FERROELECTRIC THIN FILM MATERIALS DEPOSITION BY PHYSICAL VAPOR DEPOSITION

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DECLARATION

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ABSTRAK

Filem nipis adalah lapisan bahan yang mempunyai ketebalan yang berkisar dari beberapa nanometer kepada beberapa mikrometer dan telah digunakan dalam fabrikasi peranti MEMS. Ciri-ciri filem tipis dipengaruhi oleh jenis bahan dan salah satu bahan filem nipis adalah bahan filem nipis ferroelektrik. Bahan filem nipis ferroelektrik adalah bahan yang menunjukkan polarisasi tanpa kehadiran medan elektrik. Bahan filem nipis ferroelektrik ini digunakan secara meluas dalam fabrikasi sistem mikroelektrik mekanikal (MEMS). Terdapat beberapa kaedah pembuatan yang boleh digunakan untuk pembuatan MEMS dan salah satunya adalah pemendapan filem nipis. Dalam projek ini, teknik pemendapan filem tipis yang digunakan adalah percikkan magnetron berasaskan serbuk. Percikkan magnetron berasaskan serbuk berbeza daripada yang konvensional kerana filem nipis dimedap dari sasaran serbuk. Filem-filem nipis yang dimendapkan dicirikan dan sifat-sifat elektrik filem-filem nipis diselidik. Sifat elektrik filem nipis diselidiki melalui ujian fungsi piezoelektrik dan ujian pekali rintangan suhu (TCR). Pekali piezoelektrik dan TCR adalah ciri yang perlu dipertimbangkan ketika merancang peranti MEMS. Pencirian yang melibatkan analisis mikrostruktur dan analisis unsur telah dilakukan untuk filem nipis. Secara keseluruhan, permukaan filem nipis licin dan sesetengahnya mungkin mengandungi bahan pencemar. Filem nipis PZT telah menjalani ujian fungsi piezoelektrik dan hasilnya menunjukkan bahawa filem nipis tersebut mempunyai kesan piezoelektrik. Dalam ujian TCR, filem nipis logam (filem nipis Sb) menunjukkan nilai TCR positif manakala filem nipis bukan logam (filem nipis MgO) menunjukkan nilai TCR negatif. Terdapat juga beberapa filem nipis logam seperti filem nipis Ti dan filem nipis Cr yang menunjukkan nilai TCR negatif dan ini mungkin disebabkan oleh kehadiran kekotoran dan kecacatan.

ABSTRACT

Thin film is a layer of material that has the thickness that ranging from a few nanometers to a few micrometers and have been used in the fabrication of the MEMS devices. The properties of the thin films are affected by the type of the materials and one of the thin film materials is the ferroelectric thin film material. It is a type of material that exhibits polarization without the presence of the electric field. This ferroelectric thin film material is widely used in the fabrication of microelectromechanical systems (MEMS). There are a few manufacturing methods that can apply to the manufacture of MEMS and one of them is the thin film deposition. In this project, the thin film deposition technique used is the powder-based magnetron sputtering. The powder-based magnetron sputtering is different from the conventional magnetron sputtering because the thin film is deposited from the powder target. The deposited thin films are characterized and the electrical properties of the thin films are investigated. The characterization which involved the microstructural analysis and elemental analysis are carried out for the thin film. Overall, the surface of the thin film is smooth, and some may contain contaminant. The electrical properties of the thin films are investigated through the piezoelectric functional test and temperature coefficient of resistance (TCR) test. The piezoelectric coefficient and TCR are the characteristics that needed to consider when designing MEMS devices. The PZT thin films were undergoing piezoelectric functional test and the results indicated that the thin films exhibited piezoelectric effect. In TCR test, the metal thin film (Sb thin film) shows a positive TCR value whereas the non-metal thin film (MgO thin film) shows negative TCR value. There are also some metal thin films such as Ti thin film and Cr thin film that show negative TCR value and this may be due to the presence of the impurities and defects.

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LIST OF ABBREVIATIONS

Abbreviation

CVD	Chemical vapor deposition
PVD	Physical vapor deposition
MEMS	Microelectromechanical system
SEM	Scanning electron microscopy
EDS	Energy dispersive X-ray spectroscopy
TCR	Temperature coefficient of resistance
NEMS	Nanoelectromechanical system
HARM	High-aspect ratio micromachining
GPS	Global positioning systems
MOCVD	Metal-organic chemical vapor deposition
CSD	Chemical solution deposition
MOD	Metalorganic deposition
PPVD	Powder-based physical vapor deposition
HIPIMS	High power impulse magnetron sputtering
CTE	Coefficient of thermal expansion
FE-SEM	Field emission scanning electron microscopy

Nomenclature

d_{31}	Transverse piezoelectric coefficient
d_{33}	Longitudinal piezoelectric coefficient
Q	Charge generated
F	Force exerted
C	Capacitance
V	Voltage
R_0	Resistance value at room temperature
R	Resistance
T	Temperature

CHAPTER 1 INTRODUCTION

Thin films have been used in daily items and highly specialized technologies over a century due to their potential technical value and scientific curiosity in their properties. They can be used in superhydrophobic coatings on glass and metals, micrometer dots in microelectronic, photoelectronic, thermoelectronic, superconductivity, information-storage media, fuel cells and bio-compatible coatings (Latthe, 2012).

The application of the thin film to a product or component is known as deposition. The thin film deposition method will directly affect the properties of the thin films. Therefore, the deposition method should be selected properly to obtain the required properties and versatility. The thin film deposition method can be classified into two main groups which are chemical vapor deposition (CVD) and physical vapor deposition (PVD).

Chemical vapor deposition (CVD) is a thermochemical process that the film is deposited by the reaction and decomposition of gaseous compounds. In CVD, a wide range of material can be coated and almost any material can serve as substrate. Normally, the deposited CVD coatings are thicker than those of PVD and the cycle time also longer than PVD (Kalpakjian, Vijai Sekar, & Schmid, 2014).

Physical vapor deposition (PVD) is a deposition process that moved the particle from the target source to the substrate physically. The thin films deposited by PVD have very low thickness that is in the range of few nanometers to thousands of nanometers. Besides, the multilayer coating, graded composition deposits, very thick deposits, and free-standing structures can be deposited by using this process (Mattox, 2010).

In this project, the ferroelectric properties of the thin film deposited by physical vapor deposition are studied. The characterization of the thin film, functional test and TCR test of the thin film are carried out in this project. The ferroelectric properties of materials means that the material will exhibits polarization without the presence of the electric field. This spontaneous polarization changed when there is an external electric field applied on it (William D. Callister & Rethwisch, 2010).This ferroelectric thin film has potential application in non-volatile information storage and microelectromechanical systems (MEMS).

1.1 Problem Statement

Nowadays, the popularity of the microelectromechanical system (MEMS) is increased and is widely used. The growth of the MEMS market indicated the needs of new manufacture method in order to fulfil the market needs. The substrate is expensive, therefore, development of new method of growing thin film is required to produce alternative substrate coated thin film more efficiently.

1.2 Objective

There are few objectives obtained in this project:

1. To investigate the ferroelectric properties of thin film materials deposited by physical vapor deposition.
2. To characterize the ferroelectric deposited thin film deposited by physical vapor deposition.

1.3 Scope of Work

The focus of the project is the properties of the thin film deposited by powder-based magnetron sputtering. Firstly, the scanning electron microscopy (SEM) and energy dispersive X-ray spectroscopy (EDS) equipped with SEM are used to characterize the thin film. The functional test is then be carried out to determine the piezoelectric effect of the thin film and the temperature coefficient of resistance (TCR) test is carried out to investigate the effect of temperature on the resistance of the thin film.

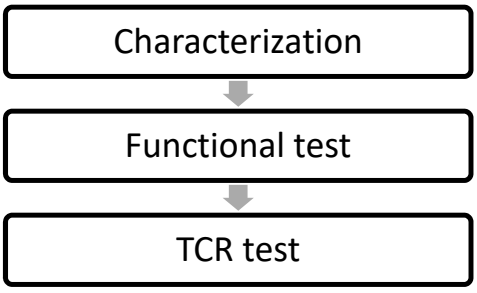

Figure 1.1: Scope of work.

1.4 Project Background

Microelectromechanical system (MEMS) is a microelectromechanical device that incorporates an integrated electrical system into a certain product.

In 1960s, MEMS was proposed but not commercialized. The MEMS applications were commercialized in 1980's and the first commercial application was the tiny nozzle assembly used in the cartridges of inkjet printer (Engineering and Technology History Wiki, 2017). MEMS-optical network components and BioMEMS are developed in the late 1990s and early 2000s. Now, the number of MEMS application is increased continually, and the size of MEMS is getting smaller. The decrease in the size of MEMS introduced a new technology which is called nanoelectromechanical system (NEMS) (Southwest Center for Microsystems Education 2017).

The accelerometers in mobile phones, gyroscopes and global positioning systems (GPS), air bag sensors in automobiles, and digital micromirror devices are parts of the MEMS (Kalpakjian et al., 2014). In the market of the smart phone, the popularity of MEMS has increased because the smart phone companies incorporated MEMS in their devices in order to enhance user experience and gain competitive advantages in the market. According to the research, the market value of MEMS is at $13 billion in the year 2015 and forecasted to reach $26.8 billion by 2022 (R. Singh, 2017).

Fabrication technique of MEMS involved bulk micromachining, surface micromachining and high-aspect ratio micromachining (HARM). Principal steps of MEMS fabrication involved deposition, lithography and etching process. The materials used for the micromachining processes involved the substrate and the thin film (Kalpakjian et al., 2014; PRIME Faraday Partnership, 2002).

Various types of thin film materials can be applied to the fabrication of MEMS, one of them is Lead Zirconate Titanate (PZT)**.** The excellent ferroelectric and piezoelectric properties of PZT thin film enable it to be widely used in microelectromechanical system (MEMS) as the integration of piezoelectric function in sensing, actuating and energy harvesting devices (Kanda, Kanno, Kotera, & Wasa, 2009).

CHAPTER 2 LITERATURE REVIEW

In this chapter, the research studies on the ferroelectric material, Lead Zirconate Titanate (PZT) material, deposition technique, coating material, and substrate are carried out.

2.1 Ferroelectric material

Ferroelectric materials are a class of material that exhibits polarization without the presence of the electric field which also called spontaneous polarization (William D. Callister & Rethwisch, 2010). Spontaneous polarization in the ferroelectric material can be reversed by an external electric field. One of the most common ferroelectric materials is barium titanate, when it is heated above the its ferroelectric Curie temperature (phase transition temperature, T_c) which is the temperature that crystal structure undergoes phase transition, its phase will change from ferroelectric phase to paraelectric phase (René & Turcotte, 2010). Ferroelectric materials have various useful properties which are ferroelectric hysteresis, high premittivities, high piezoelectric effects, high pyroelectric coefficients, strong electro-optic effects and anomalous temperature coefficients of resistivity. It can be made in various forms which included ceramic, single crystal, polymer and thin film (Whatmore, 2017).

2.2 What is PZT?

Lead Zirconate Titanate (PZT) is an important ferroelectric material which is commonly used in the manufacture of piezoelectric ceramic elements. Various possible compositions provide a wide range of dielectric constant values, piezoelectric activity and primary transition temperatures (Ceramic Industry, 2018). PZT is a perovskite ferroelectric which exhibits cubic symmetry when heated above its Curie temperature (Horchidan et al., 2016).

The PZT ceramic can be classified into two groups which are soft and hard PZT ceramic. This classification is referred to the mobility of the dipoles/domains and also the polarization and depolarization behaviour (Instrumente, 2017). PZT thin film is commonly used as the sensing and actuating component in MEMS devices as it can generate large displacement, has higher sensitivity and higher energy density with wide dynamic range and low power requirements.

2.3 Deposition technique

Thin film deposition is one of the manufacturing methods which can apply to the manufacture of MEMS (Kalpakjian et al., 2014). There are two major groups of thin film deposition techniques can be used in the manufacture of MEMS which are chemical vapor deposition (CVD) and physical vapor deposition (PVD). PVD is chosen as the thin film deposition technique because it has a few advantages over CVD. One of the advantages is that it can produce film thinner than that of the CVD. Besides, CVD will produce precursors and by-products which is toxic and will cause material handling and storage problems, whereas PVD will not cause these kinds of problem. Due to this environment friendly properties of the PVD, the manufacturers show preference to it and thus increase the market of PVD.

The increased demand for microelectronics also affected the PVD market. According to the research, the demand for the microelectronic is $3831.30 million in the year 2015 and forecasted to reach $7724.50 million in the year 2019 (See Figure 2.1). Furthermore, according to the research, the value of the PVD market is at US$ 14261.90mn in the year 2016 and forecasted to reach US$21898.10 mn by the year 2024 (See Figure 2.2).

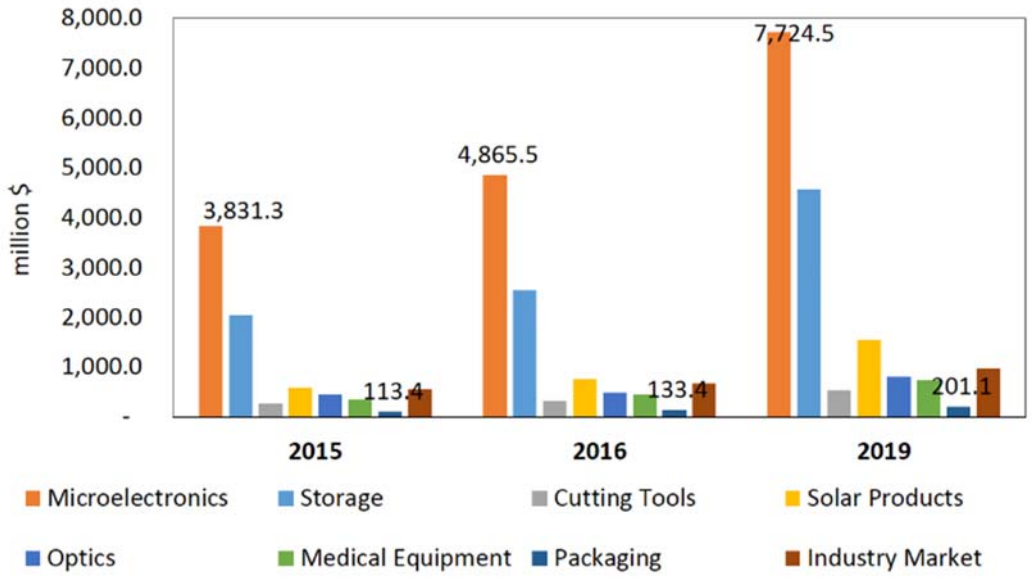

Figure 2.1: Market revenue share of PVD equipment (Transparency Market Research, 2017).

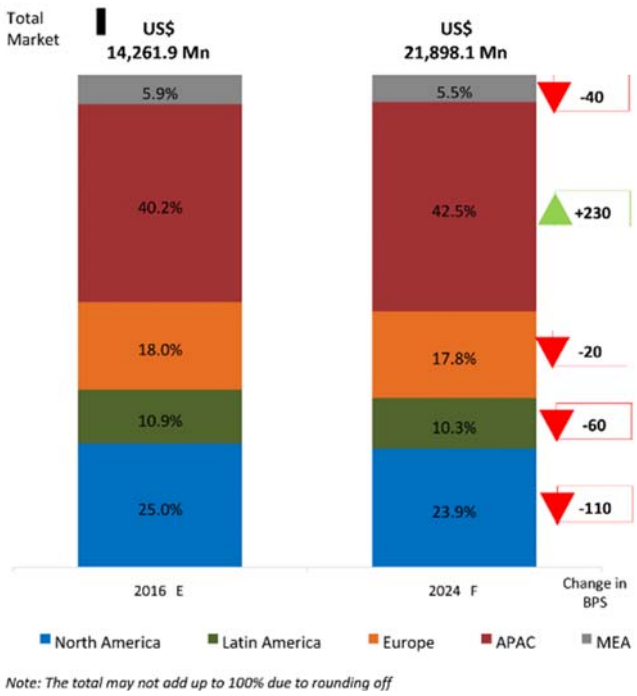

Figure 2.2: Market of physical vapor deposition (Transparency Market Research, 2017).

The physical-based deposition technique used included pulsed laser deposition, vacuum deposition, sputtering deposition, and magnetron deposition, while the chemical-based deposition included metal-organic chemical vapor deposition (MOCVD) and chemical solution deposition (CSD).

2.3.1 Pulsed laser deposition

Pulsed laser deposition used the laser beam to ablate the material for depositing thin films inside the vacuum chamber. This deposition technique has short deposition time and its compatibility to oxygen and other inert gas (Jilani, Shaaban Abdel-wahab, & Hammad, 2017).

2.3.2 Chemical Vapor Deposition

Chemical vapor deposition (CVD) consists of a few types of processes which are metal-organic chemical vapor deposition (MOCVD) and chemical solution deposition (CSD).

2.3.2.1 Metal-organic chemical vapor deposition (MOCVD)

Metal-organic chemical vapor deposition (MOCVD) involved the growth of thin layers of compound semiconducting materials by the co-pyrolysis of various

combinations of organometallic compounds and hydrides. It can be used in the fabrication of opto-electronic and high-speed electronic devices (L.Zilko, 2012).

2.3.2.2 Chemical solution deposition (CSD)

Chemical solution deposition (CSD) involved bringing the metal ion into the metalorganic solution and then deposited on the substrate by spinning. CSD processes are less expensive, easy to change the composition and can produce a smooth layer (Whatmore, 2017). There are two major classes of CSD process which are metalorganic deposition (MOD) and sol-gel.

2.3.2.2.1 Metalorganic deposition (MOD)

Metalorganic deposition (MOD) involved the metal complexes with long-chain carboxylic acids dissolved in solvent such as tolune. The solution of MOD is quite stable and resistant to hydrolysis (Whatmore, 2017).

2.3.2.2.2 Sol-gel

Sol-gel technique is work under lower temperature and gives excellent homogeneity for multi-component materials (Jilani et al., 2017). Sol-gel technique is inexpensive and able to control the stoichiometric chemical composition of the film. Therefore, it is widely used in the fabrication of high quality PZT thin films (Lu, Dong, Chen, & Cheng, 2017).

2.3.3 Physical vapor deposition

Physical vapor deposition (PVD) has a few basic types which are vacuum deposition, sputtering deposition and magnetron sputtering. In PVD, the target material is vaporized from solid in the form of atoms or molecules. The atoms or molecules are then transferred in the form of vapor through a vacuum environment and condensed on the substrate to form the thin film (Mattox, 2010). In conventional PVD, the target material used is a disc shape solid material. Whereas in powder-based PVD (PPVD), the thin film is deposited from the blended powder target.

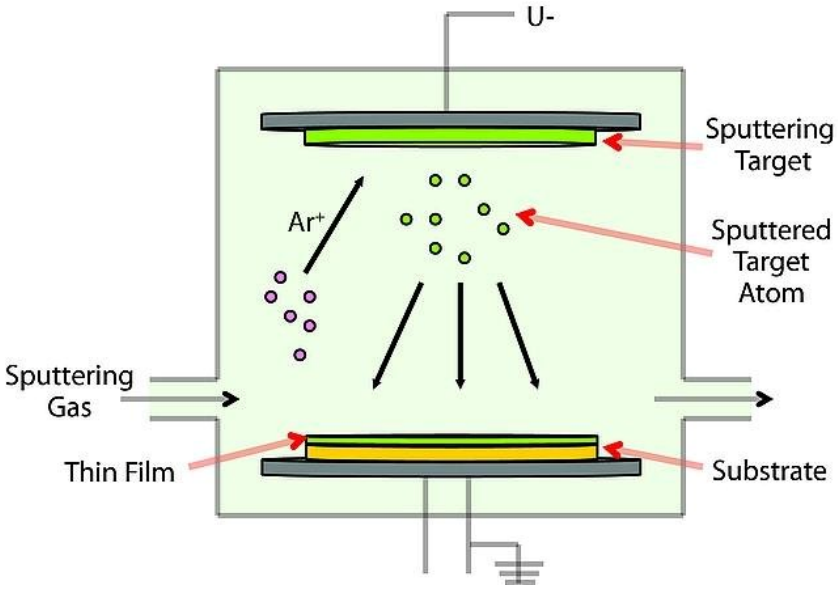

Figure 2.3: Illustration of physical vapor deposition (Koçak, 2018).

2.3.3.1 Vacuum deposition

In vacuum deposition, the target metal is evaporated at high temperature in a vacuum and then deposited on the substrate without collision with gas molecules. Uniform thickness of coating can be deposited in both simple shapes and complex shapes (Kalpakjian et al., 2014).

2.3.3.2 Sputtering deposition

The sputtering deposition is the process that the atoms ejected from the target and then deposited on the substrate. The evacuated chamber of the sputtering system contains metallic anode and cathode, and a power supply. The sputtering process can form high melting point material easily and the deposited film have composition which is similar to that of starting material (Jilani et al., 2017).

2.3.3.3 Magnetron Sputtering

Magnetron sputtering used magnetron source to grow thin film. There is various type of magnetron sputtering processes which are radio frequency (RF), direct current (DC) Pulsed direct current (DC) and high power impulse (HIPIMS) magnetron sputtering (Lundin & Sarakinos, 2012; Martin, 2009).

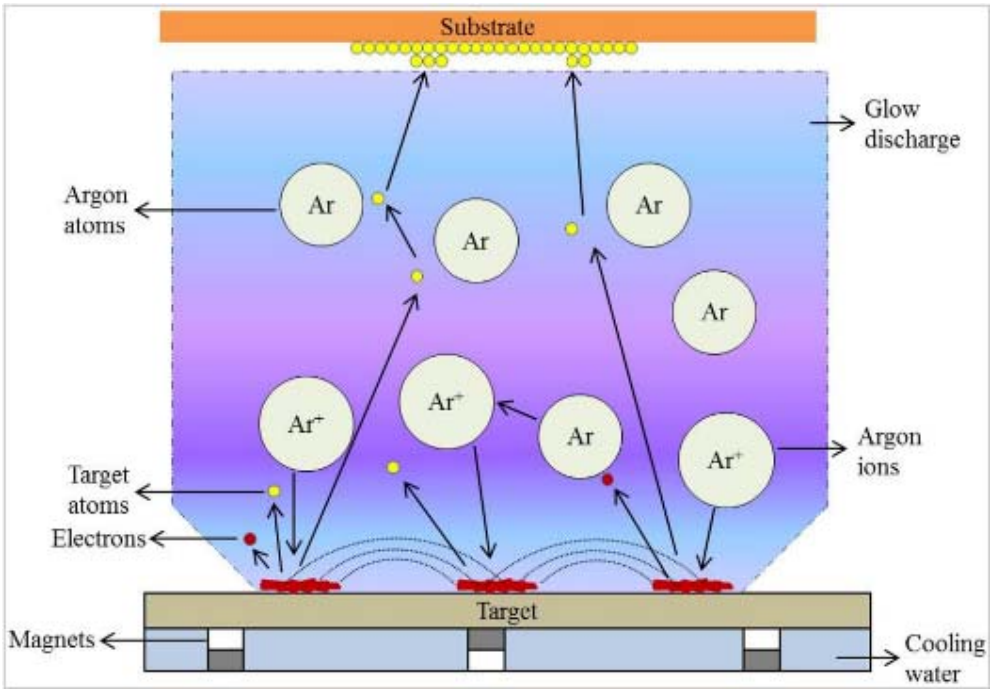

Figure 2.4: Illustration of magnetron sputtering (Maurya, Sardarinejad, & Alameh, 2014).

Table 2.1: Advantages and disadvantages of different types of magnetron sputtering (Lundin & Sarakinos, 2012; Martin, 2009).

Type of magnetron sputtering	Radio frequency (RF) magnetron sputtering	Direct current (DC) magnetron sputtering	Pulsed direct current (DC) magnetron sputtering	High power impulse magnetron sputtering (HIPIMS)
Advantage	Suitable for both conducting and non-conducting target	Simplest and least expensive	Arcing is avoided.	High energy pulsed caused high ionization degree of sputtered target.
Disadvantage	Low deposition rate	Arcing occurred will damage target and degrades quality of deposited film. Only conductive target can be used.		Low deposition rate

2.4 Coating material

There is various type of materials can be used as the coating material in thin film deposition to produce thin film with unique properties that can be used in some application. One of them is the lead zirconate titanate (PZT) which produced thin film that is commonly used in microelectromechanical system (MEMS) devices. Besides, there is titanium nitrate which is used in diffusion bearing coating, tool coating, and decorative coating, silicon which is used in semiconductor devices, titanium oxide which is used in high index optical coating and so on (Mattox, 2010).

2.5 Substrate

In thin film deposition, the films/coatings will form on the surface of the substrate. The selection of the type of substrate is very important as it will affect the dielectric and piezoelectric of the thin film (Kumar, 2011). Besides, a substrate surface must be reproducible in order to produce a reproducible thin film (Mattox, 2010). To obtain a reproducible substrate surface, the process of breaking the bond between contaminant and substrate without damaging the substrate surface which is known as substrate cleaning is required.

Substrate cleaning can help to obtain a reproducible film as it affects the smoothness, adherence, uniformity, and porosity of the film (Latthe, 2012). It can combine two or more cleaning method to clean the substrate completely. Film must apply immediately after substrate cleaning so that the dust and contaminant will not gather on the surface of the substrate (Dennison, 2018). There is various type of material can be used as the substrate in thin film deposition, such as glass, metal, polymer, magnesium oxide, Strontium Ruthenium (SRO), and Strontium Titanate (STO) substrate.

2.5.1 Glass substrates

The deposition of the thin film on the glass substrate is suitable for the application in data storage, electronic display and adaptive optics (Nguyen et al., 2017). The epitaxial growth of buffer layer on the glass substrate can optimize the piezoelectric response of the film as the piezoelectric response depends on the composition, growth quality, orientation and also size of the fabricated devices (Chopra et al., 2017).

2.5.2 Metal substrates

A metal substrate such as stainless steel and titanium can be used in thin film deposition has high fracture toughness and good mechanical elasticity (Choudhary & Iniewski, 2016). The PZT thin film deposited on the metal substrate can integrate with the engineering system easily and has potential applications as flexible electronic devices and energy harvesters (Li et al., 2018). The difference in the coefficient of thermal expansion (CTE) between PZT and metal substrate can change the electrical properties of the thin films (Hirotaka et al., 2017).

2.5.3 Polymer substrate

The thin film deposited on polymer substrate may have good flexibility. This flexible thin film is useful for the application in sensing, actuating and energy harvesting (Liu, Wallace, Trolier-McKinstry, & Jackson, 2017). The polymer substrates such as polyvinylidene fluoride (PVDF) nanofibers have been widely used in fabricating flexible and stretchable energy harvesting devices due to low elastic stiffness of these substrates (K. I. Park et al., 2014).

2.5.4 Magnesium oxide (MgO) substrate

Magnesium oxide (MgO) can be made in different degrees of purity and this depends on the raw material used (Ceramic Industry, 2018). The magnesium oxide with high quality is widely used for thin film applications. MgO has been proved that it is stable under the exposure experiment and thus is suitable to use as substrate material for Lanthanum Tungstate (LWO) membrane (Deibert et al., 2017).

2.5.5 Strontium ruthenium (SRO) substrate

Strontium ruthenium ($SrRuO_3$ (SRO)) is the oxide of strontium and ruthenium with perovskite structure. At low temperature, it is a Fermi liquid and it exhibits bad metal behavior at high temperature. The growth of SRO thin film on substrate required the matching in-plane lattice parameter to those of the substrate. Mismatch of it (change in thickness) introduces strain which will affect structural, electrical and magnetic properties of SRO thin film (Kaur, Sharma, Pandit, Choudhary, & Kumar, 2014; Koster et al., 2012).

2.5.6 Strontium Titanate (STO) substrate

Strontium Titanate ($SrTiO_3$) (STO) is the oxide of strontium and titanium with cubic perovskite structure in room temperature. At low temperature, it approaches

ferroelectric phase transition and undergoes quantum paraelectric phase transition at the lowest temperature. Its high dielectric constant, low temperature coefficient of dielectric constant, high charge storage capacity, good insulating properties, good optical transparency, and chemical stability show that it has potential applications in electronic and microelectronic industries (Fang, 2013; Marques, 2009; Mohanta, 2015).

CHAPTER 3 RESEARCH METHODOLOGY

In this chapter, the methodologies for powder-based magnetron sputtering, characterization techniques, functional test, and temperature coefficient of resistance (TCR) test are developed.

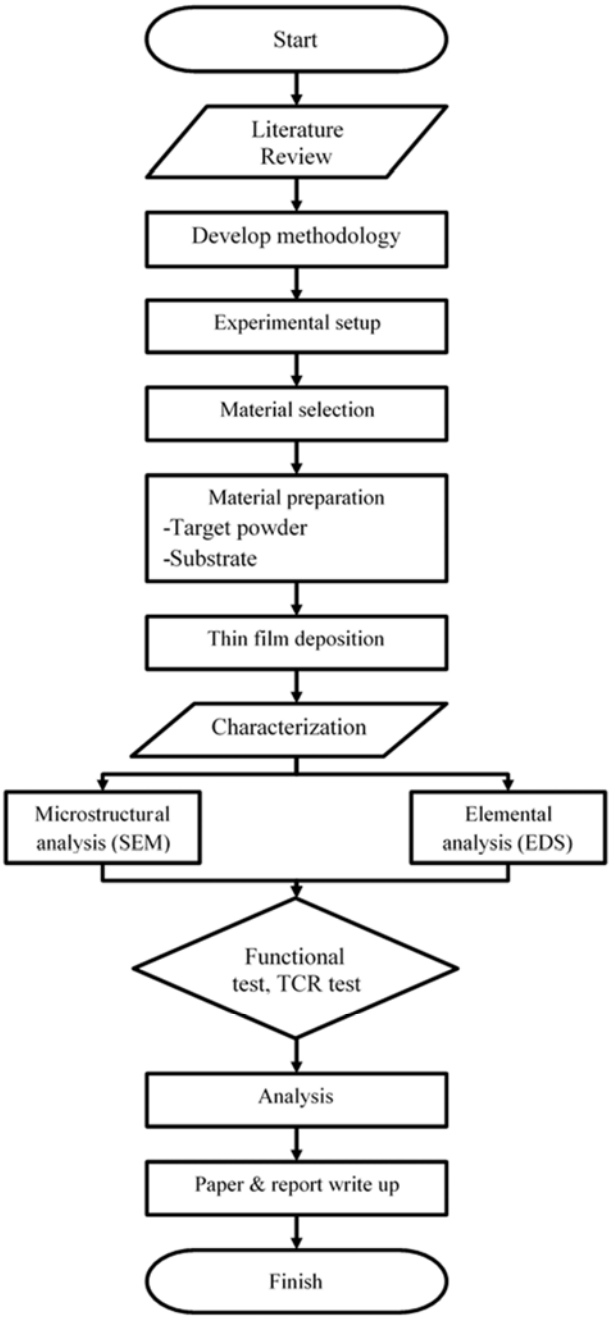

Figure 3.1: Methodology flow.

The methodology flow is listed as shown in Figure 3.1. A research study is conducted to understand the deposition technique and the thin film materials. The methodology for thin film deposition is then developed and few deposited thin films are produced. After that, the characterization, functional test, and temperature coefficient of resistance (TCR) test are conducted for the deposited thin films and the results obtained are analysed.

3.1 Powder-based magnetron sputtering

In the powder-based magnetron sputtering process, thin film deposited on the substrate by magnetron sputtering system using powder target. Before the main sputtering process, pre-sputtering is needed to remove the contaminant from the target. The sputtering condition is shown in Table 3.1 below.

Table 3.1: Powder-based magnetron sputtering condition.

Argon gas flow rate	10 ccm
Oxygen flow rate	0.5 ccm
Sputtering power (RF power)	80-100W
Substrate temperature	600 °C
Pre-sputtering time	30 mins
Sputtering time	1-1.5 hr

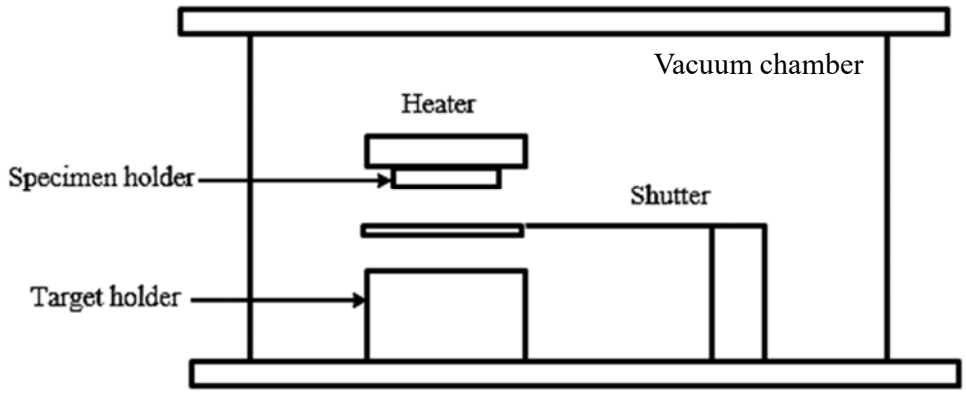

Figure 3.2: Powder-based magnetron sputtering system.

Figure 3.2 displays the sputtering system which involves a heater used to heat the substrate up to 600 °C, a specimen holder used to hold the substrate, a shutter which used for pre-sputtering process and a target holder used to hold the powder target.

Shutter plays an important role in pre-sputtering process as it was closed in between the target and substrate material in order to remove contaminant from target material. After that, the shutter was opened for the main sputtering process.

Table 3.2: Target and substrate materials for the samples.

Sample name/ID	**Target material**	**Substrate material**
MU1	Lead Zirconate Titanate (PZT)	Silicon (Si)
MU2	Lead Zirconate Titanate (PZT)	Strontium Titanate (STO)
MU3	Lead Zirconate Titanate (PZT)	Magnesium oxide (MgO)
MU4	Lead Zirconate Titanate (PZT)	Strontium Titanate (STO)
MU5	Lead Zirconate Titanate (PZT)	Silicon (Si)
Sample 1	Titanium (Ti)	Silicon (Si)
Sample 2	Titanium (Ti)	Glass
Sample 3	Chromium (Cr)	Glass
Sample 4	Antimony (Sb)	Glass
Sample 5	Magnesium oxide (MgO)	Glass

The target and substrate materials used for each sample that deposited by powder-based magnetron sputtering process.are shown in Table 3.2 above.

3.2 Characterization

The characterizations of the thin films are carried out by using scanning electron microscopy (SEM) and energy dispersive X-ray spectroscopy (EDS) technique attached to SEM's.

3.2.1 Scanning electron microscopy (SEM)

SEM can produce a high resolution image of a specimen surface and thus it is normally used for microstructure analysis. The cross section of the

specimen can be imaged by using SEM in order to see the thickness of a certain layer and the adherence of the layers (Hagerty, 2016). In this experiment, Hitachi S-3400N SEM is used to observe the microstructure of the specimens.

3.2.2 Energy dispersive X-ray spectroscopy (EDS)

EDS technique that attached to SEM's was used for elemental analysis of the thin film. It identifies the elemental composition and shows the elemental mapping of the thin film (D. Y. Park et al., 2017).

3.3 Functional test

The functional test is carried out to obtain the piezoelectric coefficient of the thin films. Piezoelectric coefficient d_{ij} is one of the constants that is important to know in order to design sensor and actuator in MEMS. In d_{ij} ,the first subscript i refers to the direction of polarization whereas j refers to the direction of the applied stress. There are two types of piezoelectric coefficient which are the longitudinal (d_{33}) and transverse (d_{31}) piezoelectric coefficient.

d_{33} shows that the direction of polarization and the stress applied are aligned in the same direction which is direction 3, whereas d_{31} shows that the polarization is aligned in direction 3 and the stress applied is aligned in direction 1 (Kivirand, 2015). There common methods that can be used to measure piezoelectric coefficient such as frequency method, laser interferometry method and quasi-static method. These methods have good accuracy but expensive devices are required for measurement (Fialka, 2010).

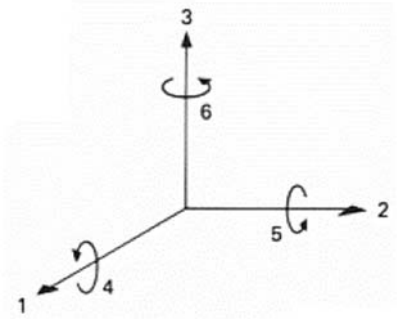

Figure 3.3: Illustration of direction of polarization and stress applied (Kivirand, 2015).

In this project, the longitudinal piezoelectric coefficient (d_{33}) is measured by applying a known force (5 N and 10 N) on thin film and measured the corresponding capacitance and voltage.

Formula used to calculate d_{33}:

$$d_{33} = \frac{q}{F} \quad \text{(Kivirand, 2015)} \qquad (1)$$

The value of q can be obtained by using the formula below:

$$q = C \times V \quad \text{(Phillips, 2019)} \qquad (2)$$

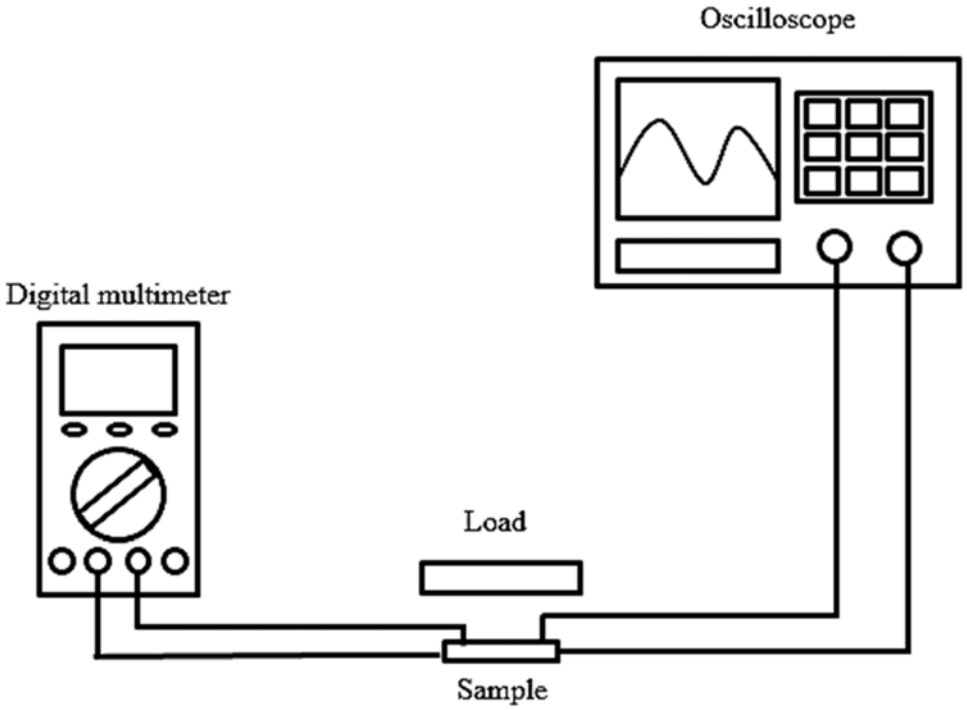

Figure 3.4: Experimental set up for functional test.

Figure 3.4 displays the experimental set-up for the functional test. An oscilloscope (model: Agilent InfiniiVision DSO-X 2002A) is used to measure the voltage when a load is applied and a digital multimeter is used to measure the capacitance of the sample.

3.4 Temperature coefficient of resistance (TCR)

The temperature coefficient of resistance (TCR) is one of the characteristics that needed to consider when designing MEMS devices. TCR value of the sensing material is important for a flow sensor, magnesium is a good material for flow sensor as it has

high TCR value (Sharma, Sharma, & Barman, 2009). TCR shows the change in the resistance of a material when there is a change in temperature.

For the pure metals, the TCR will be a positive value which means that the increased temperature will increase the resistance. The positivity of the value is due to the increasing number of collisions between the charge carrier with atoms when the temperature increasing (Zhai et al., 2012).

Whereas, for the elements such as carbon and silicon, TCR will be a negative value which means that the increased temperature will decrease the resistance (Kuphaldt, 2006). The negativity of TCR value is due to the increasing significant number of free electrons that crossing the energy gap and enter the conduction bands (Electrical 4 U, 2019).

In this project, the resistance and temperature of samples at room temperature (25 ^{o}C) are measured at the beginning of the TCR test. After that, the samples are heated up to 50 ^{o}C, 75 ^{o}C and 100 ^{o}C by using hair dryer and the corresponding resistance and temperature of the samples are measured (Defense Logistics Agency, 2013). Then, the TCR values are obtained by using the formula as shown below:

$$TCR = (\Delta R/R_0)/ \Delta T \ (ppm/^{o}C) \text{ (Defense Logistics Agency, 2013)} \quad (3)$$

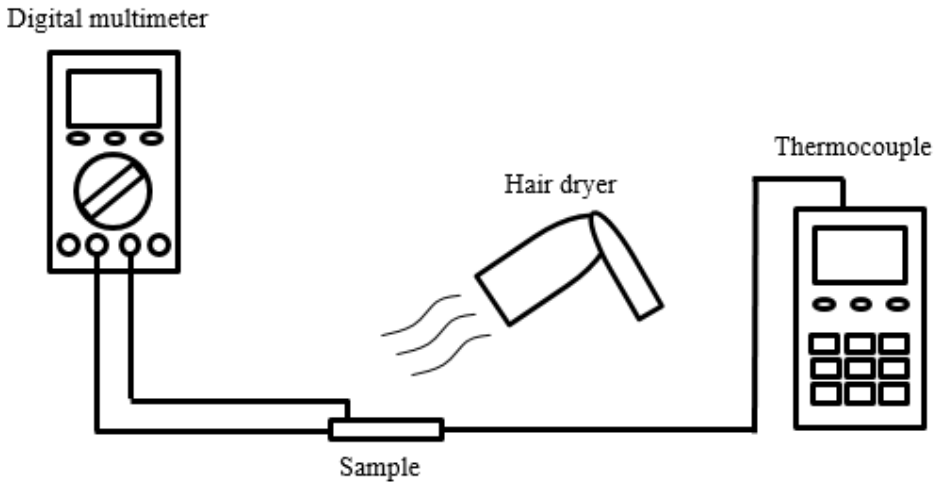

Figure 3.5: Experimental set up for TCR test.

Figure 3.5 shows the experimental set up for TCR test. A hair dryer used to heat the sample, a thermocouple used to measure the temperature and a digital multimeter used to measure the resistance of the sample.

CHAPTER 4 RESULTS AND DISCUSSION

In this chapter, the most significant results and findings are presented and discussed. The microstructural analysis, elemental composition and electrical properties of the developed coatings are provided.

4.1 Microstructural analysis

In this section, the scanning electron microscopy micrograph images of the samples are presented and the most significant findings are discussed.

4.1.1 Uncoated STO substrate

Figure 4.1 shows the surface and cross section images of the uncoated STO substrate. The surface is smooth and clean as it has not undergoing any deposition process.

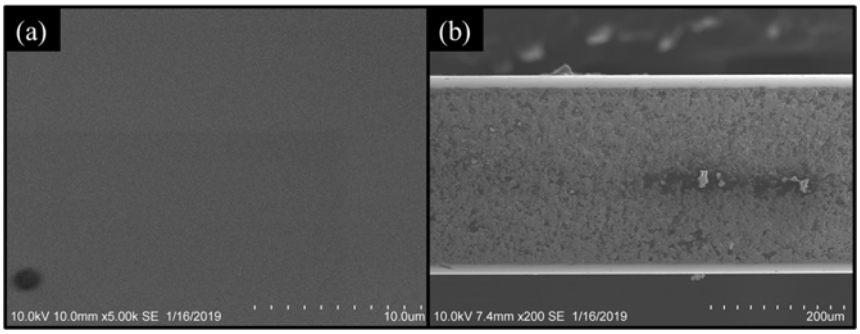

Figure 4.1: Scanning electron microscopy micrograph image of uncoated STO substrate (a) Surface image, (b) Cross section image.

4.1.2 MgO thin film coated on glass substrate (Sample 5)

Figure 4.2 shows the surface image of the MgO thin film which has presence of irregular particles with various size on the surface.

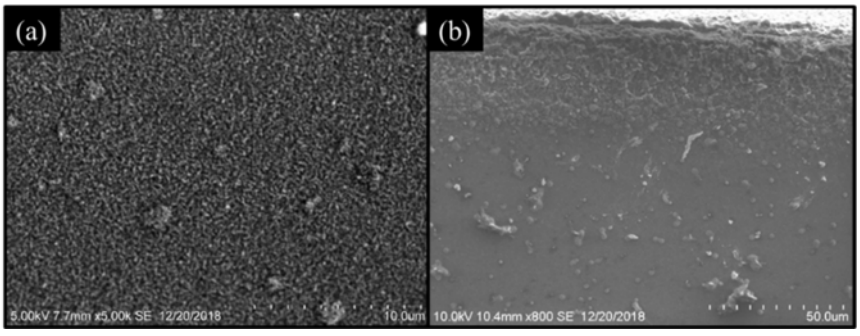

Figure 4.2: Scanning electron microscopy micrograph image of MgO thin film (Sample 5) (a) Surface image, (b) Cross section image.

4.1.3 PZT thin films coated on different substrates

The scanning electron microscopy micrograph images of PZT thin film coated on different substrates such as Si, STO and MgO are presented in this section.

4.1.3.1 Si substrate (MU1)

Figure 4.3 shows the surface and cross section images of PZT thin film coated on Si substrate. The surface is smooth, but there are some irregular particles present on surface. Besides, the thickness of the thin film cannot be identified because the layer of thin film and substrate cannot be identified from the cross section image.

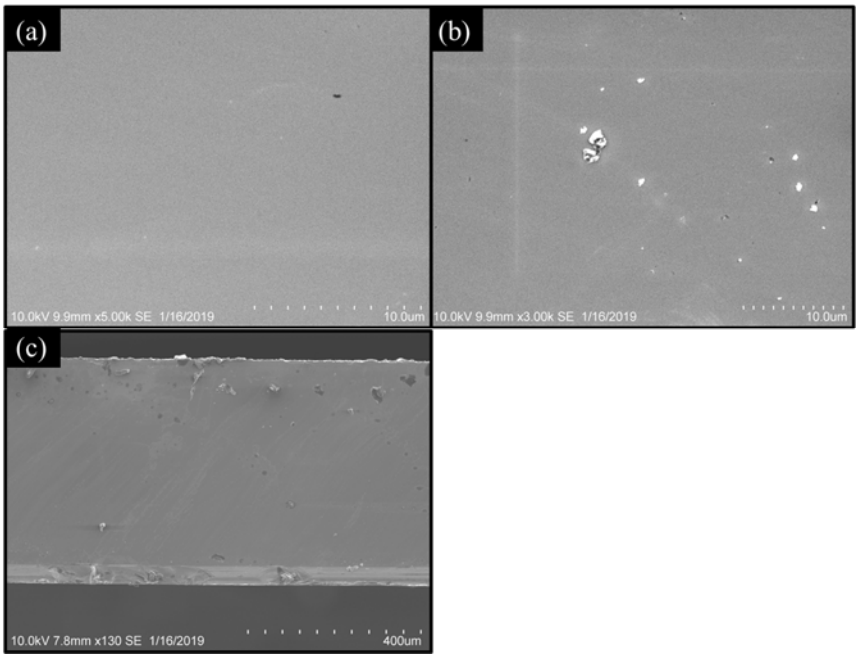

Figure 4.3: Scanning electron microscopy micrograph image of PZT thin film coated on Si substrate (MU1) (a) and (b) Surface image, (c) Cross section image.

4.1.3.2 STO substrate (MU2)

Figure 4.4 shows surface and cross section images of PZT thin film coated on the STO substrate. The surface appears to be smooth comparable with other thin film deposition. Besides, the thickness of the thin film cannot be identified because the layer of thin film and substrate cannot be identified from the cross section image.

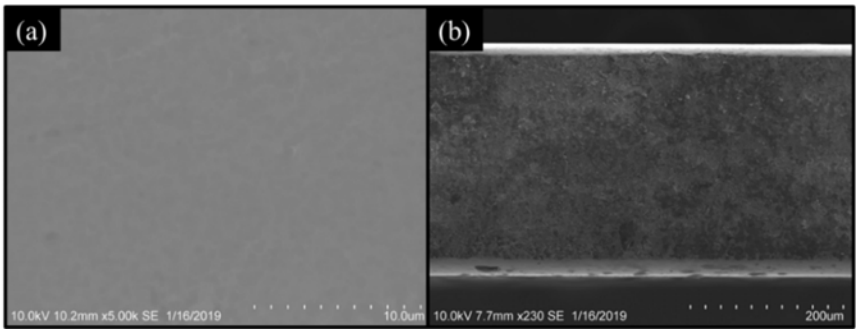

Figure 4.4: Scanning electron microscopy micrograph image of PZT thin film coated on STO substrate (MU2) (a) Surface image, (b) Cross section image.

4.1.3.3 MgO substrate (MU3)

Figure 4.5 shows smooth surface of PZT thin film coated on MgO substrate with some small irregular particle present on the surface. Besides, the thickness of the thin film is unable to identify because the layer of thin film and substrate cannot be identified from the cross section image.

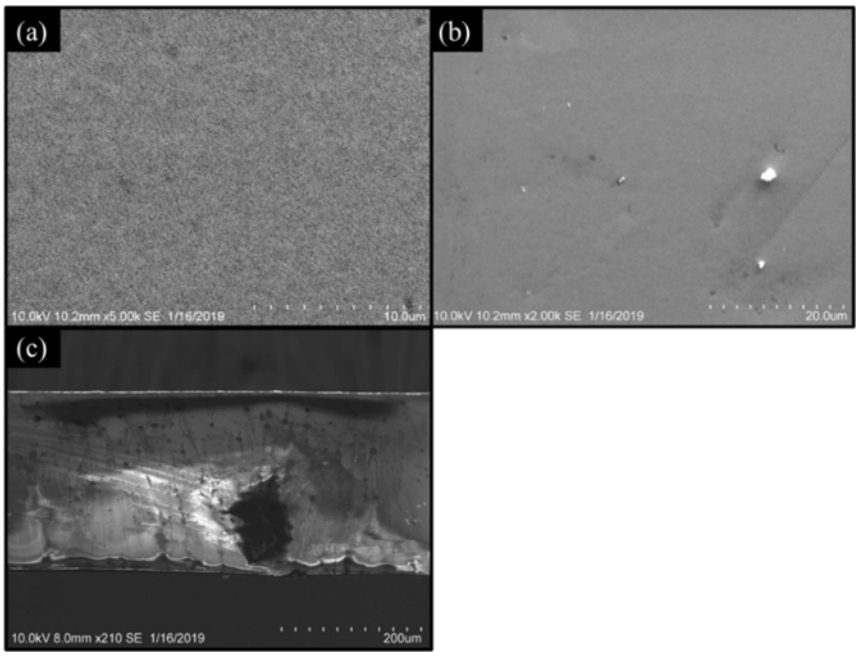

Figure 4.5: Scanning electron microscopy micrograph image of PZT thin film coated on MgO substrate (MU3) (a) and (b) Surface image, (c) Cross section image.

4.1.3.4 STO substrate (MU4)

Figure 4.6 shows the smooth surface of the PZT thin film on STO substrate with some irregular particles and the cross section image of the thin film is not able to use

for identifying the thickness of thin film because the layer of thin film and substrate cannot be identified from it.

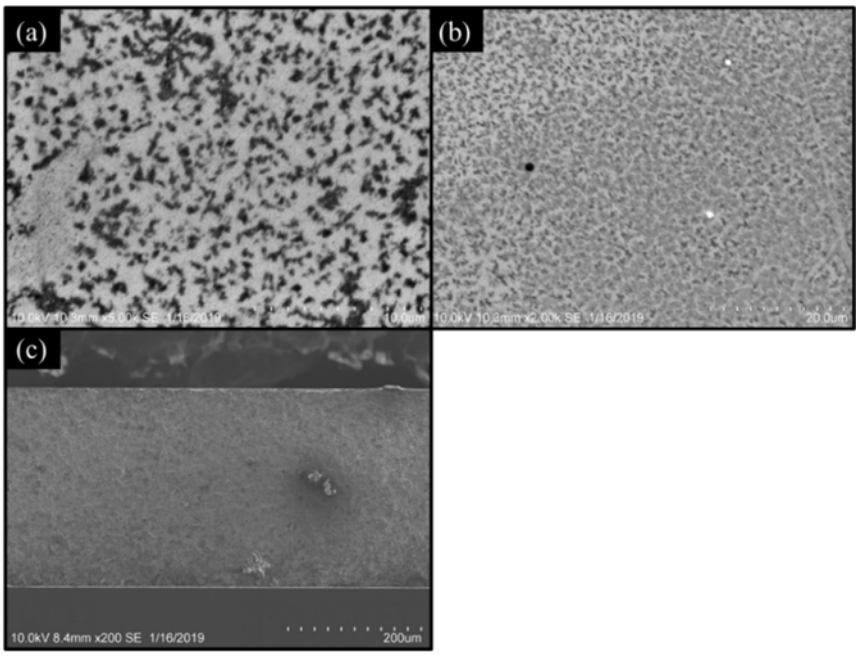

Figure 4.6: Scanning electron microscopy micrograph image of PZT thin film on STO substrate (MU4) (a) and (b) Surface image , (c) Cross section image.

4.1.3.5 Si substrate (MU5)

Figure 4.7 shows the presence of irregular particles on the surface of PZT thin film on STO substrate. Besides, the cross section image of the thin film is unable to use for identifying its thickness because the layer of thin film and substrate cannot be identified from it.

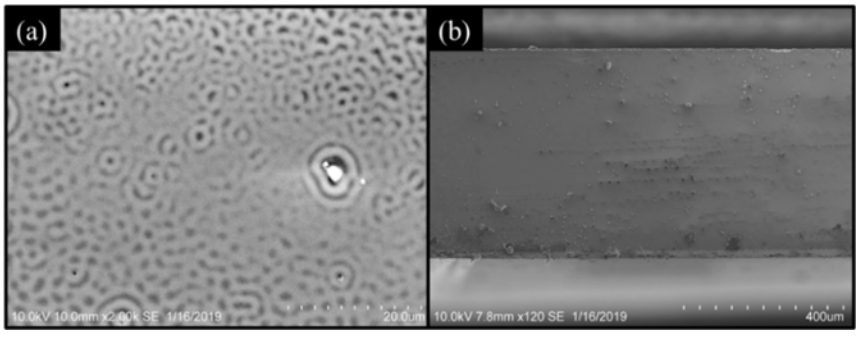

Figure 4.7: Scanning electron microscopy micrograph image of PZT thin film coated o Si substrate (MU5) (a) Surface image, (b) Cross section image.

Based on the microstructural analysis, the surface of the PZT thin film coated on STO substrate (MU2) found to have a smoother surface as compared with the other samples. Besides, most of the samples found to have irregular particles on the surfaces that perhaps are the contaminant on the thin film surface.

In addition, the layers and the thickness of the samples are unable to identify from the cross-section image due to insufficient resolution of SEM. Therefore, in order to obtain a clearer image and measure the thickness of thin film, instruments with higher resolution such as the field emission scanning electron microscopy (FE-SEM) and the high resolution multi-technique x-ray spectrometer is suggested to use.

4.2 Elemental analysis

In this section, the SEM-EDS analysis results of the samples are presented and the most significant findings are discussed.

4.2.1 Uncoated STO substrate

The SEM-EDS analysis performed in the Spot 1 of thin film revealed the presence of the Sr, Ti and O elements whereas the SEM-EDS analysis performed in the Spot 2 revealed the presence of the C element. The results indicated the sample tested is STO substrate and the C element perhaps is the contaminant in thin film (Refer to Figure 4.8).

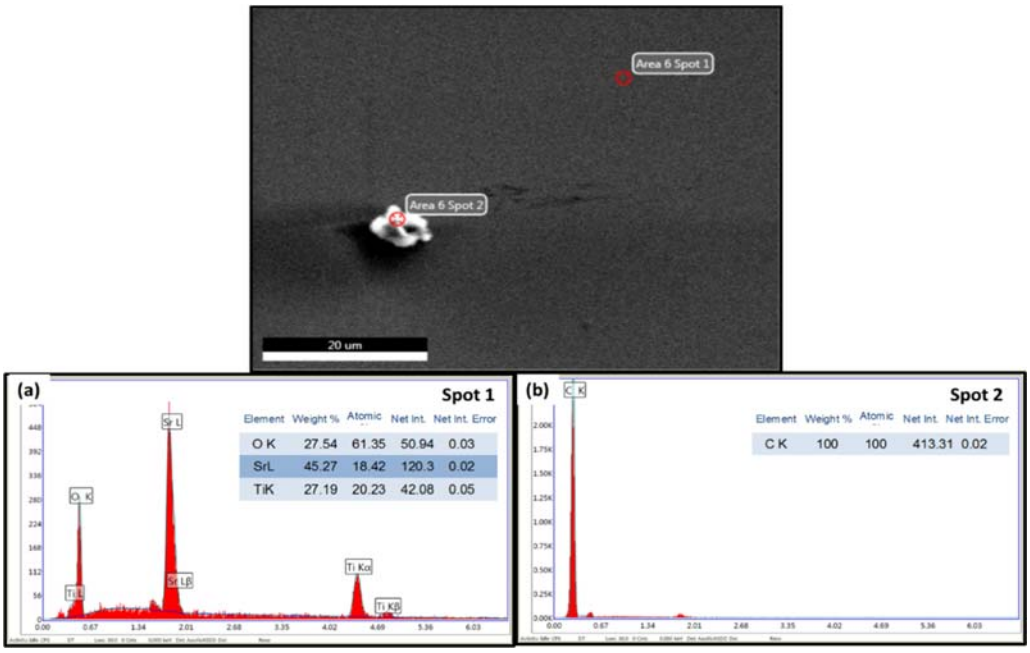

Figure 4.8: SEM-EDS analysis result for uncoated STO substrate (a) Spot 1, (b) Spot 2 .

4.2.2 MgO thin film coated on glass substrate (Sample 5)

The SEM-EDS analysis performed in the selected areas of thin film revealed the presence of the O and Mg element. The results revealed that the elements present in the area of the particle (Spot 1) is same as the elements present in the area of the flat surface (Spot 2) which are Mg, O and C elements. The results indicated that the sample tested is MgO thin film and the C element perhaps is the contaminant in the thin film (Refer to Figure 4.9).

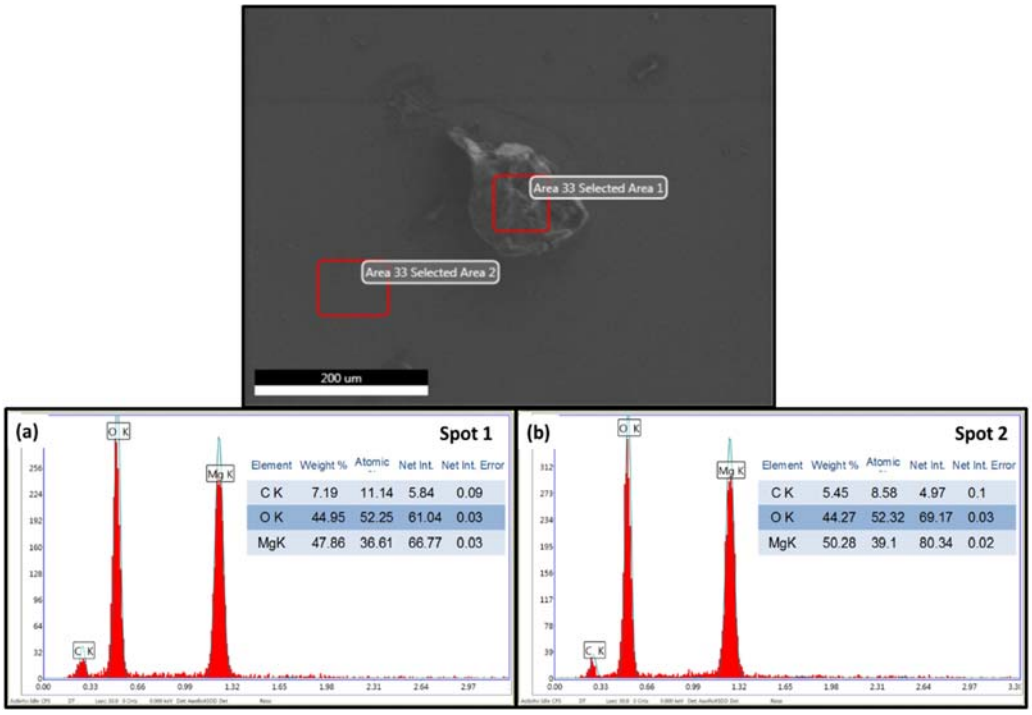

Figure 4.9: SEM-EDS analysis result for MgO thin film coated on glass substrate (a)Spot 1, (b) Spot 2.

4.2.3 PZT thin film coated on different substrates

The SEM-EDS analysis results of PZT thin film coated on different substrates such as Si, STO and MgO are presented in this section.

4.2.3.1 Si substrate (MU1)

The SEM-EDS analysis performed in the selected areas of thin film (Spot 1, 2 and 3) revealed the presence of the Si element. This result shows that the sample tested is Si substrate (Refer to Figure 4.10).