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[Sputtering of Nitride Materials]

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ABSTRACT

Zinc Nitride now becomes an interest towards the semiconductor potential material on photovoltaics and optoelectronics application. The deposition method based on DC magnetron sputtering technique had been successfully developed in University of Sheffield using glass substrates. The activities towards Nitrogen flow rate and various glass substrate temperatures had been successfully evaluated. Results showed starting at 45 sccm Nitrogen flow rate provide a positive Zinc Nitride development and the increment of substrate temperature had maximizing the mobility performance. The developed Zinc Nitride refractive index had been calculated with results in the range of 2.28 to 3.13. The deposited Zinc Nitride thickness was in the range of 522 nm to 683 nm with 70 minutes deposition time in in each run. The refractive index for the deposited Zinc Nitride had been calculated which ranging from 2.28 to 3.13. Annealing with temperature 320°C for 1.5 minutes in Nitrogen environment had shown consistent electronic properties performance and no signs of physical oxidation.

Keywords: DC magnetron sputtering, Zinc Nitride, Nitrogen flow rate, substrate temperature.

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Chapter 1: Introduction

1.0 Zinc Nitride

Zinc Nitride becomes an interest towards semiconductor materials due to its convincing electrical and optical properties. Zinc Nitride is a combination of Zinc and Nitrogen which physically in opaque appearance and has been developed commercially towards the thin film transistor including solar panel applications. Deposited Zinc Nitride being reported to have a band gap with the range of 1.01 eV to 3.2 eV which depends on the deposition method and the growth condition [1]. Due to high electron mobility and high electrical conductivity performance it being used for optoelectronics applications as well. Zinc Nitride reported been developed with high mobility of $100 \text{ cm}^2 \text{ V}^{-1} \text{ s}^{-1}$ and carrier concentration with the value of 10^{20} cm^{-3} [2]. The activities on developing Zinc Nitride thin film deposition is become an interesting trend among the researchers since the material identified as new semiconductor potential material. The push towards low fabrication cost and green technology add a value towards it as well. Zinc Nitride will be developed first for Zinc Oxy-Nitride epitaxy and then being combined with Zinc Oxide to have very important components towards photovoltaic and optoelectronic fabrications [3]. DC magnetron sputtering was one of the lowest cost approached sputtering technique that being used for depositing Zinc Nitride thin layer process.

In this project, the initial target was to develop Zinc Nitride deposition layer on a glass substrate using DC Sputtering machine. In order to understand the parameter towards Zinc Nitride development, the Nitrogen flow rate had been assessed first. Once the Nitrogen rate had been defined, then the annealing and substrate temperature will be assessed to understand the effects on the deposition electrical properties performance. The transmittance and reflectance performance will be analyzed using J.A Woollam Vase Ellipsometer while deposition thickness will be measured using Dektak 150 surface profiler. The refractive index will be manually calculated based on the transmittance results. The electrical properties from Hall Effect measurement will be monitored weekly by using four probe Van der Pauw techniques. This is to understand the effect of annealing process towards the electrical properties stability and oxidation behavior.

1.1 Project background

Denton Explorer sputtering machine is one of the DC sputtering equipment which available in the University of Sheffield. Since the initial idea was to develop Zinc Nitride capability on the glass substrate based on the available source here, a lots of review had been done to ensure the important parameter properly being selected for

evaluation process. According to literature review, the sputtering equipment has the capability to develop Zinc Nitride but most of the activities were based on RF magnetron. The setup was based on combination of Argon and Nitrogen gas using Zinc as the sputtering target. The available sputtering equipment here however has some process limitation which only allows one type of gas during its operation. This will not allow gas mixture such as Argon and Nitrogen as reviewed in the literature review. However based on small evaluation Nitrogen had been selected towards this activities due to its convincing results towards Zinc Nitride thin layer deposition. The standard Denton Explorer sputtering machine has the RF/DC sputtering capabilities which connected with 3 cathodes, which one of the cathode being energized by Radio Frequency (RF) while the remaining two cathodes are powered by Direct Current (DC). Since the existing Denton Explorer sputtering machine for this project do not equipped with the RF generator, the DC sputtering method will be used. In this project setup, the cathode will be link with the Zinc target and the anode will react as the placement of glass substrates. From the literature review, there are only few activities using the DC magnetron sputtering and this will be a challenge towards Zinc Nitride deposition referencing which require a lot of evaluation activities in order to develop Zinc Nitride capability.

In these project activities, a backup plan had been considered if the Zinc Nitride not able to generate purely on the glass substrate using Zinc target. The proposal of having Zinc Nitride sputter target as a backup plan had been raised and included in the flow chart. Cathode shielding being applied for these activities to avoid cross zinc contamination in the sputter machine and this protection is very important to ensure the unwanted Zinc component from their sputtering samples as well. All risk assessment had been listed earlier which could be refer in Appendix 2 and the necessary training had been completed from Appendix 3.

Chapter 2: Literature review compilation

2.0 Previous evaluation activities

Deposition of Zinc Nitride had been successfully reported on unheated 0.75mm thickness glass substrate however the activities were based on RF magnetron sputtering. The sputtering gas involved were based on Argon (99.999% purity) and Nitrogen (99.999% purity) source gas with the 1:1 mixing ratio [4]. The Argon gas being keeps flowing for 30 minutes to ensure the chamber free from oxygen. The base parameter for the RF magnetron sputtering being set using the range of 2×10^{-6} Torr to 5×10^{-6} Torr and the RF power density was set at 300 Watts. The deposition time took 30 minutes during the experiments and observed that annealing at 300°C for 1 hour will create oxide phase to the glass substrate. The experiment had shown results on the optical properties which shown as deposited Zinc Nitride and Zinc Nitride films after annealing process. Both of the thin films had a black and opaque appearance with wavelength for deposited Zinc Nitride at 750 nm and annealed Zinc Nitride at 900 nm which could be referred to Figure 1 below. The band gap of 1.15 eV and 1.4 eV were reported for the as deposited and anneal films towards the evaluations activities.

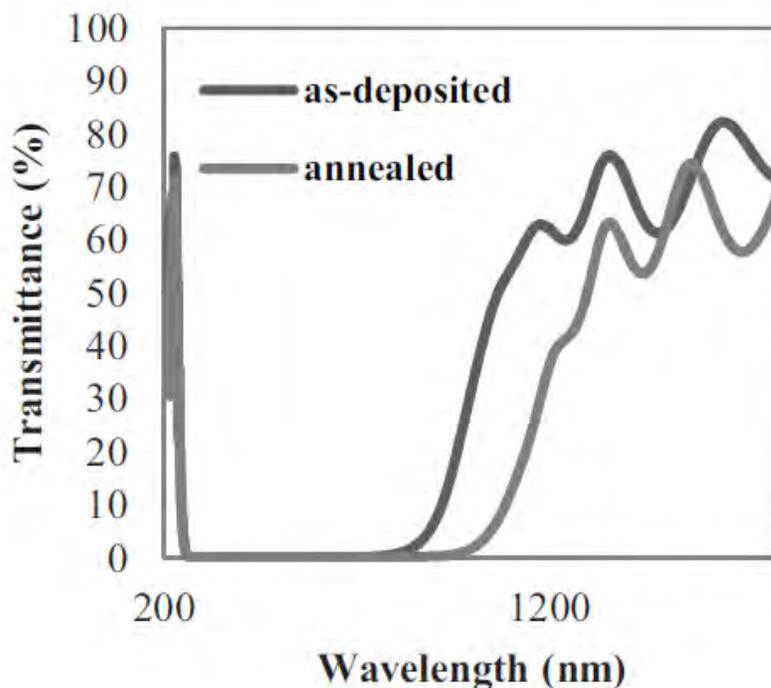


Figure 1: Wavelength performance on as deposited and annealed Zinc Nitride [3]

There were activities for Zinc Nitride deposition on RF sputtering by optimizing the RF power and the deposition time. RF power being evaluated from the range of 50 W to 250 W and deposition time setting from 5 minutes to 45 minutes with mixture of Argon (50%) and Nitrogen (50%). Zinc Nitride deposition which developed up to 150 W reported having transparent appearance with transmittance up to 90% and the optical transmittance wavelength results were in the range of 200 – 2500 nm [5].

Temperature evaluation had been assessed on Silicon and glass substrates using RF magnetron sputtering. The evaluated temperature in the range of 298 K to 523 K (25°C to 250°C) and results showed that the deposition thickness had inversed proportional effect towards the substrate performance which can be referred to Table 1.

Zinc Nitride deposition thickness (μm)	2.9	2.5	2.1	1.2	0.3
Substrate Temperature (K)	298	373	423	473	523

Table 1: Layer thickness performance versus substrate temperature

The reduction of the deposition thickness caused by the evaporation of Zinc thickness at higher temperature which limits the formation of its phase from growing. The Hall Effect measurement being carried out using four probe Van der Pauw techniques and results shown n-type deposition character with mobility ranging from 10 to 30 cm^2/Vs . The grain size being analyzed and results showed the grains size increased as temperature increased. The overall electrical properties had been analyzed in Figure 2 [1] with fluctuation results when the substrate temperature was being increasing. The inconsistent results occurred due to the fluctuation of deposition thickness which could be referred in Figure 3.

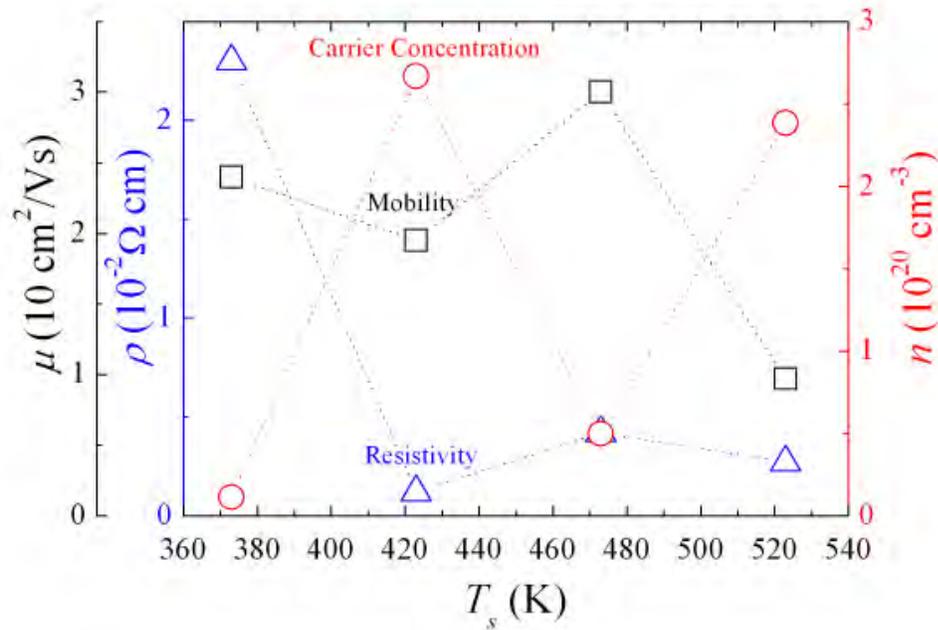


Figure 2: Electrical properties results towards the substrate temperature

The refractive index n had been calculated as well using the transmittance data based on the two lower valley observed in the graph shown in Figure 3. The refractive index were calculated based on below formula which in the range of 2.51 to 2.69 with the layer thickness d in the range of 1300 – 2300 nm.

$$n = [2 d (1/\lambda_2 - 1/\lambda_1)]^{-1}$$

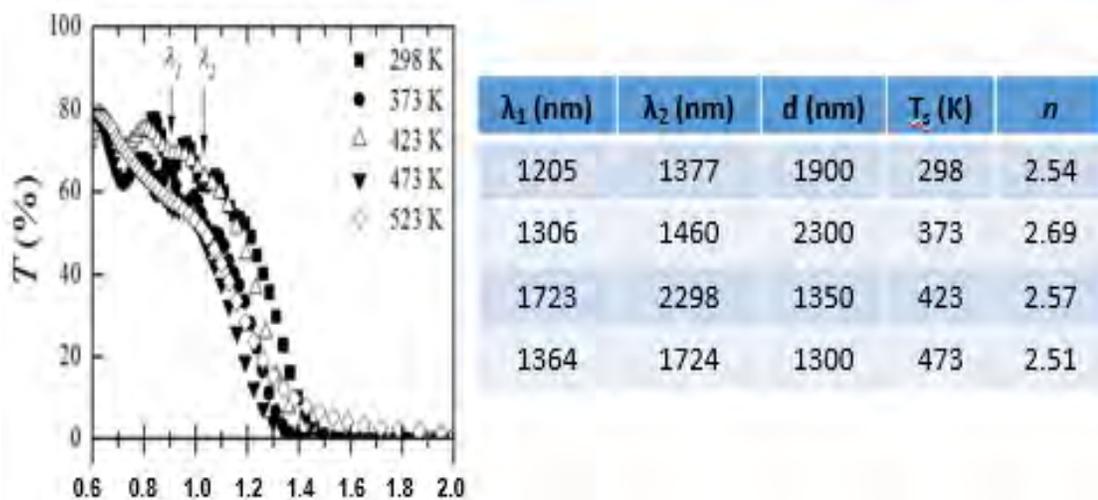


Figure 3: Calculation of refractive index n using transmittance data

Based on Zhang June [6] even though the objective of the project to generate Zinc Oxide on glass substrate, the method that being used was developing the Zinc Nitride first using the reactive magnetron sputtering approach. The Zinc Nitride being deposited first by using metallic Zinc disc with Gallium grain as the target which initially being sputter by Argon gas for 30 minutes to remove any target contamination at 40 sccm flow rate. The $N_2 - Ar$ were based on 50% mixture and sputtering time setting was 30 minutes with 120 W DC Power. The substrates temperature being set with two setting which room temperature and $200^\circ C$ with setting towards high temperature Zinc Nitride claimed has smoother surface performance.

There was some analysis towards oxidation on the Zinc Nitride films towards the oxidation time. In 1 hour the result showed that the structure of Zinc Nitride has partially changing to Zinc Oxide with increasing of oxidation time at $500^\circ C$ and at 2 hours the Zinc Nitride was completely changed to Zinc Oxide. This data could be referred to Figure 4 for reference. This is a very important information in order to avoid the oxidation occurred during the sputtering process.

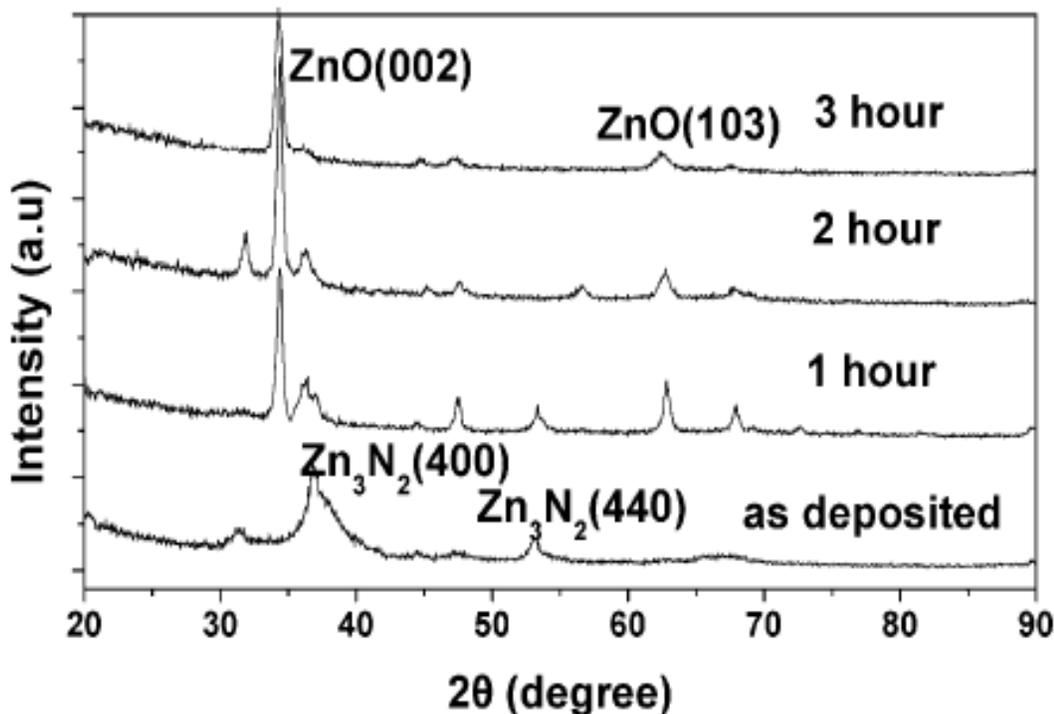


Figure 4: X-ray diffraction results of Zn_3N_2 substrates at $500^\circ C$ with different oxidation time [7]

According to Nanke Jiang [8], the Nitrogen has significant effects towards the Zinc Nitride surface performance. The higher the Nitrogen composition in sputtering gas mixture, the surface will get smoother with bigger and clearer grain size will be developed. For the planned evaluation process, the individual Nitrogen flow rate will be assessed on the sputtering machine. However to develop the Zinc Nitride, 10 cm of Zinc target distance had been used with silicon and glass substrate temperature ranging from

room temperature up to 400°C. The base pressure was set low at 10^{-6} Torr range with 80W RF Power at 5 minutes with Argon gas environment.

The usage of Zinc Nitride sputtering target had been assessed [9] by V.Kampylafka which was the backup plan that had been identified in this project if the Zinc target is not working. Results showed n-type Zinc Nitride deposited in pure Argon gas using RF Magnetron sputtering. The optical band gap of Zinc Nitride been reported having a range between 1 to 3eV depending the deposition method .The Zinc Nitride target was initially pre sputtered using Argon plasma with 5mTorr pressure, 100W RF power within 15-20 minutes deposition time. The Zinc Nitride then being deposited at 100W RF power, 5mTorr total pressure setting with mixture of Argon and Oxygen gases. The distance between target and substrate was 11 cm. Annealing was done at 400°C convert most of the Zinc into Zinc Nitride but small amount of oxygen residuals detected in the annealing process. Conductivity reported in the range of 10^{-2} to 10^1 Ω cm which depending on the deposition thickness. Zinc Nitride improved the conductivity after annealing at 300°C however annealing at higher temperature claimed to have higher resistivity and transparent physical appearance.

Another method being used for getting wider band gap (1.01 eV to 3.02 eV) Zinc Nitride from V.Kambilafka [10] which using the RF magnetron sputtering with Argon and Nitrogen gas mixture. The wide band gap material being produced to make it a suitable candidate for optoelectronics transparent devices. The Zinc Nitride deposition was based on 100W RF power with 5 mTorr pressure on unheated glass substrates. The deposited materials then went through thermal treatment with temperature up to 550°C. The results reported that with gas mixture of 20% Nitrogen and 80% Argon, substrates thermal treatment at 150 °C provided higher conductivity and mobility during the investigation while using 100% Nitrogen plasma the films were too resistive to be measured by the Hall effect measurements.

There was an approach using Ammonia (NH_3) gas which being mixed with Argon gas to generate the Zinc Nitride deposition method using RF magnetron sputtering. The deposition was done on unheated glass substrate with the Zinc target distance 60mm from the substrate. The mixture of NH_3 and Ar gas being varied with the total flow rate 20 sccm. The NH_3 flow rate setting were 1-5 sccm and balance mixture with Argon will based on the total flow rate mentioned earlier. From the results showed that Zinc Nitride crystallization depends on the Ammonia ratio. The Zinc Nitride crystallization becomes more dominant when the Ammonia ratio was being increased. The result could be referred clearly in Figure 5 for reference.

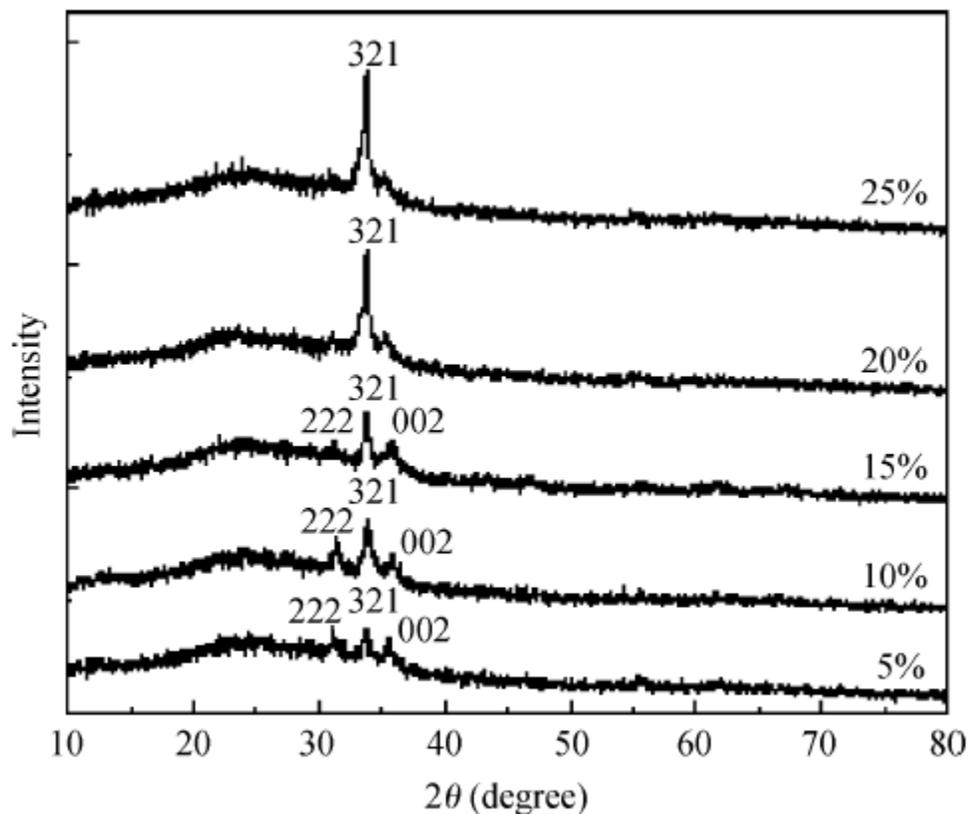


Figure 5: Effects of NH₃ ratio towards Zn₃N₂ (321) on X-ray diffraction

From the listed literature reviews, all the parameters that being used towards the sputtering activities had been compiled for referencing purpose. It is important to understand the key parameters that being used for the Zinc Nitride deposition process. The evaluation should be carried out to understand the capability of DC magnetron sputtering in The University of Sheffield and verify the effects of each defined parameters.

2.1 List of previous parameter compilations

Compilation of parameter from the literature review had been done in Table 2 to understand the previous work to develop Zinc Nitride or related sputtering activities. From the table, most of the works were using glass substrate for deposition activities and RF magnetron sputtering is the most common method used towards the Zinc Nitride deposition evaluation. This is an opportunity to develop Zinc Nitride using DC magnetron sputtering based on various Nitrogen flow rate and substrate temperatures to understand the existing sputtering capability.

Type	Target	Substrate	Substrate Temp	Target-Substrate distance	Working gas	Power (W)	Pressure (mTorr)	Deposition time	Reference
RF/M	ZnN	Glass	250-300 °C	N/A	Ar 100 sccm	50	8	15-20 min	[3]
RF/M	Zn	Glass	Unheated	N/A	1:1 Ar/N ₂	300	2-5 x 10 ⁻⁶	30 min	[4]
RF/M	Zn	Glass	Unheated	N/A	1:1 Ar/N ₂	50-250	N/A	5-45 min	[5]
RF/M	Zn	Glass	25 to 250°C	N/A	N ₂ 30 sccm	200	10	30 min	[1]
RF/M	Zn+Ga	Glass	Unheated & 200 °C	6.5 cm	1:1 Ar/N ₂ 40 sccm	120	15	30 min	[6]
DC/M	Zn	Glass	100-300 °C	6cm	1:1 Ar/N ₂ 40 sccm	100	15	30 min	[7]
RF/M	Zn	Silicon % Glass	Unheated up to 400 °C	10 cm	NA	80	0.01	5 min	[8]
RF	Zn	Thin Film Transistor (TFT)	NA	11 cm	Ar:O ₂	100	5	NA	[9]
RF/M	Zn	Glass	Unheated	NA	Ar:N	100	5	NA	[10]
RF/M	Zn	Glass	Unheated	6 cm	NH ₃ : Ar (1-5):(15-19) sccm	50	7.5	NA	[11]

Note: RF = Radio Frequency

DC = Direct Current

M = Magnetron Sputtering

Table 2: Literature review parameter compilation

2.2 DC magnetron sputtering operating process

In general, DC magnetron sputtering was using high voltage application which consists of anode and cathode for operating process. The sputtering target (which in this evaluation using Zinc) connected to the cathode which represented as the negative output of the DC power supply while the substrate (glass substrate) will be connected to anode in a vacuum chamber. The selected gas atoms then will be hitting the target and the target material will deposit a thin layer onto the glass substrate which being connected to anode. The magnetron function ensures the gas atom which was previously in random movement attracted and then being directed to the sputter target. The magnet actually being allocated behind the sputtering target to create the magnetic field around the target. With low gas pressure and high voltage which flows from the magnetron, it generated high energy plasma to bombard the target resulting to target coating layer to deposit on the glass substrate. The magnetron process functioning could be clearly seen below in Figure 6 for reference.

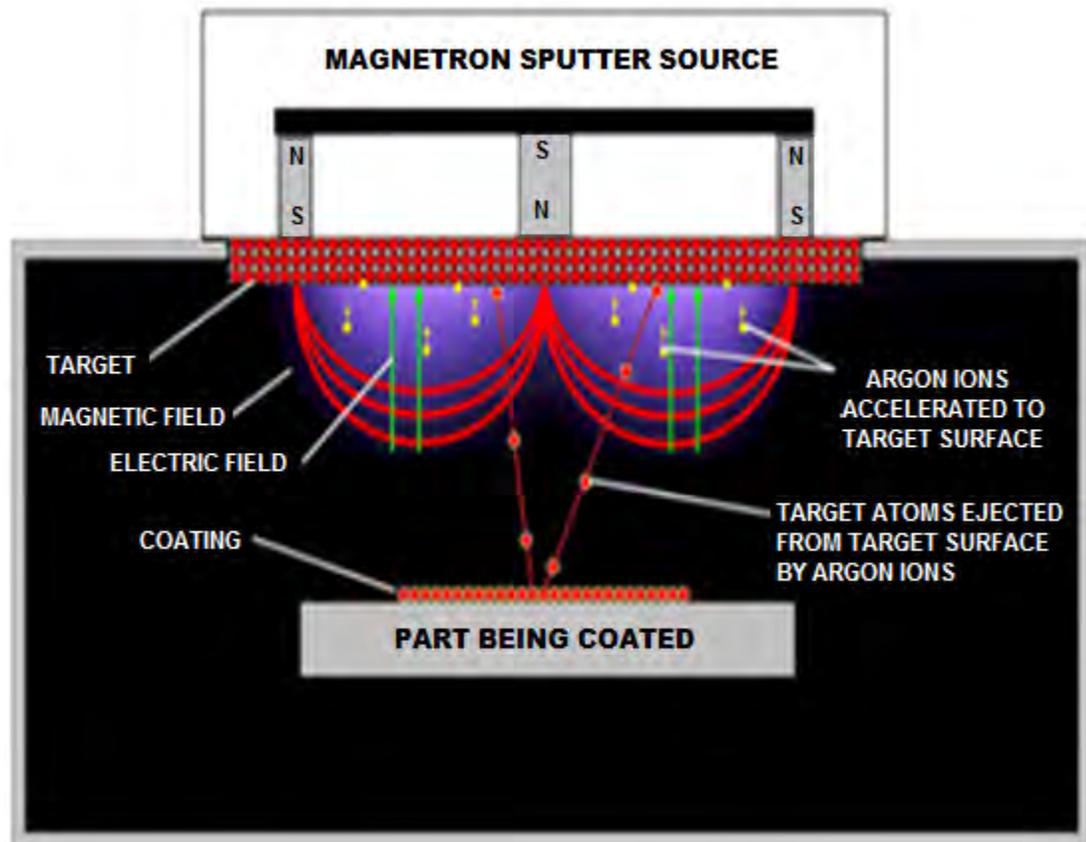


Figure 6: Magnetron process functioning

(Source from: http://www.marcoating.com/index_html_files/72.png)

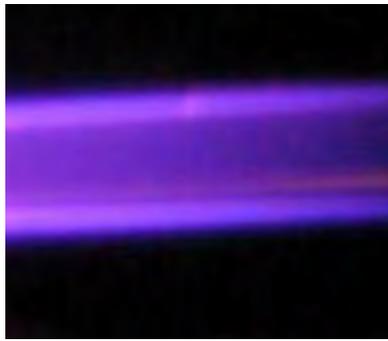
From earlier reference, Argon or Nitrogen gas added to the vacuum chamber and bombarding on Zinc target occurred through a Nitrogen plasma environment. The magnetron react as a directional for the bombarding towards the target which improves the deposition layer. The challenges start when there is a potential of an oxide layer combining with Zinc Nitride which effect the glass substrate performance. This is where the parameter optimization is required in order to produce a good Zinc Nitride deposition layer based on the defined parameters that being explained earlier. Further analysis will be done to check the results towards the performance of the Zinc Nitride on the glass substrates. This project will provide the ability of student to design an experiment with various parameter optimizations and learn a range of analysis techniques towards the semiconductor materials development which will be useful method for low fabrication cost. It is also an opportunity to setup the capability of depositing Zinc Nitride on glass substrate using DC magnetron sputtering machine in The University of Sheffield itself.

Chapter 3: Overall evaluation methods and activities

3.0 Methodology

The evaluation methodology had been reviewed and discussed with supervisor prior starting the data collection. The Gantt chart for the overall activities being finalized and can be referred to Appendix 1 for details. The key objective of this project was to develop Zinc Nitride sputtering process using the existing DC magnetron sputtering equipment and study its electronic properties stability behavior based on Hall Effect measurement. Once the Zinc Nitride successfully developed, then the deposition layer will be analyzed to understand the performance versus time. Since the deposition results will need for Hall Effect measurement the glass substrates were cut into 8 x 8 mm from the original 22 x 22 mm size. The 8 x 8 mm being selected because to fit the Hall effect measurement board contact pin fitting which according to the 8 x 8 mm size. While for original glass substrate 22 mm x 22 mm will be used on Ellipsometer and Dektak surface profiler for Zinc Nitride thickness measurement activities. The glass substrate being manual cut using diamond pen to shape it.

Once the glass substrate for mentioned sizes being prepared, the samples then being soaked in n-Butyl Acetate which being heated on the hot plate with temperature of 400°C. Then the glass substrates being soaked in Acetone and Isopropyl Alcohol in order to clean and avoid it from any contaminations. The glass substrates then being dried using air gun. To further clean the glass substrates all the substrates will be cleaned using Plasma barrel etcher. However the target will be cleaned based on pre sputter process using Argon gas with 30 sccm flow rate and 600 ms duration. To identified the gas that being used whether Argon or Nitrogen gas type can be recognized through naked eyes. The Argon gas emit violet colour while Nitrogen with pink colour. This is a type of visual check to be done on every process to ensure correct type of gas being selected throughout the evaluation process. The pre sputter process will not affect the glass substrate since the sputtering will be done only on the target only and the sputtering equipment has a feature by blocking the samples area from deposition to occur on top of it. Below in Figure 7 are some references towards the Argon and Nitrogen gas glowing colour during the sputtering process for identification check.



Argon violet emitting colour



Nitrogen pink emitting colour

Figure 7: Comparison of Argon and Nitrogen emitting colour

The sputtering operating process for DC Magnetron equipment can be referred to Figure 8 below. Zinc target which being connected to cathode being bombarded and the deposited Zinc will falls on the glass substrate that connected to anode in Nitrogen environment.

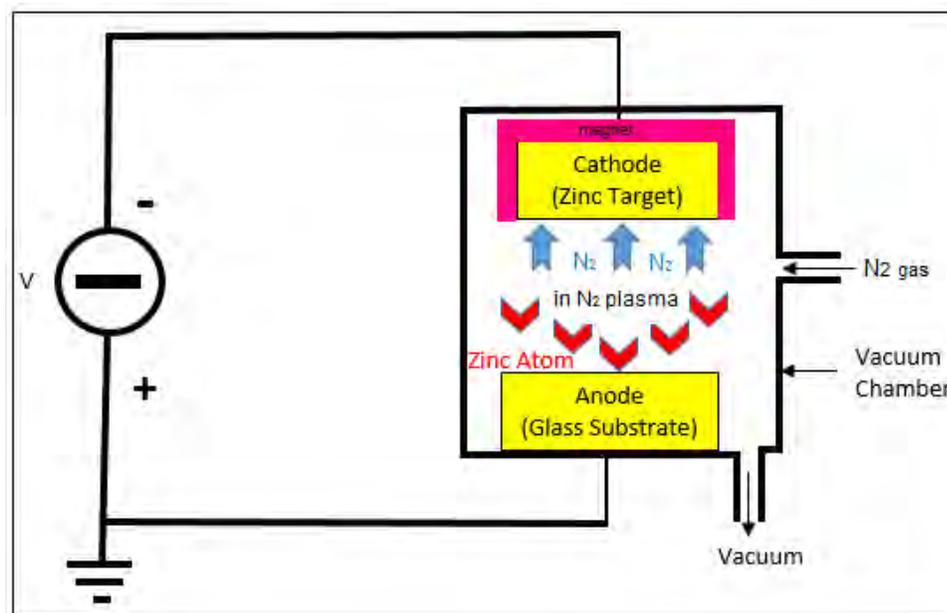


Figure 8: Illustration of process setup on DC Magnetron Sputtering equipment

The parameters that being assessed towards the deposition of the Zinc Nitride towards the glass substrate and had been identified and below are the list for reference

1. Gas Flow rate (sccm)
2. Power (Watts)

3. Substrate temperature (°C)
4. Nitrogen gas (fixed)

The Nitrogen flow rate being evaluated first to understand its performance towards Zinc Nitride deposition behavior. The parameter setting had been defined below in Table 3 for reference.

Run	Nitrogen gas flow	Temperature	Power
1	15 sccm	20°C	100 W
2	30 sccm		
3	45 sccm		

Table 3: Nitrogen flow rate assessment towards Zinc Nitride deposition performance.

The substrate temperature then being assessed by referring to Table 4 for reference. The Nitrogen flow rate and power being fix for this parameter setting. These evaluations were continuation analysis from run 1, 2 and 3 performance which involve various Nitrogen flow rate assessment.

Run	Substrate temperature	Nitrogen flow	Power
4	50°C	45 sccm	100 W
5	100°C		
6	150°C		

Table 4: Substrate temperature assessment towards Zinc Nitride deposition performance.

The overall process flow can be referred in Figure 9 for reference.

3.1 Process flow chart

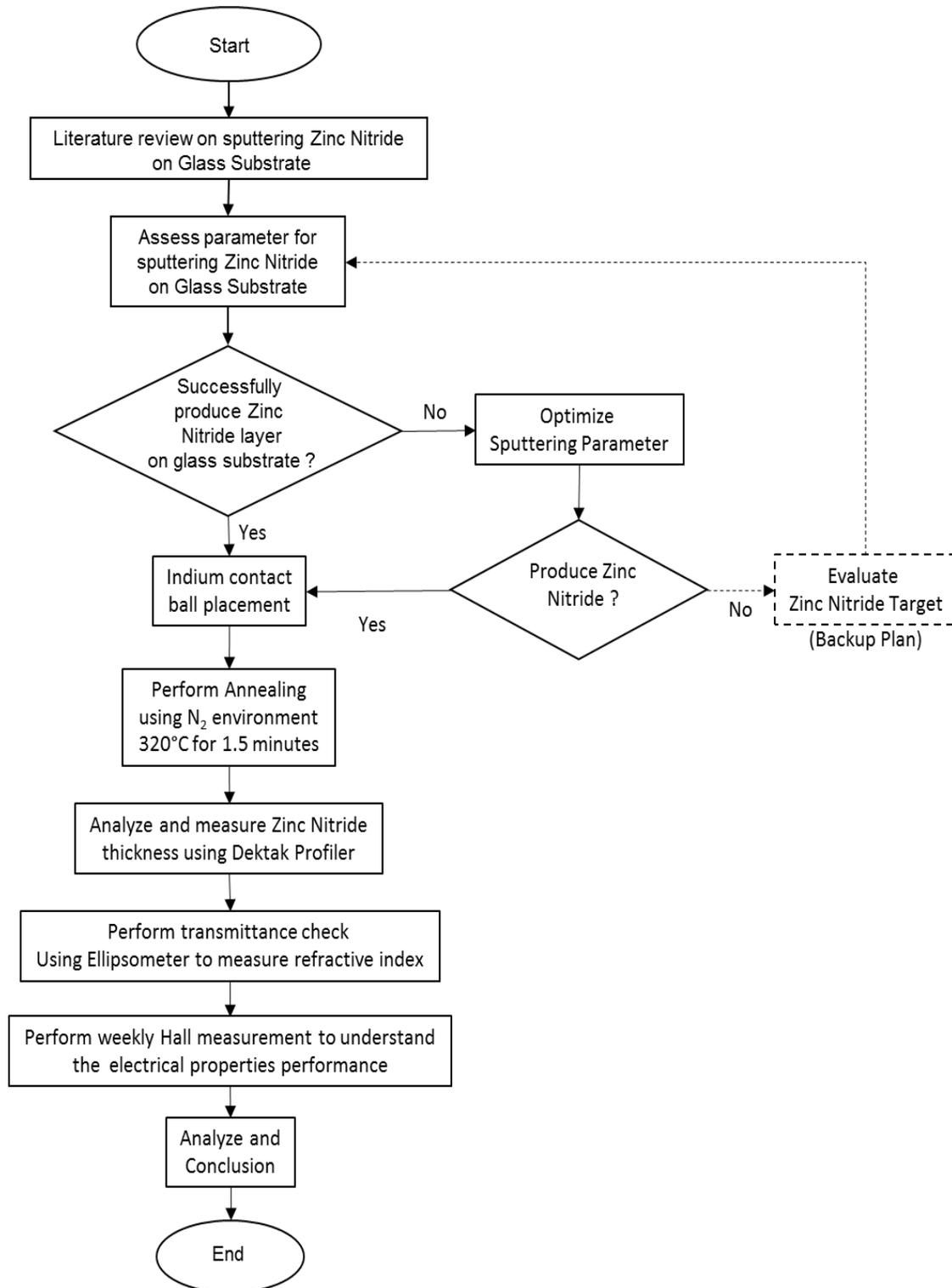


Figure 9: Process Methodology for Zinc Nitride thin film development

3.2 Indium ball placement

Indium ball placement being prepared for Hall Effect measurement activities. It being setup for jig contact point which indicated in Figure 10 below for Van der Pauw technique.

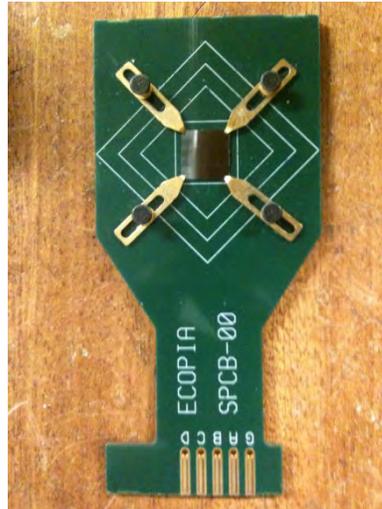


Figure 10: Four probe jig for Van der Pauw technique

Below are the steps on how the Indium ball placement being done. Big chunk of Indium had been cut manually using tweezers and then roll the Indium with glass substrate until it become thin and long. This method being done to simplify the process by cutting into small pieces. The rolled Indium samples will be soaked in n-Butyl Acetate which being placed on the hot plate with temperature of 400°C. Then the rolled Indium sample being soaked in Acetone and Isopropyl Alcohol in order to clean it and avoid from any contaminations. It will be dried using air gun and being cut into small pieces using tweezers. Then allocate the small pieces of Indium at 4 corner of the deposited glass substrate as illustrated on Figure 11.



Figure 11: Indium ball placement illustration

Once the Indium balls placement completed then the annealing process took place. Annealing was performed in Nitrogen environment with temperature 320°C for 1.5 minutes. All samples then went for Hall Effect measurement using four pin probe Van der Pauw technique.

3.3 Hall Effect definition

Edwin Hall had discovered in 1879 when an electrical current being applied through a metal/semiconductor samples, a difference of voltage difference known as Hall voltage will be produced with the presence of magnetic field. The voltage transverse to the electric current while the magnetic the magnetic field perpendicular to the current. Hall Effect measurement is a method to extract important electrical properties such as sheet resistance, sheet carrier density, doping type (n or p type material) and mobility.

3.4 Hall Effect measurement configuration setup

The electrical properties for the Zinc Nitride checking were based on the Hall Effect measurement. The Hall Effect systems consists of 5 components which can be referred in Figure 12 below.

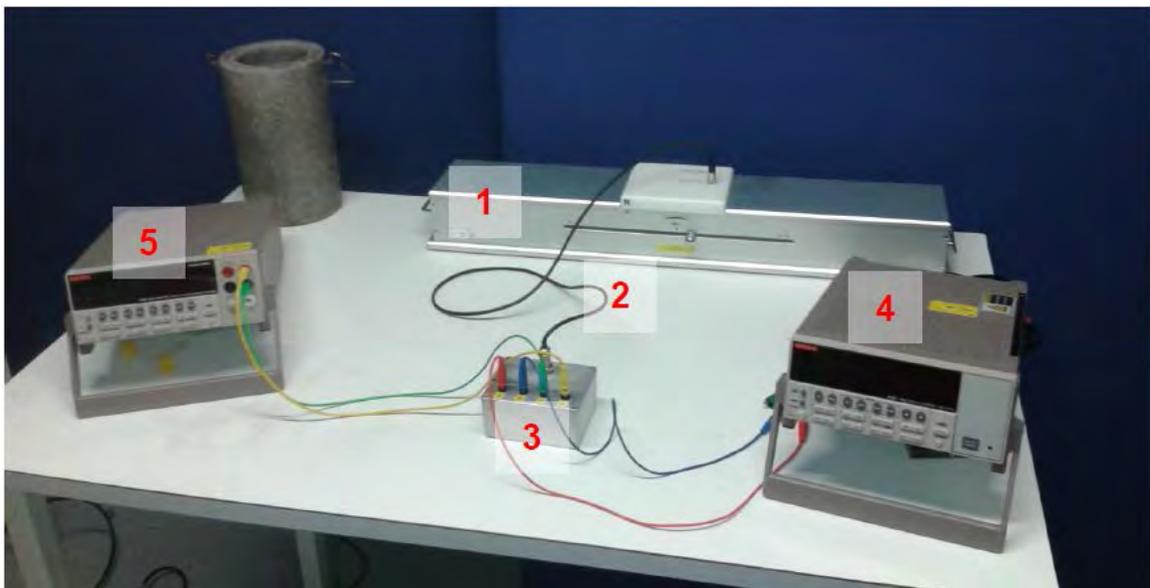


Figure 12: Hall Effect measurement configuration setup

Component 1 (Ecopia HMS5000 dual magnet assembly)

- contains a permanent magnet with different polarity setting. The evaluation samples with the board will be allocated in this area and there is a liquid nitrogen reservoir which can be used in this component as well.

Component 2 (Connection cable)

- functioning as electrical connection from the evaluation sample towards the connector box for measurement purpose

Component 3 (Connector box)

- consist of 4 bananas socket which being labelled as A, B, C and D. Functioning as a connector for current and voltage source. The positioning will be changed according to the contact point for current supply and voltage measurement.

Component 4 (Keithly 6220 precision current source)

- deliver a constant DC with current source ranging of 0.1 pA to 0.1 A which connected to the samples by the red and blue connectors.

Component 5 (Keithly 2700E multimeter)

- being connected with yellow and green banana connectors and used for voltage measurement from the samples.

The deposited glass substrates by Zinc Nitride which completed Indium ball placement being attached to the PCB sample board contact and the connector box which could be referred to Figure 13 for reference.



Figure 13: PCB sample board (left) and connector box (right)

The connector box linked by banana clips which differentiate by its colour which can be referred in Figure 14.

For voltage source → Yellow banana clip (represents positive terminal) and
Green banana clip (represents negative terminal)

For current source → Red banana clip (represents positive terminal) and
Blue banana clip (represents negative terminal)

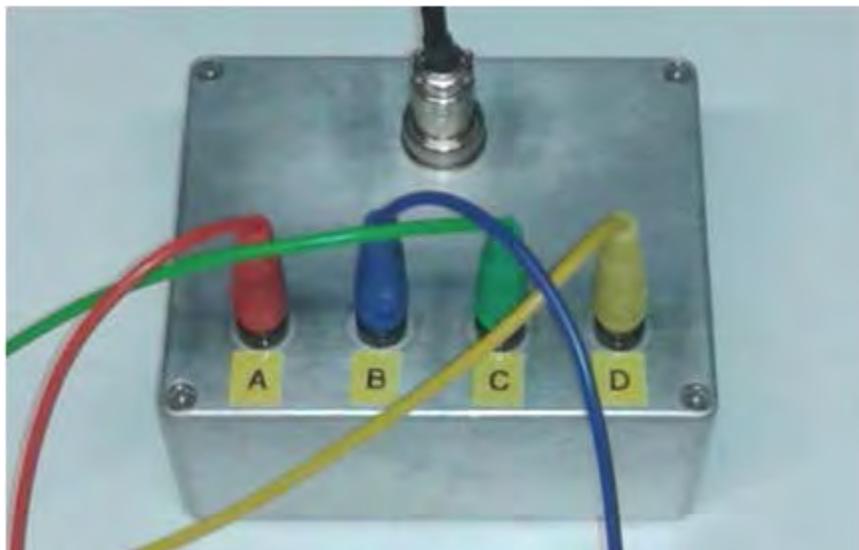


Figure 14: Banana clip link with connector box according to colour orientation