperature, there is a transition from a bulk-dominated transport regime to a finite bulk population that allows metallic surface states to emerge as the dominant transport channel.
- **Enhanced Magnetotransport** : In fig.2.1.c), the results for the MR% at various thicknesses of Bi_2Te_3 , at 3K, are presented. We observe the presence of the WAL effect, a signature of a possible topological regime (or at least of a material with high SOC). Two main conclusions can be drawn from the data: first, for each thickness, there is a noticeable increase in the MR% after annealing, showing an enhancement of the topological properties. Second, as the film thickness decreases, the MR% increases for both the as-grown and as-cast samples. This increase may be attributed to the higher surface-to-bulk ratio in thinner films, leading to a stronger metallic contribution from surface states.

At this stage, progress towards realizing a potential logical device or magnetic sensor has been made: we have identified signatures of a topological insulator material, that might promise large SOT efficiencies. The next step requires the incorporation of a ferromagnetic material, which is where our work begins.

2.2 Magnetron Sputtering

To drive a coherent work with the previous mentioned results for sputtered Bi_2Te_3 samples, the system for deposition chosen was the same utilized for the TI. This way, we can follow a more industrial approach by doing a sequential deposition, where on the same chamber we have different target materials in various sputtering heads.

As a result, all thin film samples were fabricated through DC Magnetron-Sputtering (fig.2.2.a)) on rigid silica glass substrates. We started by fabricating 110 nm FeCo reference samples for morphological and magnetic characterization of the material, advancing later to thinner FeCo/ Bi_2Te_3 heterostructures of 11nm/7nm with and without annealing *.

*Annealing at 300 °C, 1 hour, 150 mmHg Argon atmosphere, without applied magnetic field. See Appendix A for information about the annealing procedure.

For the target of FeCo, pieces of a commercial FeCo foil were cut according to the magnetron heads shape, as illustrated in fig.2.2.b). In fig.2.2.c), it is presented a test sample of deposited FeCo(110)/Bi₂Te₃(14) nm bilayer on glass substrate following a sequential deposition.

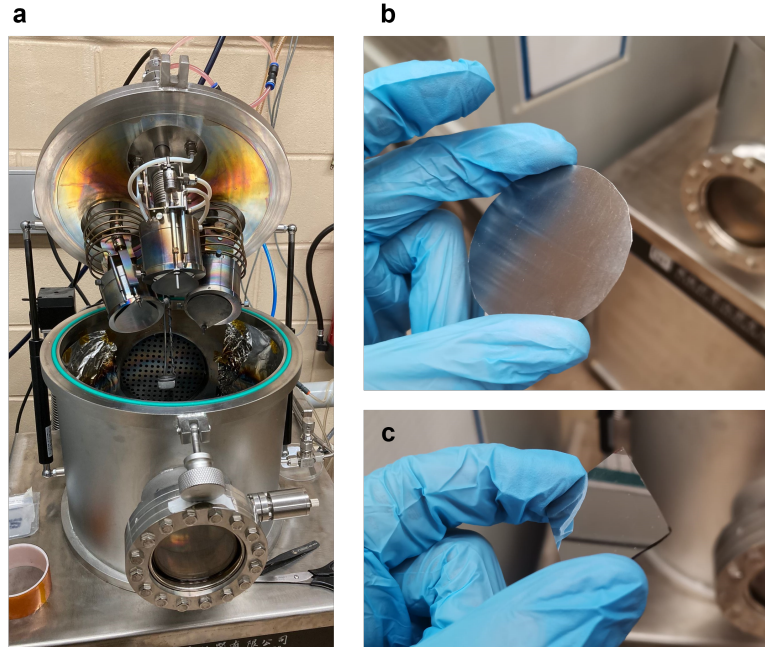


FIGURE 2.2: Magnetron Sputtering chamber a), FeCo target b) and deposited FeCo/Bi₂Te₃ thin films in glass substrate c).

Experimental details: FeCo and Bi₂Te₃ thin films were fabricated using a *CY Scientific Instrument* Sputtering device, under constant deposition conditions: 20 mA of applied current, Ar flux of 50 sccm, and table rotation speed of 60 rpm. The deposition process only started when the pressure inside the chamber achieved values inferior to 9×10^{-4} Pa. Prior to deposition, the substrates were washed with commercial dish soap to remove any grease. Then thoroughly rinsed with acetone, followed by a rinse with isopropanol.

2.3 Morphological Characterization

2.3.1 X-Ray Reflectometry

To determine the thickness of the FeCo thin films X-Ray Reflectometry (XRR) was employed. The technique is widely used to analyze surfaces, thin films, and multilayered structures, as it provides a highly precise evaluation of interface effects. In fig.2.3, is represented the XRR patterns obtained for a 5 min deposition of FeCo by DC Magnetron

sputtering. A modified version of Bragg's law (eq.2.1) was used to calculate the thickness from the position/spacing of the fringes [66]:

$$m = -\frac{1}{2} + \frac{2t}{\lambda} F(\theta_m) \quad (2.1)$$

where θ_m is the angle of the fringe maximum and m its order index; θ_c is the critical angle at which the fringes become visible. The wavelength of the X-ray radiation is given by the λ parameter, t is the film thickness and $F(\theta_m) = \sqrt{\cos^2(\theta_c) - \cos^2(\theta_m)}$.

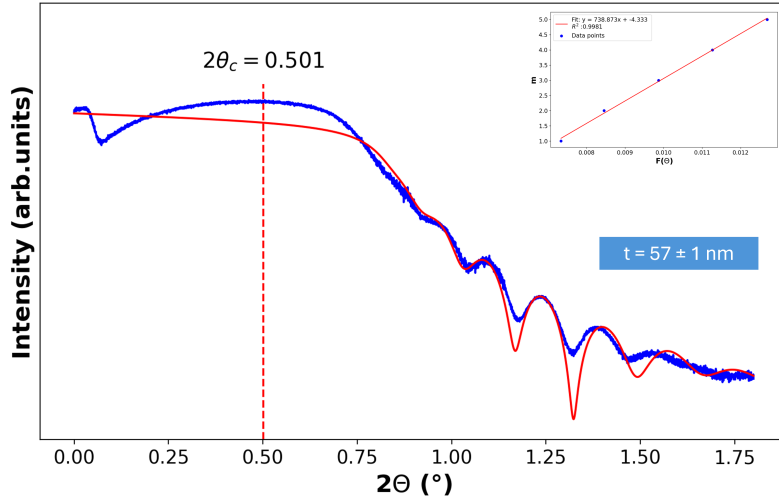


FIGURE 2.3: XRR spectrum and fitting of 5 min magnetron sputtering deposition of FeCo.

By performing a linear regression (inset graph on fig.2.3) the value of the thickness obtained for a 5 min deposition of FeCo was $t = 57 \pm 1$ nm, yielding a rate deposition of $r = 11.4$ nm/min .

Experimental details: A Rigaku SmartLab Diffractometer was used to collect the XRR patterns using the Parallel-Beam (PB) geometry, in an angle range of 0-6°. The system operates at a 9 kW power, 45 kV and 200 mA. The X-Rays were obtained using Cu K α radiation with a wavelength of $\lambda = 0.15404$ nm.

2.3.2 X-Ray Diffraction

X-Ray Diffraction (XRD) was used to study structural, preferential, crystal orientations, and grain size of the sputtered thin films, alongside with the original FeCo target, which spectra are shown in fig.2.4.

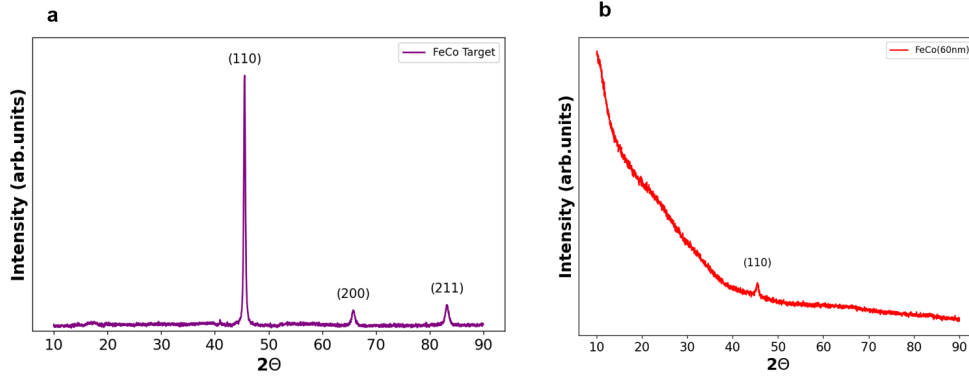


FIGURE 2.4: a) XRD spectrum of original FeCo target and b) XRD spectrum of sputtered 110 nm FeCo thin film.

Comparing the XRD spectrum of the target with the sputtered thin film, we can observe a broadening in the intensity of the peak around 45°, corresponding to the crystallographic plane (110). This enlargement of the peak's width for the 110 nm thin film can be seen as the presence of amorphous formations. In its turn, this implies that from all the deposited material not all will contribute to the magnetic behavior of the samples. The remaining peaks displayed at the target's spectrum are eliminated or in alternative, obscured by the background noise.

The crystallite size of the observed Bragg peaks was determined through the Debye-Scherrer equation:

$$D = \frac{k\lambda}{\beta \cos\theta} \quad (2.2)$$

where β represents the broadening (full width at half maximum) of the Bragg peaks, k is the Scherrer constant (shape factor $k = 0.9$ assuming spherical crystals) and $\lambda = 0.15414$ nm the incident X-ray wavelength. *Origin 2024* software was used to fit the XRD spectrum of the FeCo (110nm) sample, obtaining the peak center positioning and FWHM using a gaussian fit: $2\theta = (45.46 \pm 0.01)^\circ$ and $\beta = (0.80 \pm 0.03)^\circ$. To note that usually one has to consider a correction based on instrumental broadening* as $\beta = \sqrt{\beta_{observed}^2 - \beta_{instrumental}^2}$. However, in this case we will consider this parameter negligible. Converting both measures to radians, the crystallite size according to eq.2.2 is $D = (10.8 \pm 0.4)$ nm. As a result from this analysis, our material instead of crystalline thin film, it is rather composed by nanocrystals embedded on a matrix of non-magnetic material.

*Instrumental broadening refers to the broadening of diffraction peaks in XRD patterns that arises from the limitations and imperfections of the XRD instrument itself.

Experimental details: The diffraction patterns of the sputtered samples were acquired from XRD analysis, using the *Rigaku SmartLab Diffractometer* in the Bragg-Brentano ($\theta - \theta$) geometry, in an angle range of 10-90 °. The X-Rays were obtained using Cu $K\alpha$ radiation with a wavelength of $\lambda = 0.15404$ nm. The system operates at 9 kW power, 45 kV and 200 mA.

2.4 Magnetic Characterization

2.4.1 SQUID Magnetometry

We studied the magnetic characteristics of FeCo thin films by employing a Superconducting Quantum Interference Device (SQUID) magnetometry technique.

During these measurements we were interested in determining the magnetic nature of our sputtered FeCo thin films, that were suspected to be ferromagnetic. Additionally, we wanted to assess the effect of a TI layer on the overall magnetic response of the heterostructure.

To this end, we measured, at 300 K, the in-plane magnetic moment of FeCo(110), FeCo(11)/Bi₂Te₃(7) and annealed FeCo(11)/Bi₂Te₃(7) (nm), as illustrated in fig.2.5.a). The magnetic properties of each sample are present in table 2.1.

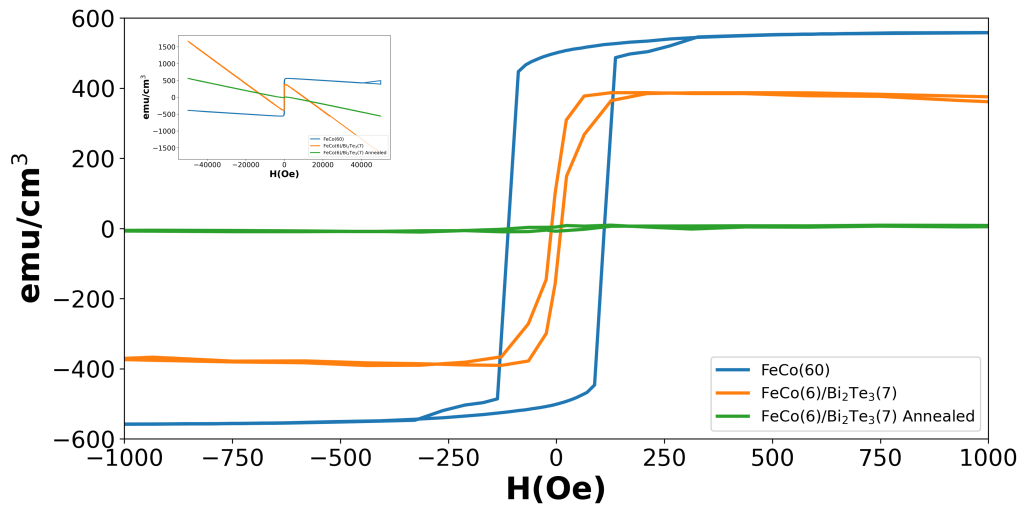


FIGURE 2.5: In-plane $M(H)$ loops for FeCo(110nm) and FeCo(11nm)/ Bi₂Te₃(7nm), with and without annealing. On the inset graph it is represented the full range of magnetic field (-5T to 5T), in which the data were collected.

The hysteresis curves in fig.2.5, measured under an in-plane magnetic field, suggest the presence of an easy-plane. To determine if the material is in-plane isotropic, we would have to perform the same measurements for various directions within the sample's plane.

However, due to the aspect ratio of the cut samples (see experimental details at the end of the subsection), and being the longer axis aligned with the applied magnetic field, we infer the potential presence of an easy axis along this direction. Moreover, since no magnetic field was applied during deposition and the FeCo thin films exhibit an amorphous structure, as indicated by the XRD results, the primary factor contributing to a preferred magnetic direction is likely the sample's geometry.

TABLE 2.1: Magnetic properties of sputtered thin films

Sample (nm)	H_c (Oe)	M_s (emu/cm ³)
FeCo(110)	111.20	558.3
FeCo(11)/ Bi ₂ Te ₃ (7)	11.06	388.1
FeCo(11)/ Bi ₂ Te ₃ (7) Ann.	NA	NA

By observing the inset graph on fig.2.5, that represents the measured signal for the entire field range, the incorporation of Bi₂Te₃ led to great diamagnetic contributions at large magnetic fields. On the other hand, through the values presented in table 2.1, we observe a decrease on both the material's coercivity and saturation magnetization, from $H_c = 111.20$ Oe to $H_c = 11.06$ Oe, and from $M_s = 558.3$ emu/cm³ to $M_s = 388.1$ emu/cm³, respectively. These low values for H_c and M_s point to the soft ferromagnetic nature of the produced samples.

The result of annealing the heterostructure is an almost complete elimination of the magnetic behaviour. These results might indicate a possible diffusion of Bi₂Te₃ into FeCo, changing lattice configuration and eventually leading to almost complete loss of ferromagnetism behaviour. Morota *et al.* [67], when annealing sputtered-grown bilayers of CoFeB with Sb₂Te₃ (also a topological insulator material), came to the conclusion that these two materials react at the interface, resulting in a loss of the ferromagnetic properties of the CoFeB layer. For Bhattacharjee *et al.* [68], by studying the crystalline structural disorder of TI on the interfacial and magnetic properties of sputter-deposited Bi₂Te₃/Ni₈₀Fe₂₀, they found that Ni, and a smaller amount of Fe, diffuse across the interface and react with Bi₂Te₃, leading to a general loss of the permalloy's magnetic moment.

Also, compared to the values reported in the literature for sputtered FeCo thin films, the saturation magnetization (M_s) of our thin films shows a significant deviation. Literature values for sputtered FeCo thin films typically range from 15 000 emu/cm³ to 23 000 emu/cm³ [69–71], while ours are two orders of magnitude lower. The reason for this can be related to the presence of an amorphous matrix that does not contribute to the ferromagnetism

of the FeCo thin films. Also, the further decrease on the M_s is a consequent of Bi_2Te_3 incorporation.

Experimental details: For the SQUID measurements, the samples were cut into rectangular shapes with lengths of 0.9–1.5 cm and widths of 0.5 cm. The samples were secured inside plastic straws using linen and paper, with holes made at the straw's center to hold the samples in place and prevent movement. The SQUID data was collected by performing DC scan in a Magnetic Property Measurement System (MPMS) from Quantum Design. The measurements were conducted at room temperature, in a range of applied in-plane magnetic field from -5 T to +5 T, in steps of 100 Oe/s.

2.4.2 VNA Ferromagnetic Resonance

For further magnetic characterization, specially in terms of magnetization dynamics, a Vector Network Analyzer (VNA) technique was used. The full description of this technique can be found in Appendix B.

For each sample, the resonance frequency, f_{res} , for a given applied external field H_{ext} was collected, by obtaining the minimum value of f in a single FMR spectrum. In fig.2.6, some of the FMR spectra for the 110 nm FeCo reference sample are presented.

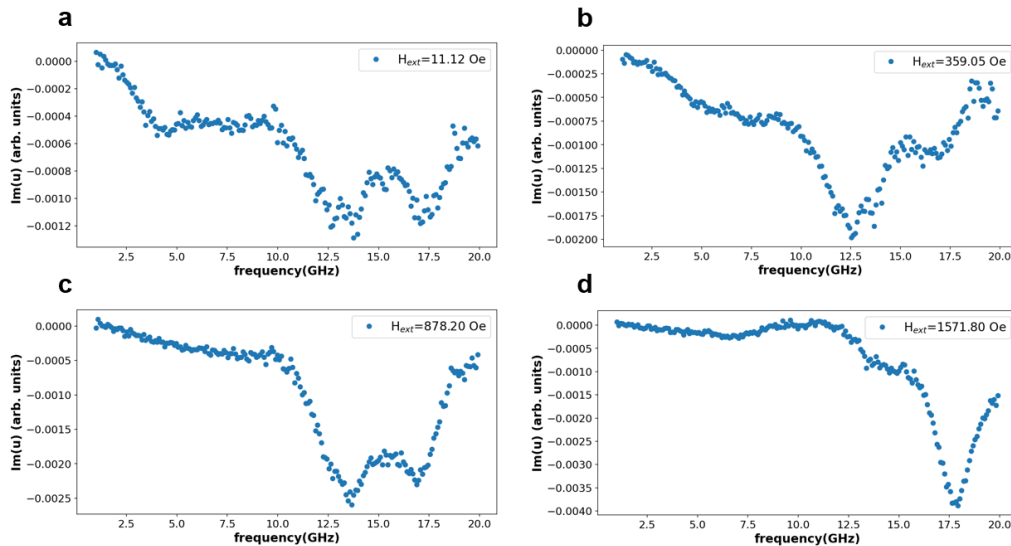


FIGURE 2.6: FMR spectra for FeCo(110) nm at different external magnetic fields H_{ext} .

We can observe broad and complex FMR spectra, opposite to the usual signals observed for good crystalline ferromagnetic materials [72, 73], that can be usually fitted through a

Lorentz shape curve. The complexity of these curves may be related to the presence of nanocrystals in an amorphous matrix. For example, in fig.2.6.a) and c), two resonance peaks are observed. This can be attributed to clusters of nanocrystals with different sizes, leading to additional resonance peaks caused by distinct local demagnetizing fields [74]. Additionally, such behavior can be also attributed to non-uniform modes, alongside the main resonance mode, resulting from the random orientation of nanocrystals with varying easy axis [75], since during deposition no external magnetic field was applied to determine a preferential magnetization direction.

Given the complex nature of the curves, the resonance frequency was not determined by performing the traditional Lorentz fit, but rather by taking the minimum of each spectrum, for a given field. Fig.2.7, illustrates the values of f_{res} obtained for each applied field, from 10-5000 Oe. Initially, we attempted to measure three samples: FeCo(110), FeCo(11)/Bi₂Te₃(7) and annealed FeCo(11)/Bi₂Te₃(7) (nm). However, for the latter two samples, the signal intensity was insufficient for accurate measurements, as illustrated in fig.2.7.c) and fig.2.7.d). To overcome this limitation we measured an additional thicker sample of FeCo(110)/Bi₂Te₃(14) (nm) (fig.2.7.b)), in order to study the effect of the topological insulator on the magnetization dynamics of the FeCo thin film.

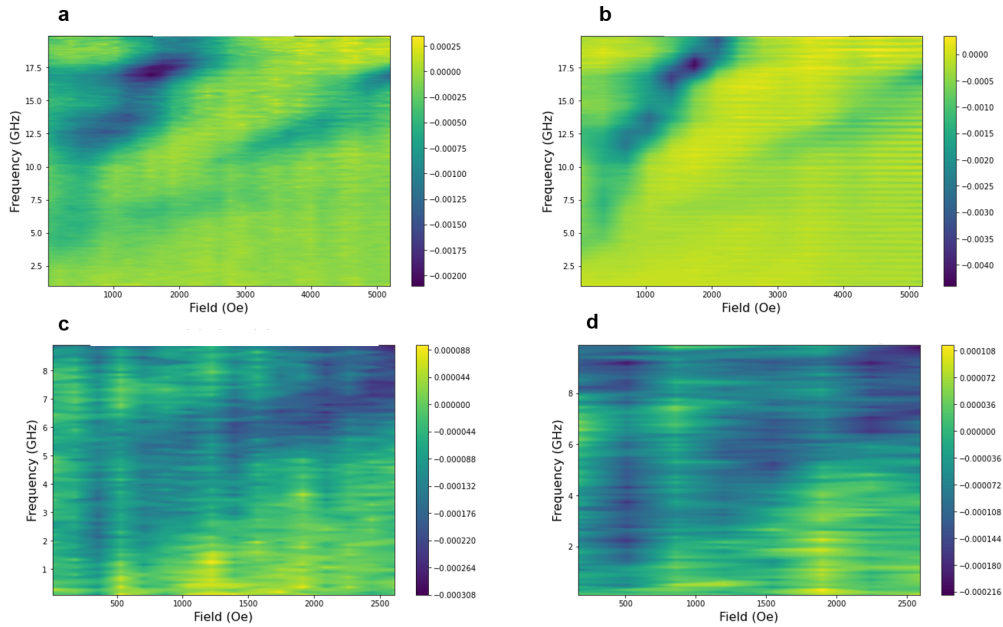


FIGURE 2.7: Counterplots of Resonance frequency, f_{res} for each applied magnetic field H_{ext} for a) FeCo(110), b) FeCo(110)/Bi₂Te₃(14), c) FeCo(11)/Bi₂Te₃(7) and d) annealed FeCo(11)/Bi₂Te₃(7) (nm).

Typically, to analyze such results the Kittel's formula [76] for thin films with an in-plane uniaxial anisotropy can be used. Ferromagnetic resonance parameters such as the Landau-Lifshitz damping constant and in-plane anisotropy can be obtained from this analysis. However, in our case, when attempting to fit the data shown in fig.2.7.(a) and fig.2.7.(b), using Kittel's formula, the fit failed to converge. Instead, the results closely resemble those observed for ferrimagnetic nanoparticles, particularly in terms of the complexity of the FMR spectrum [74, 75]. Additionally, the f_{res} vs. H_{ext} curves exhibit a more linear relationship, similar to that measured in ferrimagnetic nanoparticles, rather than the elliptical behavior predicted by Kittel's model.

Experimental details: The VNA-FMR measurements were conducted in the 1–20 GHz frequency range, employing a coplanar waveguide (CPW) connected to a Vector Network Analyzer. Samples were positioned on the CPW, and the complex S21 parameter was measured across the entire frequency range as a function of the external magnetic field.

2.5 Conclusions

Through XRR measurements it was possible to determine the deposition rate of DC Magnetron Sputtered FeCo thin films. All morphological and magnetic characterization data point for the amorphous behavior of the FeCo thin films. The XRD analysis suggests the presence of nanocrystals with a calculated spherical diameter of $D = (10.8 \pm 0.4)$ nm. From the SQUID magnetometry data we observe low magnetization of saturation, compared to other FeCo thin films [69–71], supporting the hypothesis that the majority of the deposited material is amorphous. The data also reveal that the inclusion of a Bi_2Te_3 layer on the FeCo thin films, leads both to a reduction in the coercive field and saturation magnetization of the material. Notably, an annealing process, which typically enhances the topological properties of Bi_2Te_3 [65], leads to the suppression of ferromagnetic behavior in the FeCo/ Bi_2Te_3 heterostructure. Finally, the complex FMR spectrum observed in FeCo thin films is consistent with those reported for ferrimagnetic nanoparticles [74, 75], further supporting the existence of nanocrystals within the thin films.

3 Micro-patterning of SOT heterostructures

This chapter aims to describe the journey toward creating realistic, functional, devices. It covers the micro-fabrication techniques used and the mistakes encountered along the way. The chapter is divided into two sections, each focusing on a different generation of devices. In the first section, test devices are fabricated to observe the possible experimental limitations. This section is primarily dedicated to optimizing the lithography process and concludes with the fabrication of actual Hall bars. The second section covers new design devices with incorporated improvements, informed by the lessons learned from the first generation's errors.

Additionally, the fabrication of micro-devices in two different environments will be discussed: one in a clean room and the other in a standard laboratory.

3.1 Optical Lithography Methodology

Optical lithography is a technique used to transfer desired patterns onto a substrate. There are many variants of this kind of lithography process. Nonetheless, we will be describing only the general procedure used throughout this thesis, illustrated in fig.3.1, following a top-down approach of micro-lithography. The experimental steps to micro-fabrication are the following:

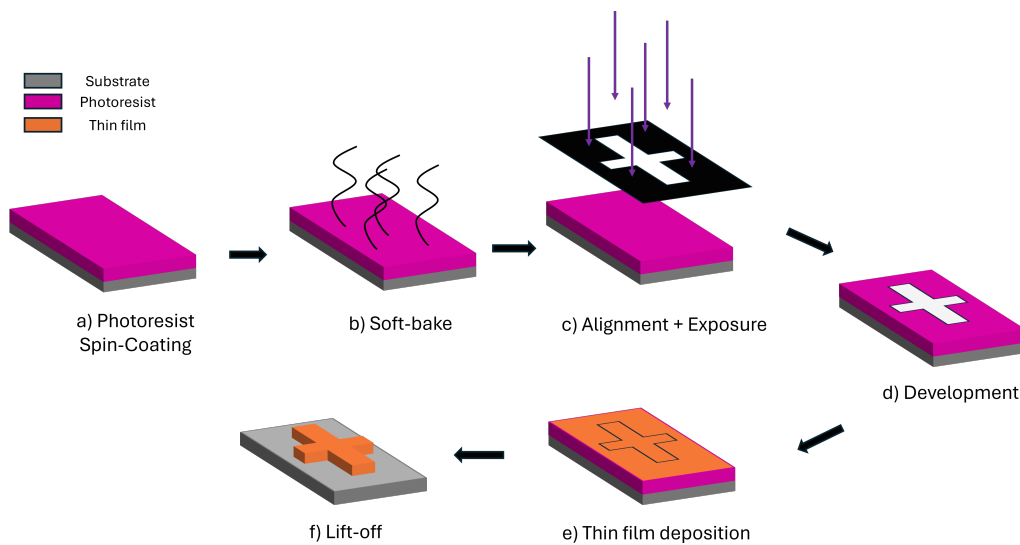


FIGURE 3.1: Scheme of optical lithography procedure.

- **Prior Step:** Before initializing the lithography process the cleaning of the substrates must be executed. For glass substrates, we start by washing them with commercial dish soap to remove any grease. Next, they are rinsed with acetone, followed by isopropanol. After rinsing, the substrates are dried using a compressed nitrogen gun if working in a clean room, or they are left to air dry if working in a benchtop setup.
- **a) Photoresist Spin-Coating:** A small amount of photoresist * is deposited onto the center of the substrate, until it covers the entire surface. This is done manually using a pipette. In our case, we used the MICROPOSIT S1818 G2 positive photoresist. A spinner is used to fix the substrate and accelerate it at 1000 RPM/s, until it reaches steady state speed at 2500 RPM and maintains it for 35s. This results in a $2.5\ \mu\text{m}$ thin photoresist layer.
- **b) Soft Bake:** After coating, a soft bake is used to evaporate most of the solvents in the photoresist. This step enhances the adhesion to the substrate and by partially curing the photoresist, soft-baking reduces its flow during the exposure step, which helps maintain the integrity of fine features. The soft-bake was set to 115°C , during 1 min, using commercial hot-plates.
- **c) Alignment and Exposure:** The next step involves aligning a patterned mask with the substrate and expose it to UV light, to make the pretended regions soluble. In our case, the regions of interest in the mask are transparent while the rest of the area is opaque. During the exposure, the substrate and the mask are in direct contact. For that reason, the features on the mask must be identical in size to the ones we wish to produce.
- **d) Development:** After exposure, the substrate is immersed on solution of Microposit 351 developer, and agitated to remove the photoresist in the target areas.
- **e) Thin film Deposition:** To deposit thin film materials, a DC Magnetron Sputtering technique is employed.
- **f) Lift-off:** To reveal the patterns, the substrate is immersed in a ultrasonic bath of acetone, during 5 min is used for this purpose. As the photoresist dissolves, it lifts

*Light sensitive polymer that becomes soluble upon the actuation of a developer after being exposed to UV light. It can be either positive or negative depending on whether we want to remove the exposed or unexposed area, respectively.

off the overlying material, leaving behind the material only in the target areas. This creates the desired pattern on the substrate.

3.2 First Generation Devices

3.2.1 Mask Design and Production

Generally, photomasks can be either hard masks, made of glass or quartz, or soft masks made of flexible polyester films. Hard masks can provide longer use, no temperature induced expansion and solvent resistance. One way to create such masks is through the use of a direct writing machine. However, in this work, we opted for a more economical choice and acquired flexible polyester masks from a local screen printing store.

Since we had no information about the resolution of the prints made at the local screen printing store, our first step was to create simple designs to determine the smallest feature size we could achieve and gather details about the mask quality. To evaluate the quality of the masks the following parameters were used:

- **Resolution:** The ability of the photomask to accurately reproduce fine details and small features.
- **Critical Dimension (CD) Uniformity:** Consistency of the feature sizes across the entire photomask.
- **Defect Density:** The number and size of defects, such as pinholes, spots, or scratches, on the photomask.
- **Edge Roughness:** Smoothness of the edges of the patterns on the photomask.

To note that this is more of a qualitative assessment than quantitative. The drawings were made in AutoCAD and masks were elaborated in order for the devices and alignment marks to fit in a single glass coverslip (26 mm x 76 mm). A larger substrate, which would offer higher throughput in a single workflow, wasn't chosen due to malfunctioning of the substrate cutting machine. Consequently, glass substrates are cut manually with a tungsten pen, which often fails to cut in the intended direction, jeopardizing the created devices. Therefore, it is necessary to minimize the number of cuts and consequently smaller substrates help in that direction.

The alignment marks on the masks consist of four Greek crosses, with a width of 1 mm, as shown in fig.3.2. Each mask contains four target patterns and has two axes of symmetry:

one horizontal, between the two central crosses, and one vertical, between the two pairs of target patterns. These symmetry axes, combined with the four alignment marks, enable the substrate to be divided into smaller, alignable parts. This feature will be crucial for the annealing procedure (see Appendix A).

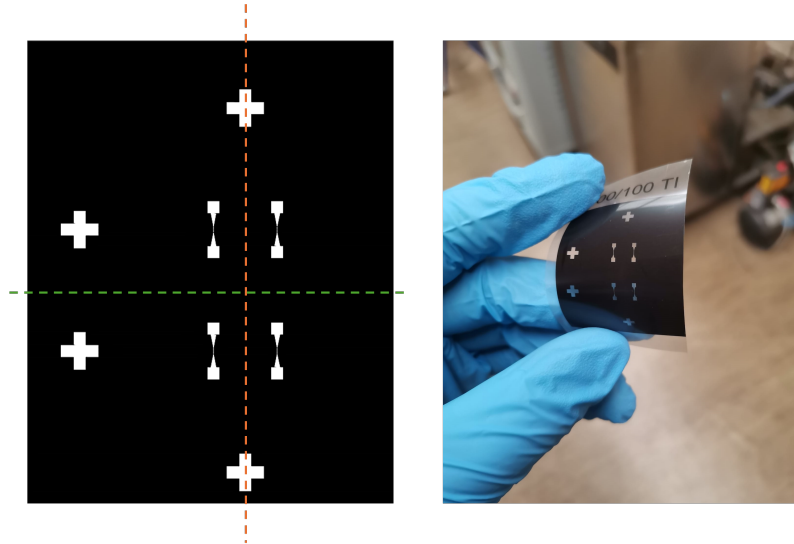


FIGURE 3.2: Left: Mask design elaborated in AutoCAD; in orange and green virtual symmetry lines. Right: Example of Printed mask.

As the first test design, two contact pads are connected by a central stripe - fig.3.3.a) - with variable width ($W = 100 \mu\text{m}$, $50 \mu\text{m}$) and length ($L = 1000 \mu\text{m}$, $500 \mu\text{m}$). Microscope observation revealed good resolution and critical dimension uniformity - fig.3.3.b). However, as shown in fig.3.3.c), which illustrates the mask pads at a higher magnification, there are defects in the form of spots, likely due to the polyester film itself.

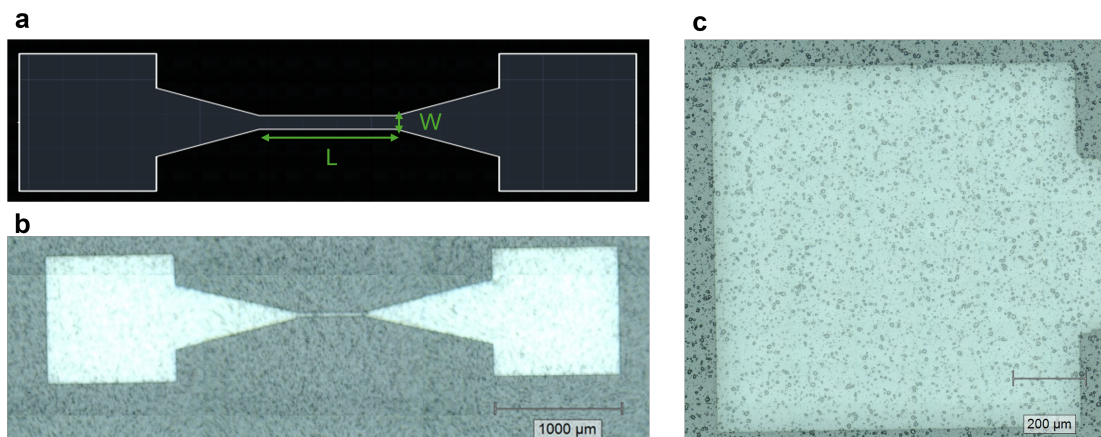


FIGURE 3.3: a) AutoCAD drawing of test device. b) Microscope observation of printed mask. c) Region of the contact pads of the printed mask, at a higher magnification.

An additional test pattern, found in Appendix C, was created in a second lithography process to test the alignment between the two masks.

Next, for intended device fabrication, Hall Bar (HB) patterns were created. The design is of a simple cross with four arms. Two structures were considered: a 20- μm width and a 100- μm width arm HB. As show in fig.3.4.a), for the design with the smaller feature (20- μm width arm), there is bad resolution, making it unfit for use. On the other hand, the 100- μm HB mask, in fig.3.4.b), showed good resolution, good critical dimension uniformity and good edge roughness, maintaining, nevertheless, the defect density of the previous test masks. Also, it was noted that the real mask feature size did not correspond to the one in the drawing. It was planned to create a mask with 100- μm width, instead we got 73- μm width. The discrepancy was later explained when we measured the width of a single line, which was set to zero linewidth in AutoCAD. However, in reality, lines have three dimensions and not one. Microscope observations revealed that the line width was 13 μm . Considering this, 26 μm (two lines) was subtracted from the intended feature width, resulting in the 73 μm width.

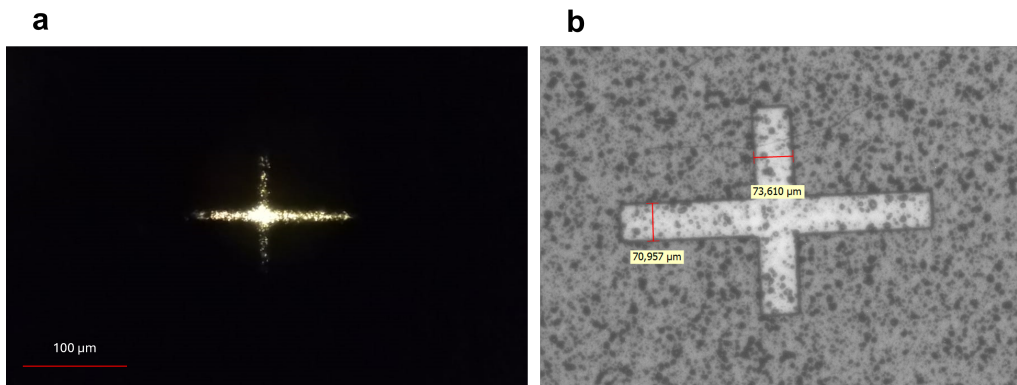


FIGURE 3.4: a) 20- μm width arm HB mask and b) 100- μm width arm HB mask

3.2.2 Benchtop Lithography Set-up

The next step was to test the feasibility of a lithography process using the polyester masks. For that, we also pretended to test the lithography process in a traditional laboratory, with a custom-built set-up. This system was already in use by some researchers at IFIMUP and provided a quick method to pattern simple, long structures in cases where the second alignment step could be easily performed. The lithography procedure encountered and already optimized for the set-up is presented in fig.3.5.

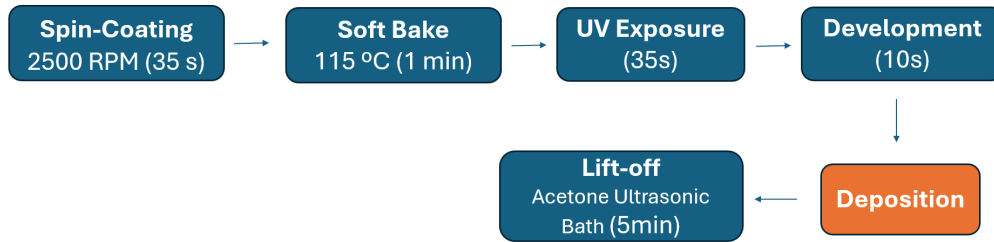


FIGURE 3.5: Lithography process encountered for Benchtop set-up.

As shown in fig.3.6.a), the setup includes a custom-built spinner station, for photoresist coating, controlled by an Arduino and powered by an external power source. The spinner is connected to an external vacuum pump to fix the samples. The Arduino is connected to a computer, allowing the programming of rotation parameters (2500 RPM) and ramp speed (1000 RPM/s) for the spinner. When in use, the spinner, like all experimental procedures involving photoresist, must be placed inside a fume hood due to the photoresist's toxicity to human health. A common hot plate is used for the soft-bake.

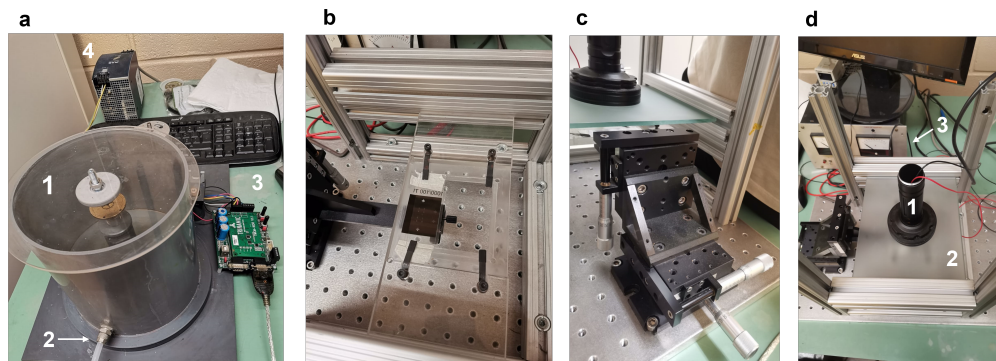


FIGURE 3.6: Benchtop Lithography set-up: Spinner Station. a) 1-Spinner, 2- Connection to vacuum pump, 3- Arduino, 4- Spinner motor. b) 1- Sample holder, 2- Mask holder. c) Micropositioners. d) 1- UV lamp, 2- Diffusive Glass, 3- External power source.

An acrylic support with clamps on the extremities - fig.3.6.b) - is used to fix the mask. Since the mask is made of flexible film, it had to be glued to a glass coverslip with tape. This ensures that during the contact with the substrate, the mask remains fixed and flat. To move the substrate and align it with the mask we have a station with micrometric screws - fig.3.6.c) -, that allows movement in the three spacial directions. Also, there is the possibility of rotation in the xOy plane.

The exposure is done with a UV lamp (365 nm) placed on top of a diffusive glass, as represented in fig.3.6.d), for 35 s. The diffusive glass ensures uniform illumination over the mask's surface. The distance between the mask's suport and the diffusive mirror had

already been optimized for the lithography process. For powering the UV lamp an external power source is used. The lamp turns on when the power source is activated by pressing a switch. It is important to use special UV-filtration glasses to protect the eyes when doing the exposure step.

For the development stage, a 1:4 solution of Microposit 351 and H₂O is used. The substrate is agitated in the solution during 10s. To stop the reaction, the substrate is immersed in a glass dish containing deionized water.

3.2.2.1 Errors and Adaptions

While for the test devices, the produced results using the lithography procedure described in fig.3.5, were reasonable, that was not the case for the Hall Bar devices. In fig.3.7.a), we have a microscope image of the photoresist after development, for the test device. After 10 seconds of development, the pattern in the photoresist exhibits good resolution and sharp edges. Following deposition and lift-off the material has clearly been deposited in accordance with the target patterns, as shown in fig.3.7.b).

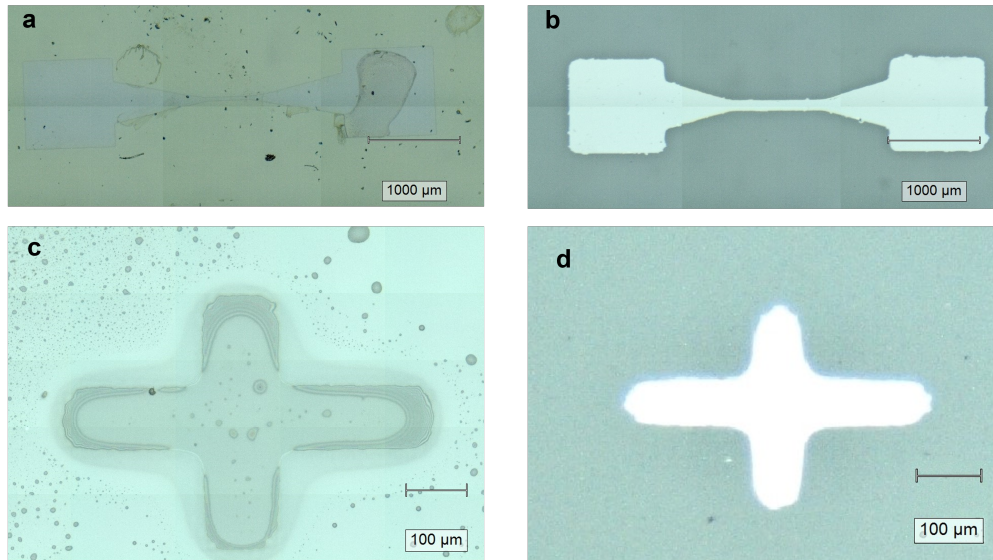


FIGURE 3.7: Microscope images: a) Test device Photoresist pattern after development. b) Test device after Bi₂Te₃ deposition and lift-off. c) Hall Bar photoresist pattern after development. d) Hall Bar after Bi₂Te₃ deposition and lift-off.

However, the same outcome was not observed for the Hall Bar devices. As shown in fig.3.7.c), which depicts a photoresist after 10 seconds of development, the edges are rounded, and the resolution is low, indicating underdevelopment. This issue is not related to the shape of the pattern but rather to the time of development for the pretended scale and resolution.

In fig.3.7.a), there appears to be no underdevelopment since the features exhibit good resolution. However, the pads in fig.3.7.b) are $1000\text{ }\mu\text{m} \times 1000\text{ }\mu\text{m}$ in size, so underdevelopment on the scale of less than $100\text{ }\mu\text{m}$, as seen in fig.3.7.c), is not relevant to the intended goal. In contrast, for patterns with a width of $100\text{ }\mu\text{m}$, such underdevelopment significantly impacts the resolution of the resulting devices. Therefore, while 10s seconds of development for long, large designs induces sub- $100\text{ }\mu\text{m}$ underdevelopments, that are not critical for the intended devices, for smaller patterns it has great influence.

Moreover, the analysis and observation of the features has to be taken carefully when dealing with micro-sized devices. In fig.3.8, it is represented two images of the same photoresist after development. The image on the left might suggest successful development. However, upon closer inspection, in fig.3.8.b) it's clear that underdevelopment has occurred. There is a gradient of photoresist along the edges of the pattern. This accumulated photoresist prevents the proper deposition of material in those areas, being washed away after lift-off.

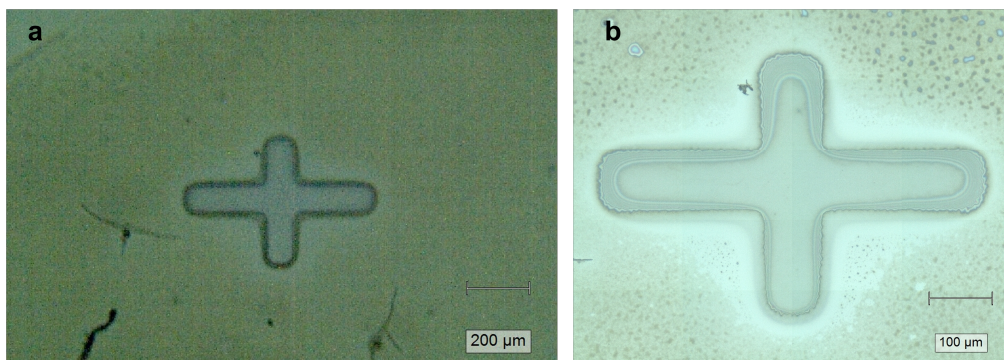


FIGURE 3.8: Microscope images: a) Photoresist pattern after development at $200\text{ }\mu\text{m}$ scale and b) Photoresist pattern after development at $100\text{ }\mu\text{m}$ scale.

As a result, the development time was increased to 20 seconds, resulting in the deposited material, in this case Bi_2Te_3 , conforming to the photoresist patterns after the development process.

3.2.2.2 Alignment

In fig.3.9.a), it is represented the AutoCAD drawing for the electrical contacts. While the alignment of the test devices was completed without using a microscope or camera, for the Hall bar devices, a camera was necessary for precise alignment. Otherwise, most of the lithography procedures would result in some form of misalignment, as observed in

fig.3.9.b). The camera was connected to the computer which allowed to see the alignment marks with some magnification.

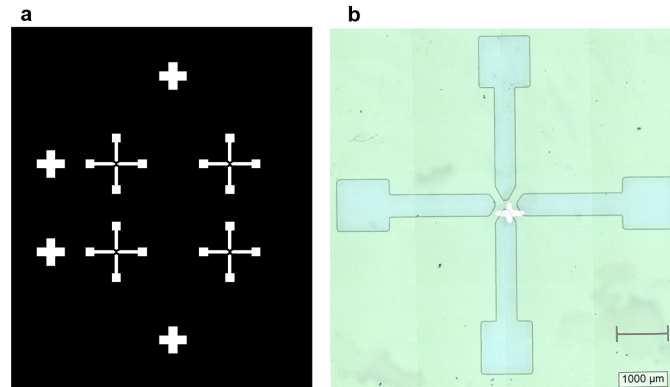


FIGURE 3.9: a) AutoCAD mask of contacts. b) Microscope image of patterned contacts in photoresist misaligned with deposited material from previous lithography process.

The electrical contacts patterns consist of $400\ \mu\text{m}$ width and 3 mm long stripes, with 1mm x 1mm pads at the ends. For all devices, we deposited 100 nm of sputtered Ag as the material for the electrical contacts.

The final parameters for the benchtop set-up are illustrated in table 3.1, with the optimized development time highlighted in yellow.

TABLE 3.1: Lithography process parameters for Benchtop set-up

Lithography Step	Parameters
Photoresist Coating	35s at 2500 RPM, 1000 RPM/s
Soft-Bake	115°C for 1 min
UV exposure	35s
Development	20s (1:4 V/V) Microposit 351/ H ₂ O
Lift-off	5 min acetone bath + isopropanol rinsing

3.2.3 Clean Room Fabrication

In a clean room the concentration of airborne particles, microbes, and parameters like temperature, humidity, and pressure are controlled to minimize contamination. In Appendix C are described common methods that allow the monitoring of these parameters.

In our case, the cleanroom used for this work was the one located in the Faculty of Sciences of the University of Porto, from MNTEC. Within this facility, two areas were primarily utilized: Lithography Room I and Lithography Room II.

The first room is classified as ISO 6 (Class 1000)*, which specifies that the concentration

*See Appendix C for information about cleanroom classification.

of particles larger than $0.5\ \mu\text{m}$ must be below 1000 particles per cubic foot of air[†]. This room features a mixed airflow system, combining turbulent displacement of contaminated air within the room with a laminar flow in the critical processing area, such as the workbenches. Inside one of these workbenches is a Spinner *PWM50* from *Headway Research Inc.*, along with several hotplates and nitrogen guns for substrate drying.

In the second room, classified as ISO 7 (Class 10,000), there is a *MJB3* mask aligner from *Karl Suss*, offering a line/space resolution of $1.5\ \mu\text{m}$ and alignment accuracies of $0.25\ \mu\text{m}$.

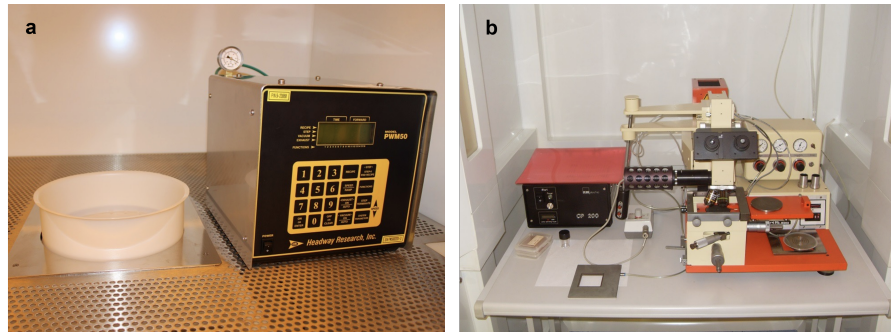


FIGURE 3.10: a) Spinner PWM50 in Lithography room I and b) Mask aligner in Lithography room II.

In both rooms, the temperature and humidity should be controlled. However, during the time of this thesis, the chiller was broken. Therefore, these two parameters were taken into account when deciding whether or not to conduct procedures in the cleanroom. Specially, if the relative humidity was above 60/65%, no experimental work should take place as it strongly influenced the photoresist performance, in terms of substrate adhesion and development rate [77].

3.2.4 Exposure Time Optimization

The lithography parameters used in the clean room are the same as those described for the benchtop setup in section 3.2.2, with one exception: the exposure time had to be optimized. Typically, exposure times in the clean room, using the MJB3 mask aligner, range from 5 to 10 seconds, for glass hard masks. However, using this standard time resulted in the features disappearing after lift-off. To address this issue, various exposure times were tested with $t = 20, 30, 35, 40\text{s}$, as shown in fig.3.11, for photoresist after development:

[†]Note that old FS 209E classes were measured in particles per cubic foot, while ISO classes are measured in particles per cubic meter of air.

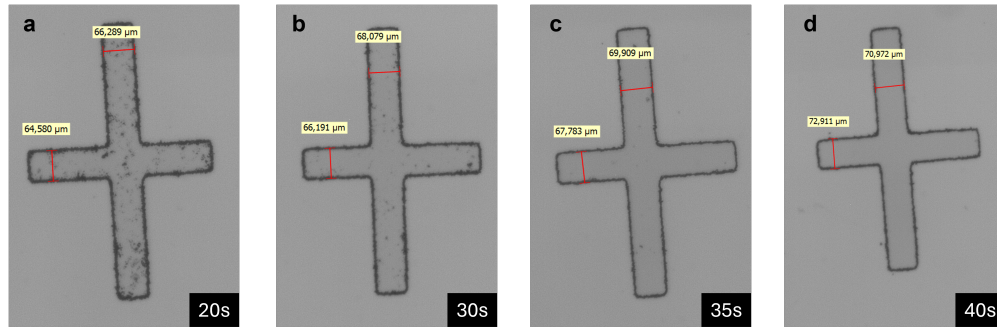


FIGURE 3.11: Microscope images of photoresist after development for exposure times of: a) 20s, b) 30s, c) 35s and d) 40s.

For exposure times of $t = 20s$ or lower, the photoresist prints the defects that we encounter on the polyester masks, discussed previously. Therefore, we might assume that the range of exposure time of $5s$ to $10s$ provides high resolution, since it can even print defects in the order of the $5-10 \mu m$. However, in our case the presence of these defects has a negative impact on the electrical transport of the deposited thin films, since most of the material vanishes after lift-off. The solution to this problem was to increase the time of exposure long enough in order to make the areas covered by the small spots sensible, and consequently be removed after development.

Regarding the alignment step for patterning the electrical contacts, several challenges arose during the process and are described in Appendix C.

The final parameters for the cleanroom are illustrated in table 3.2, with the optimized exposure time highlighted in yellow.

TABLE 3.2: Lithography process parameters for cleanroom

Lithography Step	Parameters
Photoresist Coating	35s at 2500 RPM, 1000 RPM/s
Soft-Bake	115°C for 1 min
UV exposure	40s
Development	20s (1:4 V/V) Microposit 351/ H ₂ O
Lift-off	5 min acetone bath + isopropanol rinsing

3.3 Second Generation Devices

3.3.1 Mask Design and Production

In the second generation of devices, instead of a four arm Hall Bar, we have now a different design with 6 arms, known as double cross or "1-2-2-1" Hall Bar, illustrated in fig.3.12. The fundamental difference from the previous design is the possibility of measuring both transverse and longitudinal resistances. Electrical current is employed through contacts 5 and 6, while tension is either measured transversely (between contacts 1 and 2, or 3 and 4), or longitudinally (between contacts 1 and 4, or 2 and 3).

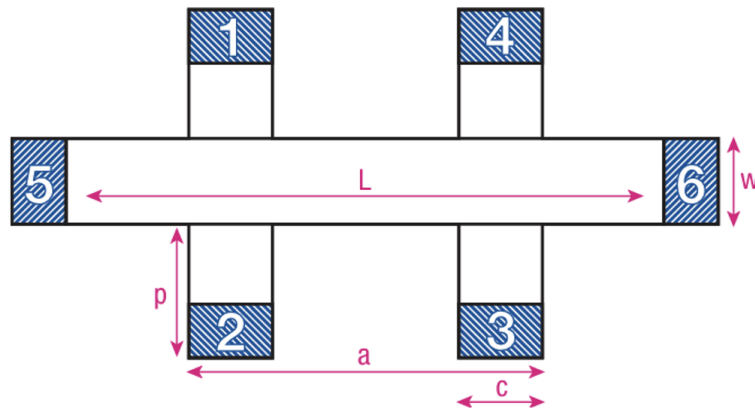


FIGURE 3.12: Double-cross Hall bar geometry: The structure consists of a channel with length L and width w , featuring four arms for voltage measurements. The distance between consecutive arms is denoted by a , while the perpendicular distance from the arms to the channel is p . The voltage contacts width is represented by c . From [78].

The aspect ratio of the Hall Bar is something to take into account when performing electrical measurements, since it can influence the deviation of experimental values from the ideal. The following aspect ratio [78] presents small deviations from the ideal: $p \approx c$, $c \leq w/3$, $L \geq 4w$.

3.3.2 Adaptions

We designed a double cross Hall bar with the parameters, as represented in fig.3.13.a), using AutoCAD.

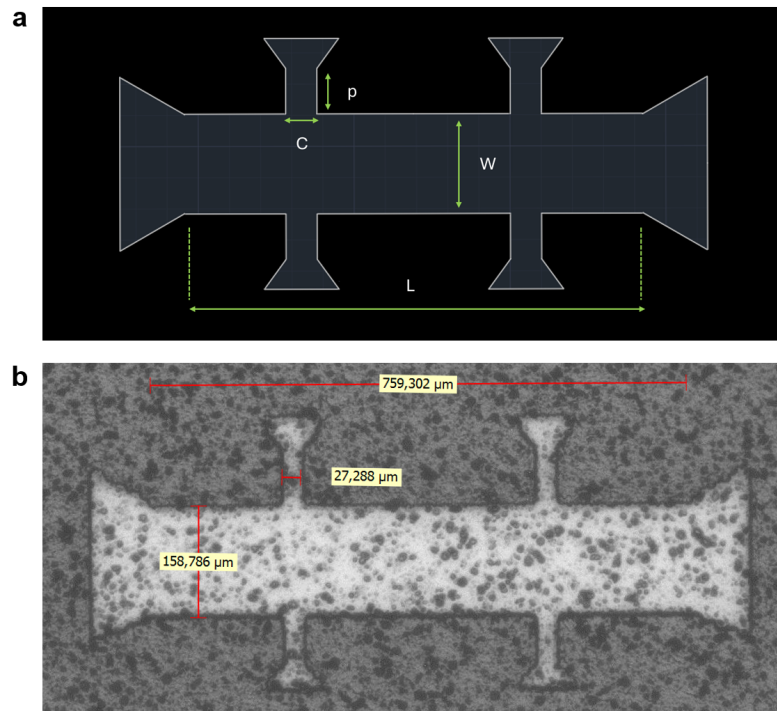


FIGURE 3.13: a) AutoCAD drawing of elaborated double cross Hall Bar of parameters $L = 734 \mu\text{m}$, $W = 160 \mu\text{m}$, $c = 50 \mu\text{m}$ and $p = 73 \mu\text{m}$; and b) Produced flexible polyester photomask.

Given the geometry chosen and informed by the errors committed on the first generation of devices, some adaptations were made in the mask design.

The first is the creation of additional, smaller, alignment marks. These consist of $250 \mu\text{m}$ width Greek crosses. By considering this additional marks, the misalignments that were not notorious with the 1 mm width Greek crosses, will now be visible and can provide an alignment with greater accuracy. This assumption is visible in fig.3.14, where the alignment marks, one with a width of 1 mm and the other with a width of $250 \mu\text{m}$, exhibit similar deviations from previous deposited marks. However, the deviation is more noticeable in the smaller Greek cross, what allows a better alignment accuracy.

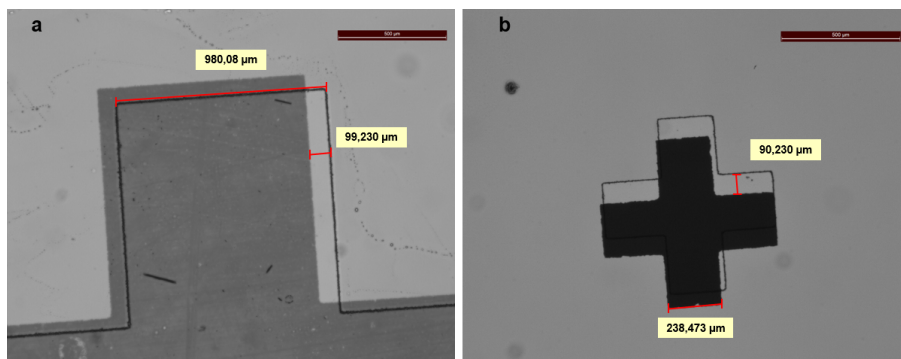


FIGURE 3.14: Microscope images of a) Second Alignment mark of 1 mm width and b) Second alignment mark of $250 \mu\text{m}$ width.

Another increment was a previous lithography step, where only the alignment marks are patterned. This way, by depositing a sufficiently thick material at this stage, typically 50 – 100 nm of Ag by DC Magnetron Sputtering technique *, we can address the issue mentioned earlier, where the alignment marks could not be visually detected after photoresist coating.

Additionally, to compensate for any experimental alignment inaccuracies, the overlap areas between active region and electrical contacts were designed with greater tolerance, foreseeing potential errors.

3.3.3 Clean Room Fabrication

The lithography process for the second generation of devices was carried out solely on the cleanroom. The need for accurate alignment determined the unfeasibility of the process using the benchtop set-up. The experimental procedure was the one used after the optimization of the exposure time, resumed in table 3.2.

By comparing the patterns in fig.3.15, we can see that there is a difference in the feature size. The pattern after lift-off, illustrated in fig.3.15.b), presents the smaller features. This is explained as the photoresist boundary also vanishes after lift-off.

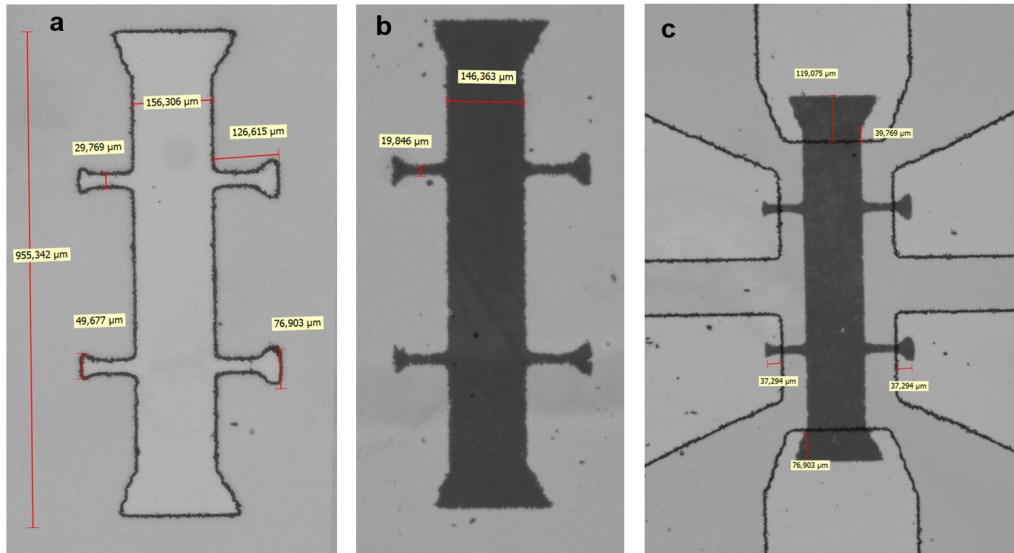


FIGURE 3.15: Double Cross Hall Bar through different stages :a) Photoresist pattern after development and b) Deposited material (14 nm Bi_2Te_3) after lift-off, and c) second lithography step with the photoresist pattern of the contacts after development.

*Deposition Parameters: $I = 20$ mA, Ar Gas flow of 50 sccm.

In fig.3.15.c), it is presented the photoresist pattern of the electrical contacts aligned with the double cross Hall Bar. The contacts are much wider than the Hall Bar arms in order to minimize alignment failure, as mentioned before.

3.4 Conclusions

We evaluated the feasibility of a lithography process using flexible polyester masks. These masks demonstrated good resolution down to 30 μm , though they exhibited defects inherent to the polyester film itself. While the overall quality was slightly lower compared to glass hard masks, we successfully patterned sub-100 μm features with satisfactory resolution by applying a few process optimizations.

Our tests in various environments showed that benchtop lithography setups are well-suited for creating longer, easily alignable patterns but require significant expertise for smaller, more intricate designs. For sub-100 μm patterns, which demand precise alignment, cleanroom conditions are preferable.

The first optimization focused on the development process. For long, nearly millimeter-scale patterns, a development time of 10 seconds was sufficient; however, for sub-100 μm features, this led to pronounced underdevelopment. Increasing the development time to 20 seconds provided the necessary accuracy for these finer features. Additionally, for simple Hall bar geometries fabricated in the cleanroom, we optimized the exposure time to 40 seconds to mitigate the small point defects caused by the polyester masks.

Finally, learning from initial errors, we produced a second generation of devices with improved contact tolerance and smaller alignment marks, enabling the fabrication of finer structures with greater precision.

4 Magnetotransport

To evaluate the potential of the fabricated devices for efficient spin-orbit torque (SOT) applications, we characterized the simple FeCo(11)/Bi₂Te₃(7) (nm) Hall bar structures using two distinct methods, as outlined in section 1. These methods include: magnetization switching by varying the applied magnetic field and magnetization switching by performing a current sweep.

For all measurements, a consistent geometry was used, as illustrated in fig.4.1: the current was applied along the long axis of the device (x-direction), while the transverse voltage was measured along the short axis (y-direction). The orientation of the applied magnetic field varied depending on the specific measurement.

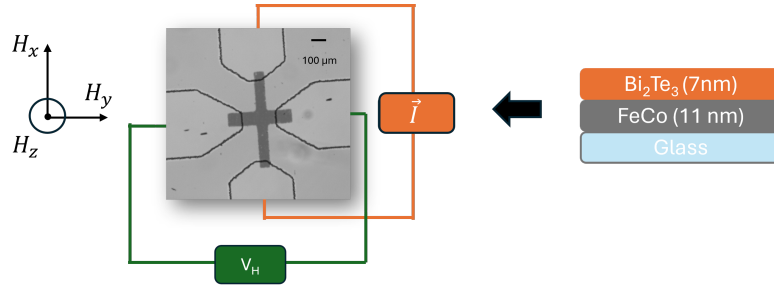


FIGURE 4.1: Scheme of employed geometry for magnetotransport measurement.

4.1 Anomalous Hall Effect

The Hall effect occurs when a transverse voltage is generated in a conductor subjected to a perpendicular magnetic field, driven by the Lorentz force acting on charge carriers (fig.4.2.a)). On the other hand, in the anomalous Hall effect (AHE), charge carriers experience a transverse deflection due to additional mechanisms tied to the material's magnetization and spin properties. Namely, due to spin-orbit coupling, electrons with different spin orientations scatter asymmetrically, as seen in fig.4.2.b), what also produces a measurable voltage signal. The Hall resistivity in both cases can be given by:

$$\rho_{xy} = \frac{V_{xy}}{I} t \quad (4.1)$$

where V_{xy} is the transverse voltage, I the applied current and t the thickness of the sample [79]. This chapter focuses on AHE measurements and their dependence on temperature, as a way to study the fabricated heterostructures.

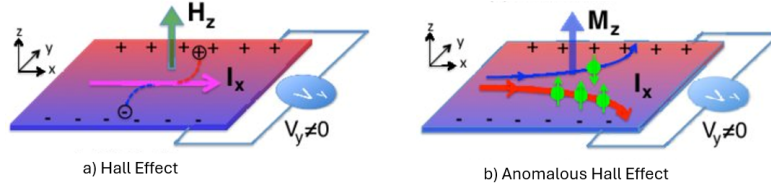


FIGURE 4.2: a) Ordinary Hall Effect and b) Anomalous Hall Effect. From [80].

The initial magnetotransport characterization of the samples was performed using a Physical Property Measurement System (PPMS) 6000 from *Quantum Design*. To measure the transverse resistance as a function of different magnetic field directions, an AC electrical current, with amplitude of 1mA and frequency of 0.5 Hz, was applied. Hall curves were obtained for various temperatures in the [3-300] K range. The samples were mounted in a Horizontal Rotator (fig.4.3), which allows for 360° rotation of the sample.

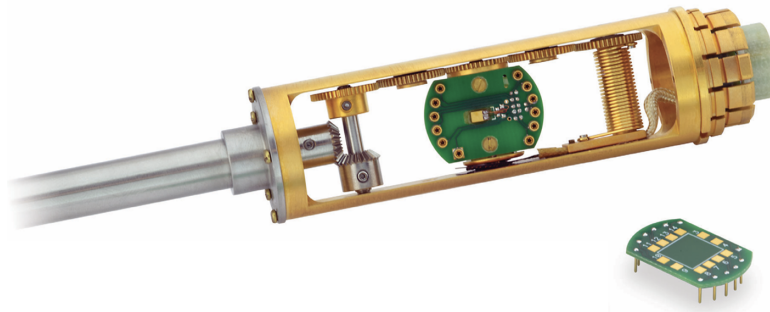


FIGURE 4.3: Horizontal Rotator and respective dockable sample board for ETO resistivity module from *Quantum Design*.

To establish electrical connections between the samples and the rotator board, TTO H20E Silver Epoxy (*Quantum Design*) was used to attach 0.01 mm wires to the patterned contact pads, with the other ends soldered to the board.

4.1.1 In-plane X Field

The first measurements for the Anomalous Hall resistance consisted on collecting the transverse voltage (V_{xy}), with the applied magnetic field aligned with the electrical current. These measurements were from 3 K to 150 K, in a HB device fabricated at the benchtop setup. Beyond 150 K, signal detection was lost due to one of the contacts breaking off

the sample.

In the forementioned experimental display, we expected to observe a hysteresis loop, according to the results in Chapter 3. SQUID data indicated a coercive field of $H_c = 11$ Oe, for the Bi_2Te_3 heterostructures, for an in-plane applied magnetic field. However, in the presented in-plane AHE measurements the field step was set to 100 Oe, what might have been too large to observe a hysteresis behavior. Instead, we observed an almost paramagnetic curve. This way, in order to confirm the presence of hysteresis we would need to perform a more precise measurement around zero field.

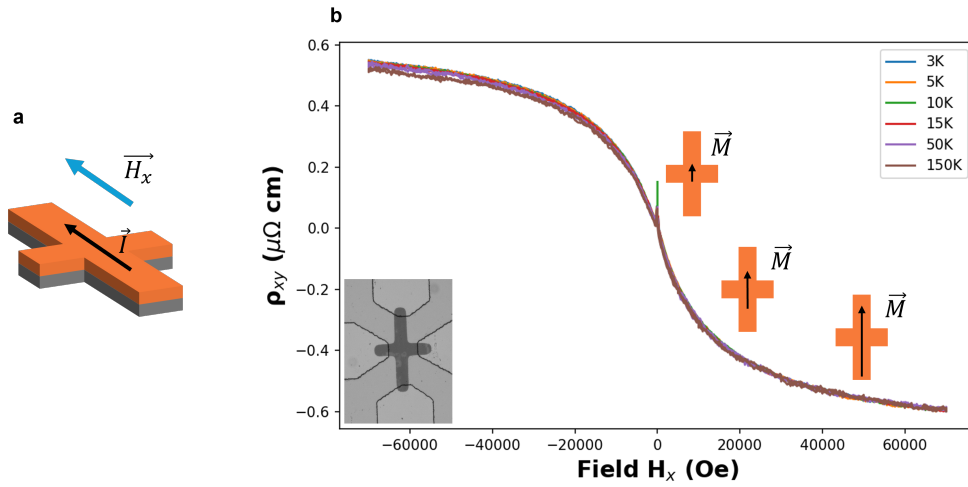


FIGURE 4.4: a) Geometry used: applied magnetic field parallel to the current direction and b) Transverse resistivity as function of applied in-plane field. The Hall Bar illustrations along the curve represent the increase of the magnetization vector in the X-axis, according to the applied field.

4.1.2 In-plane Y Field

To further investigate the AHE, we applied an in-plane magnetic field, perpendicular to the electrical current, as shown in fig. 4.5.b), for HB devices fabricated in both environments. In contrast to the previous measurement, the results in fig.4.5.a) and fig.4.5.c) display a sharp peak around zero applied field. This can likely be attributed to the presence of shape anisotropy. From the SQUID results in Chapter 3, we observed the presence of an in-plane easy axis and low coercive fields for all samples, indicating the soft ferromagnet nature of our samples. However, these results were obtained from continuous, homogeneous thin films, while the heterostructures analyzed in this section are patterned into a Hall bar geometry. As a consequent, during deposition the magnetic material is now constricted to a much smaller area with a different aspect ratio, leading to a structural and morphological organization dictated by the geometry of the pattern. This constriction leads to the creation of shape anisotropy, which can significantly increase the saturation

magnetization. Since the Hall bar has a longer axis, in the X-direction, the magnetization tends to align along this axis to minimize the demagnetization field. Consequently, when no external magnetic field is applied, the AHE arises from the material's intrinsic magnetization. As the field is increased, the magnetization gradually rotates to align with the applied field, producing the behavior depicted in fig.4.5.a) and fig.4.5.c).

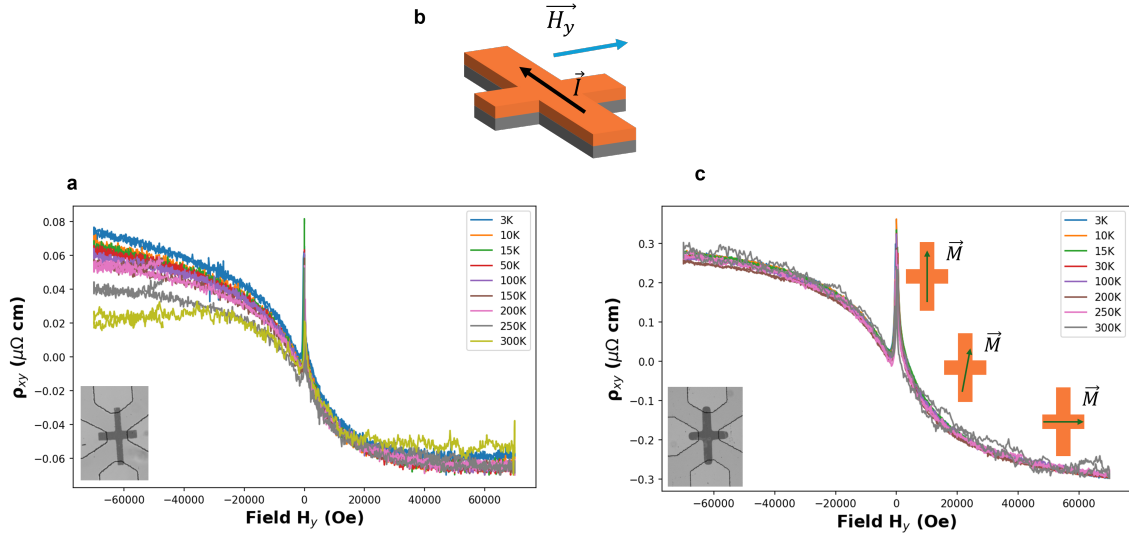


FIGURE 4.5: Transverse resistivity as a function of in-plane Y field for a) clean room device and c) benchtop set-up device; b) geometry used for this measurement. In c) the HB scheme shows the rotation of the magnetization from the easy to the hard axis.

4.1.3 Out-of plane Z Field

The out-of-plane characterization consisted on sweeping the magnetic field perpendicular to the samples's plane, and measuring the transverse voltage for various temperatures between [3-300]K. The results for the Hall Bars fabricated in two different environments are shown in fig.4.6. The behavior displayed approaches the ideal shape of an Anomalous Hall Effect curve, with negligible contribution from the Ordinary Hall Effect (OHE). To understand this, we first must note that this phenomenon can be analyzed according to eq.4.2, when the magnetic field is applied perpendicular to the sample's plane:

$$\rho_{xy}(H) = R_O H + 4\pi R_s M(H) \quad (4.2)$$

with ρ_{xy} being experimentally obtained by measuring the transverse voltage and the bias current, according to eq.4.1, previously presented at the beginning of this chapter.

The right side of eq.4.2 represents the resistivities due to the Ordinary Hall Effect (OHE) and the Anomalous Hall Effect (AHE), respectively. Here, R_O is the OHE coefficient which

can be used to calculate the number of carriers in a material. The R_s is the AHE coefficient and M is the sample's magnetization.

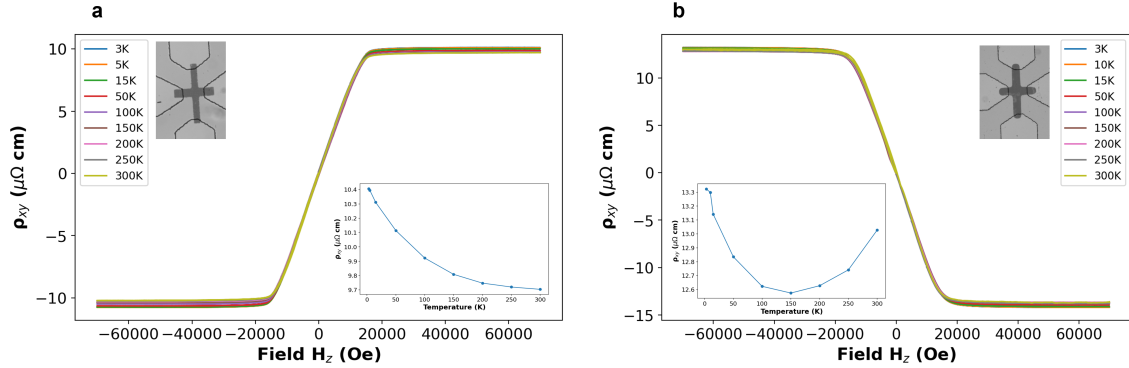


FIGURE 4.6: Anomalous Hall Curves for a) Clean room device and b) Benchtop setup. The inset graph shows the saturation resistivity as a function of temperature.

The insets on fig.4.6.a) and fig.4.6.b) represent the value of the transverse saturation resistivity, ρ_s , in each isothermal curve. We can observe a small temperature dependence for both devices. According to some authors [81], the temperature dependence of the AHE can be associated with the magnetization dependence on temperature. In our case, performing SQUID measurements as presented in chapter 2, at different temperatures for the Hall Bar devices, would allow us to assess whether the magnetization follows a similar trend and determine if this line of thinking is correct for our case.

Concerning the Hall curves in fig.4.6, the observed non-linearity corresponds to the magnetization response to the applied field: it increases up to a certain threshold, after which it reaches saturation. At this point, the AHE resistivity must remain constant, as described by eq.4.2. Consequently, if after the threshold the value of the slope is different from zero, then we have a field-dependent contribution, that is precisely from the OHe. To note that the different shape between curves a) and b), in fig.4.6, are solely due of the direction of the applied current for each device relative to the applied field direction.

We performed an analysis for the case of AHE curve at 300K, for the clean room device (fig.4.7), and separated the OHe contribution by considering the following steps:

- **Finding the Threshold Point:** to find the point where the magnetization reaches saturation we performed a fit on the linear region of the curve. Then, we find the point where the curve starts to significantly deviate from the linear fit, and identify where the difference exceeds a small threshold, defined by the user.

- **Resistivity of Saturation:** the saturation resistivity, ρ_s , was taken as the y correspondent of the threshold point.
- **Obtaining the slope after Threshold Point:** we performed a linear fit on the region after the threshold point to determine the OHE coefficient R_0 .

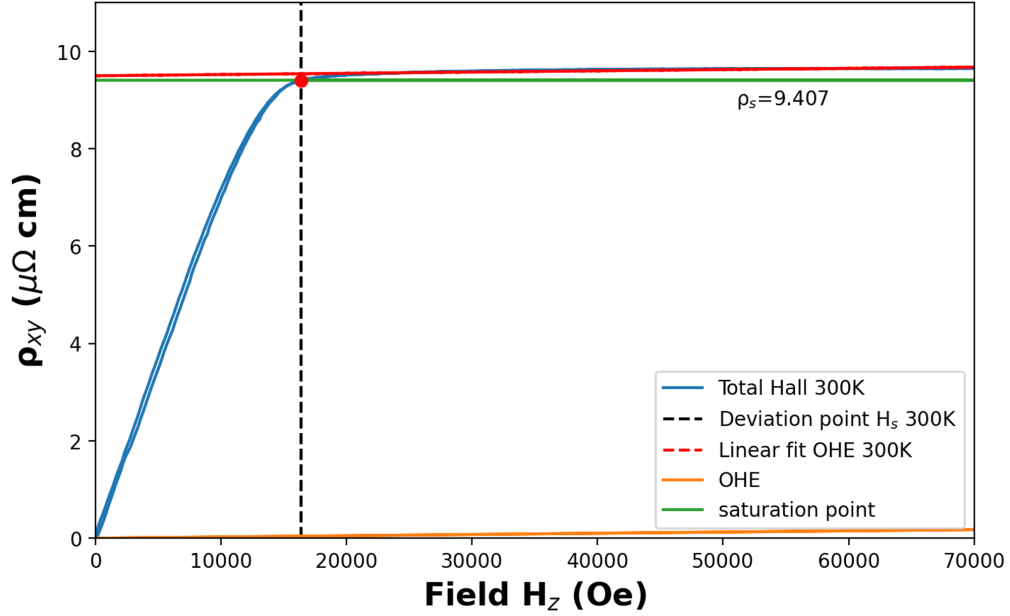


FIGURE 4.7: Deconstruction of the measured signal into OHE and AHE components for the clean room device.

The carrier density can be extracted from the Hall measurement by using: $n = (|R_O|q)^{-1}$, where q is the electron charge [79]. These steps were implemented on a python script that produced the values presented in table 4.1. These results were obtained for the device fabricated in the clean room and for the one produced at the benchtop lithography setup.

TABLE 4.1: Parameters from the Anomalous Hall Effect analysis for clean room device and benchtop setup device, at 300 K

Parameters	Clean Room Device	Benchtop setup Device
H_s (kOe)	16.353	16.559
ρ_s ($\mu\Omega \text{ cm}$)	9.407	11.668
$ R_0 $ ($\Omega \text{ m T}^{-1}$)	2.53×10^{-10}	4.21×10^{-10}
n (cm^{-3})	2.46×10^{22}	1.48×10^{22}

The difference between the two devices is not very pronounced, they share similar values for all parameters. Regarding the anisotropy field H_s , this value is large for both structures $H_s \approx 16$ kOe. Comparative with similar structures, this value surpasses the one obtained for FeCo(0.8)/MgO(1.0)/BiSb(10) (nm) by *Huy et al.* [82], of $H_s = 9$ kOe. However, in their

case, the incorporation of a MgO interface layer led the generation of perpendicular magnetic anisotropy at the interface with FeCo, reducing the H_k value.

The values for resistivity of saturation, in the order of $\rho_s \approx 10 \mu\Omega \cdot \text{cm}$, are one order of magnitude superior to the ones reported for sputtered Fe thin films [79].

The carrier concentration in both devices, measured to be on the order of $n = 10^{22} \text{ cm}^{-3}$, indicates a predominantly metallic transport behavior in the heterostructure. Typically, the carrier density for semiconductors ranges from 10^{17} to 10^{18} cm^{-3} , while for metals, it can reach up to 10^{22} cm^{-3} . In our case, the carrier concentration falls within the metallic regime, suggesting that electrical transport primarily occurs within the ferromagnetic layer, likely due to a shunting effect. In this scenario, current flows from the contacts into the topological insulator (TI) and because the FeCo layer has a much higher conductivity, electrons preferentially follow a path through this metallic layer before returning through the TI.

Finally, the OHE contribution for both devices is very low, being the signal mainly attributed to the AHE. This can be a surprising result since due to the low values of M_s , obtained from the SQUID measurements, one would expect not such an ideal behavior in terms of AHE.

4.1.4 Temperature dependence

Finally, we measured the temperature dependence of the Hall resistance in the **absence of an applied magnetic field**, after first characterizing the anomalous Hall resistance as a function of various field directions over a specific temperature range. The results are presented in fig.4.8.

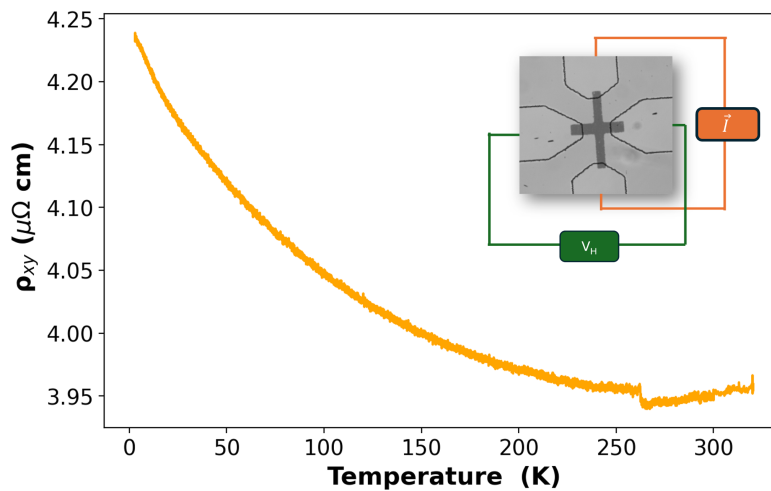


FIGURE 4.8: Temperature dependence of transverse voltage, under absence of an applied magnetic field. On the inset is the clean room device used for the measurement.

The data display a profile somewhat similar to the inset graph of fig.4.6. To interpret these results, we must consider that the HB exhibits magnetic shape anisotropy, which leads to spontaneous magnetization even in the absence of an external magnetic field. Consequently, when an electric current is applied, the transverse voltage arises from both the ordinary Hall effect (OHE) and the anomalous Hall effect (AHE), as described by eq. 4.2. From the previous results, we know that the contribution from magnetization is more significant than that of the OHE. Therefore, the data shown in fig. 4.8 reflect the temperature dependence of the AHE, that arises from the in-plane magnetization.

Nonetheless, the Hall resistivity dependence on temperature is reduced: as illustrated in fig.4.8 for a range of [3-300] K we obtained a variation of $\Delta\rho_{xy} \approx 0.30 \mu\Omega\cdot\text{cm}$. As mentioned before, the temperature dependence of the AHE can be correlated with the magnetization's dependence on temperature. However, we can also obtain contributions from the adjacent materials that are being employed, in our case from Bi_2Te_3 . For example, Ni *et al.* [83] reported an ultrahigh sensitivity of Anomalous Hall Effect sensor based on Cr-doped Bi_2Te_3 TI thin films. On the other hand, Chen *et al.* [84] synthesized $\text{Bi}_2\text{Te}_3/\text{MnTe}$ magnetic heterostructures and observed pronounced AHE signals from each layer individually and an hybrid effect when combining both together, as illustrated in fig.4.9.

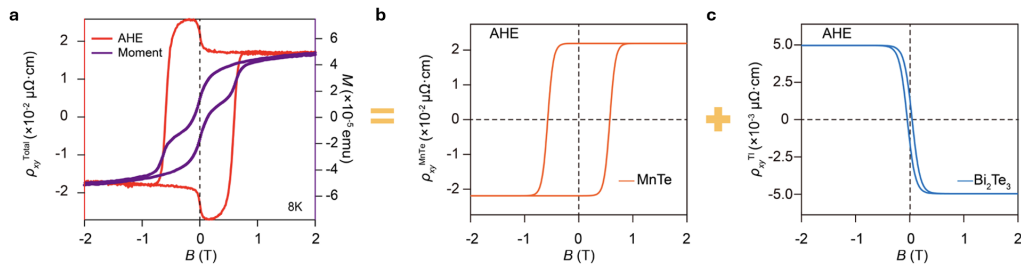


FIGURE 4.9: Work from Chen *et al.* [84]: a) Hysteresis loops at 8 K for anomalous hall resistance and magnetization, for $\text{Bi}_2\text{Te}_3/\text{MnTe}$ heterostructure; b) and c) individual contributions of AHE for MnTe and Bi_2Te_3 , respectively.

As a result, in order to understand further the various contributions we would need to measure individually, FeCo and Bi_2Te_3 single layers. This way, accompanied also with the measurement of the magnetization dependence with temperature of the heterostructure we could distinguish the role of the topological insulator in the AHE signal.

4.2 Second Harmonic Measurement Set-up

One of the aims of this thesis was to determine the effective spin hall angle, θ_{SH} , for the FeCo(11)/Bi₂Te₃(7) (nm) heterostructures. However, the desired results were not obtained at the time of this document, as a proper measurement setup had to be developed for this purpose. In second harmonic measurements, a sinusoidal current of a given fixed frequency f is applied while the external magnetic field is varied, allowing the second harmonic of the voltage to be measured. Experimentally, this is commonly achieved by using a lock-in amplifier to detect the first and the second harmonic Hall voltages, under sine wave excitation generated by a proper low current source, which is most papers is achieved by using a Keithley 6221 DC/AC current source [57, 85].

In our case, we had to build our own AC current source. This was achieved by using a set of resistors and two OPAMPs, as depicted in fig.4.10.

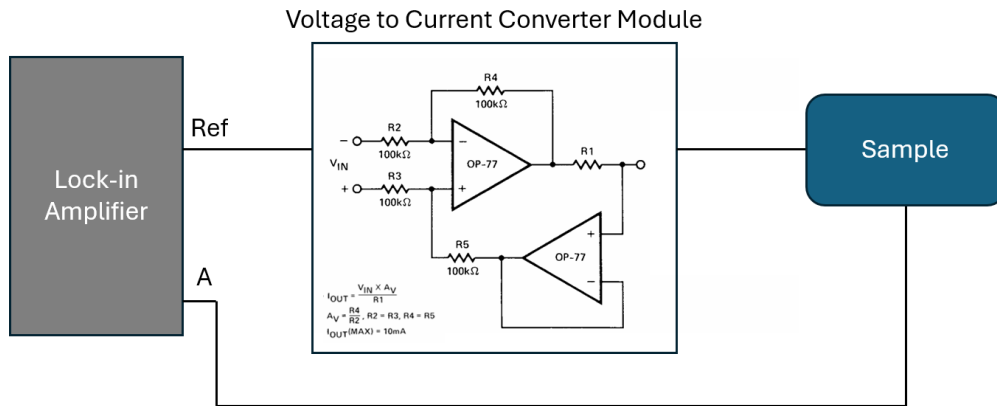


FIGURE 4.10: Experimental set-up created for the measurement of the spin Hall angle. It consists of a lock-amplifier that acts both as the source and detector for the second harmonic voltage; a voltage to current converter module composed by two OP-77 and five resistors, and finally a test sample.

Here, we use the reference sinusoidal signal generated by a SR830 Lock-In Amplifier, that can go up to 5V, as the input voltage. To convert the reference signal to a sinusoidal current, two OPAMPs and a set of resistors are used. Each OPAMP is connected to +/- 15V and to the ground. We use capacitors in parallel to filter and stabilize the voltage signal from the power source. With one OPAMP we could still achieve our goal of producing a steady current. Nevertheless, by using a noninverting voltage follower in the feedback loop, the accuracy of the I_{out} is improved. The resistors were set to $R2 = R3 = R4 = R5 = 100 \text{ k}\Omega$ and $R1 = 1 \text{ k}\Omega$. This way the output current would be given by $I_{out} = \frac{V_{IN}}{R1}$. However, this relation only stands as long as we don't reach the OPAMP's saturation.

Despite providing accurate electrical current output, the set-up is limited by the value of the sample's resistance. For example, if aimed to provide 1mA current to the sample, we could just set the $V_{IN} = 1V$. However, if the sample/charge resistance $R_{sample} = 100 \text{ k}\Omega$, then we would have 100 V at its terminal, what is not possible since the OPAMP reaches saturation way before at 15V. So the real current in this sample is $I_{out} = 0.15 \text{ mA}$. In our case, for the fabricated Hall Bars the resistance in long and short arms is $R = 2\text{k}\Omega$ and $R = 1\text{k}\Omega$, respectively. Therefore, we don't reach the OPAMP's saturation and can go up from 1-5 mA.

4.3 Magnetic devices

While the spin Hall angle and SOT efficiency of our heterostructures have not yet been fully characterized, the results from the AHE measurements indicate promising features for their application in magnetic devices. Crucial observations include:

- **Minimal Temperature Dependence of Hall resistivity**
- **Large out-of-plane Anisotropy field H_s**
- **Magnetic Shape Anisotropy**

In the following subsections, we explore into how these properties can be leveraged in the development of next-generation magnetic devices.

4.3.1 High Field Hall Sensor

In the produced $\text{FeCo}(11)/\text{Bi}_2\text{Te}_3(7) \text{ nm}$ heterostructures we observe a reduced variation with temperature $\Delta\rho_{xy} \approx 0.30 \text{ }\mu\Omega\cdot\text{cm}$, for a range of temperatures from 3 to 300K, in fig.4.8. This is a behavior transversal to all AHE measurements. Such minimal temperature dependence is particularly advantageous for Hall sensors [86]. In practical applications, Hall sensors are often exposed to varying environmental conditions, including temperature fluctuations. A small temperature coefficient (TC) of sensitivity ensures that the sensor's performance remains stable and predictable over a wide temperature range. The temperature coefficient can be given by eq.4.3:

$$TC = \frac{1}{s_0} \frac{\Delta s}{\Delta T} \quad (4.3)$$

where s_0 is the sensor's sensitivity (in Ω/T units *), given by the slope of the linear region in the out-of plane AHE curve (fig.4.6), at a reference temperature; Δs is the change in sensitivity over a given temperature range and ΔT is the change in temperature. In our case the sensitivity variation with temperature is also quite low, as depicted in fig.4.11.

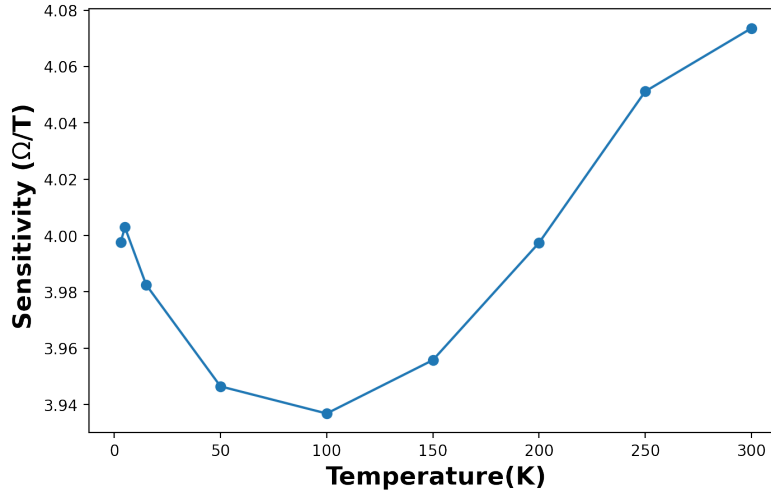


FIGURE 4.11: Sensitivity of the out-of plane AHE curve dependence with temperature, for clean room device.

In our case, given the results of fig.4.11 and a sensitivity of $s_0^\dagger = 4.07 \Omega/T$ at room-temperature, the corresponding average TC gives out 0.006%/K, considering $\Delta T = 300K$. These findings suggest the potential for a robust Hall sensor application across a wide temperature regime. Also, to note that the sensitivity of an Hall sensor reflects how effectively the sensor converts an external magnetic signal into a measurable electrical signal. For most applications this value is aimed to be as high as possible [83]. For our case, despite the low sensitivity rounding $S = 4.07 \Omega/T$, the large anisotropy field, $H_s = 1.6 T$ (point in which we reach the saturation), could be indicated for high magnetic field applications such as Magnetic Resonance Imaging (MRI) [87].

4.3.2 Room-Temperature Current Driven Magnetization Switching

Concerning the potential of the fabricated $FeCo(11)/Bi_2Te_3(7)$ nm heterostructures as SOT-efficient devices, one fundamental parameter to obtain is the applied critical density of current, J_c , to which the magnetization reverts its state.

The data obtained in subsection 4.1.2, for an applied Y field, indicate the presence of

*In accordance with the literature on anomalous Hall devices, SI units are also used for magnetic values from this point onward.

[†]Here to calculate s_0 we transitioned from resistivity to resistance in the out-of plane AHE curve by dividing by the heterostructure thickness.

magnetic shape anisotropy, along the x axis. According to the theory exposed in chapter 1, we are in conditions of realizing type-X SOT switching by applying a small out-of-plane magnetic field H_z and sweeping the electrical current, as illustrated in fig.4.12.a). As a consequence, we attempted to perform type-X SOT switching at room temperature (300 K).

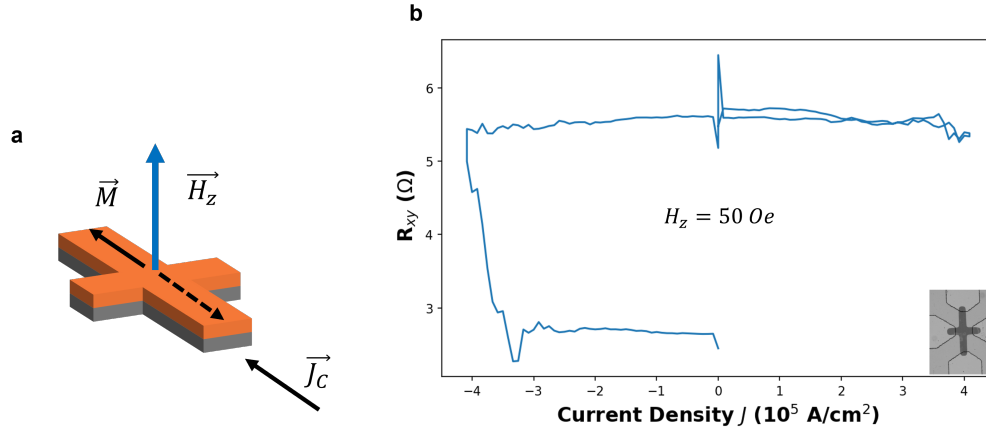


FIGURE 4.12: a) Geometry used for type X SOT switching and b) Hall resistance results for a current sweep between -5mA,5mA, under $H_z = 50 \text{ Oe}$ applied field.

The results, represented in fig.4.12.b). for an external magnetic field of $H_z = 50 \text{ Oe}$, show the transition from a low resistance state to high resistance one, for a sweep starting at negative current. This result yields magnetization switching of $J_c = -3.3 \times 10^5 \text{ A/cm}^2$, at room-temperature.

However, for positive currents, the switching does not occur at the same value, being the resistance trapped in high resistance state. For magnetization reversion, higher density of currents would have to be applied. In our case, this experiment was performed on PPMS and the limit current was 5mA for the used module, therefore we could not go beyond this value and determine the positive switching current density. For that we need to measure in a different set-up that allows a higher current range.

Nevertheless, the asymmetric profile has been reported in other studies involving type X SOT switching [30, 88]. The reason behind it may be from asymmetric coercivity, due to asymmetric domain nucleation during fabrication. One way to assess this would be by doing a R-H loop similar to the one in fig.4.4, but for low fields with a higher precision, to observe a potential shift in the hysteresis cycle.

4.4 Conclusions

In this chapter, the magnetotransport behavior of FeCo(11)/Bi₂Te₃(7) nm heterostructures, patterned into Hall bar structures, was systematically studied. By applying magnetic fields in different spatial directions, a pronounced Anomalous Hall Effect (AHE) was observed. Hall resistance measurements as a function of the out-of-plane magnetic field revealed that the AHE dominates over the Ordinary Hall Effect (OHE) in all samples, regardless of the fabrication environment. Fundamental parameters such as anisotropy field (H_s), saturation resistivity (ρ_s), and carrier density (n) were consistent across the different Hall bar geometries, indicating that the slight geometric variations (e.g., rounder shapes in the benchtop-fabricated device) have minimal influence on AHE measurements.

In the case of in-plane field characterization, sharp peaks in the Hall resistance were observed around zero field when the applied magnetic field was perpendicular to the long axis of the Hall bar. This behavior was absent when the field was aligned parallel to both the long axis and the current flow, suggesting the presence of shape anisotropy in the Hall bar structures.

Also, temperature-dependent measurements indicated a reduced variation in Hall resistivity, which could be linked to either the temperature-dependent magnetization of the ferromagnetic layer or the contribution from the topological insulator (Bi₂Te₃). Further investigation, including separate measurements on individual FeCo and Bi₂Te₃ layers, is required to isolate these contributions.

Finally, with respect to spin-orbit torque (SOT) efficiency, we were able to switch the magnetization of the heterostructure using a low current density (J_c) in the order of 10^5 A/cm², with at an out-of-plane magnetic field (H_z) of 50 Oe. This performance surpasses those of other heavy metals[43, 61], and is comparable to topological insulator thin films used for SOT switching [57, 59]. These findings highlight the potential of Bi₂Te₃ as a highly efficient material for SOT applications, despite additional data being needed to assert the average value of J_c .

5 Final Remarks

5.1 Conclusions

This thesis presents a comprehensive study of FeCo/Bi₂Te₃ heterostructures, focusing on their structural, magnetic, and magnetotransport properties, as well as the microfabrication techniques used in their development. The work addresses both fundamental and practical aspects of these heterostructures, highlighting their potential for spintronic applications, particularly in the context of spin-orbit torque (SOT) efficiency and Hall sensors. The structural and magnetic characterization of FeCo thin films revealed the predominantly amorphous nature of the material, with the presence of nanocrystals $D = (10.8 \pm 0.4)$ nm of diameter, as suggested by XRD analysis. SQUID magnetometry demonstrated the reduced saturation magnetization, $M_s = 558$ emu/cm³, compared to other FeCo thin films, reinforcing the dominance of an amorphous structure. Also, a coercive field of $H_c = 111$ Oe was obtained. The addition of the Bi₂Te₃ layer further reduced the coercive field by a factor of ten and decreased the saturation magnetization by approximately 30%, reassuring the soft ferromagnetic nature of the heterostructure. An annealing process that typically enhances Bi₂Te₃'s topological properties led to the suppression of ferromagnetism in the heterostructure. The observed complex ferromagnetic resonance spectra are consistent with those found in ferrimagnetic nanoparticles, further indicating the presence of nanocrystals within the films.

A challenge on this thesis was the design, testing and fabrication of sub-100 μ m Hall Bar structures, in order to provide a way to rigorously characterized topological insulator/ferromagnet bilayers. The employed flexible masks proved to be a low-cost and fast way to produce the desired devices, despite a few optimizations of the lithography process, with the micro-fabrication tested in two different environments. Comparing the two setups utilized, the Benchtop lithography setup proved being suitable for long patterns, but for more intricate designs requiring precise alignment, cleanroom conditions were essential. A second generation of devices with improved design features enabled finer and more accurate structures. Moreover, this work highlights that lithography, while consisting of standard procedures, requires adaptations when small changes are imposed. In our case, adjustments to development time, exposure duration, and mask design were essential for successfully produce sub-100 μ m patterns using flexible polyester masks.

Concerning the magnetotransport behavior of the FeCo/Bi₂Te₃ heterostructures, a pronounced Anomalous Hall Effect (AHE) was observed, dominating over the Ordinary Hall Effect (OHE) in all samples. Key parameters such as anisotropy field of $H_s = 16$ kOe, an average saturation resistivity of $\rho_s = 10 \mu\Omega \cdot \text{cm}$, and carrier density in the order of $n \approx 10^{22} \text{ cm}^{-3}$ were consistent across devices originated from different set-ups. These findings suggest that slight geometric variations had minimal impact on AHE measurements. Sharp peaks in Hall resistance under in-plane magnetic fields revealed shape anisotropy in the Hall bar structures, leading probably to a higher saturation magnetization than for the measured continuous films.

The temperature dependence, in our samples, of the Anomalous Hall effect is reduced, $\Delta\rho_{xy} \approx 0.30 \mu\Omega \cdot \text{cm}$, for a [3-300] K temperature range. The temperature coefficient (TC) of sensitivity, calculated as $\text{TC} = 0.006\%/K$ indicates that the sensor's performance remains consistent even with temperature fluctuations. This low TC makes these heterostructures ideal for applications requiring reliable operation over a broad temperature range. Additionally, the low sensitivity calculated $S = 4.07 \Omega/T$ and large anisotropy field $H_s = 1.6T$ suggest suitability for high magnetic field applications, such as Magnetic Resonance Imaging (MRI). Overall, these heterostructures offer a robust solution for Hall sensors needing both thermal stability and performance in a wide dynamic range.

With respect to SOT efficiency, we were able to perform magnetization's switching with a current as low as $J_c = -3.3 \times 10^5 \text{ A/cm}^2$. This is a value that surpasses those for heavy metal systems and is in agreement with those achieved for low energy topological insulator systems. These findings underscore the potential of Bi₂Te₃ as a promising material for spintronic applications, although further data is needed to fully quantify the average current density required for SOT switching, following a type X geometry.

Overall, this thesis demonstrates that the produced FeCo/Bi₂Te₃ heterostructures offer valuable insights into the interplay between ferromagnetic and topological materials, while also providing practical guidance for their fabrication, and use, in spintronic devices and Hall sensors.

5.2 Future Work

Building on the promising results obtained for the produced heterostructures, we aim to advance in three directions: employing new materials, testing the reproducibility and reliability of the produced magnetic devices, and develop a new kind of device at a nanometric scale.

5.2.1 Material Engineering

Given the amorphous nature of our FeCo thin films we would like to employ crystalline ferromagnetic materials alongside with Bi_2Te_3 . However, some magnetic properties such as the coercive field could be enhanced, what would difficult the magnetization switching process. We would have to explore different magnetic materials such as NiFe, or even ferrimagnetic such as CoTb. Also, an unachieved goal was the production of functional ferromagnetic bilayers with annealed Bi_2Te_3 , as in our case, annealing led to the loss of magnetic behavior. To address this, material engineering could be employed by adding a Pt,Ru or Ta interface layer [89] (no thicker than 1 nm) between the ferromagnetic layer and the topological insulator to prevent diffusion during annealing. However, care must be taken to avoid potential shunting effects caused by the interface layer.

5.2.2 Magnetic Devices Testing

To complete the presented work we would like to perform the measurements of AHE, type-X SOT switching, and second harmonic voltage for these material engineered samples. Regarding the already fabricated structures, additional data must be gathered to assess both the reproducibility and accuracy of the magnetic devices. Further measurements will be crucial not only for validating the device's performance but also for ensuring consistency across multiple trials. This will provide deeper insights into the stability of the high field Hall Sensor, for example, allowing us to fine-tune the material engineering process. For SOT switching, also establishing the reliability of the measurements is essential for advancing the practical applications of these devices in fields like SOT-MRAM, where precision and repeatability are crucial to developing next-generation memory and logic technologies.

5.2.3 New Concept of Magnetic Device

As a continuation of this work, we plan to implement the materials studied throughout this thesis in a Lateral Spin Valve (LSV) device, at the nanoscale. The primary goal is to use a sputtered Bi_2Te_3 thin film as the channel, which will support two ferromagnetic stripes with different aspect ratios, as illustrated in fig.5.1. By applying a DC bias current, we aim to leverage the spin-momentum locking property of the topological insulator (TI) to generate a spin-polarized current. This current is expected to reverse the magnetic state of one of the ferromagnetic stripes, thus enabling the switching of the device. The resistance of the

LSV will be determined by measuring the voltage across the stripes and calculating the ratio using Ohm's law, $R = \frac{V}{I}$.

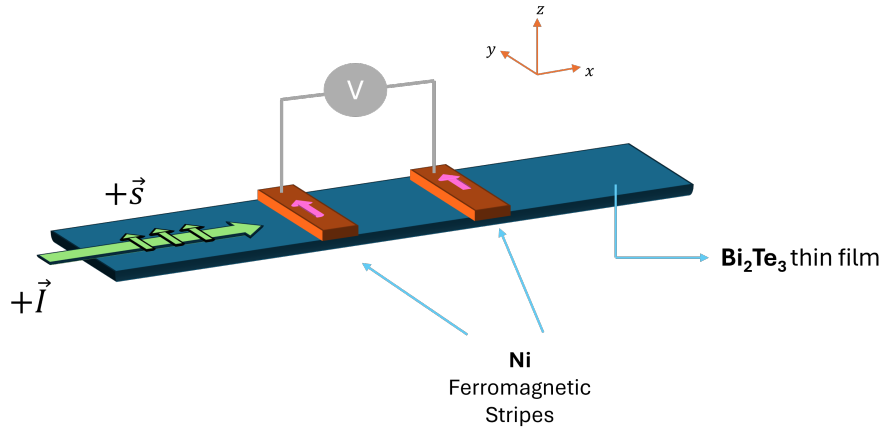


FIGURE 5.1: Representation of Lateral Spin Valve. A spin polarized current is created at the topological insulator channel. By passing through the ferromagnetic stripes it is intended to revert one of the stripes magnetization.

From a technological perspective, once the design demonstrates the existence of topological surface states and efficient spin current production, more advanced geometries can be explored. For example, instead of using a ferromagnetic stripe, a single ferromagnetic domain dot could be implemented, with an antiferromagnetic layer beneath it, both positioned on top of the topological insulator channel. By applying DC bias current pulses, this setup could emulate the behavior of artificial neurons and synapses, enabling the development of asynchronous spiking neural networks [10, 90].

Progress towards manufacturing such device has already been made. The geometry of the device has been designed: for the active region, as depicted in fig.5.2.b), the Bi_2Te_3 channel will have a width of $w_{TI} = 1 \mu\text{m}$ and a length of $L_{TI} = 10 \mu\text{m}$. Both ferromagnetic stripes will be $11.5 \mu\text{m}$ long. One stripe will have a fixed width of $w_{FM} = 1 \mu\text{m}$, while the other stripe's width will be reduced to $w_{FM} = 0.5 \mu\text{m}$ to create a different aspect ratio. This difference in aspect ratio is intended to ensure that each stripe reverses its magnetization at different coercive fields.

The fabrication of this device will be conducted in collaboration with Eindhoven University, and the process has already begun. We have completed the DC magnetron sputtering deposition of 7 nm Bi_2Te_3 on a silicon wafer, as illustrated in fig.5.2.c). The next steps will involve patterning and the subsequent deposition of the ferromagnetic stripes using e-beam lithography, at Eindhoven University.

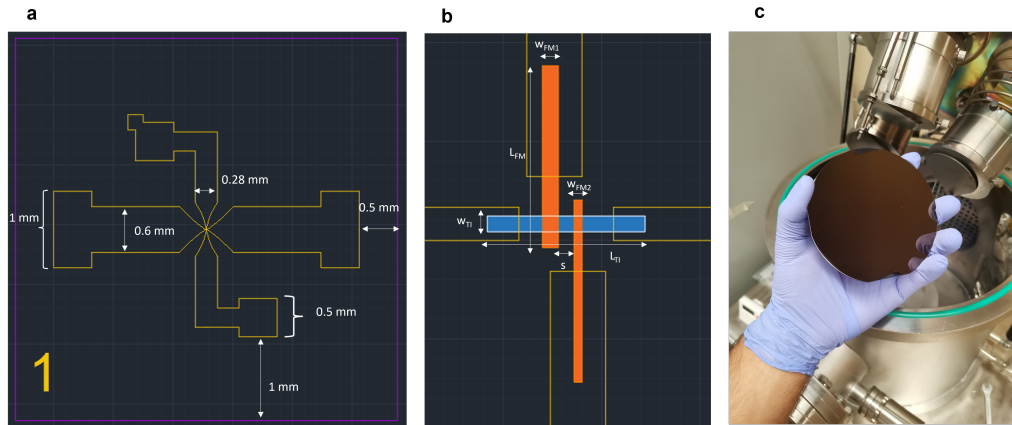


FIGURE 5.2: a) Contacts view; b) active region of the device containing the topological insulator channel and the ferromagnetic stripes; c) Deposited Bi_2Te_3 on silicon wafer.

References

- [1] G. E. Moore, "Cramming more components onto integrated circuits, Reprinted from Electronics, volume 38, number 8, April 19, 1965, pp.114 ff." *IEEE Solid-State Circuits Society Newsletter*, vol. 11, no. 3, pp. 33–35, 2006. [Cited on page 1.]
- [2] CNET, "Intel will outpace Moore's Law, CEO Pat Gelsinger says," available at: <https://www.cnet.com/tech/computing/intel-will-outpace-moores-law-ceo-pat-gelsinger-says/>. Accessed September 6, 2024. [Cited on pages x and 1.]
- [3] K. S. K. Chandra, Kakarla Hari, "Design and analysis of low power FinFET SRAM with leakage current reduction techniques," *Wireless Personal Communications*, vol. 131, no. 15, pp. 1167–1188, 2023. [Cited on page 1.]
- [4] S. K. Mohapatra, K. P. Pradhan, and P. K. Sahu, "A new nanoscale DG MOSFET design with enhanced performance – a comparative study," V. V. Das and P. Elkafrawy, Eds. Springer International Publishing, 2014, pp. 76–81. [Cited on page 1.]
- [5] F. Zahoor, M. Hanif, U. I. Bature, S. Bodapati, A. Chattopadhyay, F. A. Hussin, H. Abbas, F. Merchant, and F. Bashir, "Carbon nanotube field effect transistors: An overview of device structure, modeling, fabrication and applications," *Physica Scripta*, vol. 98, no. 8, pp. 082 003–33, Aug. 2023. [Cited on page 1.]
- [6] D. Serpanos and T. Wolf, "Chapter 13 - Networks on chips," in *Architecture of Network Systems*, ser. The Morgan Kaufmann Series in Computer Architecture and Design, D. Serpanos and T. Wolf, Eds. Boston: Morgan Kaufmann, 2011, pp. 239–248. [Cited on page 2.]
- [7] M. N. Baibich, J. M. Broto, A. Fert, F. N. Van Dau, F. Petroff, P. Etienne, G. Creuzet, A. Friederich, and J. Chazelas, "Giant magnetoresistance of (001)Fe/(001)Cr magnetic superlattices," *Physical Review Letters*, vol. 61, no. 21, pp. 2472–2475, Nov. 1988. [Cited on pages 2 and 10.]
- [8] G. Binasch, P. Grünberg, F. Saurenbach, and W. Zinn, "Enhanced magnetoresistance in layered magnetic structures with antiferromagnetic interlayer exchange," *Physical Review B*, vol. 39, no. 7, pp. 4828–4830, Mar. 1989. [Cited on page 2.]

- [9] B. A. Everitt and A. V. Pohm, "Pseudo spin valve magnetoresistive random access memory," *Journal of Vacuum Science & Technology A*, vol. 16, no. 3, pp. 1794–1800, May 1998. [Cited on page 2.]
- [10] A. Kurenkov, S. Fukami, and H. Ohno, "Neuromorphic computing with antiferromagnetic spintronics," *Journal of Applied Physics*, vol. 128, no. 1, pp. 010 902–12, Jul. 2020. [Cited on page 62.]
- [11] M. N. Leuenberger and D. Loss, "Spintronics and quantum computing: Switching mechanisms for qubits," *Physica E: Low-dimensional Systems and Nanostructures*, vol. 10, no. 1, pp. 452–457, 2001. [Cited on page 2.]
- [12] M. Nadeem, I. Di Bernardo, X. Wang, M. S. Fuhrer, and D. Culcer, "Overcoming boltzmann's tyranny in a transistor via the topological quantum field effect," *Nano Letters*, vol. 21, no. 7, pp. 3155–3161, Mar. 2021. [Cited on page 2.]
- [13] X. Li and F. Liu, "Topological field-effect transistor with quantized on/off conductance of helical/chiral dislocation states," *Physical Review B*, vol. 107, no. 22, pp. 224 101–6, Jun. 2023. [Cited on page 2.]
- [14] M. Eschbach, E. Młyńczak, and J. Kellner, "Realization of a vertical topological p-n junction in epitaxial Sb₂Te₃/Bi₂Te₃ heterostructures," *Nature communications*, vol. 6, pp. 8816–23, Nov. 2015. [Cited on page 2.]
- [15] T. M. Philip, M. R. Hirsbrunner, M. J. Park, and M. J. Gilbert, "Performance of topological insulator interconnects," *IEEE Electron Device Letters*, vol. 38, no. 1, pp. 138–141, Jan. 2017.
- [16] Y. G. Semenov, X. Duan, and K. W. Kim, "Electrically controlled magnetization in ferromagnet-topological insulator heterostructures," *Physical Review B*, vol. 86, no. 16, pp. 161 406–5, Oct. 2012. [Cited on page 2.]
- [17] J. Slonczewski, "Current-driven excitation of magnetic multilayers," *Journal of Magnetism and Magnetic Materials*, vol. 159, no. 1, pp. L1–L7, Jun. 1996. [Cited on pages 5 and 10.]
- [18] Y. Suzuki, A. A. Tulapurkar, and C. Chappert, "Spin-Injection Phenomena and Applications," in *Nanomagnetism and Spintronics*. Elsevier, 2009, pp. 93–153. [Cited on pages x, 5, and 6.]

- [19] M. Dyakonov and V. Perel, “Current-induced spin orientation of electrons in semiconductors,” *Physics Letters A*, vol. 35, no. 6, pp. 459–460, Jul. 1971. [Cited on page 6.]
- [20] M. I. D’Yakonov and V. Perel, “Possibility of orienting electron spins with current,” *Soviet Journal of Experimental and Theoretical Physics Letters*, vol. 13, no. 11, pp. 467–469, Jun. 1971. [Cited on page 6.]
- [21] Y. K. Kato, R. C. Myers, A. C. Gossard, and D. D. Awschalom, “Observation of the Spin Hall Effect in Semiconductors,” *Science*, vol. 306, no. 5703, pp. 1910–1913, Dec. 2004. [Cited on page 6.]
- [22] P. Wang, Z. Feng, Y. Yang, D. Zhang, Q. Liu, Z. Xu, Z. Jia, Y. Wu, G. Yu, X. Xu, and Y. Jiang, “Inverse orbital Hall effect and orbitronic terahertz emission observed in the materials with weak spin-orbit coupling,” *npj Quantum Materials*, vol. 8, no. 1, pp. 28–6, May 2023. [Cited on pages x and 7.]
- [23] J. M. D. Coey, “History of magnetism and basic concepts,” in *Handbook of Magnetism and Magnetic Materials*, J. M. D. Coey and S. S. Parkin, Eds. Cham: Springer International Publishing, 2021, pp. 3–51. [Cited on page 7.]
- [24] F. Bottegoni, C. Zucchetti, G. Isella, M. Bollani, M. Finazzi, and F. Ciccacci, “Spin-charge interconversion in heterostructures based on group-IV semiconductors,” *La Rivista del Nuovo Cimento*, vol. 43, no. 2, pp. 45–96, Feb. 2020. [Cited on pages 7 and 8.]
- [25] Q. Shao, G. Yu, Y.-W. Lan, Y. Shi, M.-Y. Li, C. Zheng, X. Zhu, L.-J. Li, P. K. Amiri, and K. L. Wang, “Strong Rashba-Edelstein Effect-Induced Spin–Orbit Torques in Monolayer Transition Metal Dichalcogenide/Ferromagnet Bilayers,” *Nano Letters*, vol. 16, no. 12, pp. 7514–7520, Dec. 2016. [Cited on page 8.]
- [26] Y. Liu, C. Zeng, J. Zhong, J. Ding, Z. M. Wang, and Z. Liu, “Spintronics in Two-Dimensional Materials,” *Nano-Micro Letters*, vol. 12, no. 1, pp. 93–26, Dec. 2020. [Cited on page 8.]
- [27] C. Zacchetti, “Spin-charge interconversion in Ge-based structures,” Ph.D. dissertation, Politecnico di Milano, 2019. [Cited on pages x and 8.]
- [28] V. Edelstein, “Spin polarization of conduction electrons induced by electric current in two-dimensional asymmetric electron systems,” *Solid State Communications*, vol. 73, no. 3, pp. 233–235, Jan. 1990. [Cited on page 8.]

- [29] J. C. R. Sánchez, L. Vila, G. Desfonds, S. Gambarelli, J. P. Attané, J. M. de Teresa, C. Magén, and A. Fert, “Spin-to-charge conversion using Rashba coupling at the interface between non-magnetic materials,” *Nature Communications*, vol. 4, pp. 2944–7, Dec. 2013. [Cited on page 8.]
- [30] S. Fukami, T. Anekawa, C. Zhang, and H. Ohno, “A spin–orbit torque switching scheme with collinear magnetic easy axis and current configuration,” *Nature Nanotechnology*, vol. 11, no. 7, pp. 621–625, Jul. 2016. [Cited on pages x, 9, and 57.]
- [31] S. Fukami, T. Anekawa, A. Ohkawara, Chaoliang Zhang, and H. Ohno, “A sub-ns three-terminal spin-orbit torque induced switching device,” in *2016 IEEE Symposium on VLSI Technology*. Honolulu, HI, USA: IEEE, Jun. 2016, pp. 1–2. [Cited on page 9.]
- [32] W. Thomson, “XIX. On the electro-dynamic qualities of metals:—Effects of magnetization on the electric conductivity of nickel and of iron,” *Proceedings of the Royal Society of London*, vol. 8, pp. 546–550, Dec. 1857. [Cited on page 10.]
- [33] T. Miyazaki and N. Tezuka, “Giant magnetic tunneling effect in Fe/Al₂O₃/Fe junction,” *Journal of Magnetism and Magnetic Materials*, vol. 139, no. 3, pp. L231–L234, Jan. 1995. [Cited on page 10.]
- [34] J. S. Moodera, L. R. Kinder, T. M. Wong, and R. Meservey, “Large Magnetoresistance at Room Temperature in Ferromagnetic Thin Film Tunnel Junctions,” *Physical Review Letters*, vol. 74, no. 16, pp. 3273–3276, Apr. 1995. [Cited on page 10.]
- [35] E. B. Myers, D. C. Ralph, J. A. Katine, R. N. Louie, and R. A. Buhrman, “Current-Induced Switching of Domains in Magnetic Multilayer Devices,” *Science*, vol. 285, no. 5429, pp. 867–870, Aug. 1999. [Cited on page 10.]
- [36] Y. Huai, F. Albert, P. Nguyen, M. Pakala, and T. Valet, “Observation of spin-transfer switching in deep submicron-sized and low-resistance magnetic tunnel junctions,” *Applied Physics Letters*, vol. 84, no. 16, pp. 3118–3120, Apr. 2004. [Cited on page 10.]
- [37] S. Yuasa, A. Fukushima, T. Nagahama, K. Ando, and Y. Suzuki, “High Tunnel Magnetoresistance at Room Temperature in Fully Epitaxial Fe/MgO/Fe Tunnel Junctions due to Coherent Spin-Polarized Tunneling,” *Japanese Journal of Applied Physics*, vol. 43, no. 4B, pp. L588–L590, Apr. 2004. [Cited on page 10.]
- [38] S. S. P. Parkin, C. Kaiser, A. Panchula, P. M. Rice, B. Hughes, M. Samant, and S.-H. Yang, “Giant tunnelling magnetoresistance at room temperature with MgO (100)

- tunnel barriers,” *Nature Materials*, vol. 3, no. 12, pp. 862–867, Dec. 2004. [Cited on page 10.]
- [39] S. Yuasa, T. Nagahama, A. Fukushima, Y. Suzuki, and K. Ando, “Giant room-temperature magnetoresistance in single-crystal Fe/MgO/Fe magnetic tunnel junctions,” *Nature Materials*, vol. 3, no. 12, pp. 868–871, Dec. 2004. [Cited on page 11.]
- [40] F. Iga, Y. Yoshida, S. Ikeda, T. Hanyu, H. Ohno, and T. Endoh, “Time-Resolved Switching Characteristic in Magnetic Tunnel Junction with Spin Transfer Torque Write Scheme,” *Japanese Journal of Applied Physics*, vol. 51, no. 2S, pp. 02BM02–5, Feb. 2012. [Cited on page 11.]
- [41] S. Amara-Dababi, R. Sousa, H. Béa, C. Baraduc, K. Mackay, and B. Dieny, “Break-down mechanisms in MgO based magnetic tunnel junctions and correlation with low frequency noise,” *Microelectronics Reliability*, vol. 53, no. 9-11, pp. 1239–1242, Sep. 2013. [Cited on page 11.]
- [42] N. Sakimura, T. Sugibayashi, T. Honda, H. Honjo, S. Saito, T. Suzuki, N. Ishiwata, and S. Tahara, “MRAM Cell Technology for Over 500-MHz SoC,” *IEEE Journal of Solid-State Circuits*, vol. 42, no. 4, pp. 830–838, Apr. 2007. [Cited on page 11.]
- [43] Q. Shao, P. Li, L. Liu, H. Yang, S. Fukami, A. Razavi, H. Wu, K. Wang, F. Freimuth, Y. Mokrousov, M. D. Stiles, S. Emori, A. Hoffmann, J. Akerman, K. Roy, J.-P. Wang, S.-H. Yang, K. Garello, and W. Zhang, “Roadmap of Spin–Orbit Torques,” *IEEE Transactions on Magnetics*, vol. 57, no. 7, pp. 1–39, Jul. 2021. [Cited on pages x, 11, 12, 20, and 58.]
- [44] A. Van Den Brink, S. Cosemans, S. Cornelissen, M. Manfrini, A. Vaysset, W. Van Roy, T. Min, H. J. M. Swagten, and B. Koopmans, “Spin-Hall-assisted magnetic random access memory,” *Applied Physics Letters*, vol. 104, no. 1, pp. 012403–3, Jan. 2014. [Cited on page 12.]
- [45] N. Sato, F. Xue, R. M. White, C. Bi, and S. X. Wang, “Two-terminal spin–orbit torque magnetoresistive random access memory,” *Nature Electronics*, vol. 1, no. 9, pp. 508–511, Sep. 2018. [Cited on page 12.]
- [46] M. Wang, W. Cai, D. Zhu, Z. Wang, J. Kan, Z. Zhao, K. Cao, Z. Wang, Y. Zhang, T. Zhang, C. Park, J.-P. Wang, A. Fert, and W. Zhao, “Field-free switching of a perpendicular magnetic tunnel junction through the interplay of spin–orbit and spin-transfer

- torques," *Nature Electronics*, vol. 1, no. 11, pp. 582–588, Nov. 2018. [Cited on pages x and 12.]
- [47] X. Li, S.-J. Lin, M. Dc, Y.-C. Liao, C. Yao, A. Naeemi, W. Tsai, and S. X. Wang, "Materials Requirements of High-Speed and Low-Power Spin-Orbit-Torque Magnetic Random-Access Memory," *IEEE Journal of the Electron Devices Society*, vol. 8, pp. 674–680, 2020. [Cited on page 12.]
- [48] K. V. Klitzing, G. Dorda, and M. Pepper, "New Method for High-Accuracy Determination of the Fine-Structure Constant Based on Quantized Hall Resistance," *Physical Review Letters*, vol. 45, no. 6, pp. 494–497, Aug. 1980. [Cited on page 13.]
- [49] C. Kane, "Topological Band Theory and the $\mathbb{Z}_2$ Invariant," in *Contemporary Concepts of Condensed Matter Science*. Elsevier, 2013, vol. 6, pp. 3–34. [Cited on pages 13 and 14.]
- [50] J. Gu and K. Sun, "Adiabatic continuity, wave-function overlap, and topological phase transitions," *Physical Review B*, vol. 94, no. 12, pp. 125 111–15, Sep. 2016. [Cited on page 14.]
- [51] D. J. Thouless, M. Kohmoto, M. P. Nightingale, and M. Den Nijs, "Quantized Hall Conductance in a Two-Dimensional Periodic Potential," *Physical Review Letters*, vol. 49, no. 6, pp. 405–408, Aug. 1982. [Cited on page 14.]
- [52] M. Haki, "Magneto-optics of relativistic-like electrons in 3D solids," Ph.D. dissertation, 2017. [Cited on pages x and 14.]
- [53] S. Hikami, A. I. Larkin, and Y. Nagaoka, "Spin-Orbit Interaction and Magnetoresistance in the Two Dimensional Random System," *Progress of Theoretical Physics*, vol. 63, no. 2, pp. 707–710, Feb. 1980. [Cited on page 17.]
- [54] R. Gracia-Abad, S. Sangiao, C. Bigi, S. Kumar Chaluvadi, P. Orgiani, and J. M. De Teresa, "Omnipresence of Weak Antilocalization (WAL) in Bi₂Se₃ Thin Films: A Review on Its Origin," *Nanomaterials*, vol. 11, no. 5, pp. 1077–20, Apr. 2021. [Cited on page 17.]
- [55] J. Tian, I. Childres, H. Cao, T. Shen, I. Miotkowski, and Y. P. Chen, "Topological insulator based spin valve devices: Evidence for spin polarized transport of spin-momentum-locked topological surface states," *Solid State Communications*, vol. 191, pp. 1–5, Aug. 2014. [Cited on page 17.]

- [56] S. Mukhopadhyay, P. K. Pal, S. Manna, C. Mitra, and A. Barman, “All-optical observation of giant spin transparency at the topological insulator BiSbTe_{1.5}Se_{1.5}/Co₂₀Fe₆₀B₂₀ interface,” *NPG Asia Materials*, vol. 15, no. 1, p. 57, Oct. 2023. [Cited on page 17.]
- [57] N. H. D. Khang, S. Nakano, T. Shirokura, Y. Miyamoto, and P. N. Hai, “Ultralow power spin–orbit torque magnetization switching induced by a non-epitaxial topological insulator on Si substrates,” *Scientific Reports*, vol. 10, no. 1, pp. 12 185–12, Jul. 2020. [Cited on pages x, 19, 54, and 58.]
- [58] J. Han, A. Richardella, S. A. Siddiqui, J. Finley, N. Samarth, and L. Liu, “Room-Temperature Spin-Orbit Torque Switching Induced by a Topological Insulator,” *Physical Review Letters*, vol. 119, no. 7, pp. 077 702–5, Aug. 2017. [Cited on page 19.]
- [59] Q. Shao, H. Wu, Q. Pan, P. Zhang, L. Pan, K. Wong, X. Che, and K. L. Wang, “Room Temperature Highly Efficient Topological Insulator/Mo/CoFeB Spin-Orbit Torque Memory with Perpendicular Magnetic Anisotropy,” in *2018 IEEE International Electron Devices Meeting (IEDM)*. San Francisco, CA: IEEE, Dec. 2018, pp. 36.3.1–36.3.4. [Cited on pages 20 and 58.]
- [60] S. N. Kajale, T. Nguyen, N. T. Hung, M. Li, and D. Sarkar, “Field-free deterministic switching of all–van der Waals spin-orbit torque system above room temperature,” *Science Advances*, vol. 10, no. 11, pp. eadk8669–7, Mar. 2024. [Cited on page 20.]
- [61] M. Wang, J. Zhou, X. Xu, T. Zhang, Z. Zhu, Z. Guo, Y. Deng, M. Yang, K. Meng, B. He, J. Li, G. Yu, T. Zhu, A. Li, X. Han, and Y. Jiang, “Field-free spin-orbit torque switching via out-of-plane spin-polarization induced by an antiferromagnetic insulator/heavy metal interface,” *Nature Communications*, vol. 14, no. 1, pp. 2871–10, May 2023. [Cited on pages 20 and 58.]
- [62] L. You, O. Lee, D. Bhowmik, D. Labanowski, J. Hong, J. Bokor, and S. Salahuddin, “Switching of perpendicularly polarized nanomagnets with spin orbit torque without an external magnetic field by engineering a tilted anisotropy,” *Proceedings of the National Academy of Sciences*, vol. 112, no. 33, pp. 10 310–10 315, Aug. 2015. [Cited on page 20.]
- [63] Z. Zhao, A. K. Smith, M. Jamali, and J.-P. Wang, “External-Field-Free Spin Hall Switching of Perpendicular Magnetic Nanopillar with a Dipole-Coupled Composite Structure,” *Advanced Electronic Materials*, vol. 6, no. 5, pp. 1 901 368–9, May 2020. [Cited on page 20.]

- [64] M. Moreira, “Topological Insulators: Flexing the Properties of Bismuth Telluride,” Master’s thesis, FCUP, 2023. [Cited on pages x and 21.]
- [65] M. Moreira, A. L. Pires, S. Ferreira-Teixeira, O. Iurkevich, R. Vilarinho, E. V. Castro, and A. M. Pereira, “Promoting Surface Conduction through Scalable Structure Engineering of Flexible Topological Insulator Thin Films,” *Advanced Functional Materials*, pp. 2405057–13, Aug. 2024. [Cited on pages x, 21, and 30.]
- [66] A. M. Miller, M. Lemon, M. A. Choffel, S. R. Rich, F. Harvel, and D. C. Johnson, “Extracting information from X-ray diffraction patterns containing Laue oscillations,” *Zeitschrift für Naturforschung B*, vol. 77, no. 4-5, pp. 313–322, May 2022. [Cited on page 24.]
- [67] M. Morota, S. Hatayama, Y. Shuang, S. Mori, Y. Sutou, P. Fons, and Y. Saito, “Interfacial reaction behavior between ferromagnetic CoFeB and the topological insulator Sb₂Te₃,” *Surfaces and Interfaces*, vol. 51, pp. 104486–8, Aug. 2024. [Cited on page 27.]
- [68] N. Bhattacharjee, K. Mahalingam, A. Fedorko, A. Will-Cole, J. Ryu, M. Page, M. McConney, H. Fang, D. Heiman, and N. X. Sun, “Effects of Crystalline Disorder on Interfacial and Magnetic Properties of Sputtered Topological Insulator/Ferromagnet Heterostructures,” *ACS Applied Electronic Materials*, vol. 4, no. 9, pp. 4288–4297, Sep. 2022. [Cited on page 27.]
- [69] W. Yang, J. Liu, X. Yu, G. Wang, Z. Zheng, J. Guo, D. Chen, Z. Qiu, and D. Zeng, “The Preparation of High Saturation Magnetization and Low Coercivity FeCo Soft Magnetic Thin Films via Controlling the Thickness and Deposition Temperature,” *Materials*, vol. 15, no. 20, pp. 7191–9, Oct. 2022. [Cited on pages 27 and 30.]
- [70] Y. Fu, X. Cheng, and Z. Yang, “Magnetically soft and high-saturation-magnetization FeCo films fabricated by co-sputtering,” *physica status solidi (a)*, vol. 202, no. 6, pp. 1150–1154, May 2005.
- [71] X.-L. Liu, L.-S. Wang, Q. Luo, L. Xu, B.-B. Yuan, and D.-L. Peng, “Preparation and high-frequency soft magnetic property of FeCo-based thin films,” *Rare Metals*, vol. 35, no. 10, pp. 742–746, Oct. 2016. [Cited on pages 27 and 30.]
- [72] Z. Wei, D. Navas, S. A. Bunyaev, M. Abellan, C. Garcia, G. N. Kakazei, and M. Vazquez, “Static and dynamical behaviour of magnetically coupled Co/Cu/CoFeB trilayers,” *Journal of Magnetism and Magnetic Materials*, vol. 589, pp. 171584–12, Jan. 2024. [Cited on page 28.]

- [73] G. B. G. Stenning, L. R. Shelford, S. A. Cavill, F. Hoffmann, M. Haertinger, T. Hesjedal, G. Woltersdorf, G. J. Bowden, S. A. Gregory, C. H. Back, P. A. J. D. Groot, and G. V. D. Laan, "Magnetization dynamics in an exchange-coupled NiFe/CoFe bilayer studied by x-ray detected ferromagnetic resonance," *New Journal of Physics*, vol. 17, no. 1, p. 013019, Jan. 2015. [Cited on page 28.]
- [74] B. K. Kuanr, V. Veerakumar, A. V. Kuanr, K. Lingam, S. R. Mishra, R. E. Camley, and Z. Celinski, "High frequency study of core-shell and uncoated Fe₃O₄ nanoparticles," *Journal of Applied Physics*, vol. 111, no. 7, pp. 07B542–3, Apr. 2012. [Cited on pages 29 and 30.]
- [75] B. K. Kuanr, S. Mishra, L. Wang, D. DelConte, D. Neupane, V. Veerakumar, and Z. Celinski, "Frequency and field dependent dynamic properties of CoFe 2-x Al x O 4 ferrite nanoparticles," *Materials Research Bulletin*, vol. 76, pp. 22–27, Apr. 2016. [Cited on pages 29 and 30.]
- [76] S. S. Kalarickal, P. Krivosik, M. Wu, C. E. Patton, M. L. Schneider, P. Kabos, T. J. Silva, and J. P. Nibarger, "Ferromagnetic resonance linewidth in metallic thin films: Comparison of measurement methods," *Journal of Applied Physics*, vol. 99, no. 9, pp. 093 909–7, May 2006. [Cited on pages 30 and 76.]
- [77] J. A. Bruce, S. R. Dupuis, R. Gleason, and H. Linde, "Effect of Humidity on Photore-sist Performance," *Journal of The Electrochemical Society*, vol. 144, no. 9, pp. 3169–3174, Sep. 1997. [Cited on page 40.]
- [78] J. Lindemuth and B. Dodrill, *Hall Effect Measurement Handbook: A Fundamental Tool for Semi-conductor Material Characterization*. Lake Shore Cryotronics, Incorporated, 2020. [Cited on pages xi and 42.]
- [79] Wenjin. Xu, "Anomalous Hall Effect in Ferromagnetic Metallic Thin Films." Ph.D. dis-
sertation, 2010. [Cited on pages 47, 51, and 52.]
- [80] H. Weng, X. Dai, and Z. Fang, "From Anomalous Hall Effect to the Quantum Anoma-
lous Hall Effect," 2015. [Cited on pages xi and 47.]
- [81] C. Zeng, Y. Yao, Q. Niu, and H. H. Weitering, "Linear Magnetization Dependence
of the Intrinsic Anomalous Hall Effect," *Physical Review Letters*, vol. 96, no. 3, pp.
037 204–4, Jan. 2006. [Cited on page 50.]

- [82] H. H. Huy, J. Sasaki, N. H. D. Khang, S. Namba, P. N. Hai, Q. Le, B. York, C. Hwang, X. Liu, M. Gribelyuk, X. Xu, S. Le, R. Nagabhirava, M. Ho, and H. Takano, "Large Spin Hall Angle in Sputtered BiSb Topological Insulator on Top of Various Ferromagnets With In-Plane Magnetization for SOT Reader Application," *IEEE Transactions on Magnetics*, vol. 59, no. 3, pp. 1–4, Mar. 2023. [Cited on page 51.]
- [83] Y. Ni, Z. Zhang, I. C. Nlebedim, and D. C. Jiles, "Ultrahigh Sensitivity of Anomalous Hall Effect Sensor Based on Cr-Doped Bi₂Te₃ Topological Insulator Thin Films," *IEEE Transactions on Magnetics*, vol. 52, no. 7, pp. 1–4, Jul. 2016. [Cited on pages 53 and 56.]
- [84] P. Chen, Y. Zhang, Q. Yao, F. Tian, L. Li, Z. Qi, X. Liu, L. Liao, C. Song, J. Wang, J. Xia, G. Li, D. M. Burn, G. Van Der Laan, T. Hesjedal, S. Zhang, and X. Kou, "Tailoring the Hybrid Anomalous Hall Response in Engineered Magnetic Topological Insulator Heterostructures," *Nano Letters*, vol. 20, no. 3, pp. 1731–1737, Mar. 2020. [Cited on pages xii and 53.]
- [85] P. He, H. Isobe, D. Zhu, C.-H. Hsu, L. Fu, and H. Yang, "Quantum frequency doubling in the topological insulator Bi₂Se₃," *Nature Communications*, vol. 12, no. 1, pp. 698–7, Jan. 2021. [Cited on page 54.]
- [86] K. Wang, Y. Zhang, S. Zhou, and G. Xiao, "Micron-Scale Anomalous Hall Sensors Based on FePt_{1-x} Thin Films with a Large Hall Angle and near the Spin-Reorientation Transition," *Nanomaterials*, vol. 11, no. 4, pp. 854–10, Mar. 2021. [Cited on page 55.]
- [87] J.-B. Schell, J.-B. Kammerer, L. Hebrard, E. Breton, D. Gounot, L. Cuvillon, and M. De Mathelin, "Towards a Hall effect magnetic tracking device for MRI," in *2013 35th Annual International Conference of the IEEE Engineering in Medicine and Biology Society (EMBC)*. Osaka: IEEE, Jul. 2013, pp. 2964–2967. [Cited on page 56.]
- [88] F. Xue, S.-J. Lin, M. Song, W. Hwang, C. Klewe, C.-M. Lee, E. Turgut, P. Shafer, A. Vaillonis, Y.-L. Huang, W. Tsai, X. Bao, and S. X. Wang, "Field-free spin-orbit torque switching assisted by in-plane unconventional spin torque in ultrathin [Pt/Co]N," *Nature Communications*, vol. 14, no. 1, pp. 3932–9, Jul. 2023. [Cited on page 57.]
- [89] J. Sasaki, H. H. Huy, N. H. D. Khang, P. N. Hai, Q. Le, B. York, X. Liu, S. Le, C. Hwang, M. Ho, and H. Takano, "Improvement of the Effective Spin Hall Angle by Inserting an

Interfacial Layer in Sputtered BiSb Topological Insulator (Bottom)/Ferromagnet With In-Plane Magnetization,” *IEEE Transactions on Magnetics*, vol. 58, no. 4, pp. 1–4, Apr. 2022. [Cited on page 61.]

[90] A. Kurenkov, S. DuttaGupta, C. Zhang, S. Fukami, Y. Horio, and H. Ohno, “Artificial Neuron and Synapse Realized in an Antiferromagnet/Ferromagnet Heterostructure Using Dynamics of Spin–Orbit Torque Switching,” *Advanced Materials*, vol. 31, no. 23, pp. 1900636–7, Jun. 2019. [Cited on page 62.]

[91] M. Cleanrooms, “Clean Room Classifications (ISO 8, ISO 7, ISO 6, ISO 5),” available at: <https://www.mecart-cleanrooms.com/learning-center/cleanroom-classifications-iso-8-iso-7-iso-6-iso-5/>. Accessed August 13, 2024. [Cited on pages xii and 79.]

A Thermal annealing

Thermal annealing is a cost-effective and scalable technique widely used to enhance the structural organization and transport properties of materials. In this study, annealing was performed on both continuous thin film samples and patterned Hall bar samples (without electrical contacts to prevent diffusion). The annealed continuous films were primarily used for morphological and magnetic characterization, while the patterned Hall bars were prepared for Hall resistance measurements.

The samples underwent slow-cooling thermal annealing, in absence of applied magnetic field, at 300°C for 1 hour, under constant Argon flow of 150 mmHg to prevent oxidation. The annealing process was conducted in the cylindrical furnace illustrated in fig.A.1.

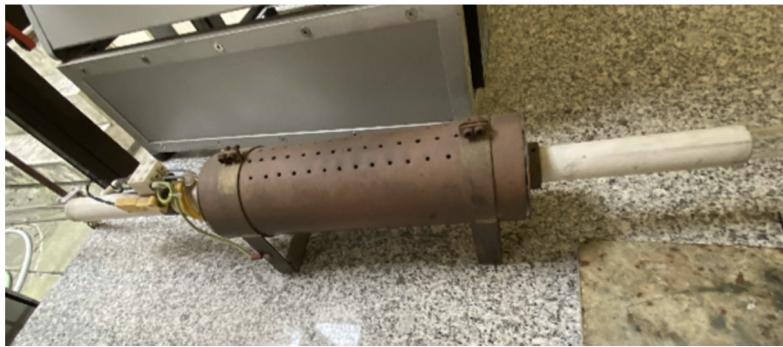


FIGURE A.1: Annealing furnace

It is important to note that for the annealing inside the furnace, the samples were placed inside a quartz tube with a diameter of 1.7 cm, requiring the samples to be cut slightly smaller than this diameter for proper insertion. For the Hall bar samples, the masks were designed symmetrically, allowing each sample to be cut in half horizontally. This design enabled the fitting of two Hall bar structures within the quartz tube without compromising space for subsequent lithography steps, such as the patterning of electrical contacts.

B VNA-FMR Experimental Set-up

Vector Network Analyzers (VNA)s characterize high-frequency devices, normally in the radio-frequency range. The VNA uses an impedance matching procedure, where a known signal is send to a device under test, and measurements are taken of the incident, transmitted and reflected signal, calculating the different ratios between these waves ^{*}.

For this study, the components were already assembled in a proper table, which allowed only unidirectional movement for once the circuit was connected no changes in the cables shapes would be made. This is an important consideration as the experiment deals with very small amplitude signals, and the cables presented in the laboratory were Teflon cables which means that different set-up shapes of the cables need different calibrations parameters run by the VNA.

A VNA *ANRITSU 37247D*, illustrated in fig.B.1.a), was connected to the sample by a three contact way. The cables were chosen to have 50Ω impedance to match the VNA own impedance and this way minimize reflected signals. A pair of Helmholtz coils were used to create the applied static magnetic field $\vec{H}_{ext}$. As the diameter of the coils were at the order of half a meter (fig.B.1.b)), heating effects were taken into account : water was used to fill the coils chamber with the power supply monitoring it's temperature. The samples were placed at the center of a Coplanar Wave Guide (CPW) (fig.B.1.c)), facing down, in a way that the changing microwave field $\vec{h}$ was perpendicular to the applied external field $\vec{H}_{ext}$ and to the sample magnetization $\vec{M}$.

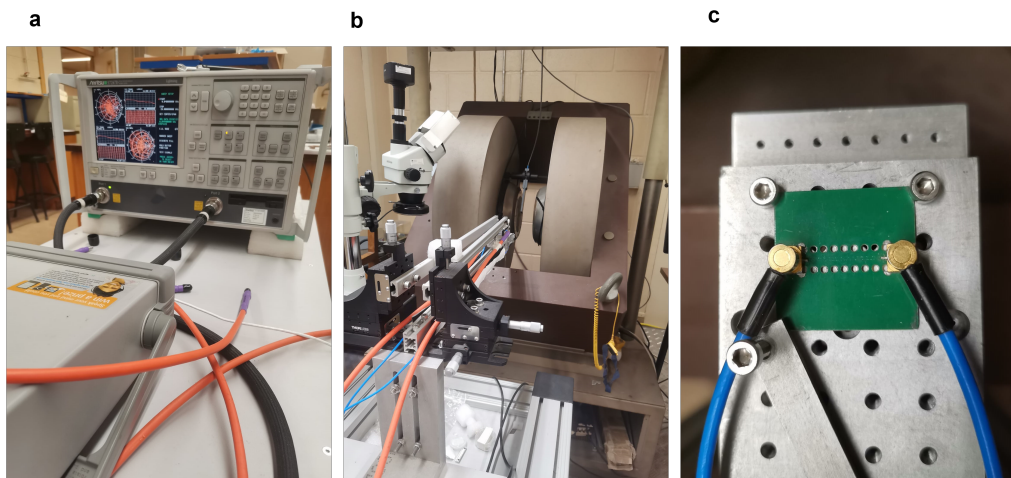


FIGURE B.1: a) VNA ANRITSU 37247D; b) Helmholtz coils to produce static external magnetic field and c) Coplanar Waveguide.

^{*}More information about the technique can be found in [76].

C Lithography

In fig.C.1, it is represented the test device B through the many stages from drawing to deposited material. This pattern served to test the alignment stage, in the benchtop set-up, after depositing the test device A.

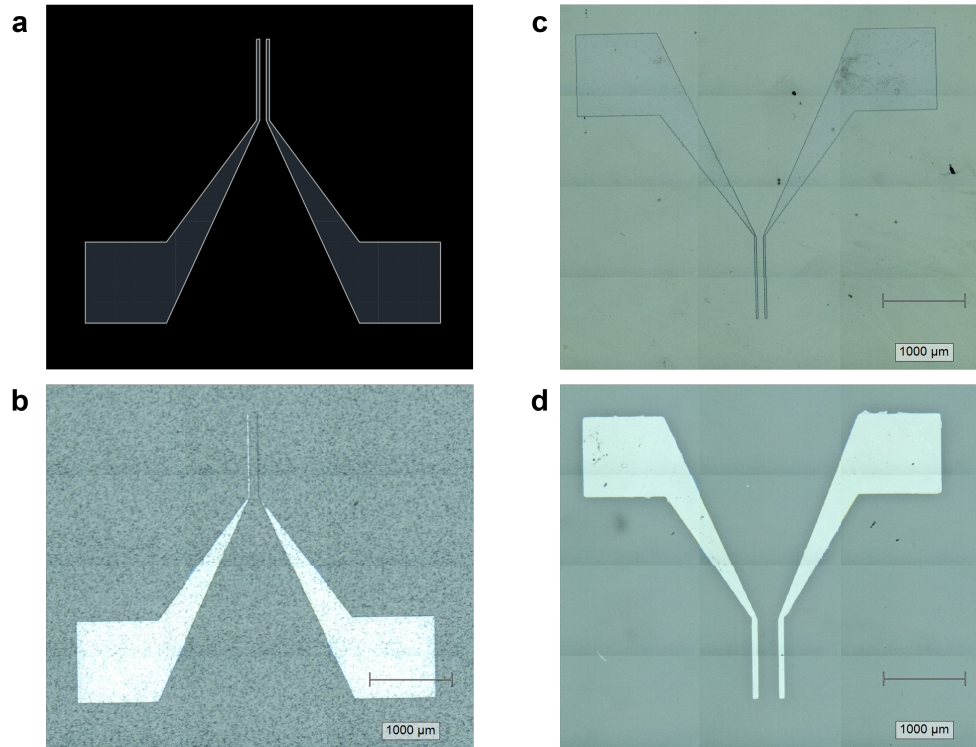


FIGURE C.1: Test device B. a) AutoCAD drawing. b) Microscope image of printed mask. c) Patterned photoresist. d) Deposited Bi₂Te₃.

C.1 Clean Room Parameters and Classification

In fig.C.2 are illustrated commonly employed methods to achieve low level contamination, crucial for the proper functioning of a clean room.

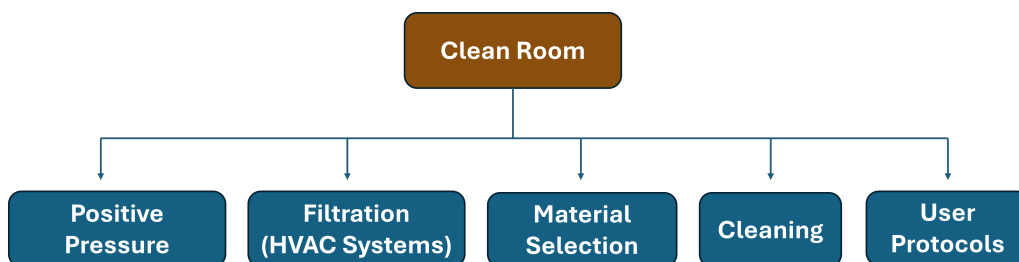


FIGURE C.2: Scheme of clean room strategies to prevent contamination.

The positive pressure*, prevents contaminants from entering a cleanroom by maintaining the pressure inside the room higher than in the surrounding areas. Air is continuously supplied to the cleanroom, creating a higher pressure that causes air to flow out of the room whenever doors or other openings are used.

The HVAC (Heating, Ventilation and Air Conditioning) systems allow the filtration of airborne particles and the employment of turbulent or laminar flow of air. It is also responsible to circulate clean filtered air through the cleanroom multiple times per hour and to control the humidity (between 45- 55 % relative humidity), and temperature (between 18-22°C). In our cleanroom, HEPA (High-Efficiency Particulate Air) are used. These filters employ fiber glass in a paper like-medium and can capture particles as small as 0.3 micrometers with an efficiency of 99.97%.

In terms of material selection, these are carefully selected to minimize particle shedding. For example, walls, floors, and ceilings are made from smooth, non-porous, and easily cleanable materials like epoxy-coated surfaces and stainless steel. The cleaning follows a regular weekly protocol.

Finally, personnel entering a cleanroom must follow user protocols such as wearing sterile suits, gloves, masks, and shoe covers to prevent contamination. Controlled entry and exit protocols require the use of airlocks or gowning rooms to don and remove cleanroom vestments, minimizing the introduction of contaminants.

Cleanrooms can be classified based on the concentration of airborne particles allowed, according to the ISO 14644-1, that is an international standard for industries that require controlled environments with specific air cleanliness levels.

It defines several cleanroom classes: ISO 1, ISO 2, ISO 3, ISO 4, ISO 5, ISO 6, ISO 7, ISO 8, and ISO 9. ISO 1 represents the highest level of cleanliness, while ISO 9 is the least stringent. However, even an ISO 9 cleanroom is significantly cleaner than a typical room. There is an equivalency between the ISO standards and the United States Federal Standard 209E (FS 209E), that was for many years the standard widely used. Fig.C.3 shows a table that defines the classification of a cleanroom based on the maximum concentration of particles of a specified size. For example, for an ISO 3 (Class 1 in FS 209E) cleanroom, the concentration of particles with size equal or above 0.1 μm should be lower than 1000 particles/ m^3 .

*Typically +25 Pa (relative to adjacent areas) for Class 100, 1000, 10,000 and +75/ +100 Pa for Class 1 and 10.

ISO Class	Maximum Particles/m ³						Particles/ft ³	FS 209E Equivalent
	≥0.1μm	≥0.2μm	≥0.3μm	≥0.5μm	≥1μm	≥5μm	≥0.5μm	
ISO 1	10							
ISO 2	100	24	10					
ISO 3	1,000	237	102	35			1	Class 1
ISO 4	10,000	2,370	1,020	352	83		10	Class 10
ISO 5	100,000	23,700	10,200	3,520	832	29	100	Class 100
ISO 6	1,000,000	237,000	102,000	35,200	8,320	293	1,000	Class 1,000
ISO 7				352,000	83,200	2,930	10,000	Class 10,000
ISO 8				3,520,000	832,000	29,300	100,000	Class 100,000
ISO 9				35,200,000	8,320,000	293,000		

FIGURE C.3: ISO clean room standards and FS209E equivalent. From [91].s

C.2 Alignment Errors

For patterning the electrical contacts a second lithography process is needed. During this, several challenges arose:

- Thickness mismatch between photoresist and thin film:** The photoresist layer, after spin-coating at 2500 RPM, has a thickness of 2.5 μm, while the deposited films are only 10-20 nm thick. As a result, the alignment marks viewed through the Mask Aligner's microscope were obscured by a layer of photoresist that is 1000 times thicker, despite being almost completely transparent. Additionally, the thin films, being only 10-20 nm thick, exhibited some degree of transparency themselves, further complicating the visual alignment process.
- Experimental Accuracy and Contact Area:** When designing the masks, it was assumed that the contacts would not deviate much from the drawing. However, as later found in the fabrication of the devices, experimental accuracy*, can affect the process. Consequently, to account for potential alignment failure, the design of the contacts has to be done with greater tolerance, to promote a larger area of contact between active device region and contacts.

*It refers to the experimentalist's ability to execute the alignment.

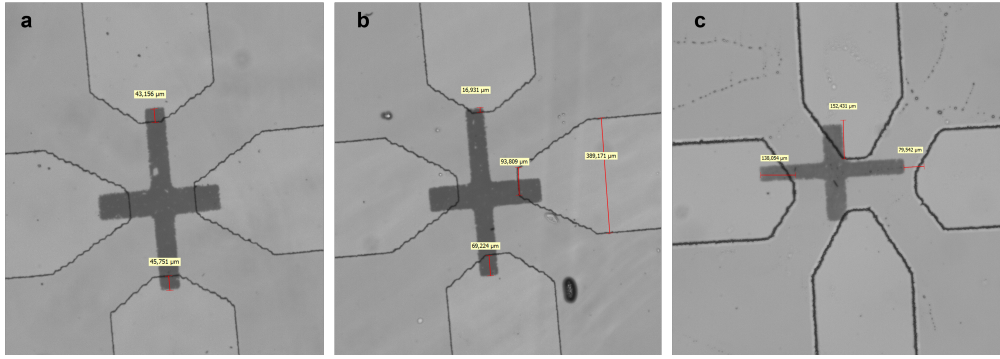


FIGURE C.4: Microscope Image of photoresist after development in second lithography process, for electrical contacts: a) Good Alignment, b) Vertical Misalignment and c) Vertical and Horizontal Misalignment.

For example, in fig.C.4 we have three different devices with patterned contacts in photoresist. In fig.C.4.a), we have a vertical misalignment of about $2 \mu\text{m}$ (which is not bad given the dimensions of our device). On the other hand, for the device in fig.C.4.b) there is a vertical misalignment of about $27 \mu\text{m}$, creating a small contact area on the top end, and a large one at the bottom. In this case, the unbalance could be undone just by designing the contacts with greater tolerance, in such a way that it would not influence the electrical measurements.

Finally, in fig.C.4.c), we have both vertical and horizontal misalignment in the order of $75 \mu\text{m}$ and $95 \mu\text{m}$, respectively. However, this time even with contacts that own larger tolerance, the misalignment would be pronounced.