Characterization of Dielectric Films for Electrowetting on Dielectric Systems

by

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Dedication

To My Parents
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Characterization of Dielectric Films for Electrowetting on Dielectric Systems

Ajay Rajgadkar

Abstract

Electrowetting is a phenomenon that controls the wettability of liquids on solid surfaces by the application of electric potential. It is an interesting method to handle tiny amounts of liquid on solid surfaces. In recent times, researchers have been investigating this phenomenon and have reported some unexplained behavior and degradation in the Electrowetting system performance. Electrowetting systems include the presence of electric field and different materials from metals to dielectrics and electrolytes that create an environment in which corrosion processes play a very important role. With the small dimensions of the electrodes, corrosion can cause failure quickly when the dielectric fails.

In this work, commonly used dielectric films such as silicon dioxide and silicon nitride were deposited using Plasma Enhanced Chemical Vapor Deposition and characterized on the basis of thickness uniformity, etch rate measurements, Dry current – voltage measurements and Wet current – voltage measurements. Sputtered silicon dioxide films were also characterized using the same methods. The correlation between Dry I – V and Wet I – V measurements was studied and a comparison of dielectric
quality of films based on these measurements is presented. Also, impact of different liquids on the dielectric quality of films was studied.
Chapter 1 Introduction

1.1 Thesis Statement

Electrowetting on dielectric (EWOD) systems include the presence of an electric field along with different materials like, metal electrodes, dielectric layers and liquids. The material properties play an important role in the performance and reliability of these systems. The thesis addresses some methods for characterization of dielectric materials for Electrowetting applications and studies the dependence of dielectric quality on voltage polarity, different liquids and different thicknesses of dielectric films. The dielectric quality of Silicon dioxide and Silicon nitride films deposited by Plasma Enhanced Chemical Vapor Deposition (PECVD), and sputtered Silicon dioxide are studied in terms of film thickness uniformity, etch rate measurements, Dry current – voltage measurements and Wet current – voltage measurements and a comparison based on these measurements is presented to determine the optimum film thickness and process that can be used for depositing the dielectric films for Electrowetting on dielectric applications.

This chapter will review the background, basic concept and various applications of Electrowetting on dielectric systems.
1.2 Background

Microfluidics is an emerging field in microsystems engineering, in which small amounts of liquids are handled to realize various applications. In particular, “Microfluidics” also known as “Lab-on-a chip” is a concept that is used for total chemical analysis on smaller scale where only a few microliters of reagents and chemicals can be used for the analysis [23]. The advantage of this is faster analysis and need of fewer amounts of chemicals and reagents, also as the devices used for such analysis being small; they are portable and can be used at the site of interest rather than taking samples to the laboratory. Microfluidic devices have networks of microchannels generated in planar substrates through which fluids can be moved, mixed and separated to carry out various chemical reactions [4]. These microchannels with cross-sectional dimensions of the order of few micrometers are made into flat substrates with well established techniques used in MEMS and Microelectronics industry [23]. Although, the devices can be fabricated using these techniques, the challenge lies in precise control over the fluid movement along desired paths.

Over the last decade, different methods have been explored for manipulating tiny amounts of liquids on solid surfaces for microfluidic applications [20]. More commonly, microfluidic systems use microchannels, micropumps and microvalves to achieve fluid motion along desired paths. A few disadvantages encountered in this approach are formation of micro bubbles at channel junctions and clogging of channels by micro bubbles [4]. Also, integration of micropumps and microvalves to form a complex geometry in small microfluidic devices is another challenge [17]. Electrowetting is a phenomenon which has shown great promise for actuating small amounts of liquids, of
the order of few microliters on solid surfaces [11]. It is a phenomenon that controls the wettability of liquids by application of an electric field [20].

In the next sections, basic concept of Electrowetting will be reviewed and some of the applications will be briefly discussed.

1.3 Electrowetting

Electrowetting is an electrocapillary phenomenon in which the contact angle of a liquid droplet is changed by the application of an electric potential. The application of electric potential causes a reduction in the surface tension at the liquid–solid interface which results in a decrease in the contact angle of the droplet and as the contact angle decreases, the liquid droplet wets the surface [15]. This phenomenon can be used to move, split, merge and mix liquid droplets on solid surfaces for realizing lab on a chip and various other applications [20].

Different Electrowetting configurations have been studied in recent years for the control of droplet motion [15], [5]. Electrowetting more commonly referred to as Electrowetting on Dielectric (EWOD) consists of a droplet of a conducting liquid placed on a dielectric layer which acts as an insulating layer as well as a hydrophobic layer. The dielectric layer is deposited on a metal electrode and a Platinum electrode is introduced in the conducting liquid. As voltage is applied across the metal electrode and the droplet, there is a redistribution of charge that causes a change in free energy of the drop. This change in free energy decreases the contact angle (θ) and the drop spreads and thus wets the surface. This configuration is referred to as Grounded Droplet Configuration and is illustrated in Figure 1 below,
Figure 1 EWOD - Grounded Droplet Configuration.

In the absence of an external applied voltage, the equilibrium contact angle ($\theta$), at the three-phase contact line is given by the Young equation,

$$\gamma \cos \theta = \gamma_{SV} - \gamma_{SL}$$

Equation 1 Young Equation.

where $\gamma$, $\gamma_{SV}$ and $\gamma_{SL}$ are the free energies of the liquid/vapor, solid/vapor and solid/liquid interfaces. On the application of voltage, the contact angle ($\theta$) will depend on the voltage (V) applied across the electrode and the droplet and the liquid wetting angle ($\theta_0$) without the applied voltage. This can then be related to the dielectric thickness (d) and dielectric
strength \((\varepsilon_0, \varepsilon_r)\) by the Young Lippman equation [5].

\[
\cos \theta_v = \cos \theta_0 + \frac{\varepsilon_0 \varepsilon_r V^2}{2\gamma d}
\]

**Equation 2 Young Lippmann Equation for Electrowetting on Dielectric.**

Another configuration which is referred to as Floating Droplet Configuration is illustrated in Figure 2. Here the potential is applied across the two metal electrodes which lie underneath the dielectric layer and the liquid droplet is placed over the dielectric layer. In this configuration a conducting liquid droplet is used and the system acts as two capacitors in series.

![Figure 2 EWOD – Floating Droplet Configuration.](image-url)
As we see from the Young-Lippman equation, it is clear that the change in contact angle is influenced by the dielectric strength, applied voltage and thickness of dielectric layer. Increasing the dielectric thickness to avoid dielectric failure would require higher actuation voltage. If a dielectric with higher dielectric constant is used this would reduce the voltage required for Electrowetting and thinner layers could be used with less chances of dielectric breakdown.

1.4 Applications of Electrowetting

The applications range from biotechnology related microfluidic devices to adjustable lenses, display technology and fiber optics [2], [3], [9]. Microfluidic devices use an array of individually addressable electrodes that allow for moving droplets along desired paths. Small droplets can be extracted from larger reservoir of different reagents and can be merged and mixed to carry out required chemical reaction.

In Electrowetting, the application of electric potential causes the change in contact angle of the droplet and hence the shape of the droplet. This can be used in the design of optical systems by using liquid lenses whose curvature and hence their focal length can be tuned by adjusting their shape [2], [7]. Electrowetting was also used as a possible mechanism for switching light in fiber optic applications. The transmission of light through the fiber can be tuned by surrounding the active part of the fiber with liquids of suitably chosen refractive index [15]. Some of the other applications include Electrowetting based reflective display with potential use in the field of electronic paper, thermal management of electronic systems, handling of small parts for micro level self assembly, etc [13], [18].
1.5 Thesis Outline

The thesis will proceed as follows: Chapter 2 will review the previous work done on Electrowetting and different challenges in Electrowetting systems. Different dielectric used and their characterization methods and different liquids used in Electrowetting systems. Chapter 3 will give the details about different processes used to deposit thin dielectric films and methods used to characterize the dielectric quality of these films. Chapter 4 will discuss the results obtained and compare the quality of dielectric films deposited by different processes. Chapter 5 will present the conclusion based on results obtained and will recommend future work to be done.
Chapter 2 Literature Review

2.1 Contact Angle Saturation and Charge Trapping

At low voltage the change in contact angle is well predicted by the Lippmann equation but as higher voltages are applied the contact angle change saturates i.e. there is no further change in contact angle with increase in voltage. This phenomena which limits the application of Lippmann equation is known as contact angle saturation and the mechanism causing this are not clearly understood but different explanations are provided [16].

Kilaru et al. have reported and explained strong charge trapping and bistable Electrowetting on nanocomposite fluoropolymer: BaTiO₃ dielectrics. The droplet rests at Young’s angle at zero applied voltage. When voltage is applied the droplet wets the surface as charge accumulates near the liquid/droplet interface. When the droplet is held at voltage corresponding to contact angle saturation the charge injects into the dielectric layer. The increased injection and screening of charge causes the droplet to dewet the surface. The liquid droplet is then grounded and the injected charge causes a reverse polarity charge accumulation near the liquid/dielectric interface. As a result of this the droplet again wets the surface and stable wetting is achieved for several minutes without application of voltage [10].
Verheijen and Prins developed a model for electrowetting based on the principle of virtual displacement. Their model proposed the possibility of charge being trapped in the insulating layer or at its surface during electrowetting experiments. They verified the model with experiments and found that up to a certain voltage the charge remains in the liquid and is not trapped in the insulating layer, but once the voltage exceeds a threshold value, the charge gets trapped in the insulating layer or at its surface. They relate this trapping of charge in the insulating layer to contact angle saturation [22].

Papathanasiou et al. did some simulation and analysis using finite element method to study the connection between dielectric breakdown strength, trapping of charge and contact angle saturation. They suggest that the dielectric material breakdown strength is connected to the charge trapping in the material [6].

2.2 Dielectric Materials

The quality of dielectric materials has been found to be of great importance in EWOD systems. The performance of EWOD systems is directly related to the properties of dielectric materials. Different dielectric materials have been used for EWOD systems.

Seyrat and Hayes studied amorphous fluoropolymer (AF 1600, Dupont) as dielectric as well as hydrophobic layer for electrowetting applications. They characterized different thicknesses of this polymer and concluded that thicknesses above 0.4 μm had good electrical properties and could be used for electrowetting applications [19].
Moon and Cho experimentally demonstrated the dependence of applied voltage on dielectric thickness by using different thicknesses of spin coated amorphous fluoropolymer (Teflon® AF, Dupont), thermal silicon dioxide and parylene deposited by vapor deposition. They also demonstrated the dependence of voltage on dielectric constant by using silicon dioxide and barium strontium titanate [14].

Cahill and co-workers presented a comparison of dielectric characteristics of silicon dioxide, silicon nitride and Parylene C by leakage current measurements and resistance characteristic curves. They used a top coat of PECVD coated Teflon like fluoropolymer as a hydrophobic layer over these dielectric layers. They found that silicon dioxide showed poor resistance to dielectric breakdown while Parylene C and silicon nitride showed good resistance. The thickness of Parylene was relatively more than that of silicon nitride. They suggested the use of silicon nitride layers for Electrowetting systems as thin layers of silicon nitride can be easily obtained [1].
Chapter 3 Dielectric Material Deposition and Characterization Procedures

The properties of thin dielectric films are very important for the reliability of Electrowetting systems. Low pinhole density, high dielectric breakdown strength, high dielectric constant, good thickness uniformity and good adhesion are some of the properties of prime importance [12]. Dielectric films can be deposited by various processes like Chemical Vapor Deposition (CVD) which include Atmospheric Pressure CVD (APCVD), Low Pressure CVD (LPCVD) and Plasma Enhanced CVD (PECVD). Also, physical vapor deposition technique like sputtering and electron beam evaporation can be used. In this work PECVD was used to deposit Silicon dioxide and Silicon nitride films of different thicknesses. Also, sputtering process was used to deposit different thicknesses of Silicon dioxide films. The details of process parameters will follow after a brief description of these two processes used for this work.

3.1 Plasma Enhanced Chemical Vapor Deposition

The working principle of CVD involves the flow of a gas with diffused reactants over a hot substrate surface. The gas that carries the reactants is called carrier gas. As the gas flows over the hot substrate, the energy supplied by the substrate temperature provokes chemical reaction of the reactants that form films during and after the reaction. The by-products of the chemical reaction are then vented. The plasma enhanced CVD utilizes the RF plasma to transfer energy into the reactant gases, which allows the
substrate to remain at lower temperature than that in APCVD or LPCVD. An inert gas like helium or argon is used to create the plasma. A typical PECVD reactor is shown in Figure 3 below [21].

![PECVD Reactor](image)

**Figure 3 PECVD Reactor.**

In PECVD process, the deposition parameters strongly influence the quality of the deposited film and hence deposition of good quality films requires tuning of different process parameters. The parameters of interest are the deposition pressure, temperature, RF power and the flow rate of the reactant gases. The most important factor in determining the electrical quality of deposited films is the rate of deposition. To vary the deposition rate in a controlled fashion generally requires a change in more than one parameter. For example, increasing the RF power increases the deposition rate but an increase in the reactive gas flow is necessary to maintain uniformity. Similarly for a fixed
power, decrease in flow rate generally lowers the deposition rate but requires decrease in chamber pressure to achieve good uniformity. Using a high flow of inert carrier gas to ensure uniformity enables a wide range of deposition rates to be achieved simply by varying the reactive gas flows.

### 3.2 Physical Vapor Deposition: Sputtering

The sputtering process is a low temperature process, carried out with plasma in a vacuum environment. This process is illustrated in Figure 4 below.

**Figure 4 Illustration of Sputtering Process.**

The material to be deposited – the target and the substrates on which material is to be deposited are placed in a chamber. The chamber is then evacuated to sufficiently low
pressures, typically microTorr range and then an inert gas like Argon or Helium is inserted into the chamber. Plasma is produced by either high – voltage DC sources or RF sources. The ionized positive Argon ions bombard the surface of the target, which is the cathode, at a high velocity and this impingement dislodges material from the target which gets condensed on the substrates [21].

3.3 Dielectric Material Deposition Methods

In this work a Plasma Therm Waf’r/Batch 700 PECVD system was used to deposit silicon dioxide films of 1 µm, 0.5 µm, 0.2 µm and 0.1 µm thickness and silicon nitride films of 0.5 µm, 0.2 µm and 0.1 µm thicknesses. N-type, <100>, silicon wafers (500 µm thick and 50 mm diameter) were used for all the depositions. The wafers were cleaned with acetone, methanol and DI water before the deposition. The deposition parameters for silicon dioxide and silicon nitride films are shown in Table 1 and Table 2 respectively.
Table 1 PECVD Parameters for Silicon Dioxide Films

<table>
<thead>
<tr>
<th>Parameter</th>
<th>Value</th>
</tr>
</thead>
<tbody>
<tr>
<td>RF Power</td>
<td>50 W @ 13.56 MHz</td>
</tr>
<tr>
<td>Pressure</td>
<td>900 mT</td>
</tr>
<tr>
<td>Temperature</td>
<td>250°C</td>
</tr>
<tr>
<td>Silane</td>
<td>5 sccm</td>
</tr>
<tr>
<td>N₂O</td>
<td>500 sccm</td>
</tr>
<tr>
<td>Deposition rate</td>
<td>~ 300 A/min</td>
</tr>
<tr>
<td>Thickness</td>
<td>Deposition Time</td>
</tr>
<tr>
<td>1000 Å (2 Samples)</td>
<td>3:48 min</td>
</tr>
<tr>
<td>2000 Å (2 Samples)</td>
<td>7:40 min</td>
</tr>
<tr>
<td>5000 Å (1 Sample)</td>
<td>17 min</td>
</tr>
<tr>
<td>10,000 Å (1 Sample)</td>
<td>34 min</td>
</tr>
</tbody>
</table>

Table 2 PECVD Parameters for Silicon Nitride Films

<table>
<thead>
<tr>
<th>Parameter</th>
<th>Value</th>
</tr>
</thead>
<tbody>
<tr>
<td>RF Power</td>
<td>50 W @ 13.56 MHz</td>
</tr>
<tr>
<td>Pressure</td>
<td>900 mT</td>
</tr>
<tr>
<td>Temperature</td>
<td>250°C</td>
</tr>
<tr>
<td>Silane</td>
<td>5 sccm</td>
</tr>
<tr>
<td>N₂</td>
<td>1000 sccm</td>
</tr>
<tr>
<td>Deposition rate</td>
<td>~ 130 A/min</td>
</tr>
<tr>
<td>Thickness</td>
<td>Deposition Time</td>
</tr>
<tr>
<td>1000 Å (2 Samples)</td>
<td>7:45 min</td>
</tr>
<tr>
<td>2000 Å (2 Samples)</td>
<td>15:30 min</td>
</tr>
<tr>
<td>5000 Å (2 Samples)</td>
<td>38:30 min</td>
</tr>
</tbody>
</table>

Sputtered silicon dioxide films were deposited in a standard DC magnetron sputtering system. Two samples each of 0.5 µm, 0.2 µm and 0.1 µm thickness were prepared. The process parameters for sputtering are shown in Table 3 below.
### Table 3 Sputtering Parameters for Silicon Dioxide Films

<table>
<thead>
<tr>
<th>Parameter</th>
<th>Value</th>
</tr>
</thead>
<tbody>
<tr>
<td>Chamber Pressure</td>
<td>5.4-5.6 mT</td>
</tr>
<tr>
<td>O₂</td>
<td>2.5 mT</td>
</tr>
<tr>
<td>Ar</td>
<td>2.9-3.1 mT</td>
</tr>
<tr>
<td>Temperature</td>
<td>25 °C</td>
</tr>
<tr>
<td>Power</td>
<td>200 W</td>
</tr>
<tr>
<td>Deposition Rate</td>
<td>0.9 Å/s</td>
</tr>
</tbody>
</table>

#### 3.4 Thickness Measurements and Etch Rate Measurements

#### 3.4.1 Ellipsometer

After the deposition of different thicknesses of dielectric films, Rudolph Auto EL Ellipsometer was used to measure the film thicknesses. In order to check the uniformity of films, measurements were done at 5 spots across each sample as illustrated in Figure 5 below.

![Figure 5 Thickness Measurement Spots for Ellipsometer.](image-url)
3.4.2 Profilometer

A Dektak 3030 Profilometer was also used to measure the thicknesses of dielectric films to confirm the Ellipsometer measurements. To create step height for Profilometer measurements, photolithography technique was used to pattern the dielectric films on all samples. The photolithography process included the use of Laurel Spinner and Karl Suss Mask Aligner and the following steps,

- Spin HMDS adhesion promoter at 3300 rpm for 40 sec.
- Spin Shipley 1813 photoresist at 3300 rpm for 40 sec.
- Soft bake for 1 min at 100º C on hot plate.
- Expose to UV light for 4 sec at an intensity of 25 mW/cm².
- Develop for 90 sec with MF 319 developer.
- Rinse with DI water for 3 mins.
- Dry with Nitrogen gas.

The target thickness of photoresist is about 1.3 µm. After this step, the samples had a portion of dielectric layer exposed and rest of the dielectric was masked with photoresist. An illustration of samples after the photolithography step is shown in Figure 6.
The exposed dielectric was then etched using 10:1 Buffered Oxide Etch (BOE). BOE is a mixture of Ammonium Fluoride solution (NH₄F), “the buffer” and Hydrofluoric Acid, “straight HF” that is 49 weight percent that constitutes the active agent in this etch.

Etch rates were measured as follows,

- First, the thickness of photoresist was measured across the step created after developing the photoresist from the exposed area using Profilometer.
- For PECVD SiO₂ samples, the sample with highest thickness (1 μm) was initially immersed in BOE solution for 15 seconds, a timer was used to note the time.
- The exposed SiO₂ was etched and thickness measurement across the step at approximately the same location on the sample gives the thickness of photoresist and SiO₂. The difference between the thickness before etching for 15 seconds and after etching gives the total thickness of SiO₂ etched. Based on this measurement, the etch rate was calculated.

Figure 6 Sketch of Sample After Photolithography Step.
• The calculated etch rate was then used to etch SiO2 completely from the exposed area.
• The etch rate thus determined was used as starting point for samples with other thicknesses.

After etching the exposed dielectric from the samples completely, photoresist was removed by soaking the samples in acetone and then they were cleaned with methanol and DI water and dried.

After this step, thickness measurement was done with Dektak 3030 Profilometer at 5 spots along the step created as described above. The measurement spots on the samples are illustrated in

![Figure 7 Thickness Measurement Spots for Profilometer.](image)

Figure 7 Thickness Measurement Spots for Profilometer.
3.5 Electrical Characterization Methods

Electrical characterization is done by measuring Dry Current – Voltage characteristics and Wet Current – Voltage characteristics. These methods are described below.

3.5.1 Dry Current – Voltage Measurements

For Dry Current – Voltage measurements, metal-oxide-semiconductor structures were created on the samples. A Varian Model 980-2462 Electron Beam Evaporator was used to deposit 2000 Å Aluminum through a metal shadow mask with 1 mm diameter holes. These Aluminum dots were deposited on about half of the entire surface of the samples and the remaining portion was left for Wet Current – Voltage measurements. The samples thus prepared are shown in Figure 8 below.

![Figure 8 Samples for Current – Voltage Measurements.](image)
An HP 415B Semiconductor Parameter Analyzer was used to obtain Current – Voltage characteristics. The silicon wafer was grounded and potentials were applied at the Aluminum top electrodes. A linear voltage sweep mode was used in which first the voltages were swept from negative 100 V to positive 100 V with a step of 1.333 V and currents were measured at each voltage and then from positive 100 V to negative 100 V again with a step of -1.333 V and corresponding currents were measured. Each measurement was done at a different spot. The setup for these measurements is shown in Figure 9 below.

![Figure 9 Setup for Dry Current - Voltage Measurements.](image)

3.5.2 Wet Current – Voltage Measurements

Wet Current – Voltage measurements were done using three different solutions. The solutions used were 1 mM NaCl, 1 mM Na2SO4 and DI water. A 5 μL droplet of each solution was used for all the tests. The setup for Wet tests was similar to Dry tests,
except liquid droplets were used instead of Aluminum top electrode. All measurements were done at fresh spots.
4.1 Film Thickness Uniformity

The thickness measurements with Ellipsometer and Profilometer for PECVD SiO2 films are shown in Table 4. The thickness variation, in percentage, is calculated as the ratio of the difference in maximum and minimum measured thickness to the average thickness. The target thickness for samples B1 and B2 was 1 µm and 0.5 µm respectively, but the measurements show that the thickness is off by 28 – 29 %. This variation is because before every deposition the PECVD chamber must be cleaned with oxygen plasma and it was not done before these depositions. For all other depositions, the chamber was pre-cleaned with oxygen plasma for 20 mins. The Ellipsometer data shows less variation, less than 3% for all samples. However, Profilometer data shows higher thickness variation with maximum of 10.83% for 1000 Å sample, which is acceptable. Also, Ellipsometer data and Profilometer data are in good confirmation. For thinner films, i.e. 1000 Å and 2000 Å, the measured average thickness was close to the desired thickness with less than 6% variation.

For PECVD Si3N4, the thickness measurements with Ellipsometer show some variations in the thickness. For 1000 Å films, the measured average thickness was found to be less by approximately 11.5% than the desired thickness with variation less than 2%.
For 2000 Å films, the measured average thickness was found to be more by about 20% than the desired thickness with variation of 3.39% and 8.05% for 2 samples. For 5000 Å films, the measured average thickness were close to the desired thickness, but variation was much higher, about 16.54% in 1 sample. The Profilometer measurements did not match with the Ellipsometer measurements except for 1000 Å films. For 2000 Å and 5000 Å films, the measured average thickness was less by about 12% and 11% than the desired thickness respectively. The thickness variation for all samples was less than 10% for all samples. The thickness measurement data for different PECVD Si3N4 samples is shown in Table 5.

Table 4 PECVD SiO2 Thickness Measurements

<table>
<thead>
<tr>
<th>Sample -&gt;</th>
<th>B1</th>
<th>B2</th>
<th>B3</th>
<th>B4</th>
<th>B5</th>
<th>B6</th>
</tr>
</thead>
<tbody>
<tr>
<td>Thickness (Å)</td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>12699</td>
<td>6411</td>
<td>987</td>
<td>1002</td>
<td>2114</td>
<td>2105</td>
<td></td>
</tr>
<tr>
<td>12778</td>
<td>6420</td>
<td>990</td>
<td>1007</td>
<td>2107</td>
<td>2134</td>
<td></td>
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<td>12786</td>
<td>6484</td>
<td>992</td>
<td>1012</td>
<td>2125</td>
<td>2113</td>
<td></td>
</tr>
<tr>
<td>12849</td>
<td>6523</td>
<td>998</td>
<td>1014</td>
<td>2123</td>
<td>2116</td>
<td></td>
</tr>
<tr>
<td>12999</td>
<td>6548</td>
<td>1011</td>
<td>1018</td>
<td>2126</td>
<td>2119</td>
<td></td>
</tr>
<tr>
<td>Avg</td>
<td>12822.2</td>
<td>6477.2</td>
<td>995.6</td>
<td>1010.6</td>
<td>2119</td>
<td>2117.4</td>
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<td>SD</td>
<td>112.27</td>
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<td>9.50</td>
<td>6.23</td>
<td>8.22</td>
<td>10.64</td>
</tr>
<tr>
<td>% non uniformity</td>
<td>2.34</td>
<td>2.12</td>
<td>2.41</td>
<td>1.58</td>
<td>0.57</td>
<td>0.66</td>
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</table>

<table>
<thead>
<tr>
<th>Sample -&gt;</th>
<th>B1</th>
<th>B2</th>
<th>B3</th>
<th>B4</th>
<th>B5</th>
<th>B6</th>
</tr>
</thead>
<tbody>
<tr>
<td>Thickness (Å)</td>
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<td></td>
<td></td>
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<tr>
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<td>6146</td>
<td>974</td>
<td>924</td>
<td>1978</td>
<td>2014</td>
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<td>12586</td>
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<td>982</td>
<td>962</td>
<td>2060</td>
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<td>6359</td>
<td>990</td>
<td>996</td>
<td>2064</td>
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<td>12757</td>
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<td>1028</td>
<td>2072</td>
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<tr>
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<td>1031</td>
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</tr>
<tr>
<td>Avg</td>
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<td>6304.0</td>
<td>1008.4</td>
<td>988.2</td>
<td>2063.6</td>
<td>2086</td>
</tr>
<tr>
<td>Std Deviation</td>
<td>100.63</td>
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<td>38.51</td>
<td>45.51</td>
<td>58.90</td>
<td>48.38</td>
</tr>
<tr>
<td>% variation</td>
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<td>9.02</td>
<td>10.83</td>
<td>8.04</td>
<td>6.52</td>
</tr>
</tbody>
</table>
Table 5 PECVD Si₃N₄ Thickness Measurements

<table>
<thead>
<tr>
<th>Sample</th>
<th>N1</th>
<th>N2</th>
<th>N3</th>
<th>N4</th>
<th>N5</th>
<th>N6</th>
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</thead>
<tbody>
<tr>
<td>Ellipsometer</td>
<td></td>
<td></td>
<td></td>
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<td></td>
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<tr>
<td>Thickness (Å)</td>
<td>879</td>
<td>878</td>
<td>2205</td>
<td>2146</td>
<td>4882</td>
<td>4691</td>
</tr>
<tr>
<td></td>
<td>885</td>
<td>886</td>
<td>2220</td>
<td>2153</td>
<td>5056</td>
<td>4731</td>
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<td>888</td>
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<td>890</td>
<td>2312</td>
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<td>5522</td>
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<tr>
<td>Avg</td>
<td>885</td>
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<td>2180.2</td>
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<tr>
<td>SD</td>
<td>3.67</td>
<td>5.93</td>
<td>76.49</td>
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</tr>
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<td>1.13</td>
<td>1.80</td>
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<td>3.39</td>
<td>6.58</td>
<td>16.54</td>
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</table>

<table>
<thead>
<tr>
<th>Sample</th>
<th>N1</th>
<th>N2</th>
<th>N3</th>
<th>N4</th>
<th>N5</th>
<th>N6</th>
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<tbody>
<tr>
<td>Profilometer</td>
<td></td>
<td></td>
<td></td>
<td></td>
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<td></td>
</tr>
<tr>
<td>Thickness (Å)</td>
<td>819</td>
<td>816</td>
<td>1706</td>
<td>1758</td>
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<td>4507</td>
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<tr>
<td></td>
<td>838</td>
<td>859</td>
<td>1730</td>
<td>1786</td>
<td>4392</td>
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<td></td>
<td>867</td>
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<td></td>
<td>888</td>
<td>884</td>
<td>1798</td>
<td>1837</td>
<td>4541</td>
<td>4610</td>
</tr>
<tr>
<td>Avg</td>
<td>860.8</td>
<td>863.8</td>
<td>1771.6</td>
<td>1816.6</td>
<td>4477</td>
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<tr>
<td>SD</td>
<td>31.70</td>
<td>31.48</td>
<td>60.42</td>
<td>43.60</td>
<td>86.24</td>
<td>86.40</td>
</tr>
<tr>
<td>% variation</td>
<td>8.48</td>
<td>9.61</td>
<td>8.69</td>
<td>5.95</td>
<td>4.11</td>
<td>4.71</td>
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</tbody>
</table>

The thickness measurements for sputtered SiO₂ with Ellipsometer show that the measured average thickness for 1000 Å, 2000 Å and 5000 Å samples is less by about 16%, 13% and 18% than the desired thickness respectively with variation less than 4% for all samples. The Profilometer measurements show that the measured average thickness for 1000 Å, 2000 Å and 5000 Å samples is less by about 12%, 9% and 17% than the desired thickness respectively. With Profilometer measurements, the thickness variation was found to be much higher in 1000 Å samples, above 14%. One sample of 2000 Å thickness showed higher variation, above 12%, and the other samples showed variation less than 10%. The Ellipsometer measurements show lower thickness variation as compared to Profilometer measurements; this could be due to a large spot size of the
incident beam (about half a mm) which could be averaging out the variations in thickness. The thickness measurement data for different sputtered SiO2 samples is shown in Table 6.

Table 6 Sputtered SiO2 Thickness Measurements

<table>
<thead>
<tr>
<th>Sample -&gt;</th>
<th>S1</th>
<th>S7</th>
<th>S2</th>
<th>S3</th>
<th>S4</th>
<th>S5</th>
</tr>
</thead>
<tbody>
<tr>
<td><strong>Thickness (Å)</strong></td>
<td>825</td>
<td>820</td>
<td>1738</td>
<td>1724</td>
<td>4037</td>
<td>4076</td>
</tr>
<tr>
<td></td>
<td>829</td>
<td>831</td>
<td>1759</td>
<td>1731</td>
<td>4054</td>
<td>4108</td>
</tr>
<tr>
<td></td>
<td>837</td>
<td>833</td>
<td>1768</td>
<td>1749</td>
<td>4087</td>
<td>4127</td>
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<tr>
<td></td>
<td>850</td>
<td>836</td>
<td>1771</td>
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<td>856</td>
<td>846</td>
<td>1781</td>
<td>1774</td>
<td>4135</td>
<td>4150</td>
</tr>
<tr>
<td><strong>Avg</strong></td>
<td>839.4</td>
<td>833.2</td>
<td>1763.4</td>
<td>1748</td>
<td>4088.8</td>
<td>4122.2</td>
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<tr>
<td><strong>SD</strong></td>
<td>13.32</td>
<td>9.36</td>
<td>16.23</td>
<td>20.84</td>
<td>44.20</td>
<td>31.24</td>
</tr>
<tr>
<td><strong>% non uniformity</strong></td>
<td>3.69</td>
<td>3.12</td>
<td>2.44</td>
<td>2.86</td>
<td>2.40</td>
<td>1.80</td>
</tr>
</tbody>
</table>

<table>
<thead>
<tr>
<th>Sample -&gt;</th>
<th>S1</th>
<th>S7</th>
<th>S2</th>
<th>S3</th>
<th>S4</th>
<th>S5</th>
</tr>
</thead>
<tbody>
<tr>
<td><strong>Thickness (Å)</strong></td>
<td>915</td>
<td>840</td>
<td>1745</td>
<td>1720</td>
<td>4075</td>
<td>4140</td>
</tr>
<tr>
<td></td>
<td>925</td>
<td>845</td>
<td>1805</td>
<td>1800</td>
<td>4115</td>
<td>4170</td>
</tr>
<tr>
<td></td>
<td>930</td>
<td>850</td>
<td>1840</td>
<td>1855</td>
<td>4130</td>
<td>4175</td>
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<td></td>
<td>950</td>
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<td>1880</td>
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<td>970</td>
<td>1980</td>
<td>1865</td>
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<td><strong>Avg</strong></td>
<td>954</td>
<td>881</td>
<td>1850</td>
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<tr>
<td><strong>SD</strong></td>
<td>55.16</td>
<td>55.27</td>
<td>87.96</td>
<td>62.59</td>
<td>72.59</td>
<td>22.19</td>
</tr>
<tr>
<td><strong>% variation</strong></td>
<td>14.15</td>
<td>14.76</td>
<td>12.70</td>
<td>7.96</td>
<td>4.58</td>
<td>1.44</td>
</tr>
</tbody>
</table>

From thickness measurements we see that PECVD silicon dioxide and silicon nitride films had acceptable uniformity, mostly less than 10% thickness variation. Sputtered silicon dioxide films showed a little higher variation as compared to PECVD films. Optimization of process parameters can give better results in terms of closeness to the desired values and uniformity of films.
4.2 Etch Rate Measurements

The etch rates for silicon dioxide and silicon nitride films are a measure of film quality. Films with low porosity show slower etch rates and with increasing pin hole density the etch rate increases [8]. PECVD silicon dioxide and silicon nitride films and sputtered silicon dioxide films were etched in buffered Hydrofluoric acid (10:1 BOE) and etch rates were measured. The average thickness value obtained from Profilometer measurements was used to calculate the etch rates for all samples. The etch rates for PECVD SiO2 were found to be in the range of 0.1 to 0.3 µm/min and for PECVD Si3N4 they were much lower ranging from 0.02 to 0.06 µm/min. The etch rates for sputtered SiO2 films were found to be higher ranging from 0.3 to 1.2 µm/min. For different thicknesses of PECVD silicon dioxide and silicon nitride and sputtered silicon dioxide, the etch rates are shown in Table 7 below.

Table 7 Etch Rates for PECVD SiO2, PECVD Si3N4 and Sputtered SiO2

<table>
<thead>
<tr>
<th>PECVD SiO2</th>
<th>Sample</th>
<th>B1</th>
<th>B2</th>
<th>B3</th>
<th>B4</th>
<th>B5</th>
<th>B6</th>
</tr>
</thead>
<tbody>
<tr>
<td>time (min)</td>
<td>3.4833</td>
<td>2.8667</td>
<td>0.3000</td>
<td>0.3833</td>
<td>1.3333</td>
<td>1.6667</td>
<td></td>
</tr>
<tr>
<td>Thickness (um)</td>
<td>1.2822</td>
<td>0.6477</td>
<td>0.0995</td>
<td>0.1010</td>
<td>0.2119</td>
<td>0.2117</td>
<td></td>
</tr>
<tr>
<td>etch rate (um/min)</td>
<td>0.3681</td>
<td>0.2259</td>
<td>0.3317</td>
<td>0.2635</td>
<td>0.1589</td>
<td>0.1270</td>
<td></td>
</tr>
</tbody>
</table>

<table>
<thead>
<tr>
<th>PECVD Si3N4</th>
<th>Sample</th>
<th>N1</th>
<th>N2</th>
<th>N3</th>
<th>N4</th>
<th>N5</th>
<th>N6</th>
</tr>
</thead>
<tbody>
<tr>
<td>time (min)</td>
<td>3.2500</td>
<td>2.7500</td>
<td>4.7500</td>
<td>4.0000</td>
<td>9.2500</td>
<td>6.8000</td>
<td></td>
</tr>
<tr>
<td>Thickness (um)</td>
<td>0.0860</td>
<td>0.0863</td>
<td>0.1754</td>
<td>0.1816</td>
<td>0.4477</td>
<td>0.4585</td>
<td></td>
</tr>
<tr>
<td>etch rate (um/min)</td>
<td>0.0265</td>
<td>0.0314</td>
<td>0.0369</td>
<td>0.0454</td>
<td>0.0484</td>
<td>0.0674</td>
<td></td>
</tr>
</tbody>
</table>

<table>
<thead>
<tr>
<th>Sputtered SiO2</th>
<th>Sample</th>
<th>S1</th>
<th>S7</th>
<th>S2</th>
<th>S3</th>
<th>S4</th>
<th>S5</th>
</tr>
</thead>
<tbody>
<tr>
<td>time (min)</td>
<td>0.2500</td>
<td>0.1667</td>
<td>0.2500</td>
<td>0.1667</td>
<td>0.5000</td>
<td>0.3333</td>
<td></td>
</tr>
<tr>
<td>Thickness (um)</td>
<td>0.0954</td>
<td>0.0881</td>
<td>0.1850</td>
<td>0.1821</td>
<td>0.4152</td>
<td>0.4174</td>
<td></td>
</tr>
<tr>
<td>etch rate (um/min)</td>
<td>0.3816</td>
<td>0.5286</td>
<td>0.7400</td>
<td>1.0926</td>
<td>0.8304</td>
<td>1.2522</td>
<td></td>
</tr>
</tbody>
</table>
Based on etch rate measurements it is clear that sputtered SiO2 films had higher etch rates which suggests of inferior quality as compared to PECVD films. The etch rates for PECVD silicon nitride are lower than PECVD silicon dioxide which does not necessarily mean that silicon nitride is of better quality. The chemical process between silicon dioxide and HF acid and silicon nitride and HF acid could result in different etch rates. The higher etch rate for 0.1 µm PECVD SiO2 films could be because of low thickness. The etch rates measured here are comparable to the published data. The comparison of etch rates for different thicknesses of PECVD SiO2, PECVD Si3N4 and sputtered SiO2 are graphically presented in Figure 10.

Figure 10 Etch Rate VS Thickness for Different Dielectric Films.
4.3 Electrical Characterization

Electrical characterization is done with Dry Current – Voltage measurements and Wet Current – Voltage measurements as explained in Chapter 3. These measurements give the relation between Dry measurements and Wet measurements and provide a comparison of the dielectric breakdown voltage with respect to dielectric thickness.

4.3.1 Dry Current – Voltage Measurements

Based on Dry – Current Voltage measurements for different thicknesses of PECVD SiO2 films, the breakdown voltage for each thickness was defined when the current reached the value of 1E-8 A. Comparison between breakdown voltages of different thicknesses of PECVD SiO2 are shown in Figure 11 below.

Figure 11 Breakdown Voltages for Different Thicknesses of PECVD SiO2.
For negative 100 V to positive 100 V sweep, the positive breakdown voltages for PECVD SiO2 films were found to be in the range of 70 to 75 V, 85 to 90 V, about 90 V and 95 to 100 V for 0.1 µm, 0.2 µm, 0.5 µm and 1 µm films respectively. There was no breakdown on the negative side for 0.5 µm and 1 µm films, but 0.1 µm and 0.2 µm films did see negative breakdown about 70 to 90 V and 90 to 100 V respectively. For positive 100 V to negative 100 V sweep, the positive breakdown voltages were almost in the same range except for 1 µm film, which did not see any breakdown. The negative breakdown is found to be little lower than that in case of negative to positive sweep. It is believed that when swept from one extreme to other, the breakdown on positive side effects the breakdown on negative side and vice versa. Still the breakdown voltages are high enough for such thin films.

For PECVD Si3N4 and sputtered SiO2 the current levels were higher than those for PECVD SiO2. The current limit used to define breakdown for these films was 1E-7 A. Comparison of breakdown voltages for different thicknesses of PECVD Si3N4 and sputtered SiO2 are shown in Figure 12 and Figure 13 respectively.
From the comparison of breakdown voltages for 0.1 µm, 0.2 µm and 0.5 µm PECVD Si3N4 and sputtered SiO2 films, it is seen that the breakdown voltages for 0.1 µm and 0.5 µm films are higher for PECVD Si3N4 films. For 0.2 µm film the positive breakdown voltages are almost in the same range but negative breakdown voltages are higher for sputtered SiO2. Also, it is seen that with increasing thickness, the breakdown voltage increases as expected, except in case of 0.5 µm sputtered SiO2 film. This is indicative of lower quality of 0.5 µm sputtered film. In case of 0.1 µm and 0.2 µm films a sudden breakdown is also observed at about 2 V.
For positive to negative sweep, the comparison between PECVD Si3N4 and sputtered SiO2 shows approximately the same positive breakdown voltages but the negative breakdown voltages for 0.1 μm and 0.5 μm films are higher in case of PECVD Si3N4.

Based on Dry Current – Voltage measurements, it can be concluded that PECVD SiO2 films show better dielectric quality as compared to PECVD Si3N4 and sputtered SiO2. The breakdown voltages for PECVD Si3N4 and sputtered SiO2 films are much lower than those for PECVD SiO2 films. Also note that the limiting current value for these films was set higher than that for PECVD SiO2 films.
4.3.2 Comparison Between Tungsten and Platinum Probes

In this work, Tungsten probes were used for all the Wet Current – Voltage measurements. To see if there was any effect related to corrosion or oxidation of Tungsten probes on these measurements, a similar test with same setup was conducted with Platinum wire and then again with Tungsten probe. Platinum being a noble metal is more resistant to corrosion and oxidation and hence a better option.

For this test, a 0.2 µm thick PECVD SiO2 sample was used with 1 mM NaCl solution. The comparison between the results obtained is shown in Figure 14 and Figure 15. From the results obtained, it is clear that there is a little variation in the measurements obtained by Platinum wire and Tungsten probe. For negative to positive sweep, with Platinum wire, the negative breakdown (as defined for all wet tests) occurs at a voltage about 10 V lower than that with Tungsten probe. There is no distinguishable variation in the positive breakdown voltages with Platinum wire and Tungsten probe. For positive to negative sweep, with Platinum wire the negative breakdown is again about 10 V lower than that with Tungsten probe and the positive breakdown is about 8 to 10 V lower than that with Tungsten probe. If Platinum probe is used instead of Tungsten probe, it is expected that the breakdown voltages measured in this work will be lower by about 10 V.
Figure 14 Comparison Between Tungsten and Platinum Probes (Negative to Positive Sweep).

Figure 15 Comparison Between Tungsten and Platinum Probes (Positive to Negative Sweep).
4.3.3 Wet Current – Voltage Measurements

On the basis of Wet Current – Voltage measurements, comparison of different thicknesses of PECVD SiO₂ films with 1mM NaCl, 1mM Na₂SO₄ and DI water is done and is presented in Figure 16 and Figure 17. For all the Wet Current – Voltage measurements, the current limit to define breakdown was set to 1E-6 A.

![Graph showing breakdown voltages for different thicknesses of PECVD SiO₂ films with NaCl, Na₂SO₄, and DI water.](image)

**Figure 16** Effect of NaCl, Na₂SO₄ and DI Water on Breakdown Voltages for Different Thicknesses of PECVD SiO₂ (Negative to Positive Sweep).

For negative to positive sweep, in case of NaCl solution, the positive breakdown occurs at low voltages, less than 10 V for all thicknesses. The negative breakdown occurs at much higher voltages, about 42 to 45 V, 50 to 70 V and 85 to 90 V for 0.1 μm, 0.2 μm and 0.5 μm films respectively. No negative breakdown is seen in 1 μm film. The occurrence of breakdown at lower positive potentials indicate flow of current due to
absorption of sodium ions in the dielectric films and higher negative breakdown voltages indicate less absorption of chlorine ions in the films.

In case of Na2SO4 solution, the positive breakdown occurs at higher potentials in a range from 20 to 50 V, above 75 V and about 93 V for 0.1 µm, 0.2 µm and 0.5 µm films respectively with some instances of sudden breakdown at lower potentials. For 1 µm film no breakdown was seen up to 100 V except for 2 instances where a breakdown occurred at 66 and 81 V. The negative breakdown occurred at lower potentials, mostly below 30 V for all films. The negative breakdown at lower potentials indicates flow of current due to absorption of sulphate ions and higher positive breakdown potentials suggest less absorption of sodium ions in the dielectric films. The difference in the effect of NaCl and Na2SO4 solutions on the dielectric breakdown is not clearly understood here and needs further investigation. There is a possibility of the polarity swap of the electrodes in the test setup during the tests conducted with Na2SO4.

In case of DI water, the positive breakdown occurs in the range from 16 to 54 V, above 75 V and above 95 V for 0.1 µm, 0.2 µm and 0.5 µm films respectively. For 1 µm film the breakdown occurred at lower voltages, 52 and 74 V. Negative breakdown did not occur up to 100 V for any of the films. DI water is supposed to be non conducting, but there might be some small amount of impurities which caused the breakdown. Also, since PECVD films are known to have some amount of porosity, this could explain few instances of sudden breakdown.
Figure 17 Effect of NaCl, Na2SO4 and DI Water on Breakdown Voltages for Different Thicknesses of PECVD SiO2 (Positive to Negative Sweep).

For positive to negative sweep, we see a similar trend with both NaCl and Na2SO4 solutions. With NaCl solution, the positive breakdown occurs at lower potentials and negative breakdown at higher potentials. Although, the positive breakdown voltage is little higher when swept from positive to negative potentials than those when swept from negative to positive potential. This again indicates that the failure of film at one polarity causes failure at lower potential on the other polarity.

Based on Current – Voltage measurements, the comparison of different thicknesses of PECVD Si3N4 with 1 mM NaCl, 1mM Na2SO4 and DI water is shown in Figure 18 and Figure 19.
Figure 18 Effect of NaCl, Na2SO4 and DI Water on Breakdown Voltages for Different Thicknesses of PECVD Si3N4 (Negative to Positive Sweep).

For negative to positive sweep, in case of NaCl solution, the positive breakdown occurs around 10 V, in a range from 10 to 35 V and at different voltages from 6 to 13 V for 0.1 µm, 0.2 µm and 0.5 µm films respectively. The negative breakdown occurs approximately around same voltages as positive breakdown for 0.1 and 0.2 µm films but is higher for 0.5 µm films about 60 V. In case of Na2SO4 solution and DI water also the breakdown occurs around the same voltages as NaCl solution.

For positive to negative sweep, again the positive breakdown occurs at lower voltages for all three liquids below 20 V. The negative breakdown for 0.5 µm films is higher ranging from 40 to 62 V for all three liquids. The negative breakdown for 0.1 µm and 0.2 µm films is around 10 V and 15 to 25 V respectively.
Figure 19 Effect of NaCl, Na2SO4 and DI Water on Breakdown Voltages for Different Thicknesses of PECVD Si3N4 (Positive to Negative Sweep).

In case of PECVD Si3N4 films, very less variation is seen in breakdown voltages with different liquids, which suggests an overall lower dielectric quality.

Based on Current – Voltage measurements, the comparison of different thicknesses of sputtered SiO2 with 1 mM NaCl, 1 mM Na2SO4 and DI water is shown in Figure 20 and Figure 21.
Figure 20 Effect of NaCl, Na2SO4 and DI Water on Breakdown Voltages for Different Thicknesses of Sputtered SiO2 (Negative to Positive Sweep).

For negative to positive sweep, the positive breakdown occurs at very low voltages, less than 10 V for 0.1 µm and 0.5 µm films with all three liquids. For 0.2 µm film the positive breakdown occurs at lower voltages about 3 V at some spots and it occurs at higher voltages about 40 V at other spots with NaCl, but with Na2SO4 and DI water the breakdown occurs at voltages less than 10 V. For 0.1 µm film, the negative breakdown occurs in the range from 20 to 30 V with NaCl, 25 to 35 V with Na2SO4 and 15 to 20 V with DI water. For 0.2 µm film, the negative breakdown with NaCl is seen to occur at higher potentials, ranging from 30 to 40 V and from 80 to 90 V, with Na2SO4 around 45 to 50 V and with DI water ranging from 18 to 27 V. For 0.5 µm film, negative breakdown occurs around 26 V with NaCl, ranges between 20 and 40 V with Na2SO4
and is about 20 V with DI water. Since in the negative to positive sweep, breakdown already occurs at negative potential, this causes the dielectric to fail at lower potentials on the positive side.

Figure 21 Effect of NaCl, Na2SO4 and DI Water on Breakdown Voltages for Different Thicknesses of Sputtered SiO2 (Positive to Negative Sweep).

For positive to negative sweep, the positive breakdown with NaCl and Na2SO4 solutions is seen at lower potentials, less than 10 V. But with DI water, the breakdown occurs in a range of 10 to 30 V for all thicknesses. The negative breakdown occurs at low voltages, less than 10 V for all thicknesses with all three liquids, except the 0.2 µm film which fails at different higher voltages i.e. at 34, 59 and 63 V with only NaCl solution.
4.3.4 Comparison of Different Dielectrics in NaCl

The performance of all three dielectrics, PECVD SiO2, PECVD Si3N4 and sputtered SiO2 with NaCl solution, based on Current – Voltage measurements is presented in Figure 22 and Figure 23. With negative to positive sweep, for 0.1 µm films, the negative breakdown voltage for PECVD SiO2 films is highest and for PECVD Si3N4 is lowest. For 0.2 µm films, the negative breakdown voltage for sputtered SiO2 is highest and PECVD Si3N4 is lowest. For 0.5 µm films, the negative breakdown voltage for PECVD SiO2 is highest and sputtered SiO2 is lowest. On the other hand, the positive breakdown voltage for PECVD SiO2 and Si3N is almost similar for 0.1 µm and 0.5 µm films but for 0.2 µm film PECVD Si3N4 shows higher breakdown voltage. The positive breakdown voltage for sputtered SiO2 is much lower for all thicknesses.

Figure 22 Comparison of Different Dielectrics in NaCl (Negative to Positive Sweep).
With positive to negative sweep, positive breakdown voltages for PECVD SiO2 are slightly higher than PECVD Si3N4 and sputtered SiO2 for all thicknesses, except a little variation in 0.5 µm film; in which PECVD Si3N4 show a little higher breakdown voltage. The negative breakdown voltage for PECVD SiO2 is higher in both 0.1 µm and 0.2 µm films, but in case of 0.5 µm film again PECVD Si3N4 shows higher breakdown voltage.

![Figure 23 Comparison of Different Dielectrics in NaCl (Positive to Negative Sweep).](image)

Overall, PECVD SiO2 performs better with NaCl as compared to PECVD Si3N4 and sputtered SiO2.
4.3.5 Comparison of Different Dielectrics in Na2SO4

The performance of all three dielectrics with Na2SO4 is presented in Figure 24 and Figure 25. With negative to positive sweep, the negative breakdown for PECVD SiO2 is highest and for PECVD Si3N4 is lowest for 0.1 µm films with some exceptions where PECVD SiO2 breakdown is at lower voltage. For 0.2 µm films, the negative breakdown voltage for sputtered SiO2 is highest and for PECVD SiO2 is lowest. For 0.5 µm films the negative breakdown voltage is highest for PECVD Si3N4 and lowest for PECVD SiO2. Positive breakdown voltage is highest for PECVD SiO2 for all thicknesses.

Figure 24 Comparison of Different Dielectrics in Na2SO4 (Negative to Positive Sweep).
For positive to negative sweep, the positive breakdown voltage for PECVD SiO2 is again highest for all thicknesses and for PECVD Si3N4 and sputtered SiO2, it is much lower. The negative breakdown voltage for 0.1 µm films was higher for PECVD SiO2 as compared to other two dielectrics. For 0.2 µm and 0.5 µm films, the negative breakdown voltage was higher for PECVD Si3N4. Again with Na2SO4 also, PECVD SiO2 performed best, while PECVD Si3N4 showed better performance at negative potentials for 0.5 µm films and sputtered SiO2 had the lowest dielectric quality.

Figure 25 Comparison of Different Dielectrics in Na2SO4 (Positive to Negative Sweep).
4.3.6 Comparison of Different Dielectrics in DI Water

Based on Current – Voltage measurements, the performance of these dielectrics with DI water is presented in Figure 26 and Figure 27. For negative to positive sweep, again PECVD SiO2 performed best with highest positive breakdown voltages and no negative breakdown for all thicknesses. The positive breakdown voltage for PECVD Si3N4 and sputtered SiO2 were much lower, below 20 V and negative breakdown voltages were almost similar for 0.1 μm and 0.2 μm films, in the range from 15 to 30 V. For 0.5 μm films, the negative breakdown voltage was quite high, in the range from 50 to 70 V.

![Graph](image_url)

**Figure 26 Comparison of Different Dielectrics in DI Water (Negative to Positive Sweep).**
For positive to negative sweep, PECVD Si3N4 and sputtered SiO2 were compared. The positive breakdown voltages for both the dielectrics were almost similar. The negative breakdown voltages for PECVD Si3N4 were higher and for sputtered SiO2 were much lower, less than 5 V.

**Figure 27** Comparison of Different Dielectrics in DI Water (Positive to Negative Sweep).

Based on Wet Current – Voltage measurements, the performance of PECVD SiO2, PECVD Si3N4 and sputtered SiO2 with 3 different liquids, NaCl, Na2SO4 and DI water is studied and the dielectric quality of PECVD SiO2 is found to be the best among these three dielectrics with sputtered SiO2 showing the lowest dielectric quality.
Chapter 5 Conclusions and Future Work

5.1 Conclusions

In this work silicon dioxide and silicon nitride films were deposited using PECVD process and silicon dioxide films were also deposited using sputtering. The quality of films was tested in terms of thickness uniformity, etch rates and also by obtaining I-V characteristics. The thickness uniformity for PECVD silicon dioxide and silicon nitride films was found to be good as non-uniformity was less than 10%. In case of sputtered silicon dioxide films uniformity was found to be less as compared to PECVD films. The etch rates for PECVD silicon dioxide and silicon nitride were found to be lower as compared to sputtered SiO2 films. I-V curves were used to determine the dielectric breakdown voltages for the films also a comparison between dry and wet I-V measurements was done to check the dielectric behavior. As expected, the dielectric performance in dry tests was better than that in wet tests. The performance of all dielectric films was compared with different liquids. Based on the results, PECVD films were found to have better dielectric quality as compared to sputtered films. Also, PECVD SiO2 films were found to have the best dielectric quality. Some of the slight variations in these measurement results can be attributed to non-uniformity of thickness of the dielectrics and local defects in the films.
5.2 Future Work

Different dielectric materials can be characterized by using the methods described in this work and can provide information about their quality which would be helpful to optimize process parameters and achieve better quality dielectric films.

Future work includes deposition of stacked dielectric layers for Electrowetting on dielectric experiments and characterization of these multi layered dielectric films. Different electrolytes with larger ions and different liquids could be used to see their effect on dielectric performance.
References


Appendices
Appendix A: Dry Current – Voltage Plots

Figure A1 I – V Plot for 1 µm Thick PECVD SiO2 (Negative to Positive Sweep).

Figure A2 I – V Plot for 1 µm Thick PECVD SiO2 (Positive to Negative Sweep).
Appendix A: Continued

Figure A3 I – V Plot for 0.5 µm Thick PECVD SiO2 (Negative to Positive Sweep).

Figure A4 I – V Plot for 0.5 µm Thick PECVD SiO2 (Positive to Negative Sweep).
Appendix A: Continued

Figure A5 I–V Plot for 0.2 μm Thick PECVD SiO2 (Negative to Positive Sweep).

Figure A6 I–V Plot for 0.2 μm Thick PECVD SiO2 (Positive to Negative Sweep).
Appendix A: Continued

Figure A7 I – V Plot for 0.1 µm Thick PECVD SiO2 (Negative to Positive Sweep).

Figure A8 I – V Plot for 0.1 µm Thick PECVD SiO2 (Positive to Negative Sweep).
Appendix A: Continued

Figure A9 I – V Plot for 0.5 µm Thick PECVD Si3N4 (Negative to Positive Sweep).

Figure A10 I – V Plot for 0.5 µm Thick PECVD Si3N4 (Positive to Negative Sweep).
Appendix A: Continued

Figure A11 I – V Plot for 0.2 µm Thick PECVD Si3N4 (Negative to Positive Sweep).

Figure A12 I – V Plot for 0.2 µm Thick PECVD Si3N4 (Positive to Negative Sweep).
Appendix A: Continued

Figure A13 I – V Plot for 0.1 µm Thick PECVD Si3N4 (0 to Positive Sweep).

Figure A14 I – V Plot for 0.1 µm Thick PECVD Si3N4 (0 to Negative Sweep).
Appendix A: Continued

Figure A15 I – V Plot for 0.5 µm Thick Sputtered SiO2 (Negative to Positive Sweep).

Figure A16 I – V Plot for 0.5 µm Thick Sputtered SiO2 (Positive to Negative Sweep).
Appendix A: Continued

Figure A17 I – V Plot for 0.2 μm Thick Sputtered SiO2 (Negative to Positive Sweep).

Figure A18 I – V Plot for 0.2 μm Thick Sputtered SiO2 (Positive to Negative Sweep).
Appendix A: Continued

**Figure A19** I–V Plot for 0.1 µm Thick Sputtered SiO2 (Negative to Positive Sweep).

**Figure A20** I–V Plot for 0.1 µm Thick Sputtered SiO2 (Positive to Negative Sweep).
Appendix B: Wet Current – Voltage Plots

Figure B1 I – V Plot for 1 µm Thick PECVD SiO2 with NaCl (Negative to Positive Sweep).

Figure B2 I – V Plot for 1 µm Thick PECVD SiO2 with NaCl (Positive to Negative Sweep).
Appendix B: Continued

Figure B3 I – V Plot for 0.5 μm Thick PECVD SiO2 with NaCl (Negative to Positive Sweep).

Figure B4 I – V Plot for 0.5 μm Thick PECVD SiO2 with NaCl (Positive to Negative Sweep).
Appendix B: Continued

Figure B5 I – V Plot for 0.2 μm Thick PECVD SiO2 with NaCl (Negative to Positive Sweep).

Figure B6 I – V Plot for 0.2 μm Thick PECVD SiO2 with NaCl (Positive to Negative Sweep).
Appendix B: Continued

Figure B7 I – V Plot for 0.1 µm Thick PECVD SiO2 with NaCl (Negative to Positive Sweep).

Figure B8 I – V Plot for 0.1 µm Thick PECVD SiO2 with NaCl (Positive to Negative Sweep).
Appendix B: Continued

Figure B9 I – V Plot for 1 µm Thick PECVD SiO2 with Na2SO4 (Negative to Positive Sweep).

Figure B10 I – V Plot for 1 µm Thick PECVD SiO2 with Na2SO4 (Positive to Negative Sweep).
Appendix B: Continued

Figure B11 I – V Plot for 0.5 μm Thick PECVD SiO2 with Na2SO4 (Negative to Positive Sweep).

Figure B12 I – V Plot for 0.5 μm Thick PECVD SiO2 with Na2SO4 (Positive to Negative Sweep).
Appendix B: Continued

Figure B13 I – V Plot for 0.2 μm Thick PECVD SiO2 with Na2SO4 (Negative to Positive Sweep).

Figure B14 I – V Plot for 0.2 μm Thick PECVD SiO2 with Na2SO4 (Positive to Negative Sweep).
Appendix B: Continued

Figure B15 I – V Plot for 0.1 µm Thick PECVD SiO2 with Na2SO4 (Negative to Positive Sweep).

Figure B16 I – V Plot for 0.1 µm Thick PECVD SiO2 with Na2SO4 (Positive to Negative Sweep).
Appendix B: Continued

Figure B17 I – V Plot for 1 µm Thick PECVD SiO2 with DI Water (0 to Positive Sweep).

Figure B18 I – V Plot for 0.5 µm Thick PECVD SiO2 with DI Water (0 to Positive Sweep).
Appendix B: Continued

Figure B19 – V Plot for 0.2 μm Thick PECVD SiO2 with DI Water (0 to Positive Sweep).

Figure B20 – V Plot for 0.1 μm Thick PECVD SiO2 with DI Water (0 to Positive Sweep).
Appendix B: Continued

Figure B21 – V Plot for 0.5 µm Thick PECVD Si3N4 with NaCl (Negative to Positive Sweep).

Figure B22 – V Plot for 0.5 µm Thick PECVD Si3N4 with NaCl (Positive to Negative Sweep).
Appendix B: Continued

Figure B23 I – V Plot for 0.2 µm Thick PECVD Si3N4 with NaCl (Negative to Positive Sweep).

Figure B24 I – V Plot for 0.2 µm Thick PECVD Si3N4 with NaCl (Positive to Negative Sweep).
Appendix B: Continued

Figure B25 I – V Plot for 0.1 μm Thick PECVD Si3N4 with NaCl (Negative to Positive Sweep).

Figure B26 I – V Plot for 0.1 μm Thick PECVD Si3N4 with NaCl (Positive to Negative Sweep).
Appendix B: Continued

Figure B27 $I - V$ Plot for 0.5 $\mu$m Thick PECVD Si3N4 with Na2SO4 (Negative to Positive Sweep).

Figure B28 $I - V$ Plot for 0.5 $\mu$m Thick PECVD Si3N4 with Na2SO4 (Positive to Negative Sweep).
Appendix B: Continued

Figure B29 I – V Plot for 0.2 µm Thick PECVD Si3N4 with Na2SO4 (Negative to Positive Sweep).

Figure B30 I – V Plot for 0.2 µm Thick PECVD Si3N4 with Na2SO4 (Positive to Negative Sweep).
Appendix B: Continued

Figure B31 I – V Plot for 0.1 μm Thick PECVD Si3N4 with Na2SO4 (Negative to Positive Sweep).

Figure B32 I – V Plot for 0.1 μm Thick PECVD Si3N4 with Na2SO4 (Positive to Negative Sweep).
Appendix B: Continued

Figure B33 I – V Plot for 0.5 µm Thick PECVD Si3N4 with DI Water (Negative to Positive Sweep).

Figure B34 I – V Plot for 0.5 µm Thick PECVD Si3N4 with DI Water (Positive to Negative Sweep).
Appendix B: Continued

Figure B35 I – V Plot for 0.2 µm Thick PECVD Si3N4 with DI Water (Negative to Positive Sweep).

Figure B36 I – V Plot for 0.2 µm Thick PECVD Si3N4 with DI Water (Positive to Negative Sweep).
Appendix B: Continued

Figure B37 I–V Plot for 0.1 µm Thick PECVD Si3N4 with DI Water (Negative to Positive Sweep).

Figure B38 I–V Plot for 0.1 µm Thick PECVD Si3N4 with DI Water (Positive to Negative Sweep).
Appendix B: Continued

Figure B39 – V Plot for 0.5 µm Thick Sputtered SiO2 with NaCl (Negative to Positive Sweep).

Figure B40 – V Plot for 0.5 µm Thick Sputtered SiO2 with NaCl (Positive to Negative Sweep).
Appendix B: Continued

Figure B41  I – V Plot for 0.2 \( \mu \)m Thick Sputtered SiO2 with NaCl (Negative to Positive Sweep).

Figure B42  I – V Plot for 0.2 \( \mu \)m Thick Sputtered SiO2 with NaCl (Positive to Negative Sweep).
Appendix B: Continued

Figure B43 I – V Plot for 0.1 µm Thick Sputtered SiO2 with NaCl (Negative to Positive Sweep).

Figure B44 I – V Plot for 0.1 µm Thick Sputtered SiO2 with NaCl (Positive to Negative Sweep).
Appendix B: Continued

Figure B45 - V Plot for 0.5 µm Thick Sputtered SiO2 with Na2SO4 (Negative to Positive Sweep).

Figure B46 - V Plot for 0.5 µm Thick Sputtered SiO2 with Na2SO4 (Positive to Negative Sweep).
Appendix B: Continued

Figure B47 I – V Plot for 0.2 µm Thick Sputtered SiO2 with Na2SO4 (Negative to Positive Sweep).

Figure B48 I – V Plot for 0.2 µm Thick Sputtered SiO2 with Na2SO4 (Positive to Negative Sweep).
Appendix B: Continued

**Figure B49** – V Plot for 0.1 μm Thick Sputtered SiO2 with Na2SO4 (Negative to Positive Sweep).

**Figure B50** – V Plot for 0.1 μm Thick Sputtered SiO2 with Na2SO4 (Positive to Negative Sweep).
Appendix B: Continued

Figure B51 I–V Plot for 0.5 μm Thick Sputtered SiO2 with DI Water (Negative to Positive Sweep).

Figure B52 I–V Plot for 0.5 μm Thick Sputtered SiO2 with DI Water (Positive to Negative Sweep).
Appendix B: Continued

Figure B53 I – V Plot for 0.2 µm Thick Sputtered SiO2 with DI Water (Negative to Positive Sweep).

Figure B54 I – V Plot for 0.2 µm Thick Sputtered SiO2 with DI Water (Positive to Negative Sweep).
Figure B55 – V Plot for 0.1 µm Thick Sputtered SiO2 with DI Water (Negative to Positive Sweep).

Figure B56 – V Plot for 0.1 µm Thick Sputtered SiO2 with DI Water (Positive to Negative Sweep).
About the Author

Ajay Rajgadkar was born in Nagpur, Maharashtra, India and earned a Diploma in Mechanical Engineering from Shri Datta Meghe Polytechnic, Nagpur and a B.E. Degree in Mechanical Engineering from Yeshwantrao Chavan College of Engineering, Nagpur University. He worked as a Quality Assurance Engineer at Mini Iron and Steel Pvt. Ltd., Nagpur, India while working towards his Bachelor’s Degree. He earned a Master’s Degree in Mechanical Engineering from University of South Florida, Tampa, Florida. He worked as a Graduate Assistant at Nanotechnology Research and Education Center at University of South Florida and helped with maintenance of different tools and provided training to students and industrial researchers on these tools.