**CHARACTERIZATION OF PIEZOELECTRIC AIN THIN
FILMS DEPOSITED ON Si BY RF REACTIVE
MAGNETRON SPUTTERING AT LOW TEMPERATURE
FOR ACOUSTIC WAVE DEVICES**

by

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A thesis submitted to the
Faculty of Graduate Studies and Research in
partial fulfillment of the requirements for the degree of

Masters of Applied Science (M.A.Sc.)

Ottawa-Carleton Institute for Electrical and Computer Engineering

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Carleton University

Ottawa, Ontario

April 2004

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ABSTRACT

This thesis reports the effect of deposition conditions on film orientation, piezoelectric coefficient and stress of Aluminum Nitride (AlN) deposited on Silicon (Si) wafers by radio frequency (RF) reactive magnetron sputtering system. The purpose of these experiments was to determine the best deposition conditions using the RF magnetron sputtering system in order to obtain a good quality AlN thin films for acoustic wave device applications. Experiments were completed with RF power from 100 to 300 W, pressure from 4 to 12 mT, and N₂ flow from 50% to 100% of total, with the balance being Ar. Temperature and time were kept constant. The film was then characterized to determine which conditions resulted in the highest quality thin film. Characterization included the use of X-ray diffraction (XRD) to study the film orientation; using a Stylus profiler to measure film thickness; and the use of a cantilever beam apparatus to determine the piezoelectric coefficient (e_{31}). It was determined that the optimum sputtering conditions in this set of experiments were: maximum RF power of 300 W; mid-pressure of 8 mT; and 50% N₂ flow of total, with the balance being Ar. The characteristics of this film were: film thickness of 0.5 micron; crystal orientation of (002) AlN film with the $\bar{c}$ - axis perpendicular to the substrate; tensile film strain of 0.22% expansion of lattice plane separation; and piezoelectric coefficient e_{31} of 0.795 C/m².

ACKNOWLEDGMENTS

I would like to thank everyone at Carleton University, from teaching to support staff, who helped me in the development of this thesis. Special thanks goes out to Dr. Niall Tait, for supervising and guiding me through the steps of my research. I am also grateful to Lyall Bernt, Carol Adams, and Rob Vandusen for all their help and insight into fabrication lab procedures at Carleton University. Finally, I wish to thank my friends, Oana A. Tinca and Lee Shyue Choong, who supported me through the production of this thesis.

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LIST OF SYMBOLS AND ABBREVIATIONS

Å.....	Angstrom
cm.....	centimeters
C/m ²	Coulombs per square meter
F/m.....	Farad per meter
hr.....	hour
mm.....	millimeter
mV.....	millivolts
ms.....	milliseconds
mT.....	millitorr
nF.....	nanofarads
N/m ²	Newton per square meter
rad/s.....	radians per seconds
sccm.....	standard cubic centimeters per minute
W.....	Watts
µm.....	micrometers
°C.....	degrees Celsius
IR.....	Infrared

CHAPTER 1 INTRODUCTION

1.1 Thesis Motivation

Current personal wireless communication systems operate at high frequencies of a few GHz. They require high performance frequency selective filters, which operate selectively within a channel and reject unwanted signals at nearby frequencies. Figure 1.1.1 below shows places in which such filters are used in a front-end of a wireless receiver.

Figure 1.1.1: Front-End of a Wireless Receiver.

At the present time to accomplish sufficient performance, off-chip surface acoustic wave (SAW), crystal, or dielectric resonator filters are often used. Integrating

on-chip piezoelectric thin film bulk acoustic wave (BAW) resonator filters with other transceiver components is a promising approach to reduce cost, increase performance, and reduce the size of the wireless communication systems. This on-chip filtering requires the use of materials that are compatible with VLSI technology.

Thin film resonators (TFR) consist of a thin piezoelectric film of $\lambda/2$ thickness at the resonant frequency with two electrodes placed either on both sides or on the same surface of the film. Depending on where the electrodes are located, either bulk acoustic waves or surface acoustic waves are generated. Bulk acoustic wave (BAW) devices involve either shear or longitudinal acoustic waves generated by the electric field between the electrodes situated on either side of the film. Surface acoustic wave (SAW) devices consist of alternating polarity interdigitated electrodes positioned on the surface of the film. SAW filters require many electrode pairs to operate efficiently. Due to the requirement of a large number of interdigitated electrodes, SAW filters need larger area compared to BAW filters. Also, SAW filters tend to suffer from high insertion loss. BAW filters can have low insertion loss and can be made in a small size and also can be easily fabricated using VLSI fabrication techniques [1], [2].

In acoustic devices, both BAW's and SAW's, a piezoelectric layer is required to convert electrical signals into acoustic signals and vice versa. For efficient conversion of electrical signals into acoustic signals and back, the piezoelectric layer needs to have a high electromechanical coupling. Piezoelectric coefficients of piezoelectric materials are related closely to their electromechanical coupling (this will be described in more detail in chapter 2). Therefore, a piezoelectric material with high piezoelectric coefficient is required for a good acoustic device performance.

Thin film piezoelectric materials have been studied for almost three decades and used in both BAW and SAW devices. Ideal piezoelectric materials are single crystals. Materials such as Lithium Niobate (LiNbO_3) and Quartz are often used. These materials are expensive thus increasing the cost of the device. Polycrystalline materials can exhibit good piezoelectric properties, provided they have a strong preferred crystal orientation. Materials such as Zinc Oxide (ZnO), Lead Zirconate Titanate (PZT) and Aluminum Nitride (AlN) can be effectively deposited as highly oriented thin films by reactive sputtering at low temperature. These materials can also be grown epitaxially by chemical vapor deposition (CVD) or molecular beam epitaxy (MBE), but deposition temperatures of these techniques are usually high and therefore not compatible with VLSI metalization [3]. Among all piezoelectric materials, Aluminum Nitride (AlN) is the most attractive choice since it is preferred by the semiconductor industry for integration due to its compatibility with active devices on Silicon (Si) and Gallium Arsenide (GaAs). AlN is also chemically stable, mechanically strong and has a high acoustic velocity.

For sputtering purposes, to deposit AlN , radio frequency (RF) reactive magnetron sputtering is preferred over direct current (DC) sputtering. Even though DC sputtering has higher rate of deposition, in reactively sputtered films insulating materials can form on the target and cause arcing, which can result in wafer defects. The alternating polarity of RF signals reduces charging of insulating targets and avoids target arcing, producing high quality thin films.

1.2 Thesis Objectives

The objective of this thesis is to determine which low temperature RF reactive magnetron sputtering deposition conditions produce the highest quality piezoelectric AlN thin films. Quality of the deposited piezoelectric AlN thin film is determined by various characterization methods. These characterization methods involved measurements of X-ray diffraction (XRD), film thickness, film stress, and piezoelectric coefficient (e_{31}). Highly oriented (002) AlN thin film with low stress and high piezoelectric coefficient is considered ideal.

1.3 Thesis Outline

This introductory chapter has described why inexpensive, VLSI compatible, and good quality thin film piezoelectric material is required for use in acoustic filters in wireless communication systems, and how it is possible to determine optimum deposition conditions. Chapter 2 provides some background on the topics presented in this thesis, and Chapter 3 describes current research and results in thin-film technology. Chapter 4 describes the experimental method for film deposition and characterization, and Chapter 5 summarizes and analyzes the results obtained. The thesis will be concluded in Chapter 6, including recommendations for future research.

2.1 Introduction to Thin-Film Technology

Thin-Film technology involves the process of depositing a thin layer of target material onto bulk materials (substrates) to engineer a material that exhibits new properties. This technology is not confined to single layer deposition, as additional properties can be achieved by depositing multiple layers of various materials [4].

The thin film materials are chosen according to the properties required for specific applications. Some of the many applications of thin film technology are found in memory discs (CD), very large scale integrated circuits (VLSI) and microelectro-mechanical systems (MEMS). One of the thin film properties used in MEMS, such as surface and bulk acoustic wave resonators, is the electro-mechanical transduction capability of piezoelectric materials [4]. These applications will be discussed in depth in later sections.

Discussion in this thesis is focused on thin film technology and the thick film technology is intentionally excluded. The difference between the thick and thin film technologies is that in thin film technology, film deposition is carried out at molecular

level whereas in thick film technology, film deposition is carried out at particle level.

Silk screening, plasma spraying, and painting are a few examples of thick film technology. Note that in thick film technology the film deposits are not necessarily thicker than film deposits in thin film technology [4].

Thin films used in electronic circuits are usually a few hundred angstroms ($\text{\AA}$) to tens of thousands of angstroms thick. One angstrom ($\text{\AA}$) is equal to 0.1 nm. This thickness is the same order of magnitude as the dimensions of a single atom while the substrate supporting the thin film could be 10,000 times as thick [3]. The research that is the basis of this thesis focuses on the Aluminum Nitride (AlN) thin film which is about 5000 $\text{\AA}$ (0.5 μm), with the Silicon substrate being around 5,000,000 $\text{\AA}$ (500 μm).

There are various film structures for thin films. Examples include the amorphous thin films like anodic oxide dielectrics at one extreme, and single crystal films like epitaxially grown silicon at the opposite extreme. Most film-structures fall between these two extremes and they are known as polycrystalline. Polycrystalline structures are composed of many small crystals, called crystallites or grains, which are more or less randomly located together. For very thin films, crystallite size can range from 50 $\text{\AA}$ to 2000 $\text{\AA}$. Crystallite size is independent of film thickness. Deposition parameters such as substrate temperature and deposition rate will influence the size of the crystallite. Annealing is another method to achieve bigger crystallite size. It is performed after the deposition process to enlarge the crystallites at the expense of the smaller ones [5].

There are many types of materials that are used in thin film technology. Pure elements, compounds, and alloys can all be deposited as thin films. These materials can be categorized into four main groups having conductor, semiconductor, insulator and piezoelectric properties. Aluminum (Al), Silicon (Si), Silicon Dioxide (SiO_2), and Aluminum Nitride (AlN) are examples of those groups respectively. Piezoelectric materials, specifically AlN, are the area of study in this thesis, and will be expanded on in the next section.

The best piezoelectric properties are exhibited by single crystal piezoelectric materials, but polycrystalline materials can also exhibit good piezoelectric properties provided that they have a strong preferred crystal orientation. Deposition techniques are important to ensure strong favorable crystallite orientation in polycrystalline materials. Thin film deposition techniques will be further explained in subsequent sections.

Piezoelectric properties of materials are determined by characterization of the deposited films. These characterizations are performed to determine the quality of the thin films, as well as to improve the deposition conditions and techniques. Section 2.5 covers characterization of thin films in depth.

2.2 Thin-Film Materials

Almost any material can be used as the target or source material in thin film technology. Therefore properties of various materials can be exploited to produce thin film materials with different desired characteristic. The choice of material is entirely based on what kinds of properties are required for a specific application [5], [4].

Properties of thin film material can be categorized into six basic categories: Electrical, Mechanical, Optical, Magnetic, Chemical and Thermal. For example, Tantalum is used for its resistive electrical property for the fabrication of thin film resistors. In the cutting tool industry, Titanium Nitride (TiN) is used as the thin film material for its mechanical hardness property [5], [4].

There are also other more interesting properties such as electro-mechanical properties that can be provided by piezoelectric material like Aluminum Nitride (AlN) used in the study of this thesis [6]. This will be further discussed in the following section.

Beside the thin film material properties mentioned above, film thickness and film structure also have substantial effects on the properties of the fabricated thin film material. For example, thinness of the SiO_2 film will directly influence the capacitance of the SiO_2 thin film capacitor since its capacitance increases with thinner dielectric film. Beyond the practical thinness to increase the capacitance though, film material with higher dielectric constant is required [5], [4].

As mentioned above, film structure also has a strong influence on the properties of fabricated thin film materials. Film structure can be categorized into three main groups: crystalline, polycrystalline, and amorphous. Amorphous solid is formless and it does not have a defined structure. As for the polycrystalline material, it consists of many small regions (crystallites) each having a well-defined structure but differing from its neighboring crystallites. In crystalline solids, on the other extreme, atoms are arranged in orderly three dimensional periodic arrays referred to as lattices [4], [7].

Film structure can influence all properties of a fabricated thin film material. For example, one of the effects on physical properties is that some thin films with polycrystalline structures are about 200 times stronger compared to the annealed bulk material. The robust strength is due to the disorderly nature of the polycrystalline film structure [5], [7].

In this thesis, polycrystalline Aluminum Nitride (AlN) thin film is the material under study. Polycrystalline AlN film that has better ordered crystallites exhibits improved piezoelectric characteristics - which will be discussed subsequently. The degree of similarity which crystallites, within a polycrystalline film, are oriented is known as texturing. Random crystallite orientation refers to no texturing. A material in which the crystallites are nearly aligned in all three dimensions is called a “mosaic”, and a perfect mosaic is considered to be a single crystal with perfect alignment. The degree of

texturing is best measured by x-ray diffraction. This technique will be discussed in further sections of this thesis [4].

2.2.1 Piezoelectric Materials

Piezoelectricity (“pressing-electricity”) is the linkage between electrical and mechanical phenomena. Piezoelectricity couples the mechanical properties (stress and strain) and the electrical properties of a crystal [8].

For a non-piezoelectric material, an application of an external electrical field will cause a skew in the distribution of its molecular charge. The relationship between the intensity of the electrical field, $\vec{E}$ (V/m) and electrical displacement density $\vec{D}$ (C/m²) is:

$$\vec{D} = \epsilon_0 \epsilon_r \vec{E} = \epsilon \vec{E} \quad (2.2.1.1)$$

Where ϵ_r is the relative dielectric permittivity or dielectric constant (dimensionless), and ϵ_0 is permittivity of free space, 8.856×10^{-12} F/m. [6].

This simple relationship does not hold for piezoelectric material because the mechanical and electrical parameters are coupled. Application of an external mechanical stress or electric field stimulus onto a piezoelectric material will in turn cause a surface electric charge or mechanical deformation accordingly. The surface charge density can be related to a piezoelectric coefficient matrix $[e]$ (with units of C/m²) such that the electrical displacement density $\vec{D}$ is given by the matrix equation below:

$$\vec{D}=[e][S]+[\epsilon]\vec{E} \quad (2.2.1.2)$$

Where S is strain and $\vec{E}$ is electrical field intensity. Because of the coupling between the electrical and mechanical properties, permittivity ϵ is measured at a constant strain, which is normally zero [6]. The electrical displacement density $\vec{D}$ is three-dimensional in x, y and z coordinates. The strain matrix $[S]$ has six components (which are related as follows: $S_1 = S_{11}$, $S_2 = S_{22}$, $S_3 = S_{33}$, $S_4 = 2S_{32} = 2S_{23}$, $S_5 = 2S_{31} = 2S_{13}$, and $S_6 = 2S_{12} = 2S_{21}$), therefore, the piezoelectric coefficients terms in $[e]$ will form a 3×6 matrix with 18 elements. The relating dielectric permittivity $[\epsilon]$ is a 3×3 matrix with 9 elements [9], [6].

Piezoelectric materials are widely used together with thin film technology for the excitation and detection of acoustic waves devices [10]. One of the applications is the acoustic wave filter design. Of the piezoelectric films that were described earlier, Zinc Oxide (ZnO), Lead Zirconate Titanate (PZT) and Aluminum Nitride (AlN) are often used in thin film technology [11]. Aluminum Nitride (AlN) is the material of study in this thesis. AlN thin film was chosen because it is compatible with active devices on Silicon (Si) and Gallium Arsenide (GaAs), which are widely used in the VLSI industry. AlN is also chemically stable, mechanically strong, and has a high acoustic velocity (as high as 5650 m/s surface wave velocity). As a result, AlN is preferred for acoustic device applications at higher frequency (more than 1GHz) [11]. Acoustic device applications will be further discussed in the next section.

In the field of acoustic wave filter design, wave velocity (v) and electromechanical coupling coefficient (K^2) are among the most practical material

properties used. The electromechanical coupling coefficient measures how effectively a piezoelectric material converts between the mechanical and the electrical energy asserted externally to it [6]. The relationship between electromechanical coupling K^2 , the piezoelectric coefficient e , elastic constant c and dielectric permittivity ϵ , can be expressed as follows:

$$K^2 = \frac{e^2}{c\epsilon} \quad (2.2.1.4)$$

Electromechanical coupling constant K^2 , depends on both the crystal cut and the propagation direction of the acoustic wave [6]. An acoustic wave can be categorized based on its propagation direction with respect to polarization path. Longitudinal waves have parallel propagation and polarization directions. In the case of shear waves, their propagation direction is perpendicular to the polarization path [10]. These will be further explained in Section 2.3.1.

As mentioned above, piezoelectric thin films can be used as the source and detector of acoustic waves [10]. Piezoelectric materials are naturally anisotropic, in that their properties vary depending on the direction in which they are measured as opposed to isotropic materials where the properties do not vary in different directions. The xyz axes are designated 1, 2, 3, respectively, in accordance with normal crystallographic practice as shown in Figure 2.2.1.1 below. The various properties, such as piezoelectric coefficients e_{33} and e_{31} (out of the coefficient matrix $[e]$ from Eq. (2.2.1.2) above), are described by symbols with subscripts which correspond to these directions. The piezoelectric coefficient e_{33} is measured when there is a longitudinal wave induced in the

material, in which the wave propagation and the particle polarization are in the same direction. While for an induced shear wave, in which the wave propagation and the particle polarization are perpendicular to each other, e_{31} is the relevant piezoelectric coefficient [12].

Figure 2.2.1.1: Crystal Lattice Directions in Crystallography Practice.

2.3 Thin-Film Applications

Thin film technology application is everywhere. Due to the fact that almost any material can be used in thin film technology, different kinds of thin film devices can be readily produced for various applications [4]. Application of thin film technology can be observed in different industries. In the automotive industry, for example, Chromium (Cr) thin film is coated on plastic to give metallic shine, improve hardness, shield against

ultraviolet light and lower cost. Titanium Nitride (TiN) thin film which is used in cutting tools manufacturing industry, provides hard, low frictional and chemically protected cutting edges [4].

Optically reflective thin film materials are used to produce effective and accurate reflectors. This thin film technology is important in the development of the interferometer. An interferometer is an essential instrument in the progress of spectroscopic study [5].

One of the most successful and important usages of thin film technology is in the fabrication of the integrated circuits [13]. Integrated circuit technology can be found in almost any modern machine today. Therefore through application of integrated circuits, thin film technology appears everywhere.

2.3.1 Acoustic Wave Devices

Acoustic wave devices that are built with piezoelectric thin film technology are widely applicable to many different markets. The electronics industry first used acoustic wave devices in 1965 when it was discovered that surface acoustic wave (SAW) transducers could be useful in the design of analog filters with selected frequencies in the range of 10 MHz to 1 GHz and above. SAW device research continued, and during the late 1970's SAW devices were used in many different applications. Afterwards SAW

devices continued to be developed for consumer, commercial, and military applications [6]. For example, in low-volume high-margin market like military radar signal processing systems, acoustic wave devices are used as pulse compression filters. In addition, acoustic wave devices also thrive as the Intermediate Frequency (IF) filters used in television manufacturing, a high-volume but low margin consumer electronic industry [6], [10].

There are two main categories of bulk acoustic waves (BAW) propagating in solids. They are either longitudinal (pressure) or transverse (shear) waves, as shown in Figure 2.3.1.1 below. As mentioned above, longitudinal waves have parallel propagation and polarization directions but shear waves propagation directions are perpendicular to the polarization paths [10]. On the other hand, surface acoustic wave (SAW) devices involve a wave propagating along the surface of the material. BAW and SAW are the two main types of acoustic waves commonly used in the design of acoustic wave devices.

Figure 2.3.1.1: Longitudinal and Shear Waves.

As mentioned in section 2.2.1, piezoelectric thin films can be used as the source and detector of acoustic waves in acoustic wave devices. One of the applications of BAW is the bulk-wave delay line as seen in Figure 2.3.1.2 below. An incoming electrical signal is converted into a mechanical acoustic wave through a transducer and the converted acoustic wave propagates from one to the other end of the piezoelectric material. The acoustic wave is then converted back to outgoing electrical signal through the transducer on the other end [14].

Figure 2.3.1.2: Bulk-Wave Delay Line.

SAWs can be also exploited to produce effective delay line devices. A SAW delay line device is shown in Figure 2.3.1.3 below. Unlike BAW devices, acoustic waves on a SAW device are readily accessible for further processing, for example filtering. Interdigital transducers (IDTs) are widely used in acoustic wave devices for SAW excitation and detection [14].

Figure 2.3.1.3: SAW Delay Line.

Smaller size is one of the obvious advantages of acoustic devices. The smaller dimension of acoustic devices like delay lines is due to the fact that acoustic waves are very slow compared to electromagnetic waves (about 10^5 times slower). Because the size of a delay line is directly proportional to wave velocity, acoustic wave delay lines are therefore about 10^{-5} the size (depending on the material) of the comparable electromagnetic wave devices [14]. IDTs on a SAW device required a relatively larger area therefore it is typically a larger device compare to a BAW device [14].

Due to their capability of reaching high resonance frequencies, acoustic waves are of interest in high frequency (as high as 2.5GHz) microelectronic design. One of the examples of this application is the low-loss (e.g. 1 to 3 dB) RF front end filters for mobile and wireless devices [6]. The front end of a wireless receiver is shown in Figure 1 in Section 1.1.

Research and discovery of new improved piezoelectric thin film is essential for the development of smaller, higher performance, and VLSI fabrication compatible acoustic devices.

2.4 Thin-Film Process

The thin-film process consists of two main steps, fabrication and characterization, as shown in Figure 2.4.1. Fabrication step involves the choice of the physical state of the source material (solid, liquid, vapor or gas), transportation medium (fluid, vacuum or plasma) and deposition process (physical, chemical or hybrid).

Figure 2.4.1: Thin-Film Fabrication Process.

Solid source materials are required to be vaporized to facilitate their transportation to the substrate. The vaporization process can be achieved by heat or by an

energetic beam of electrons, photons (laser ablation), or positive ions (sputtering).

These methods are identified as *physical vapor deposition* (PVD) [4].

In some cases, solid target materials are vaporized, and in other cases, liquid or gaseous precursor materials with sufficient vapor pressure at moderate temperatures are introduced and chemically react on the substrate to form a coating. *Chemical vapor deposition* (CVD) is the category that encompasses these kinds of processes [4].

Other methods known as Hybrid methods exist, which make use of both PVD and CVD techniques for deposition processes [15] but these will not be touched upon in this thesis. Among the above mentioned deposition techniques, PVD techniques specifically RF planar magnetron reactive sputtering is examined in depth in this thesis.

After the thin-film is fabricated, film characterization is an important step to determine film quality and quantity. Film characterization is also important to improve the deposition process. Deposition process conditions are correlated to the results of the film characterization for the purpose of improving the process. Characterization of the deposited films involves (to name a few) determination of their mechanical properties such as film thickness and stress, crystal orientation, and in the case of piezoelectric material, their electro-mechanical properties such as piezoelectric coefficient. These characterization studies will be the focus of later sections.

2.4.1 Physical Vapor Deposition (PVD)

Physical vapor deposition (PVD) uses physical ways to vaporize the target material. The produced vapor is later deposited as a thin film layer on the substrate. Evaporation can be physically induced using thermal sources (a hot filament or an evaporation crucible), electron-beam (E-gun), photon-beam (laser-induced), and energetic ions (sputtering). These techniques are performed under different degrees of vacuum, normally under high (10^{-6} torr) to ultrahigh (10^{-9} torr) for evaporation and moderate to low (10^{-4} to 10^{-1} torr) for sputtering [16].

Sputtering is the technique used in the experimental base for this thesis. In this study, a piezoelectric material, Aluminum Nitride (AlN) is deposited onto Silicon (Si) substrates.

2.4.1.1 Sputtering

Sputtering is the physical process of using energetic particles to bombard the surface of a material, causing material solid particles to dislodge and be ejected from the surface due to the momentum exchange. See Figure 2.4.1.1.1 below for the physical set up of the sputtering mechanism. The target material can be almost any solid (alloys or compounds, both insulating and conducting) thus making the sputtering technique advantageous to use [17]. The sputtering advantage is that any material can be deposited

stoichiometrically, and a uniform film deposition rate can be acquired over large areas [4]. As well, the kinetic energy distribution of sputtered atoms is close to the energy needed for the displacement of surface atoms, therefore there is no extra leftover energy, which could damage the deposited surface. Due to these properties, any form of sputtering is the physical vapor deposition method of most widespread use.

Figure 2.4.1.1.1: Modern Sputtering System.

One form of sputtering is diode sputtering, which uses gaseous plasma discharges to bombard a cathode (usually -200 to -5KV) target, resulting in atoms being displaced (sputtered) and going to the anode substrate (strategically placed to intercept the displaced atoms flux) where they form a coating [16]. Heavy inert gases (usually argon ions) are used to provide the energetic ions which are discharged onto the target. This

discharge results from a current flowing through the low pressure gas, and some of the discharges can glow due to light emission from excited atoms [16]. The plasma used in the experiment section of this thesis is of this glow discharge kind. Note that though the discharge particles could be either neutral or ionic, ions are preferred since they can be controlled (accelerated) by an electric field [18] – whereas neutral particles are harder to steer towards the target.

One form of sputtering involves using a gas to move one of the coating materials into the reaction chamber, and is called reactive sputtering due to the fact that the sputtered ions (from the target) react chemically with the introduced gas and form the resulting deposited coat [5], [4]. For example, sputtering of Aluminum (Al) in plasma containing Nitrogen (N_2) will result in deposition of Aluminum Nitride (AlN), which is the case of the study of this thesis. Other forms of sputtering include the use of a composition of materials to make up a single target material, or the use of more than one target during the sputtering process (during which deposition can be made at the same time on all targets, or in a predetermined order). These other forms are not generally used since the sputtered film could turn out to be stoichiometrically different than the target material, which would change the orientation properties of the deposited film, in turn changing its properties in an undesirable manner.

The purpose of the next two sections is to provide a basic physical understanding of how sputtering discharges work. Sputtering discharges can be simplified and generalized into four fundamental Diode sputtering arrangements: a direct current (DC)

discharge, a capacitive radio frequency (RF) discharge, a DC magnetron discharge, and the last and foremost, an RF magnetron discharge, which in the form of a planar magnetron is the sputtering arrangement chosen for the work of this thesis [16].

Different magnetron sputtering configurations exist with their variation arising from the alignment of the electric and magnetic fields which can control ion densities and in turn deposition rates [15]. One form of magnetron sputtering is the cylindrical configuration in which the magnetic and electric fields are uniformly aligned orthogonally, which are depicted in Figures 2.4.1.1.2 and 2.4.1.1.3 below. Other configurations include planar and conical sputtering, which is shown in Figures 2.4.1.1.4, and are based on similar but more complex magnetic and electric field alignment.

Figure 2.4.1.1.2: Cylindrical Magnetron.

Figure 2.4.1.1.3: Cylindrical Hollow Magnetron.

Figure 2.4.1.1.4: Planar Magnetron.

There are other sputtering arrangements, which are successful modifications to the basic diode arrangements mentioned above. Triode sputtering sustains the gas discharge by using a hot filament to supply additional electrons to the plasma. Another arrangement uses a vacuum instead of immersing substrates in a glow discharge. This arrangement is called ion-beam sputtering and uses an ion source with a low voltage discharge to create an ion beam, in turn biased to a high DC potential which gives it equal kinetic energy (around 1kV), which is then directed onto a sputtering target [16].

2.4.1.1.1 DC/RF Sputtering

In DC Sputtering a high voltage DC source (in the range of kilovolts) is used (to create and sustain the plasma), with the sputtering target being the cathode of the discharge, and with the substrate (and/or vacuum chamber walls) acting as the anode. The separation between these (cathode and anode) is around a few centimeters, and Argon is the most commonly used sputtering gas. See Figure 2.4.1.1.1 below for a representation of this arrangement.

Figure 2.4.1.1.1: DC Sputtering System.

DC discharges require the use of only conductive targets. This is due to the nature of the target cathode which accumulates charge, and would not allow for the flow of charge if it were non-conducting. In order to overcome this, Radio Frequency (RF) voltage can be used to create a capacitive environment around a non-conducting target in order to create a discharge. This is called RF sputtering and is physically depicted in Figure 2.4.1.1.2 below.

In RF sputtering, an RF source is used as a power supply, with 13.56 MHz being the most commonly used frequency, and with a range of 0.5 to 1kV amplitude voltage in frequency ranges higher than 0.1 MHz. While the RF current amplitude is relatively high, the mean ion current density is of the order of 1 mA/cm^2 . The circuit also contains a capacitor in order to sustain DC self-bias, and this configuration is matched to a

network resulting in optimized power transfer between the RF source and the plasma.

The plasma is created by the same processes as in DC discharge, except using an RF source instead (the ions behave approximately like in DC glow discharge since they can not respond to the high RF frequency). Figure 2.4.1.1.2 shows the set-up of the RF source sputtering device, with a matched network representation.

Besides its ability to use non-conducting or electrically insulating targets, RF sputtering is also preferred over DC sputtering since it can use lower voltages and sputtering gas pressures while achieving higher deposition rates [16], and is relatively simpler to implement than other techniques. Note that magnetron RF discharges are the most widely used (as opposed to non-magnetron arrangements) even though they can turn out to be more costly (as they require matching networks), and may also be noisy (RF noise).

Figure 2.4.1.1.2: RF Sputtering System.

Though DC/RF sputtering is the most popular technique due to its simplicity, it does lack efficiency, and care must be taken to ensure that the thin film is not damaged by the increase in temperature due to the energetic electrons bombarding the substrate. Another form of the DC/RF sputtering technique, DC/RF planar magnetron sputtering is explained in the next section.

2.4.1.1.2 DC/RF Planar Magnetron Sputtering

The planar magnetron sputtering technique is a form of sputtering which is based on a discharge influenced by a magnet. As previously discussed, either DC or RF excitation can be used, and a perpendicular (to the target) electric field exists. In addition, a strong permanent magnet is used which causes a magnetic field to form creating magnetic flux perpendicular to the electric field (and thus parallel to the target surface). The magnetic field acts to trap the electrons near the target surface, thus increasing ion bombardment of the surface, which in turn means increased sputtering rate. This discharge which is controlled by the magnetic field forms the magnetron, and results in higher efficiency than non-magnetic DC/RF sputtering. Refer to Figure 2.4.1.1.4 in Section 2.4.1.1 for the setup of a planar magnetron sputtering device.

To understand how electrons are trapped in a magnetron, electron movement must first be explained. In a magnetic field, an electron encircles the lines of flux in the $-\vec{E} \times \vec{B}$ direction with the gyro radius of their orbits being:

$$\rho = \frac{mv_{\perp}}{qB}, \quad (2.4.1.1.2.1)$$

Where $v_{\perp}$ is the perpendicular (to the flux lines) component of the electron velocity. Since electrons keep going in this orbit until they are displaced by another particle, entrapment occurs. Once escaped from the cathode, being trapped in this orbit causes an increased path of travel for the electrons before reaching the anode. As well, the gyro radius is usually in the millimeters range or less, so electrons are thus kept near the target surface for as long as they are looping the magnetic flux lines (which usually occurs when there is a closed $-\vec{E} \times \vec{B}$ path and lasts several loops). Increased electron path results in higher electron density. This improves deposition rate compare to non-magnetron systems.

When operating these planar magnetrons, argon is the gas of choice used at pressures of 0.5-30 mT. DC voltages are in the range of 300-700V [Waits, 1978] which is considerably less (an order of 10 times) than if a magnet was not used to trap electrons - since a high voltage is not needed to maintain the plasma due to increased electron usage efficiency. RF voltages are lower (usually less than 500V amplitude) than in non-magnetron RF sputtering as well. In general RF magnetrons are used for sputtering insulators or non-conductive materials only, since their efficiency is usually half that of DC magnetrons [16].

Problems with the planar magnetron configuration stem from the cathode negative ions which cannot be deflected by the existing magnetic field, as well as from non-

uniform erosion of the magnetron target, which in turn becomes the pattern imprinted on films deposited on substrates. The uniformity of the thin film is not much influenced by the deposition rate since those sputtered particles are neutral (and characterized by a broad cosine distribution pattern) [4].

The advantages of the planar magnetron configuration are that it has high deposition rates (due to the magnetic trapping influence on electrons which causes higher number of ions to be formed), and it can be used for large deposition areas. Another advantage is that some substrate damage is prevented since electrons are safely trapped and cannot easily bombard the substrate surface [16]. Some typical magnetron characteristics are denoted in Table 2.4.1.1.2.1 below.

Table 2.4.1.1.2.1: Typical Magnetron Characteristics.

	Typical Magnetron Characteristics	Diode
Cathode Current Density	20 mA/cm ²	1 mA/cm ²
Discharge Voltage	800 V	3000 V
Argon Pressure	≥0.1 Pa	≥10 Pa
Deposition Rate	2000 Å/min	300 Å/min
Important Characteristic	• Low pressure, Low voltage • High current, High deposition rate	

2.4.2 Chemical Vapor Deposition (CVD)

Chemical Vapor Deposition (CVD) uses chemical precursors as source material. A chemical reaction at the substrate produces a thin film layer on the substrate. The precursor materials can be solid, liquid, or gaseous – any physical form can be vaporized if it has sufficient vapor pressure and moderate temperatures.

The basis for CVD is that a chemical reaction (activated by heat, laser, glow discharge, or catalytic action) takes place between compounds that exist in the vapor phase which causes them to give a product. If the reaction occurs on the substrate surface, a thin film is formed. See Figure 2.4.2.1 for an example of a typical CVD setup. The resulting thin film is characterized by properties which are controlled by the chemical reaction parameters such as temperature, pressure, concentration, or flow rate.

Since the reacting materials can be controlled, either pure or doped deposits can be deposited onto the substrate. This property makes CVD an important process, especially in microelectronic manufacturing where pure or doped materials must be deposited at specific locations. In these processes temperature is usually minimized in order to preserve the substrate, so low-temperature reactants are usually chosen. These tend to be very reactive naturally (since they don't need high temperatures to jump start the reaction process), and must be handled carefully due to toxicity [19].

Figure 2.4.2.1: CVD System.

2.5 Characterization of Piezoelectric Thin-Film

Now we come to the final step, the step after the film fabrication process of Figure 2.4.1, which is to actually have a look at the film.

Piezoelectric properties of thin films are determined by characterizing them. These characterizations of the deposited thin films are also to improve the deposition conditions and techniques. Characterization of deposited piezoelectric thin films involves the determination of mechanical, physical, electrical and/or electro-mechanical properties of the films.

Some of the mechanical properties of thin films are determined by film thickness and stress measurements. Crystal orientation is linked to the physical properties of the films. The electrical and/or electro-mechanical properties of piezoelectric films are determined by piezoelectric coefficient measurement of the films which are closely related to the electromechanical coupling of the films.

The measurement techniques to analyze the above mentioned properties of piezoelectric thin films are described in more detail below.

2.5.1 Thin-Film Thickness Measurement

One method of measuring film thickness is with the stylus profiler. This instrument works by dragging the stylus over the thin film, which has been line patterned, while measuring the stylus deflection to an accuracy of approximately 1nm. The topography of the film is deduced through this vertical movement. This makes measurement of very thin films difficult since the stylus might tear the film if small crevices exist in the substrate (since the substrate acts as the underlying support of the thin film against which the stylus pushes). Diamond is usually used for the stylus, which is held by a controlling arm. The arm is then attached to a reference surface. The movement recorded against the reference surface as the stylus traverses the thin film can

be amplified and read to deduce film thickness. Figure 2.5.1.1 shows an example of how a stylus might be used and interpreted.

Figure 2.5.1.1: Stylus.

2.5.2 Thin-Film Stress Measurement

Mechanical stress exists at every point of a structure, whether it is tensile or compressive. Tensile stress (Figure 2.5.2.1) is when the material tends to contract along the surface in question, while compressive stress (Figure 2.5.2.2) is when the material expands along the same surface. Film layers are subject to both kinds of stresses since they are bound to each other as well as their supporting substrate. Usually physical vapor

deposition processes result in materials with tensile stress and chemical vapor deposition results in materials with compressive stress. Since tensile and compressive stresses are complementary, films and substrates induce one or the other stress upon each other. The interface forces that are transmitted along the materials influence the stress.

This physical property, stress, depends on the film deposition conditions [4]. Thus by measuring stress, the success of the deposition process and the quality of the film can be deduced. Note that stress is undesirable since it can lead to the deformation or complete breakage of the substrate depending on the film adhesion and nature of stress. Most stress is found to be in the range of 10^7 to 10^9 N/m² (10^8 to 10^{10} dynes/cm²).

Figure 2.5.2.1: Tensile Stress in a Thin Film.

Figure 2.5.2.2: Compressive Stress in a Thin Film.

The mechanics behind stresses are as follows. The total stress of a material is based on the external, thermal, and intrinsic stresses of the material. External stresses arise from factors such as assembly problems, or mistreatment of the substrate and operating components. Thermal stresses, as the name indicates, arise from differences of temperatures which can cause the material to expand or contract, and intrinsic stresses are those that are caused by internal material composition and crystal arrangement – which in this case is based on deposition conditions and success. The total stress can then be expressed as:

$$\sigma = \sigma_{external} + \sigma_{thermal} + \sigma_{intrinsic} \quad (2.5.2.1)$$

Where σ denotes stress.

Stress measurement can be done by two techniques, Newton's Ring Method and the Bending Beam Method [22]. Both of these are based on the simple mechanical relationship between stress and strain:

$$E = \frac{\text{stress}}{\text{strain}} \quad (2.5.2.2)$$

where E is Young's modulus representing the stress/strain ratio. Since strain on an object is considered to be the displacement of the object under a stress (for small displacements), the stress can then be determined by the measurement of this displacement.

Newton's Ring Method, as applied to thin films, involves the deposition of film on a thin, circular disc (with approximate thickness of 100 μm) which is then placed on an optical flat. The radius of curvature (r) can then be determined by Newton's rings, which is the interference pattern that appears. Figure 2.5.2.3 shows this setup. The stress is determined as:

$$\sigma = \frac{1}{6} \frac{E_s}{r(1-\nu)} \frac{t_s^2}{t_f} \quad (2.5.2.3)$$

where E_s is Young's modulus of substrate, and t_s and t_f are substrate and film thicknesses respectively. Poisson's ratio ν is another mechanical variable which is needed in the calculation of stress. This is based on the physical response of an object under stress, namely if an object is extended (stretched), in response it will normally contract in thickness (laterally), or vice versa (compression would result in a larger lateral thickness). This ratio is denoted as:

$$\nu = \frac{\text{lateral strain}}{\text{axial strain}} \quad (2.5.2.4)$$

Where the lateral strain represents the thickness change, and the axial strain represents the compression or tension. Note that this method has limited sensitivity (about 10^7 N/m²).

Figure 2.5.2.3: Newton's Ring Method.

The bending beam method is based on measuring the deflection of an external beam. In the case of thin films, a thin (typically 100 to 500 μm) glass slide may be used to measure the deflection (δ) of the free end. The setup, as shown in Figure 2.5.2.4, involves the clamping of the slide to the material to be measured, after which deflection

can be measured either by direct optical measurement, interference techniques, or capacitive techniques. Stress is then calculated based on the radius of curvature r :

$$r = \frac{l^2}{2\delta} \quad (2.5.2.5)$$

where $l = \text{beam length}$, $\sigma = \frac{E_s t_s^2 \delta}{(1-\nu) 3l^2 t_f}$ (2.5.2.6), and t is the thickness.

Note that this method has much better sensitivity than the former, approximately 10^4 N/m^2 which is a deflection of about 1 nm, and is thus the preferred method of thin film stress measurement. But since this method requires cutting the wafers and making beams, most commercial film stress measurement instruments are wafer curvature measurements.

Figure 2.5.2.4: Bending Beam Method.

2.5.3 Thin-Film Crystal Orientation Measurement

Thin-film crystal orientation measurement will be described in three subsections. Crystal structure will focus on physical characteristics of crystals and their atomic arrangement, x-ray diffraction will focus on x-ray methods and diffraction theory, and the measurement methods subsection will show how physical characteristics can be deduced from x-ray data.

2.5.3.1 Crystal Structure

A crystal is made up of imaginary cells, in which atoms are arranged in different geometries. These cells are known as Bravais lattices [23]. Note that cells can contain many atoms, and are all the same within a crystal, meaning that each cell exhibits the same symmetry as the entire crystal [21]. The Bravais lattice that describes atom arrangement exists in only 14 different geometries. These are shown in Figure 2.5.3.1.1. Each cell is constructed from a number of atoms which are attached to the “basis” atoms at each lattice point. Aluminum Nitride (AlN) crystal is hexagonal.

CUBIC

$$a = b = c$$

$$\alpha = \beta = \gamma = 90^\circ$$

TETRAGONAL

$$a = b \neq c$$

$$\alpha = \beta = \gamma = 90^\circ$$

ORTHORHOMBIC

$$a \neq b \neq c$$

$$\alpha = \beta = \gamma = 90^\circ$$

HEXAGONAL

$$a = b \neq c$$

$$\alpha = \beta = 90^\circ$$

$$\gamma = 120^\circ$$

TRIGONAL

$$a = b = c$$

$$\alpha = \beta = \gamma \neq 90^\circ$$

MONOCLINIC

$$a \neq b \neq c$$

$$\alpha = \gamma = 90^\circ$$

$$\beta \neq 120^\circ$$

TRICLINIC

$$a \neq b \neq c$$

$$\alpha \neq \beta \neq \gamma \neq 90^\circ$$

4 Types of Unit Cell**P = Primitive****I = Body-Centred****F = Face-Centred****C = Side-Centred**

+

7 Crystal Classes**→ 14 Bravais Lattices**

Figure 2.5.3.1.1: Bravais Lattices

Figure 2.5.3.1.2 shows a simple cubic Bravais lattice example based on Cesium Chloride, in which the Cs^+ and Cl^- ions form the basis of the structure.

Figure 2.5.3.1.2: Cesium Chloride Crystal Structure (Cubic Lattice).

Since each lattice has the same symmetry as the overall crystal, it also has the same crystal axis vectors $\vec{a}$, $\vec{b}$, and $\vec{c}$, as seen in Figure 2.5.3.1.3 below. These axes are not necessarily perpendicular (depending on which of the 14 lattice structures it represents). These crystal axes can be used to indicate direction of each axis. For example, the direction of the $\vec{a}$ axis is $[100]$, and the $-\vec{b}$ axis is $[0\bar{1}0]$. A crystal plane can also be defined from these crystal axes, and is denoted by its orientation direction as (hkl) . This orientation direction is called the Miller indices [23]. In order to find the Miller indices, the axes intercepts (on $\vec{a}$, $\vec{b}$, and $\vec{c}$) must first be found. The reciprocals of the intercepts are then reduced to three integers, having the same ratio, denoted as h , k , and l to form the Miller indices. For example, in Figure 2.5.3.1.3, the plane intersects the axis at $3a$, $2b$, and $2c$. Based on the above, the reciprocals of these numbers are at $1/3$,

$1/2$, and $1/2$. The smallest three integers having the same ratio are 2, 3, 3. The Miller indices of the plane shown in Figure 2.5.3.1.3 is then denoted as (233).

Figure 2.5.3.1.3: Crystal Axis Vectors.

Different crystal planes are shown in Figure 2.5.3.1.4 along with their Miller indices. As can be seen, the (200) plane is parallel to the (100) plane, but shifted to cut the $\bar{a}$ axis at $1/2 \bar{a}$ instead of at $\bar{a}$.

Figure 2.5.3.1.4: Crystal Planes with their Miller Indices.

2.5.3.2 X-Ray Diffraction

An “x-ray” can be defined as electromagnetic radiation of very short wavelength (0.01 to 100nm) which can be produced when an electron bombards a piece of metal in a vacuum [4]. In diffraction there is an incident beam (or wave) which can either scatter in different directions once it hits a single particle, or the scattering may be in random directions which could add up to a diffracted beam after it hits a solid material. In a crystalline solid, the molecules are arranged in a regular pattern, this regularity results in reinforced intermingled diffracted beams – as opposed to random scattering which can result in diffracted beams canceling each other out [21], [24]. The intensity of this diffraction is determined by the arrangement of the atoms in a cell (the structure factor), and also by the scattering power of single atoms (the atomic form factor) [4], [21], [24].

There are two diffraction theories which are based on how much of the incident beam is scattered randomly and how much is diffracted. Note that diffraction can occur multiple times if the diffracted beam itself is diffracted. Kinematic Diffraction Theory is based on the assumption that there is weak absorption of x-rays and that there are no multiple scattering effects, while Dynamic Diffraction Theory assumes strong absorption and multiple scattering [24]. The Kinematic Diffraction theory is the focus in this thesis since it is valid for x-ray diffraction, rather than the Dynamic Diffraction theory which is more complicated and usually used for electron diffraction.

For the purpose of thin film analysis, x-ray diffraction is used. For apparatus, x-ray diffractometers are most commonly used. A representation of this apparatus, called a Bragg-Brentano x-ray diffractometer, is found in Figure 2.5.3.2.1 below.

Figure 2.5.3.2.1: Schematic of a Bragg–Brentano X-ray Diffractometer.

Different diffraction techniques result in different diffraction patterns, which are records of the diffracted beams produced. Physical material characteristics can then be determined from these patterns. This includes measuring the average spacings between the layers (or rows) of atoms, determining the crystal structure, and measuring the size, shape, and internal stress of crystalline regions [4]. The fact that x-rays are diffracted from a crystal when their wavelength corresponds to periodicity in the crystal lattice leads to the ability to correlate diffraction and physical characteristics.

After deposition, x-rays can be used for film analysis. When an incident beam penetrates the crystal lattice it scatters as it interacts with each of the atoms. When the incident and scattering angles (θ) are equal, as measured from the x-ray plane, the length of the beam path will also be the same – resulting in interference which produces a wavefront. The properties of these waves, the wave vectors of the incident and reflected beams, and their wavelength λ form the basis of what is known as Bragg's equation [4]:

$$d = \frac{n\lambda}{2 \sin \theta} \quad (2.5.3.2.1)$$

where θ and λ are as described above, and d is the crystal plane spacing distance. The multiple “ n ” arises from the fact that for constructive interference, the difference between path lengths of beams reflected from successive planes (in the z direction) must be equal to a multiple of the wavelength λ .

There are other means, besides Bragg's geometry, of predicting the direction of the diffracted rays. An example is the use of reciprocal geometry, based on the relationship of reciprocal space to real space. Through this method, Miller's indices come into play as they describe crystal-plane orientations in reciprocal space. Note that this is mentioned in an informative manner as it is beyond the scope of this thesis.

2.5.3.3 X-Ray Measurement Methods

In order to interpret the incident or scattering angle (θ), a physical set-up must be used. The angle θ can be scanned from the x-y plane once the thin film is oriented in that plane, and the sample is rotated about the y axis. During the rotation, an x-ray detector is simultaneously rotated through an angle equal to 2θ in order to keep it at the correct angle with respect to the film surface. When the atomic periodicity (distance) is perpendicular to the film surface (which is supposed to satisfy Bragg's equation depending on which wavelength is used), a peak will be read (imprinted) by the detector. From this peak occurring at 2θ , the atomic periodicity can be used to determine the atomic plane, and the intensity (height) of the peak indicates the degree of texturing (meaning that the intensity increases with the fraction of crystallites with atomic planes parallel to the surface). From the width of the peak at half its maximum intensity, denoted as $B = \Delta(2\theta)$, the size of the crystal grains can be determined [4]. A detailed analysis of these measurements and their meanings is provided below.

In order to explain how measurements are read from an x-ray trace, an example will be used. This example is based on the XRD trace depicted in Figure 2.5.3.3.1 below which is taken from measurements of AlN thin film on silicon substrate.

Figure 2.5.3.3.1: XRD Trace of AlN Thin Film.

As previously mentioned, a high peak intensity indicates the most common occurring (preferred) crystal orientation. For the (002) AlN in Figure 2.5.3.3.1 the peak intensity shows a good crystal orientation of the $\bar{c}$ - axis perpendicular to the substrate. A peak at a small diffraction angle (determined from 2θ) indicates that there is large spacing between the lattice planes. Note that this lattice plane spacing can be calculated using Bragg's equation as was explained above. If there is a shift in the diffraction peak (from an expected position), this indicates the presence of a homogeneous strain in the crystal lattice, meaning that individual polycrystals are equally strained – as opposed to having strain in different directions (inhomogeneous strain). Peak broadening may be due to two effects which are hard to separate: inhomogeneous strain, or finite crystal size.

In order to calculate the magnitude of strain in a specified direction, Bragg's equation may be used again. For example, based on Figure 2.5.3.3.1, in the (002) direction, if $2\theta_1$ is the strained position angle and $2\theta_2$ is the unstrained position angle, then the strain can be calculated from $\left|\frac{\Delta d}{d}\right| \times 100\%$ where:

$$\frac{\Delta d}{d} = \frac{\frac{\lambda}{2 \sin \theta_2} - \frac{\lambda}{2 \sin \theta_1}}{\frac{\lambda}{2 \sin \theta_2}} = 1 - \frac{\sin \theta_2}{\sin \theta_1} \quad (2.5.3.3.1)$$

Note that a negative number for $\frac{\Delta d}{d}$ is an indication of a lattice plane separation or expansion, whereas a positive number is an indication of a lattice plane compression [21]. If only grain size broadening is considered, crystallite size can be estimated by using Scherrer formula [25]. This is based on the FWHM, $B = \Delta(2\theta)$, of the diffraction peak centered at θ_B as following:

$$t = \frac{0.9\lambda}{B \cos \theta_B} \quad (2.5.3.3.2)$$

Where t is the crystallite size. Note that ideally B must be corrected for instrumental broadening by using the following equation, though for most applications, one can set $B_{\text{instrument}} = 0$:

$$B^2 = (B_{\text{measurement}})^2 - (B_{\text{instrument}})^2 \quad (2.5.3.3.3)$$

As was shown above, various physical characteristics can be deduced from x-ray diffraction techniques. These are indicative of the thin film crystal orientation, which

imply the quality of the material and the success of the deposition process. The measurement of these characteristics will be performed in detail in the experimental section of this thesis.

2.5.4 Thin-Film Piezoelectric Coefficient Measurement

Once manufactured, thin films are clamped to the substrate, and are not in free form (bulk). Due to this, the piezoelectric properties cannot be directly determined. But with the help of a cantilever, piezoelectric properties of thin films can be examined [26], [27], [28]. Figure 2.5.4.1 below shows the schematic of a Cantilever Beam Structure. This structure is used to measure the piezoelectric coefficient e_{31} of piezoelectric thin films.

Figure 2.5.4.1: Schematic of a Cantilever Beam Structure.

Figure 2.5.4.2: Cantilever Oscilloscope Output.

The difference in voltage between the two electrodes which, with the piezoelectric thin film between them, make a capacitor, is registered on an oscilloscope as shown in Figure 2.5.4.2 above. In order to do this, the beam is deflected perpendicular to the film plane and then is suddenly released which in turn exhibits a decaying oscillation. The voltage across the capacitor can then be measured by the following equation:

$$V(t_1) = e_{31} \left(\frac{A}{C} \right) \left(\frac{3ha}{2l^3} \left(\frac{x_1 + x_2}{2} \right) \right) \left(\frac{\omega_d^2 \tau_E^2}{1 + \omega_d^2 \tau_E^2} \right)^{1/2} \left(e^{\frac{-\pi}{\omega_d \tau_M}} + e^{\frac{-\pi}{\omega_d \tau_E}} \right) \quad (2.5.4.1)$$

where A is the capacitor area, C is the capacitance, h is the film thickness, and x_1 , x_2 , l , and a are as shown in Figure 2.5.4.1. Note that the expression $\left(\frac{3ha}{2l^3} \left(\frac{x_1 + x_2}{2} \right) \right)$ is the relative elongation (strain) of the beam right under the top electrode, ω_d is the damped natural frequency of the beam, τ_E is the electrical time constant, and τ_M is the mechanical time constant.

As it can be observed from the Equation (2.5.4.1), the piezoelectric coefficient e_{31} of piezoelectric thin films can be extracted by a simple rearrangement of the equation. Piezoelectric coefficients of piezoelectric materials are related closely to their electromechanical coupling, as was discussed in more detail in Section 2.2.1.

For a good conversion of electrical signals into acoustic signals and back, the piezoelectric layer needs to have a high electromechanical coupling. The optimum value of the piezoelectric coefficient for bulk AlN is 1 C/m², but usually piezoelectric materials

with coefficients in the range of 0.6 to 0.8 C/m² are considered good materials. An AlN piezoelectric layer with piezoelectric coefficient in this range is expected to produce good acoustic device performance.

PREVIOUS EXPERIMENTS AND RESULTS

3.1 Introduction

Ideal piezoelectric materials are single crystals and include materials such as Lithium Niobate (LiNbO_3) and Quartz. These materials are expensive thus increasing the cost of the final product. Polycrystalline materials with a strong preferred crystal orientation can also exhibit good piezoelectric properties. Materials such as Zinc Oxide (ZnO), Lead Zirconate Titanate (PZT) and Aluminum Nitride (AlN) can be effectively deposited as highly oriented thin films by reactive sputtering at low temperature. These materials can also be grown epitaxially by chemical vapor deposition (CVD) or molecular beam epitaxy (MBE), but deposition temperatures of these techniques are usually high and therefore not compatible with VLSI metalization [29].

This chapter will summarize the findings of previous experiments in the area of thin film deposition and characterization. These will be discussed in three subsections: materials, deposition methods, and film characterization. The purpose of this will be to

present up to date research and conclusions in this field, and provide a background for comparison into similar techniques and results as presented in this thesis.

3.2 Materials

Piezoelectric materials such as ZnO, AlN, and PZT allow both acousto-electric SAW and semiconductor electronic device components to be fabricated on the same substrate. Bulk piezoelectric crystals such as LiNbO₃ and Quartz are also used in current SAW devices [29], but for the design and fabrication of monolithic acoustic sensors compatible with standard VLSI technology, a thin film is required [30]. The characteristics of these piezoelectric thin films - which are based on the different methods and conditions of growth - form the device properties. In comparing piezoelectric films for MEMS applications, some design tradeoffs considered are: the adhesion to the substrate, the resistance to temperature and humidity, and the compatibility with VLSI processes [31].

ZnO has a high piezoelectric coupling as compared to nonceramic materials, and also its pyroelectric property has been important in the research of acoustic sensors. This material was the first to be used for commercial applications (in TV SAW filters) [30] and is commonly used as the actuator element in high frequency scanning acoustic microscopes [31]. DC and RF magnetron sputtering techniques have been used to deposit ZnO films, as well as laser assisted evaporation using a CO₂ laser and a ZnO

powder source in a vacuum chamber [30]. The laser technique was found advantageous for this material since the process is clean and easily controllable. As well, this technique allows for compounds or mixtures to be involved, resulting in doped films with better qualities. An example is Lithium-doped ZnO film which has been deposited at low temperatures (150 °C) and results in excellent orientation and a smooth surface [30].

AlN has the desired properties of high acoustic velocity and high endurance to humidity and temperature, as well as a relatively large piezoelectric coupling factor (lower than ZnO but higher than most other non-ceramic materials) [30]. Its high acoustic velocity enables applications at high frequencies in the GHz range which is useful in size limited VLSI devices. It is also an excellent material for small bandwidth filters ($BW < 5\%$) [32]. As well its resistance to temperature and humidity make it compatible with silicon and GaAs processes and nonhermetic packaging. AlN is also desirable since it does not introduce new cations into the clean room, and thus has low likelihood of causing unknown contamination problems [31]. In comparison to ZnO, AlN has higher SAW velocity, lower propagation loss, and higher thermal stability [34]. These properties made AlN films sought after for high frequency bulk acoustic resonators and SAW sensors. Among all piezoelectric materials, Aluminum Nitride (AlN) is the most attractive choice since it is preferred by semiconductor industry for integration due to its compatibility with active devices on silicon (Si) and Gallium Arsenide (GaAs).

PZT thin film has $\text{Pb}(\text{Zr,Ti})\text{O}_3$ ferroelectric composition, and the thin film form of this solid solution system exhibits high piezo coupling (known to be as high as an order of magnitude larger than either of ZnO and AlN), and a very high dielectric constant [30]. As well, its large pyroelectric response makes it suitable for nonvolatile memory devices and IR detectors, as well as filters with bandwidth $> 5\%$. [32] Fabrication of PZT thin film has been investigated for over a decade [30]. RF sputtering conditions of this film have to be very tightly controlled, which makes sputtering of PZT unsuitable for industrial processes even though it results in a high quality material. Another method that has been used is the chemical sol-gel method which allows for easier control and film homogeneity, as well as being a low cost solution. The downside of this method is that it is difficult to process PZT films thicker than $1\mu\text{m}$ and can result in microscopic cracks which lead to low piezoelectric coupling, thus poor device performance. The fabrication of PZT thin film is still being investigated so they can be improved and used for low-cost production of acoustic sensors.

Piezoelectric crystals (LiNbO_3 and quartz) have excellent SAW characteristics of insertion loss, electrochemical coupling, and temperature sensitivity. They also have moderate phase velocity [29]. Due to this and also the fact that they have poor insulation properties, they are not useful in GHz range devices, and also not good for integration with semiconductor based components. Note that a bi-layer structure of AlN film and LiNbO_3 crystals would be desirable due to their combined properties (high coupling coefficient and velocity), but little is known about the growth of AlN on LiNbO_3

substrates [33], especially since LiNbO_3 crystals are very high priced. Recent studies in this combination will be further explored in Section 3.3.

Overall, the choice between thin films or crystals depends on the required temperature stability, the magnitude of the piezoelectric coefficient, and how easily the film is integrated in the MEMS fabrication process. For this thesis, AlN was investigated as it presents the most desirable characteristics for high frequency VLSI compatible devices.

3.3 Deposition Methods

This section will explain recent deposition methods, best deposition conditions as found by various researchers, as well as different substrates which have been used for thin film deposition. Note that these will be focused on AlN thin film technology.

RF magnetron sputtering has been found advantageous over the CVD method due to low temperature operation and smooth surface morphology growth, and ease of fabrication [33]. RF magnetron sputtering has been used for AlN thin film deposition on Si in previous experiments and best deposition conditions were found to be as follows: Nitrogen partial pressure of 0.5 resulted in excellent (002) preferred orientation. It was found that as the operating pressure increases, this causes the particles to scatter more thus decreasing the preferred orientation of the film. The (002) peak was strongest for an

optimum temperature of 400 °C, and optimum power in the range of 150W to 175W.

Optimum operating pressure was found at 2 mT [34]. It was noted that for film deposited at temperatures lower than 300 °C, there was no crystal orientation in the resulting material. Lower N₂ concentrations (around 30%), and high sputtering pressure (higher than 7.0 mT) were also found to be unfavorable as it resulted in none, or very low XRD (002) peaks [33]. Other experiments based on DC magnetron sputtering indicated optimum deposition conditions at 75% N₂ concentration, 400 °C, and 4.5 mT [35]. Table 3.3.1 below summarizes optimum conditions of some current AlN sputtering experiments.

Table 3.3.1: A few representative results of optimum AlN deposition conditions for different sputtering methods as presented in current research.

Sputtering Method	N₂ Concent.	Press. (mT)	Temp. (°C)	RF Power (W)	Characteristics
RF Magnetron [34]	50%	2	400	175	Excellent (002) orientation
RF Magnetron [33]	60%	3.5	400	165	Grain size of approximately 100nm
RF Magnetron [36]	100%	3	300	500	0.33 µm/hr growth rate
Helicon RF Magnetron [29]	75%	0.75	300	150	Very good crystalline structure and very smooth topology
DC Magnetron [35]	75%	4.5	400	100 DC	(002) orientation with low surface roughness and high electrical resistivity

As mentioned above, the CVD method has not been preferable for AlN (or similar compounds) growth. CVD has been used in the past for III-V compounds since it is an easily controllable method, thus useful for mass production [37]. This method is not ideal though since it relies on very high substrate temperatures (larger than 1000 °C) which would result in a high grain growth for AlN or similar materials. This would not

produce a smooth surface morphology, which in turn would result in high propagation loss if the material were to be used at higher frequencies [37].

Another researched deposition method for AlN has been helicon sputtering, which is based on a magnetron sputtering system which is enhanced by using inductively coupled RF plasma [29]. This allowed for very efficient ionization of sputtered atoms, as well as enabling sputtering at a gas atmosphere of 10^{-4} Torr which is two orders of magnitude lower than conventional sputtering methods. This method resulted in a large peak intensity at a temperature of 400 °C, and exhibited a smooth morphology, superior to that of the films produced by conventional magnetron sputtering [29]. The sputtering gas was at 0.75 mT and consisted of 75% Nitrogen in Argon. RF power was 150 W and the substrate temperature was varied from room temperature to 670 °C. Optimum growth occurred at 300 °C and the AlN films were highly textured with (002) plane parallel to the substrate, which is a good crystalline structure [29]. See Table 3.3 above for summary of optimum deposition conditions.

Sapphire was found to be a better substrate than Si in some cases since its lattice matches better with AlN, thus resulting in easier deposition of high quality thin film [34]. The SAW velocity was found to increase from 5040 m/s for Si, to 5960 m/s for sapphire [34]. Highly (002) oriented AlN was also deposited on glass substrates at room temperature [38]. This showed that it is possible to deposit high quality thin film without substrate heating. In other experiments, the effect of substrate type on the preferential orientation of AlN was investigated by comparing silicon, sapphire, and diamond. This

resulted in comparable silicon and sapphire substrates, but the diamond substrate resulted in a 25% thinner film for the same conditions [35]. As well, the diamond substrate showed some of (100) orientation due to the diamond roughness – thus a very low diamond surface roughness is needed in order to produce a film that can be used in working SAW devices. These are further investigated since diamond substrates can result in very high SAW phase velocity [35].

Glass substrates have also been investigated, with AlN films being deposited via a reactive gas-timing technique, which is a type of RF sputtering involving energetic nitrogen ions [39]. This technique resulted in cubic structure and nanocrystalline AlN sizes. The XRD spectra of this AlN film showed a high quality polycrystalline structure oriented along (111) and (200) as cubic structure [39]. As previously found, the flow rate of the N₂ determined the grain size, increasing the lattice constant (due to compressive stress in the thin film). This phenomenon resulted due to the improvement in the degree of crystallinity of thin films [39].

In this thesis, RF reactive magnetron sputtering is used for film deposition, a technique which has been previously investigated. The differentiating feature of this research is the use of low temperature (unheated substrates) as opposed to the high temperatures of previous experiments (300-400 °C as shown in Table 3.3). The desire for this investigation stems from the fact that VLSI fabrication does not permit high temperatures.

3.4 Characterization

Piezoelectric properties of materials are influenced by composition, grain size, crystallographic orientation, and mechanical conditions [31]. These characteristics represent the quality of the thin film material and determine the success of the deposition method used. Generally the piezoelectric properties of thin films are strongly influenced by mechanical clamping onto the substrate, electrodes, and also surrounding unpoled film.

A piezoelectric film's properties are influenced by an applied electric field (poling). In the characterization of thin films, the most widely adopted geometry uses top and bottom electrodes to drive the piezoelectric films. In order to model the device response (when poled by these electrodes), the piezoelectric coefficient is needed. The piezoelectric coefficient can be expressed in different forms, one being the effective e_{31} coefficient. This is linked to the mechanical material properties by the following equation:

$$P_3 = e_{31} \times (x_1 + x_2)$$

where P_3 is the polarization developed due to the electrodes, and x_1 and x_2 are the strains for the geometry.

Further describing e_{31} , it can be said that e_{31} is the convolution of the d_{31} coefficient with the elastic properties of the film, and this definition is useful because the elastic properties of films are rarely known (and can thus be deduced). In other cases,

depending on the device of interest, the figure of merit for the thin film changes. For example, sometimes the critical coefficient for analysis is the d_{33} coefficient which relates the applied electric field to a polarization in the same direction, a method usually used in high frequency ultrasound transducers. Other devices which employ interdigitated top electrodes to enable poling in the plane of the film (such as diaphragm actuators) utilize a combination of the d_{33} and d_{31} coefficients as a measure of material quality [31].

Piezoelectric characteristics can be found using direct or indirect effects. Some methods for determining the piezoelectric coefficient are based on the use of the direct piezoelectric effect (such as the normal loading method), or the use of the converse piezoelectric effect (like the interferometer). The former is based on measurements prior to the electrical field poling process, and the later is based on reorientation of the deposited film after applying voltage for poling [42]. Indirect effects are based on measuring the piezoelectric coefficient without using voltage which has the disadvantage of poling the material. These methods rely on mechanical forces (such as using a cantilever which is based on the beam bending technique) rather than electrical forces to induce elongation (strain) in the film [26], [27], [28].

A more commonly used method for determining coefficients is the AFM method, which is based on the use of Atomic Force Microscopy. This method measures film surface roughness in nanometer scale, and also small displacements, which can be used to determine d_{33} . Usually a reference material of a known d_{33} is used in the PFM (piezoresponse force microscope) [38]. In some cases, piezoelectric e_{31} coefficients for

AlN have been measured to be up to 0.6 [32], but the optimum value of 1.0 C/m^2 (or -1.0 C/m^2 for e_{13}) has also been recorded [31]. Note that a higher e_{31} coefficient is preferred, as this would indicate the best piezoelectric properties.

To determine preferred orientation and crystal properties, the most commonly used method is X-ray diffraction (XRD) based on 2θ angle patterns (as was described in Chapter 2). Some published examples of film thickness were measured using a Veeco DekTak-3 stylus profilometer, which was also used to measure the radius of curvature of the substrate before and after deposition of the film [41]. From this (radius) the residual stress could be determined (based on Stoney's equation). This method of determining stress was found to give results in close agreement with XRD results [41]. Note that it was found that with increasing thickness (when thickness was measured to be between 2.8 to $5.6 \mu\text{m}$ for an AlN thin film), there was a loss of the (002) preferred orientation [36]. With thicker films the crystallites in the columns are tilted and other additional orientations (besides (002)) are observed to appear [32].

In this thesis, the indirect beam bending method is used to determine the piezoelectric coefficient e_{31} . This technique has been previously investigated in the literature but, as will be explained in Chapter 4, the cantilever beam bending device was designed and built as part of the thesis research.

4.1 *Parameters Investigated*

The purpose of these experiments was to determine the best deposition conditions using the RF magnetron sputtering system in order to obtain a good quality AlN thin film. Deposition conditions which were varied throughout the experiment included pressure, gas flow, gas mixture, and RF power. Temperature and time were kept constant.

After deposition, a material must be tested to ensure the success of the deposition conditions used. The parameters representing the quality of the material are film thickness, crystal orientation, film strain, and piezoelectric coefficient. The next section explains the steps involved in measuring these parameters and Chapter 5 analyzes the results in order to determine the optimum AlN deposition conditions.

4.2 Experimental Method

The following describes the apparatus and method used along with the deposition conditions.

Films were deposited in a MRC 8620 sputtering system using a 75 mm diameter US Inc. source and a RFPP RF5S supply connected through an AM-5 automatic matching network. Samples for deposition were of 50 mm diameter test grade (100) *p*-type silicon wafers with a resistivity of 1–10 Ω cm. Wafers were coated with 1000 Å of Si_3N_4 (for good adhesion) and 5000 Å of Al prior to AlN deposition. The source was a 99.999% Al target. Sputtering gases were 99.999% Ar and 99.999% N_2 , with flow controlled by mass flow controllers. Pressure was monitored using a Pirani gauge, and controlled manually by throttling the pump. The wafer chuck was cooled using a chiller with a coolant temperature set to 4 °C. Wafer surface temperature was not measured. The chamber base pressure was 1.8×10^{-6} torr.

Films were deposited over a range of pressures, gas flows, gas mixtures, and RF powers. Experiments were arranged in a Box-Behnken design to efficiently cover the range of available deposition conditions. Experiments were completed with RF power from 100 to 300 W, pressure from 4 to 12 mT, and N_2 flow from 50% to 100% of total, with the balance being Ar. This range of factors was chosen as a practical operating range for the magnetron source and flow/pressure control system. All films were

deposited for 2 h, giving some variation in film thickness with deposition conditions.

These conditions are summarized in Table 4.2.1 below.

Table 4.2.1: AlN thin-film deposition conditions using MRC 8620 sputtering system.

Wafer #	Source	Gas Flow		Pressure (mT)	Base Pressure (T)	Foreline Pressure (T)	RF Power (W)	Sputtered Time (hr)
		Ar (sccm)	N ₂ (sccm)					
1	Al	0	20	4	1.8×10^{-6}	1.2×10^{-1}	200	2
2	Al	0	20	8	1.6×10^{-6}	1.2×10^{-1}	100	2
3	Al	0	20	8	0.8×10^{-6}	1.2×10^{-1}	300	2
4	Al	0	20	12	1.7×10^{-6}	1.2×10^{-1}	200	2
5	Al	6.6	20	4	0.9×10^{-6}	1.5×10^{-1}	100	2
6	Al	6.6	20	4	1.8×10^{-6}	1.5×10^{-1}	300	2
7	Al	6.6	20	8	1.8×10^{-6}	1.4×10^{-1}	200	2
8	Al	6.6	20	12	1.8×10^{-6}	1.4×10^{-1}	100	2
9	Al	20	20	4	1.8×10^{-6}	1.5×10^{-1}	200	2
10	Al	20	20	8	1.8×10^{-6}	1.6×10^{-1}	100	2
11	Al	20	20	8	1.8×10^{-6}	1.5×10^{-1}	300	2
12	Al	20	20	12	1.5×10^{-6}	1.5×10^{-1}	200	2

To assess the quality of the deposited film, the film thickness, crystal orientation, film strain, and piezoelectric coefficient parameters were then measured as follows.

AlN films thicknesses were measured using a Dektak IIA which is a stylus profilometer as was described in Chapter 2, Section 2.5.1. Films were line patterned using a standard HPR504 process. An 85% Phosphoric acid solution at 60 °C for 4 min was used to etch AlN.

AlN films were analyzed using x-ray diffraction and direct measurement of piezoelectric coefficients. X-ray measurements were made using a Phillips PW3710 Diffractometer using a Cu $K\alpha$ source as was described in Chapter 2, Section 2.5.3.2. Scans were limited in range to avoid the large Si peak from the substrate. Scans were completed from 2θ of 30° to 55° which includes AlN (100), (002), (101), and (102) peaks as well as Al (111) and (200) peaks [47].

The piezoelectric coefficient e_{31} was measured by a Bending Beam technique. The apparatus used for this technique was constructed as was described in Chapter 2, Section 2.5.4. This technique has been described in the literature; however, some minor notation errors and details of the measurement are clarified here. A capacitor was fabricated on a strip of Si by depositing and patterning a square of known dimensions (area A) of Al as the top electrode on AlN films as the dielectric and the underneath Al as the bottom electrode. Capacitance was measured using an HP 4280A meter and the capacitor was wire bonded with a 3 mil (0.003 inch) gold wire using a Unitek 8-146-06 thermal compression bonder. The beam was clamped at one end and a micrometer head on a spring-loaded stage was used to deflect the beam a known distance.

The results of all the measurements mentioned above will be given in detail in Chapter 5.

5.1 Results

This section will present the results obtained from thickness measurements, peak intensity and strain measurements that were obtained from x-ray diffraction measurements, and piezoelectric coefficient (e_{31}) measurements. Based on these results, optimum RF reactive magnetron sputtering conditions for AlN thin film will be determined.

5.1.1 Thickness Measurements

Table 5.1.1.1 below shows the thickness measurements of the 12 samples using stylus instrument as was described in Chapter 2, Section 2.5.1. Three thickness measurements were taken from three different points across each wafer and the film thicknesses were averaged among the three points. The standard deviations from the mean were then calculated. The lower the standard deviation is, the more uniform the thickness of the film is along the wafer. For most samples, the standard deviation was less than 6% of the mean. The highest standard deviation (24.5%) occurred on the

thinnest material (sample 8), which is as expected since uncertainty in the measurement forms an increasing part of the standard deviation as thickness decreases.

Table 5.1.1.1: Thickness measurements of AlN thin-films.

Wafer #	Thickness					
	Measurement 1 (Å)	Measurement 2 (Å)	Measurement 3 (Å)	Average (Å)	Standard Deviation	
					(Å)	%
1	2501	2819	2760	2693	169.16	6.3
2	1395	1508	1441	1448	56.82	3.9
3	5412	5485	5420	5439	40.04	0.7
4	1413	1848	1566	1609	220.66	13.7
5	1563	1703	1675	1647	74.08	4.5
6	4800	4909	4895	4868	59.30	1.2
7	2850	2985	2915	2917	67.52	2.3
8	583	896	619	699	171.27	24.5
9	4416	4605	4411	4477	110.59	2.5
10	4800	4909	4755	4821	79.19	1.6
11	4428	4544	4530	4501	63.32	1.4
12	2076	2553	2101	2243	268.47	12.0

It is observed from these measurements that even though the sputtering time was kept constant, the AlN film thickness is varied. This indicates dependence of the film thickness on other deposition factors such as RF power, Ar vs. N₂ gas flow and pressure. All these deposition factors influence the thickness concurrently without any one dominant condition.

5.1.2 X-Ray Diffraction, Peak Intensity Measurements

From the XRD measurements as was described in Chapter 2, Section 2.5.3.3, following plots were obtained for the 12 samples.

Figure 5.1.2.1: XRD Spectra for Wafer #1.

Figure 5.1.2.2: XRD Spectra for Wafer #2.

Figure 5.1.2.3: XRD Spectra for Wafer #3.

Figure 5.1.2.4: XRD Spectra for Wafer #4.

Figure 5.1.2.5: XRD Spectra for Wafer #5.

Figure 5.1.2.6: XRD Spectra for Wafer #6.

Figure 5.1.2.7: XRD Spectra for Wafer #7.

Figure 5.1.2.8: XRD Spectra for Wafer #8.

Figure 5.1.2.9: XRD Spectra for Wafer #9.

Figure 5.1.2.10: XRD Spectra for Wafer #10.

Figure 5.1.2.11: XRD Spectra for Wafer #11.

Figure 5.1.2.12: XRD Spectra for Wafer #12.

Table 5.1.2.1 below shows the peak intensities of Al and AlN thin-films of the 12 samples extracted from the above XRD plots. Most samples showed two peaks. For each sample, the peak at a 2θ -angle of 36 degrees corresponds to AlN (002) orientation and the peak at a 2θ -angle of 38.5 degrees corresponds to Al (111). Samples with only one peak show no deposition of AlN films but only Al film. To confirm that the Al peak came only from the bottom electrode and not from the AlN film, AlN film was deposited on Silicon Dioxide (SiO_2). For this sample there was no Al peak observed. Measured peaks for known samples are very close to tabulated d-spacing indicating diffractometer measurements are very accurate.

Table 5.1.2.1: XRD peak intensity measurements of Al and AlN thin-films.

Wafer #	$2\theta^\circ$	Peak Identified	Peak Intensity (counts/second)
1	38.52	Al (111)	762
2	38.50	Al (111)	767
3	35.74	AlN (002)	253
3	38.50	Al (111)	615
4	32.74	AlN (100)	156
4	38.52	Al (111)	835
5	38.54	Al (111)	824
6	34.80	AlN (002)	303
6	38.50	Al (111)	702
7	36.08	AlN (002)	222
7	38.54	Al (111)	697
8	38.50	Al (111)	692
9	35.62	AlN (002)	149
9	38.50	Al (111)	835
10	38.48	Al (111)	773
11	35.92	AlN (002)	1190
11	38.54	Al (111)	778
12	33.18	AlN (100)	266
12	38.52	Al (111)	835

It is observed from these measurements that only sample 11 gave a considerably high peak at 2θ -angle of 35.92 degrees. This indicates a good crystal orientation of (002) AlN film with the $\bar{c}$ -axis perpendicular to the substrate. It is apparent that using the maximum sputtering power of 300 Watts is the reason for achieving this good quality material. This will be discussed more in Section 5.2.

5.1.3 X-Ray Diffraction, Strain Measurements

Table 5.1.3.1 below shows the calculated results for film strain as was described in Chapter 2, Section 2.5.3.3, extracted from the XRD plots shown in previous section. The angles were extracted from the XRD plots using Matlab.

Table 5.1.3.1: XRD strain measurements of Al and AlN thin-films.

Wafer #	Peak Identified	$2\theta_1^\circ$ (Strained)	$2\theta_2^\circ$ (Unstrained)	Film Strain ($\Delta d/d$)
1	Al (111)	38.52	38.50	0.0005
2	Al (111)	38.50	38.50	0.0000
3	AlN (002)	35.74	36.00	-0.0070
3	Al (111)	38.50	38.50	0.0000
4	AlN (100)	32.74	33.14	-0.0119
4	Al (111)	38.52	38.50	0.0005
5	Al (111)	38.54	38.50	0.0010
6	AlN (002)	34.80	36.00	-0.0334
6	Al (111)	38.50	38.50	0.0000
7	AlN (002)	36.08	36.00	0.0021
7	Al (111)	38.54	38.50	0.0010
8	Al (111)	38.50	38.50	0.0000
9	AlN (002)	35.62	36.00	-0.0103
9	Al (111)	38.50	38.50	0.0000
10	Al (111)	38.48	38.50	-0.0005
11	AlN (002)	35.92	36.00	-0.0022
11	Al (111)	38.54	38.50	0.0010
12	AlN (100)	33.18	33.14	0.0012
12	Al (111)	38.52	38.50	0.0005

Sample Calculation:

Strain in (002) AlN film for sample 11:

Strained Angle $2\theta_1 = 35.92^\circ \Rightarrow \theta_1 = 17.96^\circ$

Unstrained Angle $2\theta_2 = 36^\circ \Rightarrow \theta_2 = 18^\circ$

From Equation 2.5.3.3.1 in Chapter 2:

$$\frac{\Delta d}{d} = 1 - \frac{\sin \theta_2}{\sin \theta_1} = 1 - \frac{\sin 18}{\sin 17.96} = -0.0022$$

This is an indication of a 0.22% expansion of lattice plane separation in the deposited (002) AlN film of sample 11. Assuming an isotropic Young's modulus of 308 GPa this corresponds to a stress of about 680 MPa, a moderate level for a sputtered thin film.

As it can be observed from the calculated strains, some films produced compressive strains (in the case of a positive $\Delta d/d$) and some films produced tensile strains (in the case of negative $\Delta d/d$). Compressive and tensile strains were explained in Chapter 2, Section 2.5.2. The strain behavior observed in these experiments will be discussed more in Section 5.2.

5.1.4 Piezoelectric Coefficient e_{31} Measurements

Table 5.1.4.1 below shows the calculated results for piezoelectric coefficient e_{31} of the 12 deposited AlN samples using the Cantilever Beam Structure as was described in Chapter 2, Section 2.5.4.

Table 5.1.4.1: Piezoelectric Coefficient e_{31} Measurements of AlN thin-films.

Wafer #	Piezoelectric Coefficient, e_{31} (C/m ²)
1	0.000
2	0.000
3	0.078
4	0.000
5	0.000
6	0.253
7	0.000
8	0.000
9	0.000
10	0.000
11	0.795
12	0.145

Sample calculation:

e_{31} of (002) AlN film for sample 11:

Figure 5.1.4.1: Cantilever Oscilloscope Output for Sample 11

From Equation 2.5.4.1 in Chapter 2:

$$e_{31} = \frac{V(t_1)}{\left(\frac{A}{C}\right) \left(\frac{3ha}{2l^3} \left(\frac{x_1 + x_2}{2}\right)\right) \left(\frac{\omega_d^2 \tau_E^2}{1 + \omega_d^2 \tau_E^2}\right)^{1/2} \left(e^{\frac{-\pi}{\omega_d \tau_M}} + e^{\frac{-\pi}{\omega_d \tau_E}}\right)}$$

$V(t_1) = 480\text{mV} \pm 1\text{mV}$ (digital oscilloscope marker)

$A = 1.200\text{mm} \times 1.200\text{mm} = 1.440\text{mm}^2 \pm 0.007 \text{ mm}^2$ (fabricated design dimensions:
mask uncertainty $2.5\mu\text{m}$, pattern transfer $0.3\mu\text{m}$)

$C = 1.650\text{nF} \pm 0.005\text{nF}$ (CV meter)

$h = 0.2870\text{mm} \pm 0.0005\text{mm}$ (micrometer)

$a = 1.3730\text{mm} \pm 0.0005\text{mm}$ (micrometer)

$l = 38.0\text{mm} \pm 0.1\text{mm}$ (caliper)

$$x_1 = 3.0\text{mm} \pm 0.1\text{mm (caliper)}$$

$$x_2 = x_1 + 1.2\text{mm}$$

$$\Delta t = 4.32\text{ms} \pm 0.01\text{ms (digital oscilloscope marker)}$$

$$\omega_d = \frac{1}{\Delta t} \times 2\pi = \frac{1}{4.32\text{ms}} \times 2\pi = 1454.441\text{rad/s} \pm 3.359\text{rad/s}$$

$$\tau_E = C \times R_{in, Oscilloscope} = 1.65\text{nF} \times 10\text{M}\Omega = 16.5\text{ms} \pm 0.00005\text{ms}$$

$$\tau_M = \frac{t_2 - t_1}{\ln\left(\frac{V(t_1)}{V(t_2)}\right)} = \frac{35\Delta t}{\ln\left(\frac{V(t_2)}{V(t_{36})}\right)} = \frac{35 \times 4.32\text{ms}}{\ln\left(\frac{632\text{mV}}{388\text{mV}}\right)} = 0.3099\text{s} \pm 0.0014\text{s}$$

$$\therefore e_{31} = 0.795\text{ C/m}^2 \pm 0.015\text{ C/m}^2$$

From Equation 2.5.4.1, $V(t_1)$, l , x_1 , and x_2 introduce the highest source of uncertainty. For e_{31} , the calculated worse case uncertainty was found to be 1.9%. This is acceptable considering the nature of the experiment and the measurement techniques.

A considerably high e_{31} of 0.795 C/m^2 is an indication that the (002) AlN of sample 11 is a good piezoelectric material. This was expected since the XRD peak intensity measurements in Section 5.1.2 above, showed that the deposited AlN on this sample is highly oriented in the preferred (002) direction. Also strain measurements in Section 5.1.3 above showed an insignificant tensile stress in AlN film of this sample. Oscilloscope measurements showed no voltage output waveform for the samples that showed either none or very insignificant XRD peaks for AlN deposition - hence zero

piezoelectric coefficient. The dependency of piezoelectric coefficient on crystal orientation and film stress will be discussed more in Section 5.2.

5.2 Discussion

A good quality AlN film is considered to have strong (002) orientation, high deposition rate, large piezoelectric coefficient, and low stress. These properties were found to improve with increasing deposition power, 8 mT pressure and a 50% N₂ gas mixture. Deposition rate of reactively sputtered materials is typically quite low, and use of thin film resonators at low frequencies has been inhibited by the fact that films several micrometers thick are required. In this work thin films (from 0.1 to 0.5 μm thick) were used for most of the characterization measurements. Results of these experiments will be relevant for thicker films, however film quality may change for very long deposition times.

In this work the piezoelectric constant e_{31} was measured, which is relevant for lateral field resonators and some sensor applications. For a more conventional transverse field resonator, e_{33} is required. However, measuring e_{33} by applying a uniform surface load is difficult, and applying an electric field and measuring the displacement using an interferometer poles the film and requires an accurate interferometer [48]. In comparison, e_{31} measurement is relatively straightforward. A film with a large value of e_{31} does not necessarily have a large value of e_{33} , however a strong (002) orientation

(from XRD) and a large value of e_{31} is a strong indication of properties similar to bulk material, and therefore a large value of e_{33} . It was determined that to obtain large piezoelectric coefficients, films with only the (002) orientation are preferred.

The optimum piezoelectric coefficient was obtained for the highest peak intensity. This is shown in Figure 5.2.1 below. Therefore, since a high coefficient is preferred, the deposition conditions will be compared against their resulting peak intensities in Figures 5.2.2 and Figure 5.2.3. This will indicate the best deposition conditions for a high peak intensity, which in turn demonstrates a high piezoelectric coefficient.

Figure 5.2.1: Piezoelectric Coefficient vs XRD Peak Intensity Trend.

Increasing power increases deposition rate, decreases adsorption of contaminants, and increases energy flux to the film surface, thus increasing surface temperature. Low deposition power of 100 W was found to result in unacceptably low deposition rate and poor orientation. Characterization of the deposited AlN films by x-ray diffraction (XRD) shows a dramatic improvement in the resolution of the AlN (002) peak with increasing power. The Al peak is similar in all the samples measured, and is attributed to the Al base electrode. Figure 5.2.2 below shows the trend of increasing peak intensity with power.

Figure 5.2.2: XRD Peak Intensity vs Power Trend. Lines are included to indicate data trends only.

When varying pressure, 8 mT was found to be the pressure that resulted in the highest peak intensity (refer to Figure 5.2.3 below). This figure also shows the trend of decreasing peak intensity as pressure increased above 8 mT.

Figure 5.2.3: XRD Peak Intensity vs Pressure. Lines are included to indicate data trends only.

Deposition at higher pressures is preferred for resonator fabrication due to the reduction in compressive stress when compared with low pressure deposition. Low film stress is critical to fabricating suspended resonator structures with high yield. Sputtered thin films have been shown to exhibit a transition from compressive to tensile stress at a pressure that depends on the system geometry, target material, and sputter gas. Low stress films can be deposited at the transition pressure, or with low tensile stress at high

sputtering pressures. Figure 5.2.4 shows the measured strain (which is also an indicator of stress since stress and strain are directly proportional as per Equation 2.5.2.2) versus the pressure. The data collected in this study do not appear to show a typical evolution of stress with increasing pressure. However, since power and gas mixture also vary between measured points the anticipated response may be hidden. Reasonably low film strain was measured for samples deposited at 8 mT and 12 mT, however only the films deposited at 8 mT combined both low strain and large piezoelectric coefficient. The films deposited at 12 mT were (100) AlN, not the (002) AlN that is of interest for resonator applications.

Figure 5.2.4: Film Strain vs Pressure.

Intermediate gas mixtures of 50%–75% N_2 were found to provide the best film properties. This can be seen from Figure 5.2.2 above where 100% N_2 resulted in the lowest overall peak intensity, and 50% N_2 the highest peak intensity. Figure 5.2.4 also shows the same trend. From the two above mentioned figures it can be seen that even though power and pressure act to increase and decrease the peak intensity as they increase respectively, the Nitrogen concentration is the determining factor for best deposition. While a high Nitrogen content in the sputtering gas mixture will provide ample Nitrogen to form stoichiometric AlN, the sputtering target will tend to become coated with an insulating reacted layer and deposition rate will drop. Adding Argon to the gas mixture will provide heavy ions that bombard both the target and the wafer, increasing deposition rate and energy transferred to the growing film. However, if Nitrogen content in the sputtering gas drops too low, sub-stoichiometric AlN will result. Generally it is desirable to operate at the lowest Nitrogen content that will still produce stoichiometric films. These results suggest that slight variations in power and pressure are not as critical as ensuring that the Nitrogen concentration is at 50%.

Based on the results and characteristics mentioned above, it was determined that the best sputtering conditions were: maximum RF power of 300 W; mid-pressure of 8 mT; and 50% N_2 flow of total, with the balance being Ar which resulted in the highest quality thin film (of the ones in this experiment). The characteristics of this film were: film thickness of 0.5 μm ; crystal orientation of (002) AlN film with the $\bar{c}$ - axis

perpendicular to the substrate; tensile film strain of 0.22% expansion of lattice plane separation; and piezoelectric coefficient e_{31} of 0.795 C/m².

**CONCLUSIONS AND
RECOMMENDATIONS****6.1 Conclusion**

In acoustic devices, both BAW's and SAW's, a piezoelectric layer is required to convert electrical signals into acoustic signals and vice versa. For a good conversion of electrical signals into acoustic signals and back, the piezoelectric layer needs to have a high electromechanical coupling. Therefore, a piezoelectric material with high piezoelectric coefficient is required for a good acoustic device performance. Among all piezoelectric materials, aluminum nitride (AlN) is the most attractive choice since it is preferred by semiconductor industry for integration due to its compatibility with active devices on silicon (Si) and gallium arsenide (GaAs). AlN is also chemically stable, mechanically strong and has a high acoustic velocity.

For sputtering purposes, to deposit AlN, radio frequency (RF) reactive magnetron sputtering is preferred over direct current (DC) sputtering. Even though DC sputtering has higher rate of deposition, in reactively sputtered films insulating materials can form on the target and cause arcing, which can result in wafer defects. RF reactive

magnetron sputtering is slower than DC sputtering but it avoids the risk of target arcing and produces high quality thin films.

In this thesis it was determined that the best sputtering conditions were: maximum RF power of 300 W; mid-pressure of 8 mT; and 50% N₂ flow of total, with the balance being Ar which resulted in the highest quality thin film (of the ones in this experiment). The characteristics of this film were: film thickness of 0.5 μm ; crystal orientation of (002) AlN film with the $\bar{c}$ - axis perpendicular to the substrate; tensile film strain of 0.22% expansion of lattice plane separation; and piezoelectric coefficient e_{31} of 0.795 C/m². These indicate a high quality thin film, which would be good for use in semiconductor high frequency devices.

6.2 Recommendations

It is recommended that future work be performed based on the optimum sputtering conditions determined above. As was seen in the results, only these optimum conditions resulted in preferred (002) AlN thin film. Further experiments should be carried out to confirm and isolate the best sputtering conditions more accurately. As well, the correlation between piezoelectric properties and Oxygen content should be investigated. This is of interest in order to determine if small Oxygen concentrations in the film have a strong effect on the piezoelectric coefficient [49].

Once a high quality Aluminum Nitride (AlN) thin film is obtained in usable quantities by a low temperature deposition process, this could be implemented and tested in high frequency (up to 2.4 GHz) TFRs (thin film resonators). These TFRs are sometimes called FBARs (film bulk acoustic resonators), and they operate with longitudinal or shear waves as was described in Chapter 2. These filters can be integrated with other transceiver functions on the same chip. This area of research is important since integrating filters with other transceiver functions (on-chip piezoelectric resonators) has the potential to reduce cost, increase performance, and reduce the size of systems.

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