UNIVERSITY OF CALIFORNIA  
Santa Barbara  

Aluminum Nitride Thin Films on Titanium:  
Piezoelectric Transduction on a Metal Substrate  

A Dissertation submitted in partial satisfaction of the requirements for the degree Doctor of Philosophy in Materials  

by  
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September 2006
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August 2006
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By

Seth E. Boeshore
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This dissertation is dedicated to Jamie. Although you were two thousand eight hundred and seventy–seven miles away, you were always in my heart.
VITA OF SETH E. BOESHORE

EDUCATION

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ABSTRACT

With many microsystems recently fabricated on titanium substrates—mechanical arrays, sensing elements, and fluidic devices—titanium has emerged as a platform for MEMS fabrication. The success of these devices relies upon the advantageous material properties of titanium: high fracture toughness, oxide biocompatibility, three–dimensional stacking ability, and the integration of micro– and macromachining techniques. However, due to the relative immaturity of this technology, the number of established processes are still limited; thus far, titanium MEMS rely on electrostatic actuation and incorporate few, if any, functional materials.

This dissertation introduces aluminum nitride thin film deposition and piezoelectric transduction to the titanium MEMS design toolkit. A process has been developed for depositing AlN films onto polished titanium wafers by middle–frequency sputter deposition. By utilizing the hysteretic behavior of reactive sputtering, thin films with strong c–axis orientation have been deposited; XRD rocking curves for these films have FHWM as low as 1.8°. Further characterization has shown that these films have piezoelectric, optical, and electrical properties that approach single crystal AlN.
Piezoelectric unimorph cantilever beams have been fabricated from the deposited films and used for the first demonstration of piezoelectric transduction on titanium. At large displacements, these beams exhibit nonlinear spring softening. An analytical model has been developed to accurately describe the frequency response of these beams and to calculate the material properties of the film. Test results from these beams show that $d_{31}$ and $k_{31}^2$ for the films are within 10% of the reported values.

Surface acoustic wave (SAW) filters have also been fabricated from AlN films on titanium. Although the conductivity of titanium results in severe electromagnetic feedthrough between the input and output ports, the transmission response of the filters has been measured using a time–gating method. The electromechanical coupling and power attenuation have been extracted from $s_{12}$ measurements; though $k^2$ is high, the attenuation is roughly an order of magnitude higher than SAW filters fabricated from AlN films on silicon.
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INTRODUCTION
This chapter introduces the research presented in this thesis. First, the motivation behind the research is given, including prior work that heavily influenced what later became this thesis. The second section is an outline of the thesis by chapter, including a brief summary of the contents therein.

1.1 Motivation of the Research

In 2004, following the reporting of the first bulk–micromachined titanium MEMS and the subsequent refinement of new microprocessing techniques, titanium emerged as a platform for MEMS fabrication [1], [2]. From microfluidic devices to sensor arrays, various new MEMS were rapidly developed using this technology [3], [4]. These devices were specifically designed to take advantage of the material properties of titanium, including its fracture resistance and oxide biocompatibility. Government interest in the new titanium MEMS technology fueled research into devices that would not
degrade in harsh environmental conditions and low-loss, radio frequency (RF) mechanical switches. Concurrent with the expansion of titanium as a MEMS platform, a strong research effort was being made to deposit aluminum nitride films onto silicon using reactive sputter deposition for the purpose of fabricating piezoelectric RF MEMS [5]. This effort proved successful when beam–supported bulk acoustic wave (BAW) resonators were fabricated from these films [6].

The research described in this thesis brings together the two paths above by developing piezoelectric MEMS on titanium. While titanium has proven to be a powerful platform for microscale device design, its scope remains limited in that most titanium MEMS rely on electrostatic device actuation; piezoelectric active layers would be a powerful addition to the titanium design toolkit. Furthermore, a thin film deposition process and new bulk micromachining techniques must be developed in order to fabricate piezoelectric MEMS on titanium, adding additional processing techniques to those available to titanium MEMS designers. The challenge requires a start–to–finish effort, from the deposition and characterization of AlN films through the bulk micromachining of the devices to finally the device testing
and analysis. To this day there remains little to no reported literature on any of these steps.

1.2 THESIS OUTLINE

Not including this introductory chapter, the thesis has five chapters. Chapter 2 discusses the material properties of titanium and aluminum nitride, comparisons with other materials commonly used in MEMS fabrication are made, and the advantageous material properties of AlN and titanium are used to justify the materials selection. Chapter 3 is a comprehensive description of the reactive sputter deposition of AlN onto titanium substrates. An optimized process for sputtering highly-crystalline AlN is detailed, and the characterization of these films is presented. Chapters 4 and 5 describe the fabrication of piezoelectric MEMS from the sputtered AlN films and the testing of these devices. In Chapter 4, piezoelectric unimorph cantilevers are fabricated using new bulk micromachining techniques adapted for use on titanium. Through analysis of the motion of the cantilevers and the use of analytical and finite element models, important material properties like the piezoelectric coefficient and the electromechanical
coupling are calculated. Chapter 5 discusses the fabrication and testing of RF surface acoustic wave (SAW) filters using AlN thin films on titanium. The frequency response of the SAW devices is used to calculate material properties and a comparison is made of the titanium filters to SAW filters fabricated on other substrates. The final chapter summarizes the results from the earlier chapters, drawing conclusions from the research and outlining new directions for future research.

REFERENCES


MATERIALS:

ALUMINUM NITRIDE AND TITANIUM
Materials selection plays a crucial role in the operation and capabilities of a fabricated device. Among the material properties that must be considered: the morphology and crystal structure of each material; thermal and electrical compatibility between materials; and ability to withstand processing conditions without unacceptable degradation. Even after a material has been selected, there is the possibility that a new deposition method or material processing step could result in better device performance. This chapter examines the material properties of the two most significant materials of the piezoelectric MEMS presented in this research: aluminum nitride and titanium.

2.1 ALUMINUM NITRIDE

AlN has physical properties desirable to both MEMS designers and the larger research community. A III–V semiconductor, many of its material properties are a result of its close-packed crystal structure. Under ambient conditions, the thermodynamically–stable structure of AlN is hexagonal
wurtzite—shown in Figure 2.1 below—although at very high pressure it can take the cubic rocksalt form [1]. The \(c/a\) ratio is 1.60 and its close-packed covalent bonds result in its very high hardness and excellent thermal conductivity, properties that it shares with the other III–V nitride semiconductors [2], [3]. AlN ceramics are found in industrial power supplies and inverters to efficiently dissipate waste heat from these devices [4]. Because of its hardness and corrosion resistance, AlN is often used as a wear-resistant and anti-corrosive coating, both in industrial and consumer applications [5]. AlN is a very chemically-resistant material; however, it can be etched by certain chemical and reactive ion plasma etches [6]. Polycrystalline AlN tends to be particularly susceptible to chlorine in gas, liquid, and plasma phases. Aluminum forms grain boundaries between AlN crystallites, and these aggregates are readily attacked by chlorine.
Electrically, AlN is a wide band gap semiconductor with a conduction band edge 6.2 eV above the valence band. This property has led to the use of AlN in a number of photonic applications [8]. Magnesium–doped AlN grown by low–pressure metal organic vapor phase epitaxy has recently been used as the active layer in a deep ultraviolet light–emitting diode with an emission wavelength of 210 nm [9]. Because there are no dopants that increase the band gap in AlN, at present there have been no reported multiple quantum well structures with AlN as the active region. However, AlN is used as a
buffer layer in the growth of multiple quantum well structures and in a highly–efficient superlattice cladding layers [10], [11].

In addition—and for the purpose of this research, more importantly—AlN is a piezoelectric material. More correctly, it is ferroelectric: it exhibits a spontaneous polarization under zero electric field. For a semiconductor, AlN has relatively high piezoelectric coefficients $d_{33}$ and $d_{31}$, permitting multiple modes of static and dynamic motion. AlN thin films are used in a wide variety of piezoelectric applications, from in vitro biosensing and wide–band pressure sensing to the actuating layer in precision components, such as complex microactuator arrays capable of simulating locomotion [12], [13], [14]. As a result of its piezoelectricity, acoustic waves can be excited in AlN using electric fields, making it a common choice for RF signal processing and filtering; it is often the active material in surface acoustic wave (SAW) and bulk acoustic wave (BAW) devices [15], [16].

**PIEZOELECTRICITY**

The piezoelectric effect is defined as the induction of a voltage within a material in the presence of an applied stress. The converse piezoelectric effect is the reverse of this process, where an applied voltage induces a stress in the
material. A crystalline material is comprised of atoms that share electron density in some manner, e.g. ionic and covalent bonds. Because of this sharing, the electron density is not uniform throughout the crystal and there are periodic electric dipoles that reflect the periodic atomic arrangement of the crystal. In most crystals, the constituent atoms of the crystal are distributed such that the sum of the individual dipoles between all of the atoms is zero, although a ferroelectric has a nonzero dipole sum at equilibrium. When an applied stress creates a strain in the crystal, there is a small change in the bond lengths between the atoms, resulting a shift in the positions or directions of the individual dipoles. Many crystals are centrosymmetric and even under the applied stress the sum of the dipoles is zero. In most non–centrosymmetric crystals, however, the stress results in the formation of a nonzero dipole—a manifestation of piezoelectricity. The converse effect is also observable; an applied electric field creates a strain in a piezoelectric material.

The piezoelectric effect can be expressed mathematically using equations that describe the electric and structural behaviors of a material. Electric displacement is defined as

$$D_i = \varepsilon_0 \varepsilon_{ij} E_j$$  \hspace{1cm} (2.1)
where $\varepsilon_g$ is the permittivity matrix of the material and $E_j$ is the applied electric field. Similarly, Hooke’s law for mechanical strain is

$$S_{ij} = s_{ijkl} \sigma_{kl}$$

(2.2)

where $s_{ijkl}$ is the elastic compliance tensor of the material and $\sigma_{kl}$ is the stress.

In a piezoelectric, these equations are combined to yield the coupled constituent equations

$$S_{ij} = s_{ijkl}^E \sigma_{kl} - d_{kij} E_k$$

$$D_i = d_{ijk} \sigma_{jk} + \varepsilon_0 \varepsilon_{ij}^\sigma E_j.$$  

(2.3)

$d_{jk}$ is the strain–charge form of the piezoelectric coefficient tensor; $s_{ijkl}^E$ is the compliance at constant electric field; and $\varepsilon_{ij}^\sigma$ is the permittivity at constant stress [17]. In AlN, crystal symmetry reduces the 18–element matrix $d_{jk}$ to

$$
\begin{pmatrix}
0 & 0 & 0 & 0 & d_{15} & 0 \\
0 & 0 & 0 & d_{15} & 0 & 0 \\
d_{31} & d_{31} & d_{33} & 0 & 0 & 0
\end{pmatrix}.
$$

The coefficients of the matrix control the response of the piezoelectric to an applied stress or voltage. For the purpose of the research, an explanation
of the coefficients \( d_{31} \) and \( d_{33} \) is sufficient. \( d_{31} \) couples the 1 and 2 axes—the basal plane—with the 3, or c, axis, such that a stress in the c direction results in an electric field along the basal plane axes. Conversely, a stress in the basal plane produces a voltage along the c axis. \( d_{33} \) is a direct effect in the c-direction: a stress in c produces a voltage in c. It is these piezoelectric coefficients that govern the operation of the cantilever beams, and SAW devices fabricated and tested in this work.

**ALUMINUM NITRIDE AND PIEZOELECTRIC MEMS**

AlN is only one of many materials used in piezoelectric applications. There are entire classes of materials—such as certain perovskites—with piezoelectric coefficients that exceed AlN, sometimes by orders of magnitude. However, many of these materials are not suitable for piezoelectric MEMS fabrication, because of either incompatibility with the fabrication process or specialized growth conditions. Figure 2.2 is a table of material properties for three piezoelectric materials that are compatible with microprocessing and are commonly used in MEMS.
<table>
<thead>
<tr>
<th>Property</th>
<th>AlN</th>
<th>ZnO</th>
<th>PZT</th>
</tr>
</thead>
<tbody>
<tr>
<td>Density ($kg/m^3$)</td>
<td>3230</td>
<td>5610</td>
<td>7570</td>
</tr>
<tr>
<td>Piezoelectric constant ($C/m^2$)</td>
<td>$e_{31} = -0.58$</td>
<td>$e_{31} = -0.57$</td>
<td>$e_{31} = -6.5$</td>
</tr>
<tr>
<td></td>
<td>$e_{33} = 1.55$</td>
<td>$e_{33} = 1.32$</td>
<td>$e_{33} = 23.3$</td>
</tr>
<tr>
<td>Thermal conductivity ($W/cm·°C$)</td>
<td>2.85</td>
<td>0.6</td>
<td>0.018</td>
</tr>
<tr>
<td>Thermal expansion ($1/°C$)</td>
<td>$\alpha_a = 4.2 \times 10^6$</td>
<td>$\alpha_a = 6.5 \times 10^6$</td>
<td>$\alpha = 2 \times 10^6$</td>
</tr>
<tr>
<td></td>
<td>$\alpha_c = 5.3 \times 10^6$</td>
<td>$\alpha_c = 3.0 \times 10^6$</td>
<td>$\alpha_c = 2 \times 10^6$</td>
</tr>
<tr>
<td>Young’s modulus ($GPa$)</td>
<td>308</td>
<td>201</td>
<td>68</td>
</tr>
<tr>
<td>Acoustic velocity ($m/s$)</td>
<td>10,127</td>
<td>5,700</td>
<td>3,900</td>
</tr>
<tr>
<td>Index of refraction</td>
<td>2.08</td>
<td>2.01</td>
<td>2.4</td>
</tr>
<tr>
<td>Band gap ($eV$)</td>
<td>6.2</td>
<td>3.4</td>
<td>2.67</td>
</tr>
<tr>
<td>Resistivity ($\Omega cm$)</td>
<td>$1 \times 10^{11}$</td>
<td>$1 \times 10^9$</td>
<td>$1 \times 10^9$</td>
</tr>
</tbody>
</table>

**Figure 2.2: Material properties of common MEMS piezoelectrics.** These three materials are used for nearly all thin film and bulk piezoelectric MEMS applications [3, 18-24].

AIN has several advantages over zinc oxide and lead zironate titanate (PZT). First, it is very compatible with standard MEMS processing techniques. It is very selective to many wet chemical and dry plasma etches but can be readily etched in a chlorine environment. Its very high hardness and melting point ensure that films will not degrade during processing. Second, AlN films with excellent crystallinity and orientation can be reproducibly deposited or grown on many different substrates and films, including dielectrics, semiconductors, and metals [25]. Third, AlN exhibits both moderate electromechanical coupling in conjunction with high acoustic
and surface velocities, making it a useful material for BAW and SAW devices [24]. Finally, although it does not have very high piezoelectric coefficients, for many applications such a large response is not required; its strong crystal qualities allow for very sensitive devices with high quality factors. Because of its low dielectric losses and high breakdown field, the figure of merit for AlN transducers can be 24 times higher than comparable PZT transducers [26].

2.2 Titanium

Traditionally, bulk micromachined MEMS have been fabricated from silicon and other single crystal semiconductors like gallium arsenide. This is due to three factors: the vast processing and engineering infrastructure of the integrated circuit and photonics industries; the sheer volume of research devoted to semiconductor and dielectric crystals; and the highly selective processes—doping, masking, and etching—that can be performed on these materials. Although these factors have been the catalysts behind the rapid growth of MEMS research and development, and the close compatibility of MEMS with complimentary metal–oxide semiconductor (CMOS) processing, the result is essentially a sole platform on which MEMS are fabricated, that being single crystal substrates. However, in recent years there has been much
new research on fabricating MEMS on other materials, including glass and metal substrates; one of the most promising metal substrates is titanium.

Early MEMS technology that incorporated titanium included microbolometers and RF relay switches [27], [28]. In both cases, these devices were fabricated by the surface micromachining of titanium thin films deposited in conjunction with other materials onto a single crystal substrate. The first reported titanium–only microdevices were cantilevers and fixed–fixed beams, although once again these devices were fabricated using surface micromachining techniques and a polyimide ashing release [29]. An advancement towards bulk micromachining MEMS on titanium substrates occurred when the first high aspect ratio devices were fabricated on polished bulk titanium substrates [30]. These devices—micromirror actuator arrays—exhibited the high fracture toughness that metal substrates had promised and proved titanium as a viable platform for MEMS design.

**Titanium as a MEMS Material**

It is instructive to begin a discussion of titanium as a platform for MEMS design by comparing it to the archetypal MEMS substrate: silicon. Figure 2.3
and Figure 2.4 compare important material properties of silicon, titanium, and their corresponding oxides and nitrides.

**Figure 2.3: Material properties of silicon and its derivatives.** The robust growth of native oxide on silicon and the strong contrasts in resistivity, dielectric contrast, and thermal conductivity have resulted in the development of a wide variety of MEMS. (Table adapted from [31].)

<table>
<thead>
<tr>
<th>Property</th>
<th>Si</th>
<th>SiO₂</th>
<th>SiN</th>
</tr>
</thead>
<tbody>
<tr>
<td>Density (kg m⁻³)</td>
<td>2330</td>
<td>2200</td>
<td>3440</td>
</tr>
<tr>
<td>Resistivity (Ω m)</td>
<td>3.2 x 10³</td>
<td>1.0 x 10⁶</td>
<td>2.82 x 10⁶</td>
</tr>
<tr>
<td>Thermal conductivity (W cm⁻¹ °C⁻¹)</td>
<td>1.24</td>
<td>0.014</td>
<td>29</td>
</tr>
<tr>
<td>Thermal expansion (°C⁻¹)</td>
<td>2.6 x 10⁻⁶</td>
<td>0.5 x 10⁻⁶</td>
<td>8.0 x 10⁻⁶</td>
</tr>
<tr>
<td>Young’s modulus (GPa)</td>
<td>170</td>
<td>90</td>
<td>300</td>
</tr>
<tr>
<td>Fracture toughness (MPa m⁻¹/²)</td>
<td>0.8</td>
<td>0.95</td>
<td>0.6</td>
</tr>
</tbody>
</table>

**Figure 2.4: Material properties of titanium and its derivatives.** The high fracture toughness and thermal conductivity of titanium allow for the fabrication of MEMS with functionality unobtainable on silicon substrates. (Table adapted from [31].)

<table>
<thead>
<tr>
<th>Property</th>
<th>Ti</th>
<th>TiO₂</th>
<th>TiN</th>
</tr>
</thead>
<tbody>
<tr>
<td>Density (kg m⁻³)</td>
<td>4.51</td>
<td>4.23</td>
<td>5.21</td>
</tr>
<tr>
<td>Resistivity (Ω m)</td>
<td>3.9 x 10⁻⁷</td>
<td>0.1</td>
<td>2.1 x 10⁻⁷</td>
</tr>
<tr>
<td>Thermal conductivity (W cm⁻¹ °C⁻¹)</td>
<td>0.22</td>
<td>0.067</td>
<td>120</td>
</tr>
<tr>
<td>Thermal expansion (°C⁻¹)</td>
<td>8.6 x 10⁻⁶</td>
<td>7.5 x 10⁻⁶</td>
<td>3.3 x 10⁻⁶</td>
</tr>
<tr>
<td>Young’s modulus (GPa)</td>
<td>108</td>
<td>282</td>
<td>600</td>
</tr>
<tr>
<td>Fracture toughness (MPa m⁻¹/²)</td>
<td>50</td>
<td>5</td>
<td>5</td>
</tr>
</tbody>
</table>

The high fracture toughness of titanium—almost two orders of magnitude higher than silicon—is often cited as one of its strongest attributes as a MEMS platform, and with good reason. In combination with its high ductility,
titanium devices can fail through yielding by plastic deformation rather than brittle fracture—often a detrimental yet unavoidable failure route for MEMS on silicon substrates. Another desirable property of titanium is that it forms a thick native oxide in the atmosphere, far thicker than the native SiO$_2$ on silicon. Surface studies of titanium have shown that roughly 30% of the surface titanium is bonded to carbon and nitrogen to form TiC and TiN; the other 70% has a $+4$ electronic valence and is bonded to oxygen as TiO$_2$ [32]. This native layer extends to a depth several nm below the surface to a gradient region that incorporates a mixture of titanium carbide, nitride, and oxide. This gradient region ends 20–30 nm below the surface. Both TiN and TiO$_2$ have high elastic moduli and provide strong interfacial toughness; in addition, the native oxide acts as a protective barrier for the titanium substrate against chemical corrosion and environmental damage [33, 34]. Bulk titanium with an oxide interface is also biologically compatible with the living organisms. Highly researched by the medical community, titanium devices have been implanted for decades at time in the human body with minimal degredation [35]. A final note regarding titanium oxide: the fabrication of MEMS often requires electrical isolation from the substrate, and a titanium surface can be easily oxidized and at a much lower temperature than silicon,
of particularly importance when critical yet thermally unstable components are part of the fabrication process.

The bulk micromachining of MEMS requires the ability to create a structure out of the substrate material, often achieved using an etching process. To that end, a powerful set of plasma etches have been developed, including the metal anisotropic reactive ion etching and oxidation (MARIO) and titanium inductively–coupled plasma (ICP) deep etch (TIDE) processes [30, 36]. Both of these etches use a chlorine–based plasma chemistry to obtain anisotropic etching of titanium microstructures. The MARIO process is a cyclic etch that alternates between oxidation and etching steps, creating the structure in a manner similar to the Bosch process in silicon. The TIDE process—highly selective to the masking oxide layer—requires only a single etching step. However, a limitation of these processes is that the fabrication of large area devices requires thin titanium foils; the etching process is used to etch the pattern completely through the substrate. An effective, highly–undercutting isotropic plasma release etch has not yet been reported. As a substrate for MEMS, titanium presents additional processing challenges; these will be discussed below. Nonetheless, while these problems can
complicate MEMS processing, a number of effective processes and fabricated devices have already been demonstrated on the titanium platform.

TITANIUM SUBSTRATES AND PROCESSING

Until recently, titanium substrates were generally available only as sheets of rolled metal in various material grades; these are listed in Figure 2.5. The most important distinction between grades is the oxygen concentration. As discussed below, oxygen can cause major problems during etching, particularly when it results in an increased amount of oxide at the grain boundaries between crystallites. Titanium substrates can be purchased commercially and come in a variety of thicknesses and polishes, though limitations in the polishing process generally dictate that thicker substrates have better polishes. Using a shearing tool, these sheets can be cut into smaller pieces suitable for microfabrication and processing. Unlike most semiconductor substrates, the material in these sheets is polycrystalline. Recently, titanium wafers have become available from Tokyo Stainless Grinding Co., Ltd. While not wafers in the traditional sense, as these are not diamond saw–cut wafers from a boule of single crystal material, titanium wafers have the same shape as a 4–inch silicon wafer, including the flat, and
have a similar thickness. These wafers are ideal—in some cases, necessary—for the fabrication of piezoelectric MEMS on titanium. Nonetheless, silicon remains a much smoother substrate, with a surface roughness an order of magnitude less than titanium. This difference, shown in Figure 2.6, can be critical during microprocessing, particularly during the deposition of piezoelectric thin films and the fabrication of SAW devices, as will be discussed in Chapters 3 and 5. The wafers are formed from sheets of rolled and polished titanium using a patterned punch. The punching process and the additional handling do not have a strong effect on the surface roughness, and the material quality is as good as any commercially available sheets. Even though the technology is very new and only at a research level, the cost of punched titanium wafers is comparable to silicon wafers.

<table>
<thead>
<tr>
<th>Grade</th>
<th>Fe</th>
<th>C</th>
<th>O</th>
<th>H</th>
<th>N</th>
<th>Pd</th>
</tr>
</thead>
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Figure 2.5: Elemental composition in percent of commercially available titanium alloy grades. Titanium composes the balance of the material in each grade. Grade 1 titanium is the purest form, with the least amount of iron and oxygen impurities. (Data courtesy of ESPI.)
Figure 2.6: Optical profilometer image comparison of silicon and titanium. The left image is the surface of a prime silicon wafer. The RMS surface roughness is less than 3 Å. The right image is the surface a polished titanium wafer with a RMS roughness of 3 nm. Both images use the scale bar for reference.

During MEMS processing, titanium wafers have a number of advantages over sheared pieces. First, many deposition and etching tools used during MEMS fabrication are production scale and have automatic robotics and sensors that require the use of a wafer. In the absence of a wafer–shaped substrate, a dummy carrier wafer must be used on which the substrate is mounted. Because many of these tools have chambers that operate at high temperatures and ion concentrations, the substrate not only needs to be mounted but also it must be in thermal and electrical contact with the carrier. There are various oils, greases, and tapes that are often used to provide contact, but these can outgas, decompose, or otherwise degrade during processing in the chamber. In other tools, process conditions and chamber geometries completely prevent the use of a substrate mounted on a carrier.
wafer. By having titanium in a wafer shape, these problems are completely avoided. Another benefit is that titanium wafers provide the maximum surface area for device layout, allowing a MEMS designer to fit more devices on a wafer than on sheared titanium pieces.

Despite their advantage over sheared pieces, titanium wafers have some key difficulties during processing that are not encountered when using silicon substrates. Because of its ductility, titanium bends under significantly less force than silicon wafers. This is manifested in a photolithographic process that requires a vacuum chuck to fix the substrate during high–speed photoresist spinning. While the chuck can very slightly and reversibly warp a silicon substrate, a titanium wafer will deform to the point that photoresist will not have a radially–uniform thickness following spinning, altering the photoresist curing, UV exposure, and development times. There can also be significant warping after the deposition of thin films if the film has a nonzero stress. The deposition of a highly stressed thin film onto titanium can curve or warp the substrate to such a degree that further processing is impossible. Also, because commercially available titanium substrates are punched, sheared, or otherwise cut from rolled sheets of metal, while the polish on these substrates can be quite good, the ductility of titanium results in burring
at the edges of the substrate that interferes with some chamber robotics. Processing equipment such as vacuum chucks can cause further permanent deformation. The burr on the outer edge can also prevent hard contact between a photomask and the substrate during photoresist exposure. A limitation of titanium regardless of substrate shape is that it is not selective to the chemistries of many common etches. For instance, a standard AlN plasma etch uses a combination of chlorine and argon. When etching through an AlN thin film, this plasma will not etch into silicon and the substrate can serve as an etch stop. Titanium etches readily in this chemistry, considerably complicating device processing.

The material quality of titanium substrates is important in relation to a phenomenon often seen during processing. Called grass, it is well–known in integrated circuit microfabrication and can be caused by the sputtering of exposed materials—most commonly a soft metal like gold; the redeposition of the material being etched; or the formation of halogenated carbon polymer byproducts. These all can have etch rates that differ from the material being anisotropically etched; the contaminants can act as point masks during etching. When viewed at the microscale, the etched surface appears to be coated with grass; macroscopically, the surface appears dull or black because
light gets scattered from the grass. In the worst cases, grass can completely impede the etch chemistry and the etch rate drops to zero. While the three factors listed above can contribute to grass formation during titanium processing, impurities in the metal substrate often result in much more significant grassing. Although the purity of a substrate can be over 99% titanium, there are always oxides that aggregate at grain boundaries and impurities such as iron. These etch at different rates than titanium and can be completely impervious to chlorine etch chemistries.

Substrate grassing can be minimized or even eliminated with proper materials selection and process characterization. The purest material commercially available is a single crystal titanium substrate grown using Czochralski or Bridgman melting techniques. These substrates have the highest purity and fewest grain boundaries but are extremely expensive and very small—unsuitable for either process characterization or large-scale device fabrication. A method more suitable for MEMS device fabrication is the use of a relatively pure substrate, such as a grade 1 titanium wafer, and a well-characterized etching process. The addition of argon to a chlorine plasma can reduce grassing by increasing the amount of physical sputtering at the substrate. Similarly, high bias power and low pressure provide
increased energy to ions arriving at the etched surface, decreasing the selectivity of impurities that cause grass. These techniques all have negative effects like low selectivity to masking layers and increased damage to the other layers, but with proper characterization substrate grassing can be reduced.

**SUMMARY**

Individually, both aluminum nitride and titanium provide substantial functionality for MEMS. The ceramic, piezoelectric, and semiconductor properties of AlN are already being used in a wide variety of devices, and MEMS that incorporate AlN thin films as a sensing or actuating layer have already been developed on other substrates. Titanium has shown considerable promise as a new MEMS platform, particularly for devices where high fracture toughness, biocompatibility, or chemical resistance is required. A fabrication and processing method incorporating both of these materials would result in devices having functionality beyond current MEMS technology.
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DEPOSITION AND CHARACTERIZATION
OF ALUMINUM NITRIDE THIN FILMS
ONTO TITANIUM SUBSTRATES
Piezoelectric thin film deposition plays a crucial role in the fabrication and operation of piezoelectric MEMS. At the expense of much process characterization, these films yield material with properties and functionality invaluable to MEMS designers. For piezoelectric MEMS fabricated from aluminum nitride films, this is most certainly the case. A sputtered AlN film requires highly optimized deposition conditions to increase its effective piezoelectric coefficients and electromechanical coupling while simultaneously controlling the thickness uniformity, residual stress, and other material properties. This chapter describes the process of depositing and characterizing AlN films on titanium substrates.

3.1 METHODS OF THIN FILM DEPOSITION

The formation of films on surfaces encompasses an entire field of science and engineering. For the sake of brevity, the discussion below will focus on the deposition methods used in electronic and photonic circuit fabrication, as these are the technologies that are commonly used for MEMS thin film deposition. These methods can be separated into two distinct processes:
chemical and physical deposition. Chemical depositions occur when reagents or precursors react at the surface of a substrate to form a film. Because the precursors are generally gases or liquids, chemical depositions tend to be more conformal to the substrate topography than physical depositions. These vary from scalable, inexpensive processes like electroplating to expensive, ultra–high purity metalorganic chemical vapor deposition (MOCVD). Physical depositions, on the other hand, use a mechanical or thermal process to transport material from a source to the substrate surface. Again, there are some processes that are relatively simple and inexpensive, such as evaporation and sputtering, and high–end epitaxial methods like molecular beam epitaxy (MBE).

AlN can be deposited by various chemical and physical means. When used in photonics, where defect–free single crystal layers must be epitaxially grown as part of an active region or as a cladding layer, both MBE and MOCVD are used. These costly processes produce very high quality films but require temperatures in excess of 1000 °C, severely limiting the materials that can be used and the processing feasible prior to film deposition [1]. Also, both MOCVD and MBE are meant for epitaxial film growth on single crystal substrates, but the titanium substrates currently available are polycrystalline
and have a relatively high surface roughness, effectively canceling any advantage that MBE and MOCVD might offer. Physical vapor deposition (PVD) is an excellent alternative for depositing AlN films on a lower–quality substrate like bulk titanium while maintaining material properties that approach single crystal values. In particular, sputter deposition has been embraced by both industry and the research community for its ability to deposit high quality films at a lower cost and with fewer design restrictions than epitaxial methods.

**Sputter Deposition**

Sputter deposition uses ion bombardment to eject atoms from a source, usually a high–purity metal, onto the surface of the substrate. Ion bombardment is usually provided by directing ions towards the substrate from a plasma generated within the sputtering chamber. However, material sputtered from a focused ion beam generated outside of the sputtering chamber can produce similar results; a combination of the two methods is called ion–assisted deposition (IAD). A brief history of the emergence and development of the sputter deposition process can be found in [2].
A plasma–based sputtering process takes place in vacuum and is initiated by the ignition of a plasma using an electrical bias inside the chamber. The plasma forming gas is often simply a noble gas such as argon, though in reactive sputtering there can be other gases introduced into flow. The bias dissociates electrons from the gaseous atoms. The electrons drift towards the anode while the newly–ionic gas species drift towards the cathode—the target. It is this ionic bombardment that causes the ejection of atoms from the target. These atoms then travel through the chamber; some land on the substrate surface, some collide with the plasma and are redirected towards the target, and others land on the chamber walls. An in–depth analysis of the sputtering process is provided by Smith [3].

While sputter deposition can occur simply with the placement of a target in a simple DC glow discharge, the need for high–quality films and fast deposition rates has introduced a wide array of sputtering technologies. Current sputtering technology includes RF–coupled power modes that increase the ion density; magnetic field sources to enhance ion bombardment and minimize film damage; geometric chamber designs that maximize the input power and facilitate film growth; and the addition of non–noble gases that alter the stoichiometry of the plasma and allow for the sputtering of new
materials. Taken altogether these form the basis for sputtering high-quality AlN at fast deposition rates: reactive sputtering.

3.2 REACTIVE SPUTTER DEPOSITION OF ALUMINUM NITRIDE

It has traditionally been difficult to sputter thin films of dielectric materials. Although it is possible to sputter AlN using a pure DC discharge, the growth rate is very low and the crystallinity of the film can require high temperatures; an RF or other alternating source is normally used [4]. Also, one of the main advantages of sputter deposition over the other methods outlined above in Section 3.1 is the high deposition rate, but dielectric sputtering often occurs at rates that are much lower than metal sputtering rates. The solution to these problems is reactive sputtering.

Reactive sputtering is, by definition, the addition of a reactive gas to the main noble plasma gas. To sputter AlN by reactive deposition, the plasma is formed with a mixture of argon and nitrogen in the presence of an aluminum target. The addition of nitrogen has a three–fold effect on the sputtering mechanism: nitridization of the aluminum target occurs, causing the target to sputter a mixture of Al, N, and Al\textsubscript{1−x}N\textsubscript{x}, rather than simply pure aluminum;
nitrogen ions in the plasma can react with passing Al species to form nitride species; and further reactions between Al and N can occur at the surface of the film. In practice, the first effect is usually the dominant mechanism when N₂ is added to the plasma.

**GROWTH PROCESS OF ALUMINUM NITRIDE FILMS**

The microstructure of a thin film is highly dependent on the process conditions during film growth, and many structure zone models (SZM) have been formulated to explain these dependencies. Movchan and Demchishin developed the first SZM to describe observations in metal and ceramic films; Thornton developed a SZM for films deposited by sputtering [5]. Pictured in Figure 3.1, this SZM uses two process conditions—normalized growth temperature and plasma pressure—in an attempt to fully describe the microstructure of a thin film. Four main zones are classified: zone 1, where structure is controlled by shadowing effects; zones 2 and T, where structure is a product of surface diffusion; and zone 3, a region dominated by bulk diffusion.
Figure 3.1: Thornton’s structure zone model for sputtered thin films. The process conditions for low temperature AlN sputtering correlate to the microstructure of zone T. (Figure taken from [6].)

Low–temperature reactive sputtering of AlN occurs at $T_x/T_m$ values between 0.1 and 0.2 and pressures well below 1 mbar. These conditions correspond to zone T growth, which was later shown to be a subzone of zone 1 [7]. In the revised model, the zone modified the axes of the SZM to reflect thermal– and bombardment–induced mobilities and added film thickness as a third process condition dependence. These modifications accurately reflect the growth mechanism and resulting microstructure of sputtered AlN films.

The growth of an AlN thin film by reactive sputter deposition occurs in four stages [8]. Because the temperature is relatively low—the external
heating element of the sputtering tool used has a maximum operating
temperature of 400 °C—the initial growth of the AlN film is a Volmer–Weber
process. Adatoms from the plasma condense onto the bare substrate, forming
nucleation sites. As more adatoms condense, these nucleation sites grow into
discrete islands of AlN thin film. (These islands are not epitaxial with the
substrate; the growth temperature is too low and, barring some exceptions
like Al₂O₃, the substrate and the AlN film are not lattice–matched.) The
islands continue to grow until impingement with neighboring islands occurs,
causing the islands to form grain boundaries and coalesce into one another to
form a single film. Because each island has a crystalline orientation
independent of other islands, the coalesced film is not a single crystal but a
collection of these crystallites, each having its own normal crystal plane and
axial orientation. The growth of the film is now determined by the growth
rates of the normal crystal faces of the crystallites. The (002) plane of AlN has
the highest growth rate of the crystal faces so as deposition continues these
grains can overtake crystallites with other crystal plane faces [9]. The entire
process is depicted below in Figure 3.2
Figure 3.2: Growth mechanism of an AlN thin film. A four stage process occurs during sputter deposition: atoms and molecules sputtered from the target travel to the surface of the substrate and form nucleation sites (1); islands grow from the nucleation sites (2); the islands coalesce into a film containing many crystallites with various orientations (3); crystal orientations with the fastest growth rates outgrow unpreferred, slower orientations and the film coalesces into a polycrystalline film with a preferred orientation (4).

There are three important considerations about this process that must be noted. First, the coalescence of islands of AlN is key to the crystalline orientation of a deposited film. The plasma conditions and the energy of the arriving adatoms have a direct correlation on the size and orientation of the islands, which in turn strongly affect the structural properties and crystalline orientation [7]. Second, the amount of material sputtered during the fourth
stage comprises almost all of the AlN film, yet by this time many of the film properties have been determined; small, randomly oriented islands will not produce a highly crystalline film, regardless of the sputtering conditions during this stage. (This is not to say that the fourth stage has no bearing on the film, as the arrival rate and energy of the plasma species will affect the film stress and final columnar alignment.) Finally, growth of an AlN film with any preferred orientation is not sufficient; growth of polar (002) crystallite faces are required for fabricating MEMS with maximum piezoelectric response [10].

The effective piezoelectric response of an oriented piezoelectric film was modeled by Lughi using a Gaussian–distributed analysis [11]. Consider a Cartesian coordinate reference frame $C = (x, y, z)$ such that the $z$ direction is perpendicular to the substrate; $x$ and $y$ lie in the plane of the substrate. On the substrate is a polycrystalline AlN film where each crystallite can have any angular orientation of its $c$ axis. Using the reference frame $R$ as a basis, the coordinate system $C_{gr} = (x_{gr}, y_{gr}, z_{gr})$ matching the orientation of an individual crystallite can then be defined, as shown in Figure 3.3. The new coordinate system is simply a transformation of the reference frame by three successive angular rotations: $\phi$, an in–plane rotation parallel to the substrate; $\theta$, a tilt off
the crystallite off of the substrate normal $z$; and $\psi$, a rotation about the $c$ axis of the crystallite. In the coordinate system $C_{gr}$, the $c$ axis of an individual AlN crystallite is parallel to $z_{gr}$ while $a_1$ and $a_2$ lie in the plane of $x_{gr}$ and $y_{gr}$.

![Figure 3.3: The transformation from the reference frame of the substrate to the coordinates of a crystallite.](image)

To model the piezoelectric response of the thin film—a collection of crystallites, each with their own orientation—a transformation to the coordinates of an individual crystallite must be defined. The black arrows represent the new coordinate axes formed by a rotation from the gray axes. The rotating arrow designates the axes about which each rotation is performed. Adapted from [11].

The entire transformation between $C$ and $C_{gr}$ described above is contained within the matrix

$$n_{ij} = \begin{bmatrix}
\cos \phi \cos \psi - \cos \theta \sin \phi \sin \psi & \sin \phi \cos \psi + \cos \theta \cos \phi \sin \psi & \sin \theta \sin \psi \\
-\cos \phi \sin \psi - \cos \theta \sin \phi \cos \psi & -\sin \phi \cos \psi + \cos \theta \cos \phi \cos \psi & \sin \theta \cos \psi \\
\sin \theta \sin \phi & -\sin \theta \cos \phi & \cos \theta
\end{bmatrix}.$$ (3.1)
By combining the transformation matrix in equation (3.1) with the piezoelectric tensor $e_{ij}$, the effective piezoelectric constant of an individual crystallite reduces to the equation

$$
e_{33,\text{crystallite}}(\theta) = (2e_{15} + e_{31})(\cos \theta \sin^2 \theta) + e_{33} \cos^3 \theta. \quad (3.2)$$

The effective piezoelectric coefficient of an individual crystallite is a function only of the tilt angle. Also note that although the above derivation uses the stress piezoelectric constants, a substitution for the strain piezoelectric constants $d_{ij}$ can be made with the same result.

To find the total piezoelectric response of the thin film, the contribution of each crystallite in the film must be summed. The distribution is assumed to be Gaussian due to experimental x–ray diffraction (XRD) results, and a modified Gaussian function that represents the physical film structure is defined as

$$p(\theta, \sigma) = \frac{1}{\sigma \sqrt{2\pi} \text{Erf}} \left( \frac{\pi}{\sigma \sqrt{2}} \right) e^{-\frac{\theta^2}{2\sigma^2}}, \quad (3.3)$$

where

$$\sigma = \sqrt{\frac{\omega_{FWHM}^2}{8 \ln 2}}. \quad (3.4)$$
\( \omega_{FWHM} \) is the full–width half–maximum (FWHM) of an XRD \( \omega \) scan centered at the (002) peak; this scan is called the rocking curve. By combining equations (3.3) and (3.4) and integrating over the entire range of tilt angles, the effective piezoelectric constant of a film is then:

\[
e_{33, \text{effective}} = \int_{-\infty}^{\infty} p(\theta, \sigma) e_{33, \text{crystallite}}(\theta) d\theta.
\]  

(3.5)

This equation shows that the piezoelectric response of the entire film, much like an individual crystallite, is a function only of the tilt angle. The theoretical result is the effective piezoelectric constant \( d_{33} \) of an AlN film can be determined experimentally using XRD to measure the tilt angle \( \theta \) – fabricating a device or structure to directly measure the value is not required. Equation (3.5) is relatively complex and an exact analytical solution to the integral in \( e_{33, \text{effective}} \) can be found using computational software. The function \( e_{33, \text{effective}} \) is plotted over the entire range of FWHM in Figure 3.4.
Figure 3.4: The effective piezoelectric constant $e_{33}$ as a function of the rocking curve FWHM. As the FWHM of the film increases from zero, the piezoelectric response of the film drops off sharply. An exact solution to equation (3.5) was calculated using Mathematica software.

The above model shows that the piezoelectric response of the film is strongly dependent on the c-axis alignment of individual crystallites to the substrate normal. However, there are certain limitations to this model. The calculations assume that there is no interaction among grains, such as motion along crystallite boundaries or the formation of voids under deformation. There is justification for this assumption if the material is highly aligned and the crystallites have a true columnar structure [11]. Also, the modified Gaussian in (3.3) does not completely represent true random orientation in the film. Nonetheless, this model was shown to be in agreement with
experimental data at low values of $\omega_{\text{FWHM}}$; agreement was also shown on BaTiO$_3$ [12]. Reported results for AlN thin films on silicon show that a rocking curve FWHM better than 5.5° is necessary for good piezoelectric characteristics and strong electromechanical coupling [13]. Clearly there was justification for finding sputtering parameters that minimized the rocking curve FWHM for the purpose of maximizing the piezoelectric properties of the AlN film.

**PARAMETERS OF REACTIVE SPUTTER DEPOSITION**

It is essential to discuss the parameters that are common to all reactive sputter deposition tools. Four basic parameters for sputter deposition are: the type of waveform used to apply power to the plasma; the amount power applied; the flow rates of the noble and reactive gases; and the pressure of the sputtering chamber. These parameters are not necessarily independent of each other—e.g. changing the applied power can affect the sputtering pressure—but rather are the four variables most intrinsic to the deposition of thin films.

The deposition rate of a film is a function of applied power because the energy of the ions impacting the target is a determining factor of the
deposition rate. Although a high deposition rate is favorable, at higher power there is also more damage to the film because impacting species can resputter the growing film and cause an increase in the residual stress in the film through high ion impact energies that create interstitials and vacancies. The deposition rate as function of chamber voltage at various deposition conditions is shown in Figure 3.5. Two trends are apparent: increased power and decreased pressure raise the deposition rate. High power imparts more energy to the plasma species, increasing the energy of ions hitting the target and sputtering more material. Low pressure increases the mean free path of both the plasma species and sputtered material, increasing the energy of both the ions hitting the target and the ejected material.
Figure 3.5: Deposition rate at various process conditions. As the applied power increases, more material is sputtered from the target, increasing the deposition rate. Also, at a given power, higher pressure—equivocal in the Endeavor 8600 to higher gas flow—decreases the deposition rate. The lines are linear fits of the data taken.

The power waveform affects the plasma response to the electrical energy from the source; without a negative cycle in the waveform, the target becomes electrically charged and the effective voltage drops. Waveforms that have been used to reactively sputter highly crystalline AlN include RF, pulsed DC, and middle frequency (MF) or AC [14], [15], [16]. Each of these technologies has advantages. RF sputtering is the most common but can be very sensitive to substrate materials, sputters at a relatively low deposition rate, and does not sputter films with the highest axial alignment. MF
sputtering requires more expensive electrical components but has the advantage of reducing the deposition of dielectric films on the target and anode by dual-cathode bipolar sputtering, reducing arcing and increasing film quality [17].

As discussed above, reactive sputter deposition is defined by the introduction of a reactive gas into the noble gas plasma during deposition. In the case of AlN sputtering, the gases are almost always Ar and N₂. (Ar is used because unlike the heavier nobles it is truly inert, yet easily adsorbs in the cryopump used to pump the deposition chamber. Likewise, other reactive gases such as NH₃ have been used, but these often have byproducts that can contaminate an AlN thin film.) The proportion of reactive gas must be above 0%, otherwise pure metal will be sputtered from the target, and can be as high as 100% in some systems. The flow proportion of Ar/N₂ is a very important parameter, as illustrated in Figure 3.6 below. Reactive sputtering occurs when the aluminum target is nitridized, or poisoned, in the presence of nitrogen gas. In the absence of N₂, the target sputters pure Al. As the N₂ flow is increased, AlN begins to form at the target but it is sputtered off faster than a stable nitride layer can form. At a critical flow rate, however, the nitrogen in the chamber has a high enough partial pressure that the target
becomes completely poisoned; increasing the flow does not change the chemistry of the target. Conversely, as the N$_2$ flow is decreased in the presence of a poisoned target, a critical flow value is reached where the target switches from poisoned to metal mode.

![Diagram](image)

**Figure 3.6: Crossover between metal and poisoned sputtering.** Hysteresis in the chamber voltage occurs because the sputtering rate of Al is higher than AlN. During a decrease in flow rate a stable mixed–composition target can be obtained., as seen in the red curve between approximately 6 and 9 sccm. The data were taken during sputtering with 3 kW of power and 7 sccm Ar flow.

There is a third regime that occurs during a decrease in N$_2$ flow: the reactive mode. It occurs prior to the crossover back to the metal sputtering
mode and is due to the differential sputtering rates of AlN and Al. In general, metals sputter at higher rates than their corresponding oxides and nitrides; this relationship holds for aluminum and is reflected on the decreasing flow curve of Figure 3.6. As the N₂ flow is decreased, the target composition changes from completely poisoned to a mixture of Al and AlN. Unlike the case of increasing flow, however, the mixed composition is stable and can be maintained over several sccm. This hysteretic behavior is characteristic of reactive sputtering and was crucial to depositing highly aligned AlN thin films on titanium, as will be discussed below.

Chamber pressure is the final parameter universal to all reactive sputtering systems. It is widely reported that the sputtering pressure can have a profound effect on the quality of sputtered films [18]. Sputtering at low pressure can cause the chamber voltage and the ion energy to increase considerably, causing substantial damage to the film. Arcing can also occur inside the chamber. At high pressures, however, the chamber voltage is lessened, reducing the amount of energy imparted to the ions. Also, the mean free path of the ions is decreased; there are many more collisions as the ions travel from the target to the substrate, further reducing the kinetic energy of
the ionic species. The result of these two effects is that impinging ions do not have enough kinetic energy to arrange into a properly aligned film.

**PARAMETERS OF THE ENDEAVOR 8600 SPUTTER CLUSTER TOOL**

All AlN films discussed in this work were sputtered in an AC bipolar chamber using a Sputtered Films Endeavor 8600 sputter cluster. This chamber has been specifically engineered for sputtering dielectric thin films such as AlN [19]. Owing to its proprietary design, there are sputtering parameters that are unique to this system and should be discussed prior to an analysis of the sputtered AlN films.

Shown in Figure 3.7, the chamber is a dual cathode magnetron source. The targets—termed S–Guns—have been designed to deposit films at a high deposition rate with no arcing, anode coating, or magnetic interference [20]. Strong magnets embedded around each target confine the plasma to a region proximate to the target, minimizing electron damage and heating of the substrate, suspended in the center of the chamber. The plasma is sustained by an AC discharge operating at 40 kHz, making the chamber a middle frequency (MF) sputtering source. It can operate at high power levels and have the highest film deposition rates of any sputter source while
maintaining film qualities that are comparable to other sources [21]. The AC circuit also includes a separate bipolar controller used to adjust the relative amounts of power applied to the two targets. Bipolar control provides independent control over film uniformity, though it can have an effect on other film properties such as residual stress and crystallite orientation.

![Diagram of AC sputtering chamber](image)

**Figure 3.7:** Cross-sectional view of the AC sputtering chamber in the **Endeavor 8600 sputter cluster**. The Endeavor is an upward-sputtering tool; the substrate is suspended above the sputtering guns and is held by a pin-loaded holder. (Adapted from [19].)

Another unique feature of the chamber is that it does not allow for independent control of the chamber pressure; there is no user-controllable butterfly valve at the output port leading to the turbomolecular pump. As a result, the pressure in the chamber depends solely upon the flow rate of the
process gases and the sputtering mode—poisoned, metal, or mixed. The dependence of chamber pressure on the mode of sputtering is shown in Figure 3.8. At zero reactive gas flow—the metal sputtering mode—the chamber pressure is controlled solely by the Ar flow rate. The addition of N₂ gas does not increase the chamber pressure because the gas reacts with the target faster than it can become part of the ambient. At a critical flow, however, the sputtering mode changes from metal to poisoned and a further increase in N₂ flow causes the pressure to increase. Decreasing the reactive gas flow in poisoned mode causes a hysteresis similar to the effect seen above in Figure 3.6. As the N₂ flow is decreased, there is a stable intermediate mode between poisoned and metal where the target composition is mixed. During this stage, the pressure continues to be roughly proportional to reactive gas flow. Once the metal sputtering mode is reached, however, the chamber pressure reverts to a constant value.
Figure 3.8: Dependence of chamber pressure on reactive gas flow. Because
the sputtering chamber does not have independent pressure control, the flow
of the process gases controls the chamber pressure. Across the entire flow
spectrum there is not a simple linear dependence because of the different
sputtering modes. These data were taken sputtering at 3 kW power and 7
sccm Ar flow.

One of the characteristics of the reactive mode is that small fluctuations in
voltage or reactive gas flow can cause unstable sputtering conditions and
force the sputtering into the metal mode—instabilities that result from the
differential sputtering rates of AlN and aluminum. Metal mode sputtering
will completely disrupt the deposition of an AlN film, so it is imperative that
instabilities not occur during film deposition. To avoid this problem, some
reactive sputtering systems use feedback–driven partial pressure control of the $\text{N}_2$ flow [19]. A drawback of the Endeavor 8600 is that it is not equipped with feedback control of the gas flows. Instead, sputtering in the reactive mode can only be maintained by either real–time control of the deposition parameters or sputtering using optimized deposition conditions that result in a very stable reactive region.

As shown in Figure 3.7 above, the substrate is suspended above the source during deposition and films are deposited on the bottom face of substrates. The robotics of the Endeavor 8600 and the pin–loading system within the chamber itself only accept substrates with exactly 4–inch diameters and with a maximum substrate thickness of approximately 700 $\mu$m. As a result, there are two ways to sputter onto a non–circular substrate such as a sheared titanium piece. The first method is to use a sputtering plate: a sheet of metal that has been machined into the shape of a 4–inch wafer with a window cut out of the center of the plate. A film can then be sputtered on a sheared piece by placing the piece over the window and loading the plate into the sputter tool. The second method is to mount a titanium piece onto the underside of a carrier wafer. However, there are severe limitations to both methods.
A sputtering plate is not economical because it can only be used once. After a sputtering run, the plate is coated with an AlN film that can only be removed by sandblasting, but sandblasting will warp the plate. The plates must be very thin to ensure that the plate height plus the sample height is less than the maximum allowable thickness of the sputter tool. If the film is not removed before reuse, the highly capacitive AlN film will disrupt the electric field within the chamber. Also, the thickness of the sputter plate has a negative effect on both the film uniformity and crystallite orientation of films deposited using a sputtering plate. The Endeavor 8600 is designed for depositing films onto standard silicon wafers; the geometry of the plasma created by the S–Guns is optimized for sputtering at the plane corresponding to the surface of a wafer. If the substrate surface is above the plane of a silicon wafer, as is the case when a sputtering plate is used, the plasma does not deposit a film under ideal conditions. Additionally, the S-Guns sputter at an angle far from the substrate normal—a requirement for highly conformal sputtering—and the thickness of the sputter plate shadows a substrate from the plasma. As a result of these two effects, the films sputtered using a sputtering plate have far poorer crystalline alignment and thickness uniformity than samples mounted on carrier wafers.
Samples mounted to a carrier wafer must adhere to a different thickness limitation than samples placed on a sputtering plate. A mounted sample cannot interfere with the movement of the robotic transfer arm. The clearance on the underside of a carrier is approximately 300 μm; the thickness of a sample plus its adhesive must be within the clearance. Because the substrate is heated by the impinging species during deposition, any adhesive used to mount the sample to the carrier must be able to maintain its viscosity at these elevated temperatures, otherwise the sample will fall into the chamber and the adhesive will be exposed to the plasma and contaminate the chamber. Outgassing from the adhesive will also contaminate the chamber and the AlN film. Deposition experiments were conducted using several different adhesives, including Santovac 5 polyphenal ether lubricant; Apiezon H and T vacuum greases; 3M 9882 thermally conductive tape; DuPont Kapton polyimide film; and Electron Microscopy Sciences carbon adhesive tabs. In fact, the only adhesive that was sufficient for mounting samples was Apiezon H. All other adhesives either lost viscosity or otherwise could not maintain sufficient adhesion during deposition. However, Apiezon H outgassed considerably and degraded to the point of failure in approximately one third of the experiments. Once titanium wafers became available, deposition of
AlN thin films onto sheared titanium pieces using sputtering plates and carrier wafers ceased, and all experiments and further depositions were carried out on wafers.

3.3 **Deposition of C Axis Oriented Aluminum Nitride Films on Titanium**

To be used for piezoelectric MEMS fabrication, an AlN thin film must exhibit: aligned columnar growth of AlN; (002) face growth parallel to the plane of the substrate; and large crystallite size. Films with strong crystallinity can have piezoelectric coefficients and acoustic velocities approaching single-crystal AlN values, and high electromechanical coupling—factors all critical for the operation of SAW and BAW devices. Additionally, AlN films with strong crystallinity can be processed with a consistency and uniformity above films with weak crystallinity. A film with small crystallites will have a higher proportion of grain boundaries and be more prone to grassing during plasma etching because of the masking oxides that aggregate at grain boundaries. Higher amounts of grain boundaries will also decrease the reflectance of the film, lowering the accuracy of the optical
profilometry used to measure the film thickness and the optical constants $n$ and $k$.

Other properties of the film must be controlled as well. There must be a mechanism for controlling stress in deposited films, as films with high compressive stress can cause buckling–induced delamination in deposited films and fracture in released devices [11]. The thickness uniformity across the diameter of the titanium wafer is also important. Sputtered films exhibit some thickness variation because of position–dependent deposition rates in the sputtering chamber. A film with a large variance in thickness can cause problems during the fabrication process, particularly when attempting a controlled etch of the thin film. Variations in film thickness will result in some areas of the substrate being exposed to the etching species before all areas of the film are etched. If the substrate reacts with the AlN etching species, problems can arise in later processing steps—e.g. the etching of titanium in a chlorine plasma will cause large step heights for AlN features; or the formation of nano–structured titania (NST) in the presence of heated H$_2$O$_x$ will increase the electrical resistance of contact pads deposited later. A well–characterized deposition process allows control of all of the film properties listed above.
CRYSTALLINE ALIGNMENT

The piezoelectric properties of an AlN film are so important to the operation of microscale devices that the first step toward characterizing a deposition process for AlN onto titanium is depositing films with good c axis alignment of the crystallites. There are many techniques for sputtering highly–aligned AlN films onto silicon for the purpose of fabricating surface and bulk acoustic wave devices. Most of these methods utilize high temperature RF or pulsed–DC sputtering systems. However, the Endeavor 8600 uses a MF sputtering system, so processes characterized on systems with other waveforms are unsuitable for use on this tool. Also, because the thermal expansion coefficients of AlN and titanium are so different, substrate heating was eliminated as a process variable; the curvature that resulted from the tension in these films was very high, and highly curved wafers proved impossible to process. A new method for sputtering AlN films was needed.

Initial deposition characterization began based upon earlier AlN sputtering research using MF deposition on the Endeavor 8600, in which AlN films with rocking curves under 2° had been obtained on silicon using a two–stage deposition process [19], [22]. The first stage is a low–power, high–
temperature deposition that provides high mobility to the surface adatoms at the expense of uncontrolled stress. The second stage is a high–power deposition at lower temperatures characterized by high deposition rates and residual stress control. This process was modified in two ways: all substrate heating was eliminated, and chamber pressure and sputtering power were instead used to impart energy to the adatoms. The first stage of the process—a high–power, low–pressure deposition—increased the surface energy to the substrate and gave impinging species higher mobility to form aligned crystals at the expense of causing damage to the growing film. The second stage was a deposition at moderate power and high pressure, depositing the bulk of the film on top of what was essentially an aligned seed layer. Because the Endeavor has a fixed exhaust system to pump the sputtering chamber, the gas flows were lowered to 20 sccm Ar and 10 sccm N₂ during the first stage and 40 sccm Ar and 20 sccm N₂—the maximum flow rates possible—during the second stage. Figure 3.9 shows the result of the initial depositions.
Despite an increase in the crystalline alignment of the films, improvements to the deposition process reached a limit at films with 5° rocking curve FWHM. The conditions that had been altered to this point were the bipolar power ratio and the pre-deposition treatment of the film as well as slight modifications to the gas flow ratios. Because these refinements could
not produce sufficient columnar alignment, a better sputtering method was needed to deposit highly crystalline AlN films.

Following the direction of some reported results of highly crystalline dielectric films sputtered in the reactive mode, a study of the sputtering modes of the Endeavor was performed [16, 23]. For a given combination of MF power and Ar gas flow, films were deposited at 1 sccm intervals of N₂ flow. 2θ x–ray scans of the films deposited in metal mode exhibited no AlN peaks, which was not surprising considering both the low proportion of N₂ flow and the very high chamber voltages. The films also had a higher than usual surface roughness due to damage from the high–energy plasma species. Past the crossover to the poisoned mode, however, the chamber voltage dropped dramatically and the films began to show the (002) peak. At lower N₂ flows, e.g. 8 sccm, XRD scans showed that there were also (110) AlN peaks, an indicator that films did consist entirely of the textured alignment necessary for strong piezoelectricity [24]. As expected, films deposited at higher flows—well into the poisoned region—showed only the (002) peak and had rocking curve FWHM within the range previously obtained. At the maximum limit of Ar and N₂ flows, the deposition conditions were essentially the same as the films deposited in Figure 3.9.
Different results were obtained as the N\textsubscript{2} flow was decreased into the region of reactive mode sputtering. Because of the hysteretic effect, as the flow is decreased into the reactive mode, there is a higher chamber voltage compared to the voltage obtained as the flow is increased—see Figure 3.6 for N\textsubscript{2} flow between 6.5 and 10 sccm. A high chamber voltage corresponds to increased plasma energy and deposition rate; these conditions can positively influence the crystallinity of the film by providing surface mobility to the species forming the film. However, above a threshold level the energy begins to have a negative effect: the impinging species achieve high energies characteristic of metal mode sputtering and begin to damage the film through the formation of vacancies and interstitials. Additionally, the temperature of the substrate will increase, resulting in a large tensional stress upon cooling due to the large thermal mismatch between AlN and titanium. Accordingly, it was critical to characterize this region to see if reactive mode sputtering could yield films with better crystallinity than previously obtained using poisoned mode sputtering.

Characterization of the reactive mode was in iterative process due to the large number and strong interdependence of the process variables. Because the Endeavor 8600 is not equipped with a partial pressure gas controller, N\textsubscript{2}
partial pressure hysteresis curves were taken at 2 kW and 3 kW power for Ar flows between 0 and 40 sccm and N\textsubscript{2} flows between 1 and 20 sccm. As mentioned earlier, sputtering in the reactive mode is prone to instabilities that decrease the crystalline qualities of an AlN film; it is imperative to find a reactive mode with a large differential in N\textsubscript{2} partial pressure coupled with a stable behavior with respect to small fluctuations in N\textsubscript{2} flow. Some trends were apparent: low Ar flow or low power caused a very narrow region of reactive flow; high Ar flow or high power washed out the partial pressure differential. Figure 3.10 shows the partial pressure hysteresis for the optimized sputtering conditions: 3 kW sputtering power, 20 sccm of Ar flow, and half–power bipolar control. The reactive mode extended across nearly 5 sccm of N\textsubscript{2} flow and the maximum partial pressure differential was 50%. There was also a gradual boundary between the reactive and metal modes; instabilities were unlikely to result in a transfer to metal sputtering.
Having found process conditions that had a suitable reactive mode, the next step was to find the particular flow in the reactive region that resulted in films with the best crystallite alignment. The center of the reactive mode, 6 sccm, proved to be the flow at which the best columnar alignment was obtained. At lower flows, the voltage was significantly higher and the AlN films began to exhibit high residual stress and visible cracking. Also, despite the stability of the reactive mode, over an hour-long deposition the gradual increase in sputtering voltage—characteristic of dielectric film sputtering—would sometimes cause a shift into the metal mode and completely ruin the
deposition. At higher reactive mode flows, the chamber voltage decreased, lowering both the deposition rate and the energy imparted to the film, reducing the crystallite orientation. Data from films sputtered in the reactive mode onto various substrates are displayed in Figure 3.11.

![Figure 3.11: Crystallite orientation as a function of film thickness and substrate for AlN sputtered in reactive mode. Films sputtered on titanium wafers exhibited much better orientation than films sputtered on titanium pieces and approached the collimation obtained on silicon wafers. The large spread in crystalline alignment of films sputtered on titanium wafers was a result of dynamic chamber conditions. The silicon data is fit using a least squares method to show how collimation improves as the film thickness increases.](image)

There are a few important trends that are evident. With these sputtering conditions it was possible to obtain AlN films on titanium with a quality equal to films sputtered on silicon. It is important to note that not only did
these films have FWHM less than 2°, but this level of collimation was obtained at a 1 μm film thickness. Second, the deposition conditions could yield AlN films on silicon with FWHM below 1°, the best collimation reported for sputtered films on silicon [11]. Third, sputtering onto sheared titanium pieces continued to yield poor quality films; however, even some of these films were within the arbitrary rocking curve FWHM limit of 5°. Finally, there is a considerable spread in the collimation of films sputtered on titanium wafers. This is a result of the data being taken from films sputtered under different chamber conditions. The chamber conditions are significantly affected by: venting and cleaning; dielectric film buildup on chamber walls; anode sandblasting; and deposition history, including prior AlN depositions and other users’ processes such as Al2O3 and Al. The silicon data was taken from one single process run, so there is a uniformity that is not present in the titanium data, which was taken from many different runs over an extended period of time.

3.4 CHARACTERIZATION OF ALUMINUM NITRIDE FILMS

Although the c axis orientation of AlN thin films was the impetus for optimizing the deposition conditions, other material properties of the films
were measured. Some characteristics, such as the optical properties of AlN, were observed to be relatively unaffected by the process conditions, varying by less than 4% over the entire range of deposition parameters. However, other properties have a very strong dependence on the growth conditions and film microstructure; film stress and crystallite size are two examples. As compared to AlN films sputtered on silicon, the ductile nature of the titanium wafers limited the range and accuracy of some characterization measurements that could be made. As received, the flatness of the titanium wafers was not within the curvature range of available optical stress characterization tools. As a result, the stress of the deposited AlN films could not be directly measured. Also, the titanium wafers would often warp following AlN deposition due to the residual stress in the film. Warping impedes measurements that use surface reflection by changing the reflection angle and reducing the intensity—e.g. the accuracy of optical interferometry depends upon the detector counts at each wavelength, and the number of counts scales with intensity.
**CROSS-SECTIONAL IMAGING**

Having measured rocking curves that indicated columnar alignment in the sputtered AlN films, a quantitative analysis of the film was performed on films with high and low rocking curves, and between highly aligned AlN films on silicon and titanium. On silicon, simply cleaving the wafer with a diamond scribe exposed the cross section of the AlN film, which could then be imaged with scanning electron microscopy. Because polycrystalline titanium cannot be cleaved, films on titanium had to be ion milled to expose the cross section. An FEI Strata DB 235 focused ion beam system was used for in situ scanning electron imaging of ion milled samples. Additionally, this system is equipped with an energy dispersive x-ray (EDX) spectrometer used to provide in situ chemical analysis. EDX probes a material with high-energy electrons to a depth of 1–2 μm. The electrons excite the electronic states of atoms in the material; subsequent relaxation causes photon emission. These photons are then collected by a detector and analyzed using computer software to determine the elemental composition of the material. A caveat to the in situ EDX is that the analysis occurs after the ion milling process; sputtered material from the ion beam can be redeposited on the sidewall of
the cross section, masking the true chemical composition. Still, EDX can reliably be used as an estimation of the composition [25].

Figure 3.12 compares the cross sections of two AlN films sputtered onto titanium using different sputtering conditions. The film shown in image A was sputtered at 3 kW with 20 sccm Ar, 6 sccm N₂, and half-power bipolar control; the film thickness was 3.9 μm and the rocking curve FWHM measured 4.0°. In this film, the crystallites are axially aligned, uniform in width, and continuous from the substrate to the surface of the film. There are very few voids, resulting in little misalignment in c–axis orientation of the crystallites. The film in image B was sputtered using conditions similar to the two–stage process described in [11], with the addition of half–power bipolar control to increase the thickness uniformity. The film thickness was 4.0 μm and had no measurable rocking curve, indicating that the columnar alignment of the film is low. Significant misalignment of the crystallites is clearly visible, as voids and angular growth disrupt the continuity of the crystallite columns. EDX also revealed differences in the elemental composition of the films. The aligned film was 53% Al, 44% N, and 3% O, with a variation of ±3% between the top and bottom of the film. However, the surface of the misaligned film was 64% Al, 33% N, and 3% O. Even less
stoichiometric was the center of the film: 79% Al, 20% N, and 1% O. From these data, it was evident that the sputtering in the reactive mode tended to produce higher quality, more stoichiometric AlN films.
Figure 3.12: Cross sections of AlN films sputtered on titanium. A highly aligned film with a rocking curve FWHM under 3° (A) exhibits fewer voids and misaligned crystallites than a film with no measurable rocking curve (B). The bands of light and dark that can be seen in the films are attributed to charging from the electron beam and slight variations in elemental composition.
Having compared aligned and misaligned films on titanium, a comparison between aligned films on titanium and silicon was made, shown in Figure 3.13. The AlN film on titanium, shown in image A, was sputtered at 2 kW with 20 sccm Ar, 6 sccm N\textsubscript{2}, and half-power bipolar control; the film thickness was 1.5 μm and the rocking curve FWHM measured 2.6°. The film on silicon—image B—was sputtered using the two-stage process referenced above. This film was 1.2 μm thick and had a FWHM of 1.5°. Clearly, these films have very similar characteristics: continuous, axially–aligned crystallites with little evidence of voids. In fact, the most visible difference between the films is the higher surface roughness of the film sputtered on titanium. In fact, it was surprising that the cross sections were so similar considering that titanium wafers have a roughness an order of magnitude higher than silicon wafers.
Figure 3.13: Cross sections of aligned films sputtered on titanium and silicon. The film sputtered on titanium (A) appears nearly identical to the film sputtered on silicon (B). The change in contrast halfway through the film thickness is an artifact of severe electrical charging in the highly–resistive AlN film.

**Crystallite Size**

Data taken from XRD was used to determine the size of the crystallites in sputter deposited AlN thin films. Consider a single crystal film that is free of
defects, grain boundaries, or any other scattering center. Reflections from the crystal planes that occur when the diffraction condition is satisfied will approach \( \theta \) functions; there is no other angle that will reflect except the exact angle at which total reflection occurs. In a crystal that contains scattering centers, the reflections that occur will be broadened over some range of angles. In a polycrystalline material—e.g. sputtered AlN films—the presence of crystallites and grain boundaries reduces the periodicity of the crystal lattice and changes the phase of the reflection, broadening the reflection peak. The Scherrer method uses an analysis of x–ray data from a \( \theta \)/2\( \theta \) scan to calculate the average crystallite size in a polycrystalline material [26]. For a textured thin film, the crystallite size is

\[
D = \frac{K \lambda}{\Delta_2 \cos(2\theta)}.
\]  

(3.6)

\( \lambda \) is the x–ray wavelength—1.54 Å for Cu \( K_{\alpha1} \); \( 2\theta \) is the location of the \( \theta \)/2\( \theta \) peak corresponding to the (002) reflection; \( \Delta_2 \) is the FWHM of the peak; and \( K \) is the appropriate shape factor—approximately 0.9 [27]. Equation (3.6) confirms that as the peak broadens, the crystallite size decreases.

From experimental results, an empirical relationship was developed for the crystallite size as a function of the crystalline alignment of the film.
This relationship is shown in Figure 3.14. The crystallite size is inversely proportional to the FWHM of the rocking curve, and for a given rocking curve the crystallite size is larger on titanium substrates than for AlN on silicon substrates. The larger crystallite size is a result of a larger island area prior to film coalescence and is attributed to the polycrystalline nature of titanium. Although the substrate is polished, the individual grains have different planar heights and are separated by grain boundaries. The boundaries serve as barriers to coalescence, resulting in the islands occupying more area before coalescing across the grain boundary. Once coalesced, these larger islands form the basis for larger crystallites once the film has been deposited.
Figure 3.14: Crystallite size as a function of the rocking curve FWHM. The data are shown for films with 1200 nm > h > 1800 nm. The AlN crystallite size scales with the collimation of the film. For a given collimation, the crystallite size for a film sputtered on titanium is larger than on silicon. The gray lines simply serve as guides for the eye.

OPTICAL AND ELECTRICAL MEASUREMENTS

The complex index of refraction for a material is given by the equation

\[ \tilde{n} = n - i\kappa. \]  

(3.7)

\( n \) is the index of refraction and is a measure of the phase velocity of light traveling in the material. \( \kappa \) is the extinction coefficient, and the imaginary term \( i\kappa \) quantifies the absorption and loss during propagation through the material. These coefficients can be determined from optical measurements and the Kramers–Kronig relations; reflectance data were taken and coefficient
measurement was performed on a Filmetrics F20 white light reflection
dielectric characterization tool. Although $n$ and $\kappa$ are frequency dependent,
for AlN the values are nearly constant over the visible wavelengths. The tool
is capable of simultaneously calculating thickness, $n$, and $\kappa$, although in
practice more accurate results are obtained from taking individual
measurements sequentially. It was intuitive to determine if the degree of
crystallinity of the AlN films affected the index of refraction. From the data
plotted in Figure 3.15, it was clear that $n$ showed no appreciable variation
with the FWHM of the rocking curve.

![Index of refraction plotted against crystalline alignment](image)

**Figure 3.15: Index of refraction plotted against crystalline alignment.** The
index of refraction has a relatively narrow distribution over the rocking
curve FWHM. Additionally, all of the indices for films on titanium substrates
shown are within the reported range for epitaxial or single crystal AlN.
The value of $n$ for AlN is dependent on the crystallinity; reported ranges are 1.8–1.9 for amorphous films, 1.9–2.1 for polycrystalline films, and 2.1–2.2 for epitaxial films and bulk single crystals [28]. From Figure 3.15, the optical properties of AlN on titanium fall within the values of high quality AlN films. In fact, there was greater variation in $n$ for films sputtered onto silicon wafers. The calculated $\kappa$ for AlN on titanium was between 0 and 0.001, indicating that there was almost no dielectric loss in the visible spectrum. Like SiO$_2$, AlN is transparent at these wavelengths, so this result was expected. Additionally, the conduction band edge of AlN is at 210 nm, a deep ultraviolet wavelength; optically excited carrier generation, a primary loss mechanism, occurs only at higher photon energies.

Electrical measurements of the AlN films were made using a HP 4280A capacitance meter and a HP 4145A semiconductor parameter analyzer. The 4280A was used to measure the capacitance of different films to compare the effect of thickness variation, while the parameter analyzer was used to take current–voltage curves and measure any relevant semiconductor properties. The 4145A is capable of voltages between −40 V and 40 V; however, undoped AlN does not exhibit diode behavior in this bias range. Nonetheless, the ohmic behavior could be used to measure the conductivity of the film.
The capacitance of a dielectric film is given by the equation

\[
C = \frac{\varepsilon_0 \varepsilon A}{d}
\]

where \( \varepsilon_0 \) is the permittivity of vacuum, \( \varepsilon \) is the relative permittivity or dielectric constant, \( d \) is the thickness of the film, and \( A \) is the contact area. Having measured the capacitance of an AlN film, equation (3.8) can be used to find the electronic dielectric constant. The results of the capacitance measurements are shown below in Figure 3.16; the relative dielectric constant of the sputtered AlN thin films is calculated to be slightly under 4. The reported value of \( \varepsilon \) for single crystal AlN deposited by MOCVD is 4.67 [29]; the experimental value is within 15%. Furthermore, \( \varepsilon \) is related to the index of refraction \( n \) through the relation

\[
n = \sqrt{\varepsilon, \mu_r}
\]

where \( \mu_r \) is the relative permeability; for nonmagnetic semiconductors, \( \mu_r \) is about 1. The measured dielectric permittivity is within 10% of the value calculated from the index of refraction.
Figure 3.16: Determination of the dielectric constant in AlN thin films. By plotting the known values from equation (3.8), the slope of the linear fit is equal to the relative dielectric constant, in this case approximately 4.

Because the AlN film exhibited ohmic behavior over the range of applied voltages, the conductivity $\sigma$ of the film could be measured using Ohm’s law and by defining resistance as

$$ R = \frac{\rho d}{A} \quad (3.10) $$

where the resistivity $\rho$ is equal to $\rho = \frac{1}{\sigma}$. Using the data shown in Figure 3.17, the conductivity of the AlN films was calculated as $4.0 \times 10^{-9} \Omega^{-1}$. 

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Edwards et al measured a magnitude of $10^{-11} \Omega^{-1}$ for undoped bulk single-crystal AlN at room temperature [30]. One explanation for the discrepancy between the measured and reported values is that sputtered AlN is polycrystalline with aluminum aggregating at the grain boundaries between crystallites. These metallic regions will be pathways for conduction. Also, because the contact pads used to make these measurements were very small, between 200 and 600 $\mu m^2$, the resistance was very high—the smallest resistance measured was 4.1 G$\Omega$. At such a high resistance the parameter analyzer was measuring currents that were roughly at the noise floor, so any
ohmic behavior intrinsic to the analyzer would be included in the measurement, increasing the calculated conductivity.

**SUMMARY**

Highly crystalline AlN was successfully deposited onto titanium substrates using an optimized reactive sputter deposition process. Reactive mode sputtering at powers between 2 and 3 kW and gas flows of 20 sccm Ar and 6 sccm N₂ yielded films with rocking curve FWHM well under 5°. These films were roughly stoichiometric AlN and had crystalline qualities comparable to films sputtered on silicon wafers. Other properties of the AlN films were measured, including the crystallite size, the optical constants n and κ, and the conductivity σ. The measured values often agreed with previously reported results for single crystal or epitaxial AlN, further demonstrating the viability of titanium substrates as a platform for fabricating piezoelectric MEMS.
REFERENCES


PIEZOELECTRIC CANTILEVERS
In this chapter, the fabrication and testing of basic piezoelectric MEMS on titanium are detailed. Piezoelectric unimorph cantilevers were fabricated on both silicon and titanium substrates. The silicon cantilevers were part of an early demonstration that the sputtered AlN films had strong piezoelectric properties. The titanium unimorphs were used as the first example of piezoelectric actuation in titanium MEMS and also for the calculation of material properties of the AlN film. Also provided are descriptions of the finite element and analytical models used to model the cantilever beam response.

4.1 Piezoelectric Cantilevers

Piezoelectric cantilevers typically fall into two general categories: unimorph and bimorph structures. A unimorph is a single piezoelectric layer adhered to a substrate, often a ceramic, with electrode contact layers on either side of the piezoelectric. A bimorph structure is two piezoelectric layers each with individual contacts; the substrate is sandwiched between the piezoelectrics, placing the neutral axis of the cantilever in the center of the
beam. (Some reported literature makes no distinction between bimorphs and unimorphs, calling any piezoelectric flexure structure a bimorph.) Electrical input power can be applied to bimorphs in series—the direction of the electric field is the same through both piezoelectric layers—or in parallel—the center electrode acts as a ground plane and the outer electrodes are at the signal voltage. As actuators, a bimorph exhibits higher tip displacements and electromechanical coupling than a unimorph comprised of the same structural materials [1]. There are other, more complicated cantilever designs that use three or more piezoelectric films but due to such limitations as thermal mismatching and surface quality of deposited electrodes, common thin film piezoelectrics like PZT and ZnO cannot be used in these structures [2].

There are many applications that use piezoelectric cantilevers. Most commonly, these are found in atomic force, scanning tunneling, and scanning probe microscopy [3, 4]. The cantilever tip angle can change considerably when displaced, a property exploited for cantilever array rastering of a video image [5]. In tandem, two unimorph structures can be combined to create a gripping structure for microsurgery [6]. A conductive cantilever can be actuated onto the surface of a waveguide to short the electrical signal,
creating a switch [7]. Bimorph structures of epitaxial Al$_{0.7}$Ga$_{0.3}$As on GaAs substrates have been fabricated for direct integration with high-speed electronics and optoelectronics [8].

**PRINCIPLES OF CANTILEVER ACTUATION**

A unimorph cantilever design was chosen to demonstrate piezoelectric film properties on silicon and the first example of piezoelectric actuation on titanium. From bottom to top, the layers of a bimorph are: the substrate, a bottom electrical contact, the piezoelectric film, and a top electrical contact. The purpose of the top and bottom contacts is to create a uniform electric field through the thickness of the piezoelectric film. Considering aluminum nitride as the piezoelectric material, a voltage applied along the z direction of the film has two main piezoelectric effects: the $d_{33}$ coefficient governs a strain in the thickness of the film and the $d_{31}$ coefficient results in strains in the $xy$ plane of the film. Although the thickness change of an AlN thin film is extremely small, on the order of femtometers, the length of a cantilever beam is sufficient to cause an appreciable strain along the beam length. Because the beam is fixed at one end and the film is constrained by the substrate layer, a voltage between the contacts causes the cantilever beam to bend. If the
substrate used is conducting—e.g. heavily doped silicon or titanium—then the substrate can also act as the bottom electrode, as shown in Figure 4.1.

![Principle of actuation in a piezoelectric cantilever beam.](image)

**Figure 4.1: Principle of actuation in a piezoelectric cantilever beam.** Because the piezoelectric effect is AlN coupled between crystallographic directions, a through–thickness voltage causes an in–plane strain via the d_{31} coefficient of the piezoelectric matrix. If the top electrode is compliant and thin compared to the piezoelectric and bottom electrode layers, the beam acts like a piezoelectric bimorph and will bend under an applied voltage.

It is important for the layers above and below the piezoelectric to have different thicknesses or compliances because otherwise the piezoelectric layer will be constrained by similar boundary conditions and the beam will not bend, only stretch. All processes for fabricating the AlN cantilevers used 100–300 nm of aluminum as the top electrode. Since this layer was both much thinner and more compliant than either the silicon or titanium bottom
electrodes, the cantilevers were expected to behave like a piezoelectric unimorph when actuated.

### 4.2 CANTILEVER BEAMS ON SILICON

As a first step towards fabricating piezoelectric MEMS on titanium, piezoelectric actuation was demonstrated using cantilevers fabricated on silicon substrates. A process for the sputter deposition of highly crystalline AlN had been established but no MEMS had been fabricated from these films; there was an impetus to prove the viability of these sputtered AlN films before further research on silicon or titanium substrates continued. Because the silicon wafers used were highly doped, the substrate acted as the bottom contact layer. A graphical representation of cantilever beam and its support structure is shown below in Figure 4.2.
Figure 4.2: Schematic of a three–layer piezoelectric cantilever. The AlN film forms the bulk of the cantilever beam, with an aluminum top contact layer and the doped silicon substrate as the bottom contact. A SiO$_2$ layer is used to provide electrical isolation between the top and bottom contacts away from the beam. Selective etching is used to release the beam while maintaining a fixed support. The x direction is along the length of the cantilever, the y direction is along the width, and the z direction is through the thickness.

Standard MEMS processes were used to fabricate cantilever beams on silicon wafers; a detailed diagram of the process appears below in Figure 4.3. The AlN films were deposited and patterned first, followed by the deposition and patterning of a dielectric SiO$_2$ layer. Metal contacts were added via a liftoff process. After the thin films were processed, the cantilever and support structures were etched into silicon and released.
Figure 4.3: Process flow for fabricating AlN cantilevers on silicon wafers.
The seven–step process begins with the sputter deposition of an AlN thin film onto a bare silicon wafer (A). A photoresist mask is then used to pattern the film with a plasma etch (B). Next is the chemical vapor deposition of a SiO$_2$ film and patterning with a photomask (C–D). The metal contacts were added using electron beam deposition and photoresist liftoff (E). Using the photoresist as a masking layer, the cantilever structure was anisotropically etched into the silicon substrate (F) and then the cantilevers were subsequently released with an isotropic plasma etch (G).

The silicon wafers used were (100) oriented and p–doped with phosphorus so the substrate layer of the cantilever could be the bottom electrode. The surface roughness of these wafers was under 1 nm; a low surface roughness is critical for depositing AlN films with high crystallinity. Although an ideal substrate is atomically smooth and lattice–matched, because sputter deposition does not produce an epitaxial film at standard deposition temperatures and silicon has a lattice mismatch with AlN, only a very low surface roughness is necessary. Before loading into the Endeavor 8600 deposition chamber, the wafers were cleaned in ultrasonic baths of
acetone, isopropanol, and deionized water for 3 min each. After chamber pump down, the wafers were treated with a 100 W RF argon plasma for 2 min prior to the AlN film deposition. A pre–deposition high–temperature heating was not performed because it was found that the Ar plasma removed any water vapor and organics that would interfere with the early stages of the AlN film growth. The AlN films were deposited with an AC chamber power of 3 kW using process gas flows of 40 sccm Ar and 20 sccm N₂. Films were deposited to thicknesses between 1 and 2 μm. Following the deposition, the film thickness was measured and θ/2θ and ω rocking curve scans were recorded. The post–deposition characterization was used to determine the parameters for later processing steps and to analyze data from fabricated devices.

Following the deposition and characterization of the AlN films, a photoresist mask was spun and patterned using contact lithography. The mask pattern was then transferred to the AlN layer using a chlorine plasma etch in a Panasonic E640 inductively–coupled plasma (ICP) etcher. A mixture of Cl₂ and Ar was used to create an anisotropic reactive ion etch (RIE). Any photoresist remaining after the RIE was removed in an 80 °C bath of Shipley microposit remover 1165.
The isolation layer, 1 μm of SiO₂, was deposited using a PlasmaTherm 790 plasma–enhanced chemical vapor deposition (PECVD) system. Generally, PECVD films are highly conformal. Conformality is necessary for a smooth transition between the piezoelectric and dielectric interface—a requirement for having an unbroken top contact layer across the interface. After deposition, another layer of photoresist was spun on and patterned. The pattern was then transferred into the isolation layer using CF₄/CHF₃ chemistry in the Panasonic E640; exposed AlN was selective to this etch and was undamaged. Remaining photoresist was removed in heated 1165.

The top contact layer was added prior to the etching and release of the cantilevers. Aluminum was chosen for the electrode material because its elastic modulus is low—Al thin films have a modulus of around 70 GPa—and Al has an inherent adhesiveness to AlN [9]. To pattern the Al contacts, a photoresist liftoff process was used. Negative photoresist was patterned using contact lithography and then 100 μm of Al was deposited using a CHA SEC600 electron beam evaporator. Following deposition, a 5 min soak in 80 °C 1165 removed all of the photoresist and Al not deposited directly onto the AlN and SiO₂ films.
Once the piezoelectric and dielectric layers were defined, the profile of the cantilevers was etched into the silicon substrate and subsequently released using a PlasmaTherm 770 SLR SiRIE fluorine etcher. To etch the cantilever structure into the silicon substrate, a time division multiplex (TDM) etch process was used. TDM etching, also called the Bosch process, is a three–step ICP cycle that can be modified to have excellent selectivity to photoresist and a high silicon etch rate [10]. The cantilever structure was etched 40 μm into the silicon substrate and the cantilever beams were released from the substrate by isotropically etching the wafer in a SF₆ plasma.

AlN cantilevers fabricated on a silicon wafer are shown in Figure 4.4. The upward curvature of the beams is a result of stress within the AlN layer. When the silicon bottom electrode is released from the substrate, the AlN film is no longer constrained by the bulk wafer and simply held by the bottom and top electrodes. The top electrode, Al, is compliant compared to AlN and silicon, so the curvature of the beam is primarily due to residual stress in the AlN film. The upward curvature of the beam indicates that the AlN film is in tension. This was a promising development, as MEMS like film bulk acoustic resonators (FBARs) require piezoelectric films that have zero stress or are slightly in tension with the substrate [11].
Figure 4.4: Released AlN cantilevers on silicon. The jaggedness of the features can be attributed to both the low feature resolution of the contact mask and thin film degradation during the isotropic plasma release. Despite these problems, however, the beams exhibited piezoelectric actuation. The bottom electrode of silicon can be seen in the thick dark line extending the length of the cantilever beam.

Many features of the cantilever structures were affected by processing, particularly the silicon etch steps. All of the chrome masks used for photolithography had address resolutions of 5 \( \mu \text{m} \), making the mask units very visible at higher magnifications. These rough edges were then transferred into the films and substrate during etching. Also, many features on the mask were improperly rastered. These problems were corrected on
later devices by using a different photomask manufacturer. Another degradation to the resolution of the features was that both the silicon deep etch and release steps were performed simultaneously. Although photoresist is selective to this etches, there was no resist mask remaining after the wafer was removed from the etcher, indicating that the resist was cleared at some point during the process. While SiO₂ and AlN are selective to these etches, Al is not, and Al was clearly etched during the silicon processing, albeit at a low rate.

**Testing of AlN/Silicon Cantilever Beams**

The cantilevers were tested using a multi-dimensional MEMS motion characterization system [12]. This system measures the Doppler shifts of a visible laser reflected off of a moving device. Because the laser is positioned perpendicular to the substrate surface, it is easiest to measure out-of-plane motion. Data from the vibrometer controller was recorded with an oscilloscope and signal analyzer for further analysis, including measurement of the frequency response and integration of the raw velocity data. Electrical contact to the cantilevers was made manually using a probe station. A waveform generator with 1 dB gain was used to pulse the cantilever beams.
The displacement of a cantilever beam at its resonance frequency is shown in Figure 4.5. Because of the cantilever dimensions, the resonant frequencies for these devices were all in the kHz range. Finite element modeling indicated that a cantilever with a resonant frequency above 1 GHz could theoretically be fabricated, but its dimensions were below the resolution obtainable from contact lithography.

![Figure 4.5: An AlN/Si cantilever at resonance.](image)

The cantilever beam, measuring 500 μm long and 25 μm wide, resonated at 8.86 kHz. The applied waveform was a 100 V sinusoid.

The cantilever beams were also tested off of resonance to observe motion in the quasi–static regime. Beams were actuated with a low frequency square wave to approximate the static behavior of the beam. At low voltages the
displacement was very small and the response time was short, as shown in Figure 4.6. At higher voltages ringing would occur, increasing the response time.

![Figure 4.6](image)

Figure 4.6: Sub–nanometer displacement of a piezoelectric cantilever. By sending a 1 V square wave pulse to the cantilever, the displacement of the cantilever tip was approximately 5 Å. This displacement was within the range of the vibrometry system.

### 4.3 Cantilever Beams on Titanium

Having successfully demonstrated the actuation of AlN cantilevers on a silicon substrate, it was determined that the next logical step was to fabricate similar structures on titanium substrates. Although a robust process for fabricating AlN cantilevers on Si had been developed, the change to a new substrate presented some new obstacles. First, at this time that this research
began, 4-inch titanium substrates were not available, so substrates were cut from polished sheets of titanium. Shearing of the samples introduced edge curvature that could interfere with contact photolithography. Second, the etch chemistry of Ti is different than Si, so new etch methods had to be characterized. Finally, unlike single-crystal Si, there was no known dry isotropic release, despite the considerable work done in that area [13].

Another motivation for fabricating cantilevers on Ti was provided by a study by Clement et al [14]. The results of their work suggest that c-axis collimation in AlN films does not necessarily result in the films having a strong piezoelectric response. A collimated film can have little to no piezoelectric response if the (002) grains grow in an anti-aligned manner such that the sum of the individual piezoelectric vectors is zero, as shown below in Figure 4.7. To that end, the actuation of titanium cantilevers could be used as proof that the films contained crystallites with aligned piezoelectric vectors.
**Figure 4.7:** A simplified schematic showing anti-aligned grain growth in a piezoelectric film. Despite the presence of strong collimation among grains, a film that grows in this manner will exhibit very little or no piezoelectric response. X-ray techniques, such as rocking curve measurements, cannot readily identify anti-aligned growth. A strongly piezoelectric film will have the polarization vectors all pointing in the same direction.

**FABRICATION OF CANTILEVER BEAMS**

The substrates were 500 μm thick Grade 1 polished titanium from Tokyo Stainless Grinding Co., Ltd. Using a shearing tool, sheets of titanium were cut into 1 in² pieces. Prior to AlN deposition, each piece was cleaned in ultrasonic solvent baths and rinsed in deionized water. After mounting the sample onto a carrier wafer with Apeizon H grease and loading the carrier into the Endeavor 8600, a pre-deposition 200 W Ar plasma treatment was used to partially remove the native oxide layer as well as any surface
contaminants. The sputtering parameters described in Chapter 3 were used to deposit the AlN film. This new recipe produced a film 1.3 μm thick with a grain alignment of 4.5°.

Another major change to the process flow was the elimination of a secondary dielectric layer. For the titanium cantilevers, the AlN film was patterned so that it formed both the piezoelectric cantilevers and the electrical isolation layer. The dielectric strength of AlN is comparable to SiO₂ and SiNx, so the removal of an additional dielectric layer was reasonable [15]. Additionally, the later acid release step requires the dielectric layer to be resistant to highly–concentrated hydrofluoric acid; neither SiO₂ nor SiNx are suitable materials for that process. A photoresist mask was patterned using contact photolithography, and then the AlN film was etched in a Panasonic E640 ICP etcher with a mixture of chlorine and argon for an anisotropic RIE. Following the AlN etch, the cantilever pattern was transferred into the titanium substrate. Because titanium is also highly susceptible to chlorine plasmas, the E640 was used, albeit with a different gas ratio and modified applied power. The selectivity of the AlN and titanium etches to photoresist was high enough that the same photoresist layer was used for both etches.
Various strategies for releasing the cantilevers were tested. Initially, research efforts focused on characterizing an isotropic dry etch similar to the release etch commonly used in the fabrication of silicon–based MEMS. The ability to undercut a deep–etched titanium feature had already been demonstrated, but a process for releasing features with dimensions consistent with MEMS structures remained elusive [16, 17]. Two approaches to finding a suitable dry etch were considered: a single step etch with process conditions that would render the etch isotropic or a cyclic oxidation and oxide removal process. An Applied Materials Centura DPS+ etching chamber was used to test an isotropic plasma release etch. Like the Panasonic E640, the Centura DPS+ is an ICP/RIE chamber, though the DPS+ chamber used was specifically for etching metals. By varying the source and bias powers, the gas flows, and the chamber pressure, features with 5 μm linewidths were partially released. Unfortunately, this process was neither stable nor repeatable, and was abandoned. XeF₂ etching of titanium was also attempted. Patterned and deep–etched cantilever beams were exposed to a XeF₂ ambient in an XACTIX e1 Series silicon etch system. Some samples had been sputtered with 1 μm of titanium in an Endeavor 8600 DC chamber prior to the AlN film deposition. The rationale behind the sputtered layer was that previously reported etching
of titanium in XeF$_2$ was only for titanium thin films deposited by electron beam deposition. Because the etching mechanism is not well-understood and there are significant differences between bulk and thin-film titanium, it was suspected that bulk titanium might not etch. In fact, neither the sputtered nor the bulk titanium exhibited any measurable etch after an hour-long exposure to 4 Pa of XeF$_2$.

It was suspected that the native titanium oxide was acting as a barrier to the XeF$_2$ gas. Because the samples were exposed to atmosphere for many days between the titanium deep etch and XeF$_2$ etch steps, a complete native oxide would have grown on any exposed titanium surface. In a final attempt to demonstrate bulk titanium etching in XeF$_2$, cantilevers were patterned as above. Prior to the gas etching, though, the pieces were exposed to a 200 W argon RF plasma for 5 min to sputter off the native oxide, then 1 μm of tungsten was sputtered onto the surface and sidewalls in an Endeavor 8600 RF chamber. The tungsten was to act as a conformal layer protecting the titanium from any oxide growth. In a XeF$_2$ ambient, it was expected that the tungsten layer would quickly etch away, exposing bare titanium that would etch at a measurable rate. Unfortunately, the samples did not etch during a 1 hr exposure to 4 Pa of XeF$_2$. It is hypothesized that the sputtering targets
contained a significant amount of oxygen impurities residual from reactive sputtering. The films sputtered then contained non-stoichiometric metal oxides that interfered with the chemistry of the XeF₂ etch.

Because a dry release method could not be found, wet etch processing was pursued. Wet etches are by nature isotropic except in certain crystallographically controlled etches, such as silicon etching in KOH. Titanium etches readily in many acids but has the highest etch rate in strong HF solutions. A solution of 49% HF yielded an isotropic etch rate of approximately 30 μm/min. Because an underlayer of titanium was required to provide the bottom electrical contact, the titanium deep etch was characterized to give sidewalls that were as reentrant as possible. Reentrant sidewalls meant that the wet etch would clear through the thinnest exposed area first, thereby releasing the cantilevers from the substrate yet still leaving some titanium on the underside of the beam.

After releasing the unimorph beams from the substrate, the final step was the deposition of the top contact layer. Aluminum was used because it had proven to be a very robust and adhesive material for the silicon cantilevers. 300 μm of Al was deposited in a CHA SEC600 electron beam evaporator. The undercut profile that resulted from the HF acid etch made it possible to
simply deposit the aluminum over the entire substrate without any subsequent liftoff or metal etch; a blanket deposition did not result in electrical shorting between the top and bottom electrodes.

On the mask used to pattern the AlN layer, the cantilever beams all had the same planar dimensions: 5 μm wide by 60 μm long. However, the thickness and geometry of the release beams was not completely uniform due to three factors. First, reactive sputtering produces films with a radially-dependent thickness profile; the thickness of the AlN layer in a cantilever beam changes depending upon its position on the wafer. Second, the anisotropic titanium deep etch exhibited a variation in depth across the surface of the titanium substrate. The etch rate was faster in the center of the piece and also more reentrant; prior to release, a beam in the center had both a greater height and inward-sloped profile. Second, the etch rate of titanium during an acid dip is grain-dependent. Different crystal faces of titanium etch at different rates. As a result of these variations, released beams had dissimilar geometries, leading to a range of frequency responses and beam displacements during testing. A fabricated cantilever beam is shown in Figure 4.8.
Figure 4.8: An AlN cantilever beam on a titanium substrate. The SEM was taken prior to the metallization step. The isotropic profile of the HF acid etch can clearly be seen.

TESTING OF CANTILEVER BEAMS

The AlN cantilever beams on titanium were tested using the same multi-dimensional MEMS motion characterization system described in Section 4.2. The first measurements taken were quasi-static displacement measurements. The cantilevers were pulsed with square waveforms at low frequencies; this approximated a measurement of the static displacement. Figure 4.9 shows the
response of three cantilevers to a square pulse. The response time is less than 10 μs. Although most of the cantilevers measured did not exhibit ringing—e.g. beams A and B—a few showed strong ringing when pulsed; beam C is included in the figure as an example. The cause of this ringing was unknown.

![Image](image_url)

**Figure 4.9: Quasi–static displacement of cantilever beams.** By pulsing the beams with at 10 kHz with a 10 V_{pp} square wave, static displacement was approximated. Although most cantilevers had equivalent responses (A & B), some cantilevers exhibited very strong ringing when pulsed (C).

To calculate the effective piezoelectric coefficient $d_{31}$, the profile of a cantilever beam was measured, as shown in Figure 4.10. Using a method similar to the measurements in Figure 4.9, a beam was pulsed with a low frequency square wave. Repositioning the laser and measuring the displacement at different
length coordinates and measuring the displacement created a static beam profile. The tip deflection was 29.8 nm.

![Displacement profile of a cantilever beam.](image)

To obtain a theoretical result for the tip deflection, reported values for single crystal AlN were used to create a FEM in ANSYS. (A detailed explanation of FEM for piezoelectric transducers appears below.) The geometry of the model was created using SEM images of cantilever beams. By using appropriate boundary conditions—anchoring one end of the beam and placing a 50 V potential across the AlN film—the solution to the model returned a
displacement map, plotted above in Figure 4.10. The tip deflection of the model was 31.9 nm. By comparing the displacement map from the model with the experimental data, the calculated effective $d_{31}$ was $-2.49 \text{ pm/V}$, 94% of the theoretical value. This compares very well with effective $d_{31}$ of 97% for highly aligned AlN on silicon substrates [18].

Figure 4.11: ANSYS simulation of the static displacement of a cantilever beam. Using a simple multiphysics analysis, the theoretical displacement of the cantilever beam was obtained.
Following the static analysis, frequency response curves were measured for different beams. These curves were used for the dynamic analysis below and to determine the variation between beams. These measurements are displayed in Figure 4.12. An interesting feature of the cantilever response was that the cantilevers exhibited nonlinear behavior at resonance. The resonant tip displacement of the beams was between 10 and 12 μm, over 15% of the beam length. At large displacements, nonlinear behavior begins to appear; these nonlinearities will be discussed in depth below. It was clear that the shape of the response curve was affected by this behavior and would need to be considered when modeling the beam response.
Using the resonant response data, the quality factor $Q$ of the cantilever beams was calculated. These results are plotted in Figure 4.13. $Q$ increases with the resonant frequency except for beams over 1 MHz. This was due to the frequency range of the spectrum analyzer; there was very sharp rolloff in signal detection that occurred above 1 MHz.
Figure 4.13: Q of the cantilever beams. The quality factor tended to increase with resonant frequency, although the beams above 1 MHz do not follow this trend. This was due to a limitation of the spectrum analyzer.

4.4 CANTILEVER BEAM MODELING

Modeling of the dynamic response of the cantilever beams occurred in three steps. First, a finite element method was used to determine if the resonant frequency of the beam was strongly affected by the nonlinear behavior. Next, an analytical model was used to incorporate the piezoelectric and damping losses that were missing from the FEM model. Finally, a numerical solution to Newton’s law for the system was used to model the nonlinear behavior of the beam and extract the electromechanical coupling coefficient.
A Finite Element Model

FEM meshes a continuous medium—in this case, the cantilever beam—into discrete elements connected by nodes. Boundary conditions and loads can be applied to this mesh. Appropriate mathematical formulas are then used to solve the system of differential equations that governs the behavior of the mesh. To properly describe the deformation of a piezoelectric cantilever beam under an applied electric load, elements must be used that reflect the constituent equations that govern the material behavior of all layers: a basic 3–dimensional stress–strain element can be used for the top and bottom electrodes, but an element that additional reflects structural changes due to the presence of an electric field is needed for the AlN thin film. This type of coupled analysis is available with FEMLAB software operating in multiphysics mode. The dynamic frequency response can be found using an iterative process that identifies the eigenmodes of the cantilever beam, then FEMLAB solves the system of equations over an appropriate frequency range. By using SEM measurements of the fabricated cantilevers, a three–dimensional geometric model was created. The model had three subdomains: the titanium bottom contact complete with isotropic etch profile, the AlN
film, and the aluminum top contact. The contact layers were meshed with \textit{solid3} elements and AlN layer was meshed with a \textit{piezo3D} element. The mesh—containing 1340 elements with a total of 22591 degrees of freedom—provided adequate resolution of the structure while being solvable within a reasonable amount of time. In defining the AlN layer, the reported matrices for elasticity and piezoelectricity were used along with the experimental values for \( d_{31} \) and the dielectric constant, reported in Chapter 3. The response of the beam was then modeled by defining the appropriate boundary conditions for one end of the beam and setting a voltage load across the piezoelectric subdomain. In determining the frequency response of the cantilever beams, the first step was to identify the resonant frequencies of the beam. Shown below in Figure 4.14 are the first six eigenmodes of a piezoelectric cantilever on titanium. While the fundamental resonance is a bending of the beam in the z direction, most higher order resonant frequencies correspond to other bending modes.
Figure 4.14: Eigenmodes of titanium piezoelectric cantilevers. The colors represent the total displacement of the beam from its undeformed shape; light blue is no deflection while bright purple is the maximum deflection. The fundamental resonance occurs at 927 kHz and is pure bending in the z direction (A). At 1.54 MHz, the beam bends purely in the xy plane (B). The second harmonic in the z direction occurs at 5.71 MHz (C). The beam bends in the xy plane with a slight bend in z at 9.34 MHz (D). A pure twist of the beam occurs at 12.21 MHz (E). The second mode of (D) is at 25.04 MHz (F).

Having identified the eigenfrequencies, a simulation of a frequency sweep about the first resonant frequency was compared with the experimental results. The theoretical and measured frequency response curves are plotted together in Figure 4.15. Two results are clear: the nonlinearity of the beam shifts the resonance peak and alters the response curve considerably; and the
model does not account for all of the damped response, yielding a peak displacement much higher than the experimental result.

Figure 4.15: Calculated frequency response from FEMLAB and the experimental response. The FEMLAB model produced a resonance peak near the correct frequency but did not account for all of the damping nor the nonlinear shape.

AN ANALYTICAL MODEL

Many models for predicting motion in piezoelectric actuators are reported in the literature. Although most models are relatively simple and not valid at frequencies near resonance or large tip displacements, a more refined model for a complete piezoelectric unimorph including the top and bottom contacts and a substrate layer was developed by Brissaud et al [19]. This model can
predict the response of the piezoelectric beam over the entire dynamic range.

The model is a powerful analytical tool—it has been used to find the
frequency response of a cantilever beam, the electrical admittance and
electromechanical coupling, and the open circuit voltage across the
electrodes.

Consider a fixed–free cantilever beam with a length $L$ much greater than
width $b$ and thickness $h$. These constraints lead to two assumptions: the in
plane stress $T_x$ and deformation $u_y$ are zero; and the thickness displacement
$u_z$ and stress $T_z$ are also zero. Using these assumptions the constitutive
piezoelectric equations reduce to:

\[
T_{x,p} = c_{11}^E S_x - e_{31} E_z \\
D_z = e_{31} S_x + e_{33}^S E_z.
\]  
(4.1)

Using Hooke’s law, the bending displacement of the beam is

\[
w = W(x) e^{\text{int}}. \tag{4.2}
\]

By defining a geometry as shown in Figure 4.16 and calculating the stress
in a beam element with length $dL$, the neutral $xy$ plane of the beam occurs at
the height

\[
z_0 = \frac{Y_{c1} h_{c1} (h_{c2} + h_z) + c_{11}^E h_p (h_{c2} + 2h_{c1} + h_z) + Y_{c2} h_{c2} (h_{c2} + 2h_p + 2h_{c1} + h_z)}{2 (Y_{c1} h_{c1} + Y_{c2} h_{c2} + c_{11}^E h_p + Y_{c2} h_{c2})}. \tag{4.3}
\]

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At \( z_0 \) the stress \( T_x \) is zero; in this coordinate system \( T_x \) is positive above \( z_0 \) and negative below \( z_0 \).

![Figure 4.16: The geometry used to model a piezoelectric unimorph.](image)

The model defines the four structural beam layers and determines the neutral plane position to accurately describe the cantilever motion. (Adapted from [19].)

The total bending moment of the cantilever beam \( M_F \) is

\[
M_F = \int_{z_0}^{z_{\text{ue}}} zT_{x,s} \, dz + \int_{z_0}^{z_1} zT_{x,s1} \, dz + \int_{z_1}^{z_2} zT_{x,s2} \, dz + \int_{z_2}^{z_{\text{ue}}} zT_{x,s3} \, dz
\]

\[
= M_s + M_{s1} + M_p + M_{s2} + M_{\text{piezo}}
\]  

(4.4)

From the constitutive equations, the stress in the piezoelectric layer is a function of both the strain and the electric field; this is the origin of \( M_{\text{piezo}} \).

The first four terms are the structural moments of the individual layers of the cantilever. The total structural moment \( M_G \) is then
\[ M_G = M_s + M_{el} + M_p + M_{e2} \]
\[ = -G_s \frac{\partial^2 W}{\partial x^2} - G_{el} \frac{\partial^2 W}{\partial x^2} - G_p \frac{\partial^2 W}{\partial x^2} - G_{e2} \frac{\partial^2 W}{\partial x^2}, \]
\[ = -G \frac{\partial^2 W}{\partial x^2} \quad (4.5) \]

where \( G \) is the global flexural rigidity for the entire beam [20]. By neglecting the influence of the electrodes, \( G \) reduces to

\[ G = Y_s \left( \frac{h_z^3}{12} + h_z^2 z_0 \right) + c_{i1} \left( \frac{h_z^3}{3} + h_e^2 \left( \frac{h_z}{2} - z_0 \right) + h_t \left( \frac{h_z}{2} - z_0 \right)^2 \right) \quad (4.6) \]

\( M_{piezo} \) is the bending moment due to the applied electric field. It is this term that initiates beam deflection, and is defined as:

\[ M_{piezo} = e_{31} E_z K \]
\[ = e_{31} E_z \left( \frac{h_p}{2} \left( h_z + 2h_{el} + h_p - 2z_0 \right) \right) \quad (4.7) \]

Having defined all of the moments of the cantilever, the differential equation for the out–of–plane displacement given by Euler–Bernoulli beam theory can be written

\[ G \frac{\partial^4 W}{\partial x^4} + \rho_{tt} \frac{\partial^2 W}{\partial t^2} = 0, \quad (4.8) \]

where \( \rho_{tt} \) is the mass per unit surface of the cantilever. Combining equations (4.2) and (4.8) yields

\[ \text{127} \]
\[
\frac{\partial^4 W}{\partial x^4} + \alpha^4 W = 0, \quad (4.9)
\]

where \( \alpha = \sqrt{\frac{\rho_n \omega^2}{G}}. \) The general solution to equation (4.9) is

\[
W(x) = A \cosh \alpha x + B \sinh \alpha x + A' \cos \alpha x + B' \sin \alpha x. \quad (4.10)
\]

There are appropriate boundary conditions that can be applied to a fixed–free beam: \( W(0) = 0; \) \( \frac{dW(0)}{dz} = 0; \) \( M(L) = 0; \) and \( \frac{\partial^3 W(L)}{\partial^3 x} = 0. \) These boundary conditions can be used to solve for the coefficients \( A, B, A', \) and \( B', \) and equation (4.10) becomes

\[
W(x) = -\frac{M_p}{\alpha^2 G} \left[ \frac{\cosh \alpha L + \cos \alpha L}{2(1 + \cosh \alpha L \cos \alpha L)} \left( \cosh \alpha x - \cos \alpha x \right) \right. \\
\left. - \frac{\sinh \alpha L - \sin \alpha L}{2(1 + \cosh \alpha L \cos \alpha L)} \left( \sinh \alpha x - \sin \alpha x \right) \right]. \quad (4.11)
\]

The resonance condition is satisfied whenever the denominator of equation (4.11) goes to zero; the fundamental resonant frequency of the cantilever beam is given by

\[
\frac{0.5598}{L^2} \sqrt{\frac{G}{\rho_n}}. \quad (4.12)
\]
The tip displacement obtained from equation (4.11) is plotted versus frequency in Figure 4.17. Also included in the plot is the experimental frequency response of a titanium piezoelectric cantilever.

![Graph showing tip displacement versus frequency](image)

**Figure 4.17: Analytical modeling of a piezoelectric unimorph.** Using the imaginary coefficient method described by Brissaud, the turquoise line of the analytical model is fitted relatively well to the experimental data. The nonlinearity is not modeled.

Clearly, the analytical model provides a better fit than a basic FEM model, but the nonlinear response is not modeled. Another limitation of the analytical model is that mechanical and electrical dampings are introduced simply by using complex values for the elastic and piezoelectric coefficients. For the analytical curve in Figure 4.17, the elastic constant $c_{11} = 410 - 6.42i$ GPa.
and the piezoelectric coefficient $d_{31}=-5.9+0.0059i$ pm/V. The piezoelectric coefficient obtained was in accordance with static displacement calculations and within 10% of the expected value [21]. Further analysis of this model has shown that without imaginary components there cannot be in agreement with experimental results [22]. The imaginary component is then assumed to incorporate all of the relevant loss mechanisms. Despite this, the model quite accurately predicts the real component of the material properties. Still, the nonlinear response was not modeled by the imaginary loss coefficients.

**ANALYSIS OF THE CANTILEVER SPRING SOFTENING**

As it was obvious that the cantilevers exhibited nonlinear spring softening when driven at the fundamental resonance with a large voltage, a new method for finding a theoretical basis for the experimental data was needed. The spring softening evident in the frequency response of the piezoelectric cantilevers was due to cubic nonlinear damping [23]. For the undamped forced oscillations of a dynamic system, the equation of motion can be written

$$\ddot{x} + \omega_0^2 x + \alpha x^3 = P \sin \omega t$$  \hspace{1cm} (4.13)
The phase angle between the forcing function and the periodic response $x(t)$ is always $0$ or $\pi$. The addition of a linear damping term $c\dot{x}$ will result in intermediate phase angles [24]. Considering a piezoelectric cantilever as this dynamic system, the complete equation of motion is then Duffing’s equation:

$$\ddot{x} + c\dot{x} + \omega_0^2 x + \alpha x^3 = P\sin \omega t \ [25].$$

(4.14)

Rather than using the forcing function $P\sin \omega t$ and $x_i = A\sin(\omega t - \phi)$ for the periodic solution, the preferred form of equation (4.14) is

$$\ddot{x} + c\dot{x} + \omega_0^2 x + \alpha x^3 = P_1 \sin \omega t + P_2 \cos \omega t$$

(4.15)

with $P^2 = P_1^2 + P_2^2$ and $x_i = A\sin \omega t$. By using the method of harmonic balance and neglecting the higher order $\sin 3\omega t$ term, the solution to (4.15) results in

$$\left(\omega_0^2 - \omega^2\right)A + \frac{3}{4} \alpha A^3 = P_1,$$

$$c\omega A = P_2,$$

(4.16)

from which

$$P^2 = P_1^2 + P_2^2 = \left(\left(\omega_0^2 - \omega^2\right)A + \frac{3}{4} \alpha A^3\right)^2 + (c\omega A)^2$$

(4.17)

is obtained [24]. Although this equation can be directly solved for $A$, the process is cumbersome and not particularly useful because the damping coefficients and spring constant are unknown. Instead, equation (4.17) was
solved in Mathematica using a numerical solver in conjunction with an iterative nonlinear fitting algorithm.

Figure 4.18 below shows the result of this analysis on the measured frequency response of four cantilever beams. The beams demonstrated fundamental resonances at angular frequencies between $5.04 \times 10^6$ and $6.30 \times 10^6$ rad. The experimental data was imported into Mathematica for nonlinear fitting with equation (4.17).

![Figure 4.18: The nonlinear dynamic response at large displacement of four AlN cantilevers on titanium. Using a numerical solution to Duffing’s equation, the frequency response of the cantilever beams could accurately be modeled and mechanical constants extracted.](image-url)
The coefficients of the fit $c', \alpha', F'$, and $\omega_0$ are related to the real mechanical coefficients $c$, $\alpha$, $F$, and $k$ through the relations $c' = \frac{c}{m_{\text{eff}}}$, $\alpha' = \frac{\alpha}{m_{\text{eff}}}$, $F' = \frac{F}{m_{\text{eff}}}$, and $\omega_0 = \sqrt{\frac{k}{m_{\text{eff}}}}$, where $m_{\text{eff}} = 0.236m$ for a cantilever beam [25]. The actual masses $m$ of the cantilever beams were calculated directly from the FEMLAB model. Additionally, using the equation

$$F = d_{31} \gamma \text{piezo} h_{\text{piezo}} V$$

(4.18)

a piezoelectric coefficient $d_{31}'$ was calculated. This value, however, is not the true effective piezoelectric coefficient as calculated above. Recall that this analysis used a spring, mass, and damper system with the addition of a cubic nonlinear term. This was not a model; rather, it was essentially a parameter fitting method, and did not account for physical properties like the dielectric constant and assumed that all of the applied voltage is used to generate piezoelectric force. However, AlN is a capacitor and energy from the electric field is stored in the film as potential energy. The coefficient $d_{31}$ calculated from equation (4.18) is actually a piezoelectric constant multiplied by a factor that accounts for the relative coupling of the electric field to the piezoelectric motion: the piezoelectric coupling coefficient $k^2$. By using the piezoelectric
coefficient calculated from the static displacement, \( k^2 \) was calculated and the disparity between the applied field and the piezoelectric response was attributed to this electromechanical coupling [26].

<table>
<thead>
<tr>
<th>Beam</th>
<th>( f_0 ) (kHz)</th>
<th>( Q )</th>
<th>( c ) (N/m)</th>
<th>( \alpha ) (kg/m²/s²)</th>
<th>( k ) (kg/s²)</th>
<th>( F ) (N)</th>
<th>( k^2_{31} )</th>
</tr>
</thead>
<tbody>
<tr>
<td>A</td>
<td>808.0</td>
<td>107</td>
<td>(-2.61 \times 10^{-8})</td>
<td>(-5.62 \times 10^9)</td>
<td>14.9</td>
<td>(1.37 \times 10^{-6})</td>
<td>4.04%</td>
</tr>
<tr>
<td>B</td>
<td>808.8</td>
<td>124</td>
<td>(-2.15 \times 10^{-8})</td>
<td>(-5.39 \times 10^10)</td>
<td>15.0</td>
<td>(1.45 \times 10^{-6})</td>
<td>4.28%</td>
</tr>
<tr>
<td>C</td>
<td>925.2</td>
<td>185</td>
<td>(-1.99 \times 10^{-8})</td>
<td>(-5.97 \times 10^10)</td>
<td>21.7</td>
<td>(1.51 \times 10^{-6})</td>
<td>3.77%</td>
</tr>
<tr>
<td>D</td>
<td>1006.5</td>
<td>155</td>
<td>(-2.07 \times 10^{-8})</td>
<td>(-6.54 \times 10^10)</td>
<td>28.1</td>
<td>(1.75 \times 10^{-6})</td>
<td>3.78%</td>
</tr>
</tbody>
</table>

**Figure 4.19: Mechanical properties of the cantilever beams.** By fitting the measured frequency response of the beams to the solution of Duffing’s equation, the mechanical coefficients of the beam were extracted.

The results in Figure 4.19 show that the mechanical constants of the cantilever beams did not vary appreciably, with the exception of \( k \). The large variation in spring constant was attributed to the variations in thin film thickness and beam geometries—the same factors that caused the spread in resonant frequencies. For thin film single crystal AlN, the reported value of \( k^2_{31} \) is 9% [27]. However, unlike \( d_{31} \), \( k^2 \) is significantly affected by even minor changes in
the columnar alignment of the film; the experimental value is in agreement with data for polycrystalline AlN films on silicon [26].

**SUMMARY**

AlN thin films were used to successfully fabricate and test the first example of piezoelectric actuation of MEMS on bulk titanium. Piezoelectric unimorphs were tested under static conditions to calculate the effective piezoelectric constant $d_{31}$, and under dynamic conditions in order to calculate the electromechanical coupling and other mechanical constants of the cantilever beam. Under large displacements, the beams demonstrate a marked nonlinear response in the form a cubic spring softening. The frequency response of the beams, including the nonlinearity, was successfully modeled using a combination of finite element, analytical, and numerical analyses. From the calculations, the piezoelectric coefficient was 94% of the reported value for single crystal AlN; the electromechanical coupling coefficients obtained were approximately half of the reported single crystal value.
REFERENCES


RF Acoustic Wave Devices on Titanium
This chapter discusses the fabrication and operation of the first surface acoustic wave (SAW) MEMS on bulk metal substrates. Following an introduction to acoustics in a piezoelectric medium, including the generation and propagation of transverse Rayleigh waves, fabrication and processing methods of SAW filters are presented. Results from the testing of the devices follow, including comparisons to SAW filters on other substrates.

5.1 Piezoelectric Waves

Motion is propagated in a solid whenever a force is applied. The internal response of the solid is dependent on many variables, including the magnitude and shape of the force, the structure of the material, and the internal forces that bind the solid. In a crystal, interatomic forces bind atoms together such that the atoms can be modeled as a collection of point masses interconnected by springs, and Newton’s law can be used to find the equations of motion. The solutions to these equations of motion are waves;
motion propagates through a crystal by periodic disturbance of the crystal lattice.

As described by the constituent piezoelectric equations, applying an electric field to a piezoelectric crystal creates a force on the lattice atoms that results in a strain of the atomic displacements. Consequently, a time–varying field generates time–varying displacements—acoustic waves. Consider a piezoelectric thin film on a substrate, as shown in Figure 5.1. A periodic series of electrodes—an interdigitated transducer (IDT)—on top of the film can be used to generate a time–varying electric field. Though there are many kinds of acoustic waves that can be generated by electric fields, these fall into two general categories: surface waves and bulk waves. The generation of bulk waves is governed by the piezoelectric coefficient $d_{33}$, the electromechanical coupling $k_{33}$, and the crystal properties of the 3–direction; the strength of surface waves is determined by $d_{31}$, $k_{31}$, and the crystal properties in the 1– and 2–directions. In basic SAW devices, the propagation of the transverse Rayleigh wave, shown in Figure 5.1(B), is the desired operating mode, with other surface and bulk waves contributing to loss in the filter response [1]. A SAW filter is made using two IDTs. The first transducer serves as the emitter, into which the time–varying electric field is sent. The second IDT, the
collector, is in propagation path of the Rayleigh wave sent from the first IDT. As the wave passes through collector, the piezoelectric wave creates a time-varying electric field across the fingers of the IDT fingers, and as a result a signal is transmitted from the emitter to the collector. The frequency at which the signal is sent from the emitter is given by

$$f_0 = \frac{v}{\lambda_{IDT}},$$

(5.1)

where $v$ is the acoustic velocity of the piezoelectric film. Because the signal is only sent when the condition in (5.1) is met, the device acts as a bandpass filter. For a more in–depth analysis of piezoelectric waves, see [2].
Figure 5.1: The generation of surface acoustic waves in AlN. An interdigitated transducer is fabricated on top of an AlN thin film, with the substrate serving as the ground plane (A). Electric field lines create thickness variations, causing the emission of transverse Rayleigh waves (B). Alternately, the electric fields can couple to in-plane strains, creating regions of expansion and compression and resulting in longitudinal wave emission (C).

The transmission properties of a SAW filter can be used to extract material properties of the AlN film. By measuring the transmission time between the emitter and collector IDTs, the acoustic velocity of the Rayleigh wave on the AlN film surface can be calculated. Deposition of a metal film onto the film surface between two IDTs results in a short-circuiting of the traveling electric fields, slowing the SAW [3]. By measuring the slowed acoustic velocity $v_m$, the
electromechanical coupling coefficient $k_{31}^2$ can be determined through the relation

$$k_{31}^2 = \frac{2(v_0 - v_m)}{v_0}$$

(5.2)

This relation is only valid when there is no conductive loss due to the substrate [4]. As will be discussed below, AlN SAW filters on titanium substrates are highly prone to large electrical and acoustic losses.

5.2 FABRICATION OF SAW FILTERS ON TITANIUM

The first step in the fabrication of a basic interdigitated transducer (IDT) is the deposition of a piezoelectric film onto a titanium substrate. Prior to deposition, titanium wafers were cleaned for 5 min in high power ultrasonic baths of acetone, isopranol, and deionized water, and then baked in atmosphere at 200 °C for 10 minutes. Substrate cleaning is very important during SAW device fabrication because any particles or residue will cause disruptions in the profile of the film; a smooth surface is crucial not only to the propagation of surface waves, but also to the minimization of SAW attenuation through effects like BAW generation and absorption by defects in the crystal lattice. The process for sputtering highly piezoelectric AlN onto
titanium is described in Chapter 3; using that procedure, AlN films with thicknesses between 1 and 2 μm were deposited on 4-inch titanium wafers.

Following the piezoelectric film deposition, a contact mask process was used to fabricate the IDTs. From equation (5.1), for AlN SAW filters that operate in the GHz range, the IDT linewidths and spacings often require the use of electron beam photolithography. However, in the MHz range standard contact lithography can be used. The contact mask used for this process had been previously used successfully to fabricate AlN SAW filters on silicon [5]. A liftoff process using AZ 5214E photoresist was used to define the IDTs. After patterning the resist, 100 nm of aluminum was deposited using electron beam evaporation and liftoff was achieved in a sonicated acetone bath. An IDT fabricated on titanium is shown below in Figure 5.2. IDTs were also fabricated using a similar process on an AlN film deposited on a silicon wafer; for comparative purposes an image of this device is also included in Figure 5.2. The most marked difference between the two SAW filters is that the surface roughness is much greater on the titanium wafer. Using high-resolution optical profilometry, the roughness of the AlN film on titanium was measured as 7.32 nm. For the AlN film on silicon, this value was only 1.10 nm. Because transverse Rayleigh waves are easily disrupted by defects
on the order of the SAW wavelength, this would have a negative influence on the response of the filter.

![Figure 5.2: Optical profilometer images of IDTs on AlN. Identical SAW filters were fabricated on titanium (A) and silicon (B). The fingers are 10 μm wide with 10 μm spacings and are grouped together in pairs; the excitation wavelength of the IDT is 40 μm.](image)

### 5.3 SAW Filter Response

Traditionally, SAW filters are fabricated from thick piezoelectric films on highly–resistive single crystal substrates. Devices fabricated in this manner exhibit characteristics necessary for good filtering: the SAW travels solely in the film, mechanically uncoupled to the substrate; there is little to no electrical loss into the substrate; and the substrate roughness is very low, causing little disruption to the motion of acoustic waves. In these filters, the electromechanical losses are very low and the filter response is high. For SAW filters fabricated on titanium substrates, however, the electrical and
mechanical properties of traditional platforms most certainly do not apply. Perhaps the most important difference is the presence of a conducting substrate. When conductive, inductive, and capacitive losses occur between the input and output IDTs, the insertion loss—the out-of-band transmission—increases and the filter response—the in-band transmission—decreases [6]. A resistance change of from 2000 Ω cm to 10 Ω cm increases the insertion loss by three orders of magnitude and drastically reduces the in-band response [4]. The thinness of the AlN film and the mechanical properties of the titanium substrate contribute additional losses. Efficient Rayleigh wave SAW filters are generally constructed either with thick piezoelectric films, distancing the surface wave from the film–substrate interface and preventing acoustic loss into the substrate; or with thin films on low-loss substrates such as sapphire, SiC, or diamond [7]. Neither of these conditions applies to the AlN filters fabricated on titanium. The AlN film is less than 2 microns thick, causing considerable acoustic coupling to the titanium substrate. The acoustic velocity of titanium is 4140 m/s, even lower than silicon, which exhibits high acoustic losses [8]. Also, the acoustic velocity in titanium is lower than in AlN, so there is no confinement within the piezoelectric thin film and it does not act as an acoustic waveguide [9].
Finally, the surface roughness of the titanium wafer is at least an order of magnitude above traditional SAW filter substrates; surface roughness provides a strong loss mechanism for transverse Rayleigh wave propagation [10]. Despite all of these inefficiencies, SAW excitation and transmission was demonstrated in the AlN films.

SAW filter response was measured using an Agilent E8394A PNA Series network analyzer. This network analyzer is capable of time–gated spectrum analysis, a technique that allows the coupling of time–domain filtering to the frequency response across an RF spectrum [11]. Because so much of the signal loss occurs at very high speed—e.g. conduction between the input and output IDTs—by measuring the frequency response in the time domain and filtering out the unwanted conduction, the true frequency response of a SAW filter on a metal substrate can be obtained [12]. Figure 5.3 shows the time domain frequency response of a SAW filter with a spacing of 3.656 mm between the IDTs. The peak transmitted power occurs shortly after 0 ns, indicating electromagnetic feedthrough between the input and output IDTs. The SAW transmission is the broad, flat peak centered at 820 nm. Neglecting the effect of the titanium substrate, the SAW will travel at the acoustic velocity of AlN, reported as 5600 m/s for single crystal AlN [5]. The time domain peak
indicates that for this filter, the acoustic velocity is 4430 m/s, 80% of the single crystal value. The delayed response indicates that some of the acoustic wave is leaking in the titanium substrate. Because AlN film is only 1.5 μm thick, there is strong coupling between the film and the titanium substrate; the acoustic velocity of titanium slows the SAW.

![Image](image)

**Figure 5.3: Time domain analysis of the SAW filter response.** Although a considerable amount of power is lost to electromagnetic feedthrough, the response of the SAW filter can be detected using time domain analysis, seen here occurring at a time centered around 825 ns.

Having measured the time domain response of the SAW filter, time–gating can be used to filter the electromagnetic feedthrough. Figure 5.4 plots the time–gated and ungated frequency response of a SAW filter on titanium. Without time–gating, the transmitted power at each frequency reflects the
large electrical losses and the SAW response is washed out; a small distortion at the resonant frequency is the only indication that there is any response from the filter. However, with time–gating enabled the conductive losses are eliminated from the spectrum and the $Sinc(f)$ response of the SAW filter is obtained.

![Gated and ungated frequency response curves for an AlN SAW filter on titanium.](image)

*Figure 5.4: Gated and ungated frequency response curves for an AlN SAW filter on titanium.* The ungated response appears as small peaks, due to high electrical losses from the metal substrate and between the IDTs. By using a time domain gate to filter out these losses, the distinctive sinc function response of a SAW filter is realized.

After a transverse piezoelectric wave has been generated, there is power attenuation during propagation due to loss mechanisms such as scattering.
from surface distortions and impurities. By performing the frequency analysis described above on SAW filters with different resonant frequencies and IDT spacings, the attenuation in power was observed. Because power loss is linearly dependent on the distance the wave travels, the attenuation $\alpha$ can be obtained for each frequency measured and is plotted in Figure 5.5.

![Figure 5.5: Attenuation in AlN SAW filters.](image)

After measuring the attenuation in the AlN films, the data could be compared to attenuation in AlN films deposited on silicon. Using previously reported values for SAW filters on silicon, Figure 5.6 compares the power attenuation on titanium and silicon. The attenuation for AlN on titanium is an order of
magnitude above attenuation in AlN on silicon. This is an expected result considering the much higher surface roughness on titanium and the higher rocking curve FWHM values, both translating into higher loss during SAW travel. A power law fit of the titanium data showed attenuation in AlN on titanium to be a function of $f^{2.6}$, only slightly higher than the $f^2$ dependence for AlN films on silicon.

![Graph showing AlN SAW attenuation on titanium and silicon.](image)

**Figure 5.6: AlN SAW attenuation on titanium and silicon.** Data taken from SAW filters on titanium was compared to previously reported results for AlN filters on silicon [13]. The line plotted is the theoretical dependence of attenuation on $f^2$ for silicon, also taken from [13].

The SAW filter response was compared to an analytical model originally developed to account for parasitic electrical losses in AlN SAW filters on
This model considers the time–gated response of a SAW filter as function of the admittances at the input and output IDTs, such that the transmission through the film is

\[ S_{21} = \frac{-2Y_{21}Y_{0}}{(Y_{0} + Y_{11})^2 - Y_{21}^2}. \]  

Equation (5.3) is plotted in conjunction with the gated response curve in Figure 5.7 below. Although the magnitude of the response is somewhat higher than expected, the shape of the response is in agreement with the theoretical result. In fact, the disparity between to the predicted and experimental results is due to the aggressive time gating used in making the measurements. From Figure 5.3, it is observed that the filter response appears as a broad, flat peak in the time domain. Because the filter response on titanium was so affected by spurious electromagnetic transmission, the gate function was applied to completely filter all out–of–band transmission. Although this results in a much purer transmitted signal, it comes at the expense of eliminating some frequency characteristics of the SAW filter, to the point that the gated in–band response of the filter cannot be used as a figure of merit for the device performance [4]. It was experimentally observed that as the width of the gate function was increased, the relative height of the
peaks began to adjust to the shape of the theoretical response. However, the noise floor of the measurement would increase dramatically due to electrical losses, eliminating the value of these measurements.

Figure 5.7: Predicted response of a SAW filter on a conducting substrate. The analytical model predicts that a sinc function will be observed if the electromagnetic feedthrough is removed (blue line). This is quantitatively observed in the experimental data (black dots).

By fitting the model to the experimental data, material properties of the AlN film were extracted. For the filter displayed in Figure 5.7, the electromechanical coupling $k_{31}^2 = 0.77\%$ and the acoustic velocity was 5150 m/s. The calculated $k^2$, however, is underestimated by a factor of up to 20 because the time–gating does not truly yield the exact ideal response of a SAW filter.
[12]. Nonetheless, \( k^2 \) values calculated from the model are often used to make relative comparisons of SAW filters on conducting substrates [1]. The \( k^2 \) value calculated above is comparable to SAW filters fabricated from AlN films on doped silicon [6].

**SUMMARY**

Sputtered deposited AlN films on titanium were used to demonstrate the first known examples of SAW filters on a bulk metal substrate. Despite electrical loss due to the metal substrate and high electromagnetic feedthrough between the emitter and collector IDTs of the filter, measurable filter responses were obtained using time–gating measurement techniques. By collecting data from SAW filters with various IDT geometries and resonant frequencies, the acoustic velocity and attenuation for AlN films on titanium were measured. These results are comparable to published data for AlN SAW filters on silicon. Although mechanical coupling to the titanium substrate decreased the acoustic velocity and increased the power attenuation by an order of magnitude, the electromechanical coupling was not as severely affected. It is expected that filters fabricated from thicker AlN films would have performed even better than the devices tested.
REFERENCES


CONCLUDING REMARKS
This final chapter concludes the work presented. First is a summary the main experimental results with an emphasis on comparing these results with reported values for single crystal AlN and AlN thin films on silicon. Following are suggested directions for future research, including possible methods for increasing the piezoelectric properties of the thin films, better transducer designs, and new piezoelectric MEMS on titanium.

6.1 RESEARCH SUMMARY

The main objective of this thesis was to demonstrate piezoelectric transduction in the materials system of aluminum nitride on titanium. A three-step approach was taken: deposit highly crystalline AlN films onto titanium substrates; fabricate MEMS from these films; and calculate the piezoelectric properties of AlN using experimental data. All three of these goals were reached.

Highly crystalline AlN was sputtered onto polished titanium wafers using an optimized deposition process that emphasized the hysteretic behavior of
reactive sputter deposition. For the sputtering chamber used, the deposition conditions were a plasma power of 2–3 kW, 20 sccm of Ar, 6 sccm of N\textsubscript{2}, and half-power bipolar control. It should be noted, however, that the efficacy with which these parameters produce crystalline films is not constant and must be regularly monitored to reflect changing chamber conditions—i.e. adjustment of the N\textsubscript{2} flow to maintain sputtering in the center of the hysteresis curve is necessary. Despite the much higher surface roughness of titanium, the crystalline alignment of the AlN films approached, and in some cases matched, results reported for sputtered AlN on silicon. The best rocking curve FWHM for an AlN film on titanium was 1.79°. Furthermore, the AlN films had other comparable material properties, including grain size and optical constants.

Piezoelectric transduction was demonstrated in unimorph cantilevers and surface acoustic wave (SAW) filters. Using static measurements of the vertical displacement of cantilevers, the piezoelectric coefficient \( d_{31} \) was calculated to be 2.49 pm/V, 94% of the value for single crystal AlN, and very close to the reported result of 2.57 pm/V for AlN cantilevers fabricated on silicon substrates [1]. The dynamic response of the cantilever beams was also measured. At large displacements, the beams exhibited a nonlinear spring
softening that shifts resonant peaks to lower frequencies. Because a finite element analysis and a reported analytical model did not sufficiently model all features of the frequency response curves, a numerical solution to Duffing’s equation was fitted to the experimental data. The solution was used to calculate the electromechanical coupling coefficient $k^2$. An average of 4%, this value was roughly half of the reported $k^2$ for single crystal AlN but in almost exact agreement with polycrystalline AlN films on silicon having the same columnar alignment [2, 3].

SAW filters were fabricated by patterning aluminum interdigitated transducers (IDT) onto the AlN films. Because of large electromagnetic and acoustic losses, the $s_{12}$ parameter was minimal compared with the noise floor. By utilizing a time–gating measurement technique, however, the SAW signal was decoupled from the electromagnetic feedthrough. Frequency–dependent attenuation of $s_{12}$ was observed; the attenuation was 3.1 dB/cm at 129 MHz and 18.3 dB/cm at 256 MHz. The power law frequency dependence compared well to the reported results for AlN SAW filters on silicon, though the amount of attenuation was an order of magnitude higher [4]. This discrepancy was attributed to acoustic losses from the relatively high roughness of titanium—and by extension, the surface of the AlN film—and strong acoustic coupling.
to the substrate. Also, the resonant response of the filters was analyzed using an admittance model to obtain values for the SAW electromechanical coupling and the acoustic velocity [5]. $k_31^2$ was calculated as 0.77% and the velocity of the film was 5150 m/s. These values were comparable to AlN SAW filters on silicon when electromagnetic feedthrough diminishes the electromechanical coupling [6].

From the experimental results, it is concluded that piezoelectric transduction has been realized on titanium substrates. This thesis contains strategies for depositing highly crystalline AlN with good piezoelectric properties and fabricating transducers from those films. Additionally, effective methods for determining the piezoelectric properties of the films have been detailed.

6.2 Future Directions

This work has contained the first examples of piezoelectric transduction on bulk metal substrates, yet by no means is it a technological limit; there are a number of improvements that can be made. Advancements in the AlN deposition method would be likely to increase the piezoelectric properties of the thin films. For instance, although the reported two–stage process for
depositing high quality AlN on silicon was ineffective on titanium substrates, the principle behind that method—an initial high energy deposition to provide a columnar seed layer—could be used to improve the film quality. This could be accomplished by finding proper chamber conditions on the hysteresis curve that provide a seed layer with maximal quality. Also, reducing the surface roughness of the titanium wafers was not investigated. Although the wafers are polished prior to delivery, there is reason to believe that individual polishing of the wafers using a well-characterized chemical mechanical polish (CMP) could further reduce the surface roughness. This would not only improve the columnar alignment of the AlN films but also have a positive effect on the fabrication and performance of piezoelectric MEMS. Additional characterization studies would be interesting, such as employing a method for tracking the quality of AlN films through Raman spectroscopy [7].

Concerning the transducers, a number of improvements could be made, in particular with respect to the SAW filters. The large electromagnetic feedthrough due to the conducting substrate could be mitigated by the incorporation of a strong dielectric layer such as polycrystalline TiO₂ between the substrate and the AlN film. Also, because of the acoustic velocity
differential between AlN and titanium, high acoustic losses are incurred during SAW transmission. The use of an acoustic waveguide structure—e.g. a Bragg reflector or an intermediate thin film with a higher acoustic velocity than AlN—would reduce these losses.

More advanced piezoelectric transducers can be fabricated on titanium. Although not outlined in this thesis, research was directed toward fabricating a film bulk acoustic resonator (FBAR) on titanium. The key step during fabrication is releasing the resonator from the substrate. Preliminary results from a high rate anisotropic plasma etch from the back side of the wafer were promising; with further characterization the successful fabrication of an FBAR should be possible. As AlN FBARs are currently fabricated on silicon, which exhibits frequency–dependent electrical nonlinearities, a resonator on titanium would be of particular scientific interest due to the linear resistivity of titanium allowing for out–of–band frequency response [8]. Further ideas for piezoelectric MEMS include in–plane shear mode transducers for mechanical amplification, integrated uni– and bimorph structures for RF waveguide switching, and thin–foil bonded cavities for microfluidic pumping.
REFERENCES


