Investigation of MIM Diodes for
RF Applications

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ABSTRACT

Metal Insulator Metal (MIM) diodes that work on fast mechanism of tunneling have been used in a number of very high frequency applications such as (Infra- Red) IR detectors and optical Rectennas for energy harvesting. Their ability to operate under zero bias condition as well as the possibility of realizing them through printing makes them attractive for (Radio Frequency) RF applications. However, MIM diodes have not been explored much for RF applications. One reason preventing their widespread RF use is the requirement of a very thin oxide layer essential for the tunneling operation that requires sophisticated nano-fabrication processes. Another issue is that the reliability and stable performance of MIM diodes is highly dependent on the surface roughness of the metallic electrodes. Finally, comprehensive RF characterization has not been performed for MIM diodes reported in the literature, particularly from the perspective of their integration with antennas as well as their rectification abilities.

In this thesis, various metal deposition methods such as sputtering, electron beam evaporation, and Atomic Layer Deposition (ALD) are compared in pursuit of achieving low surface roughness. It is worth mentioning here that MIM diodes realized through ALD method have been presented for the first time in this thesis. Amorphous metal alloy have also been investigated in terms of their low surface roughness. Zinc-oxide has been investigated for its suitability as a thin dielectric layer for MIM diodes. Finally, comprehensive RF characterization of MIM diodes has been performed in two ways: 1) by standard S-parameter methods, and 2) by investigating their rectification ability under zero bias operation.

It is concluded from the Atomic Force Microscopy (AFM) imaging that surface roughness as low as sub 1 nm can be achieved reliably from crystalline metals such as copper and platinum. This value is comparable to surface roughness achieved from amorphous alloys, which are non-crystalline structures and have orders of magnitude lower conductivities. Relatively lower
resistances of the order of 1 k ohm with a sensitivity of 1.5 V⁻¹ have been obtained through DC testing of these devices. Finally, RF characterization reveals that input impedances in the range of 300 Ω to 25 Ω can be achieved in the low GHz frequencies (from 1-10 GHz). From the rectification measurements at zero bias, a DC voltage of 4.7 mV has been obtained from an incoming RF signal of 0.4 W at 2.45 GHz, which indicates the suitability of these diodes for RF rectenna devices without providing any bias. It is believed that with further optimization, these devices can play an important role in RF energy harvesting without the need to bias them.
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<td>ADS</td>
<td>Advanced Design System</td>
</tr>
<tr>
<td>AFM</td>
<td>Atomic Force Microscopy</td>
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<tr>
<td>BPF</td>
<td>Band Pass Filter</td>
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<tr>
<td>CAD</td>
<td>Computer Aided Design</td>
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<tr>
<td>CPW</td>
<td>Co Planar Waveguide</td>
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<td>DC</td>
<td>Direct Current</td>
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<tr>
<td>EDX</td>
<td>Energy Dispersive X-Ray</td>
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<tr>
<td>F-N</td>
<td>Fowler-Nordheim</td>
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<tr>
<td>GSM</td>
<td>Global System for Mobile Communications</td>
</tr>
<tr>
<td>HAADF</td>
<td>High Angle Angular Dark Field STEM</td>
</tr>
<tr>
<td>IR</td>
<td>Infra - Red</td>
</tr>
<tr>
<td>ISM</td>
<td>Industry Science Medical</td>
</tr>
<tr>
<td>MIM</td>
<td>Metal Insulator Metal</td>
</tr>
<tr>
<td>P-F</td>
<td>Poole – Frenkel</td>
</tr>
<tr>
<td>RF</td>
<td>Radio Frequency</td>
</tr>
<tr>
<td>RMS</td>
<td>Root Mean Square</td>
</tr>
<tr>
<td>SSPS</td>
<td>Space Solar Power System</td>
</tr>
<tr>
<td>STEM</td>
<td>Scanning Transmission Electron Microscopy</td>
</tr>
<tr>
<td>WKB</td>
<td>Wentzel–Kramers–Brillouin</td>
</tr>
<tr>
<td>WLAN</td>
<td>Wireless Local Area Network</td>
</tr>
<tr>
<td>WPT</td>
<td>Wireless Power Transmission</td>
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1. INTRODUCTION

RF energy is defining the paradigm shift in smart environment where a multitude of sensors and devices are being used to deliver useful information in large quantities. This is particularly important for applications such as very small smart dust sensors or implantable sensors [1, 2], where the use of battery will be difficult. RF energy - omnipresent in the modern world in the form of mobile GSM and Wi-Fi signals - offers an interesting source of energy to power a system remotely from far-field RF technology. In most of these cases, a dedicated RF source is used, which can provide sufficient power to operate these sensors. An alternate approach is to power these devices from ambient RF energy around us. However, in this case, the RF power is low and, thus, puts a lot of constraint on the efficiency of the system. In either case, a rectenna is required which consists of rectifier (diode) to rectify the incoming RF signal and convert it into useful DC signal.

Mode of RF energy free space transmission dates back to 1950s with possible applications in microwave power aircraft [3] and Solar Power Satellite Concept[4]. More recently, interest has been triggered by Industry-Science-Medical (ISM) bands around 0.9, 2.4, 5.8 GHz[5]. It needs to be mentioned that the topic of interest here is about far-field RF energy transmission and not close contact inductive RF energy transmission. To give a more holistic introduction to this topic, three main sub-topics 1) Rectenna system for converting RF energy to DC energy, 2) Ambient RF Energy, and 3) MIM Diodes for rectification need to be explained in more detail.

1.1 Rectenna

Microwave Transmission has been under intensive development in both theoretical and application aspects such as wireless power transmission (WPT) and space solar power system (SSPS). RF energy can be gathered using a device called Rectenna. Rectenna utilizes the wave
nature of electromagnetic radiation by collecting wavelengths of the desired band and rectifying them to get DC energy[6].

A simple Rectenna system [2] is shown in Figure 1.

![Figure 1. Rectenna System to harvest RF energy [2]](image)

The antenna is used to collect RF energy. This is followed by the Band Pass Filter (BPF), which is used to filter frequencies within a certain range by suppressing unwanted high order harmonics. The next important cog of the rectenna system is the matching network required in order to provide impedance matching with the rectifying component, which consists of a diode. The rectifying component (diode) rectifies the received RF energy, which is then smoothened using the DC pass filter in order to obtain DC Voltage at the load side.

### 1.2 Ambient RF Energy

Having looked at rectenna system for converting RF energy to DC energy, we will now look at the ambient RF energy. RF energy, in the form of microwaves, is plentiful around us. The most common RF energy bands are GSM900 (900 MHz), GSM1800 (1800 MHz) and WLAN (2.45 GHz). These RF energy frequencies are omnipresent in an urban environment, and they use resonant antennas which have areas in the order of 10-50 cm². In comparison, broadcasting systems (Radio and TV) operate at much lower frequencies. As a result of this, they require large antennas, and hence, not good options for RF energy harvesting in small systems.
Power density levels data gathered in Cooperation in Science and Technology (COST) Action 244 bis “Biomedical Effects of Electromagnetic Fields”[7] have been used in the referenced work[5], and have been shown in Figure 2. This data was gathered in Austria, Germany, and Hungary between November 1996 and November 2000 in the frequency range 935 – 960 MHz (GSM900).

Figure 2. GSM900 peak power density. Code “XY-a” indicates area and measurement site characteristics. XY: IC ¼ inner city; OC ¼ outer country; IR ¼ industrial area; ST ¼ small town; R ¼ rural or countryside area; 1 ¼ outdoors on roof, terrace or balcony; 2 ¼ indoors, close to windows, 1.5 m or less; 3 ¼ indoors, not close to windows.

This figure shows power density of GSM900 and it does not include data gathered outdoors on ground level. This is because large variation would result in skewed data. It can be understood from the figure that in between 25 and 100 m from a GSM900 base station, a power density between 0.01-1 mW/m² can be obtained indoors everywhere and on elevated level outdoors. Similar power density levels can be obtained from GSM1800 base stations up to 100 m, whereas wireless local area network (WLAN) power density levels are at least one order magnitude lower [8]. These measurements show that for obtaining an incident RF power of 100 µW, an area equivalent to about one A4 page (330 cm²) is required. It is also important to mention that of this 100 µW incident RF power, efficiency limitations, antenna to rectifier mismatch, RF to DC conversion, and rectifier to load mismatch will all lead to even smaller
power levels. The rectifier which is composed of diode is required to rectify the incoming RF signal and convert it into useful DC signal. These ambient RF power levels look small and, hence, a rectifier with low or no operating bias is desirable for RF energy harvesting. If the diode does not draw any power for its own operation, it is typically known as zero bias operation. This is not feasible with typical semiconductor based diodes. In contrast, MIM diodes offer zero-bias operation by utilizing two different work function metals. Also, there is a possibility of realizing them through additive manufacturing techniques such as printing, thus enabling easy integration with low cost large area electronics. It is worth mentioning here that printing semi-conductor based diodes for GHz operation is still a big challenge for the research community.

The focus of the work presented is on the development of MIM diodes with operation at zero bias for RF energy harvesting applications. For small power applications, a frequency of 2.45 GHz offers choice of smaller antennas compared to 900 MHz. Effective zero bias rectification using MIM diodes offers a great opportunity for such RF applications. The same has been shown in this work, specifically at 2.45 GHz. A brief introduction to MIM diodes will be presented in the following sub-section with more details in chapter-2.

1.3 MIM Diode

MIM diode is a quantum mechanical junction device, which is formed by two electrodes separated by a thin-film insulating layer. The quantum tunneling effect leads to electron transfer between the two electrodes which should ideally have different work functions (labelled in Figure 3)[9, 10].

MIM diodes coupled with an antenna to create rectennas hold great promise for detecting and mixing high frequency signals[11, 12]. MIM diodes are also anticipated to outperform
counterparts, either hetero or Schottky junctions, where higher frequency rectification is required[13].

![Energy Diagram of MIM Diode](image)

**Figure 3.** Representation of energy diagram of MIM diode with two metals with different work functions[10]

### 1.4 Challenges

MIM diodes offer various challenges, ranging from the theory of operation to fabrication. Theory requires deep understanding of quantum mechanics. Fabrication of MIM diodes requires advanced nano-fabrication tools. The issue of bottom electrode roughness as detailed subsequently, in MIM diodes requires deposition methods optimized for smooth metal depositions. Additionally, for a very thin insulator (few nm), accurate deposition method technique is also required. Also, RF characterization for MIM diodes has been limited due to the difficulty in measurements.

### 1.5 Objectives

- Investigation of different metal deposition methods to achieve low surface roughness for stable MIM diode operation. In particular, crystalline metals to be compared with amorphous alloys for this investigation.
• Design and fabrication of MIM diodes to assess their DC performance, i.e. I-V response, differential resistance, and non-linearity.

• Characterize MIM diodes for RF performance through s-parameters. In addition, investigate its ability to rectify RF signals at zero bias by appropriate feeding and loading arrangements.

1.6 Thesis Organization

• Chapter-2 gives a review of MIM diodes theory. In addition, recent work on MIM diodes; which includes MIM diodes with RF characterization and the use of amorphous alloys to overcome the hypothesized metal roughness issue in MIM diodes, is covered.

• Chapter-3 provides MIM diode fabrication details and work on analysis of roughness for crystalline metals deposited using different techniques (sputtering, e-beam evaporation, and ALD). Surface roughness analysis for amorphous alloys has also been presented.

• Chapter-4 contains the results for the DC and RF characterization of different MIM diodes fabricated. This includes the demonstration of RF rectification using MIM diode.

• The final chapter presents the conclusion and possible future work.

1.7 Contributions

➢ Thorough investigation of the effects of various deposition methods on the surface roughness of metals in a MIM diode arrangement has been conducted. Surface roughness of sub 1 nm has been achieved for crystalline metals.

➢ Zero bias operation of MIM diode through different work function metals has been demonstrated.

➢ Comprehensive RF characterization for MIM diode has been performed through two methods: 1) s-parameters, and 2) rectification process via an input signal and attached load.
2. LITERATURE REVIEW

MIM diodes have been in use for some time, and various prototypes fabricated with different metals and oxides have been demonstrated. They work on an inherently fast mechanism of tunneling and can be used in very high-frequency applications. Although MIM diodes have been demonstrated for ultra-high frequencies (in the infrared range, etc), their use in RF domain is not that common. This chapter explains the basic theory and operation of the MIM diode, and also provides a summary of MIM diode work in literature with a special emphasis on MIM diodes in the RF domain.

2.1 Conduction Mechanism in Dielectric Films

As explained in the introduction, MIM diodes use a very thin insulator layer (dielectric) sandwiched between two electrodes. Tunneling across the insulator layer leads to electron transfer. Understanding the conduction mechanisms in the insulator is important in order to comprehend MIM diode operation.

Dielectric materials are nearly insulators with very low electrical conductivity and large energy band gap. The valence band for dielectric is completely filled and the conduction band is empty. For temperatures larger than 0 K, some electrons are thermally excited from the valence band and also from the donor impurity level to the conduction band. These electrons, in addition to the holes generated by the acceptor impurities and the vacancies left by the excited electrons lead to a conduction current. However, the conduction current at normal electric field will be small because of the inherent low conductivities of insulators. In case of larger electric field, the conduction current through the dielectric is noticeable.

Dielectrics have two conduction mechanisms: 1) Electrode-limited conduction mechanism, and 2) Bulk-limited conduction mechanism [14].
2.1.1 Electrode Limited Conduction Mechanism

This mechanism depends on the electrical properties at the electrode-dielectric, leading to the term electrode-limited conduction mechanism. Barrier height at the electrode-dielectric contact and the effective mass of the conduction carriers in dielectric are important parameters, which affect the conduction mechanism. Within electrode conduction mechanisms, there are four different types: 1) Schottky or thermionic emission, 2) Fowler-Nordheim (F-N) tunneling, 3) Direct tunneling, and 4) Thermionic-field emission.

Schottky emission is the conduction mechanism when electrons obtain enough energy by thermal activation to overcome the energy barrier at the metal-dielectric interface. This mechanism is the most common conduction mechanism, especially at relatively high temperature.

According to classical physics, when the energy of incident electrons is less than the potential barrier, the electrons will be reflected. However, quantum mechanics predicts that potential barrier for the case when the barrier is very thin (less than 10 nm) will not be enough to stop penetration of the electron wave function. Both F-N tunneling and direct tunneling refer to the quantum mechanical tunneling for the specific case of electrons not having energy to cross the potential barrier mentioned. For the case of high voltage or thick insulator F-N tunneling is dominant, while for the case of low voltage/thinner insulator direct tunneling is more dominant. Direct Tunneling refers to transport through the forbidden band across the entire insulator. In F-N tunneling, charges tunnel through a part of the insulator before entering the insulator conduction band. Due to the smaller tunneling distance in the case of F-N tunneling, there is a larger current than in the case of direct tunneling. The transition from direct to F-N tunneling occurs as the magnitude of bias voltage is increased in forward or reverse direction.
The last type of electrode-limited conduction mechanism is thermionic-field emission. In this type emission takes place intermediately between field and thermionic emission. In this case, tunneling electrons require energy between the Fermi level of metal and conduction band edge of dielectric[14].

2.1.2 Bulk-Limited Conduction Mechanism

Bulk limited conduction mechanism depends on the dielectric’s electrical properties. Some of the types of this conduction mechanism are: 1) Poole – Frenkel (P-F) emission, 2) hopping conduction, 3) ohmic conduction, and 4) ionic conduction.

In the case of P-F emission, thermal excitation of electrons may emit from traps in the conduction band of dielectric. Hopping conduction refers to the conduction of trapped electrons in insulator hopping from one trap site to another. Ohmic conduction, which is the usual type of conduction for metals, refers to the movement of mobile electrons in the conduction band and holes in valence band. Since the energy band gap of dielectrics is large compared to conductors, this mechanism contributes little to the overall conduction in dielectrics. In the case of ionic conduction, lattice defects in dielectric films may give rise to movement of ions.

In all the types of bulk-limited mechanism, the properties of the dielectric govern the conduction. However, this is not the case for electrode-limited conduction mechanism[14]. MIM diode operates on electrode-limited conduction mechanism; specifically F-N or direct tunneling, also defined as quantum-mechanical tunneling.

2.2 MIM Diode- Operating Principle

Having looked into the conduction mechanism for MIM diode, the next step would be to look into the operation principle of MIM diode. MIM diode is made up of two metal electrodes that are separated by an extremely thin (few nanometers) insulator. Metals having higher work
function than the electron affinity of the insulator produce a barrier at the metal/insulator interface[9].

This barrier allows charge transport across the insulator due to quantum-mechanical tunneling. Transmission probability of charge transport is associated with the possibility of an electron tunneling through the classically forbidden region of insulator bandgap, as shown in figure 4.

![Figure 4. Energy-band profile of a MIM diode][9]

The probability of electron tunneling depends on the thickness and the height of the insulator barrier, which changes shape with the voltage across the diode. This equates to the nonlinear dependence of the tunnel current on the applied voltage and diode characteristics. Wentzel–Kramers–Brillouin (WKB) approximation has been used mostly to model MIM diode characteristics. Electron tunneling, which is the dominant conduction mechanism in MIM diodes instead of bulk-limited conduction mechanism, occurs on a femtosecond timescale. This is ensured by having insulating layer of no more than a few nanometers[9].

The first ever MIM diode was reported by Fisher [15], in which thin films of aluminum oxide were sandwiched between aluminum electrode films. Aluminum films were deposited from vapor, followed by oxidation using three different ways; air, oxygen and distilled water. In
[15], the variations of junction's current and resistance with applied voltage, and oxide thickness were measured.

Simmons in 1963 [16] followed on Fisher’s work. A formula was derived for electric tunnel effect through a barrier of any arbitrary shape. The effect of the dielectric constant of insulating film was also discussed in detail in the mentioned work. Equation 2.1 for current density was obtained from Simmons’ work and was used by Nasir in [17]:

\[
J = \frac{1.1 \, q^2 \, 1}{4 \pi h \, \varphi_b} \left( \frac{V + \Delta \varphi_b}{S} \right)^2 \times \exp\left(\frac{-23 \pi \sqrt{q \, m}}{6 \, h} \varphi_b \frac{3}{2} \left( \frac{S}{V + \Delta \varphi_b} \right)\right)
\]

(2.1)

Where q is the electric charge, h is Plank's constant, V is the applied bias, \( \varphi_b \) is the barrier height of the electrode insulator interface from which electrons are tunneling, \( \Delta \varphi_b \) is the difference in barrier heights between interfaces of the insulator with the top and bottom electrodes, m is the effective electron mass, S is the tunnel barrier thickness, and J is the tunneling current density. If the equation is analyzed, it can be observed that a) current density decreases with increase in tunnel barrier thickness, and b) work function difference and Fermi level gradient is required between two electrodes for tunneling of electrons.

### 2.3 MIM Diode Performance Parameters

Various parameters are used to characterize MIM diodes which depend on the band structure diagram. Resistance - more commonly known as Differential Resistance \((r_D)\) - is obtained by differentiating current with respect to the applied voltage, with formulae shown below. Generally, low impedance is required for the case when diode is used in RF applications such as rectenna- rectifier (diode) integrated with an antenna. To obtain low resistance, barrier heights and insulator thickness need to be small.

\[
r_D = \frac{1}{I'} \quad \text{where} \quad I' = \frac{dI}{dV}
\]

(2.2)
Another important parameter is the responsivity, which is the measure of how efficiently a diode can rectify. Responsivity has various definitions but the one used in this work is shown in the equation below. This parameter is useful to measure the rectification ability of the diode. Higher the numeric value of responsivity, higher is the rectification ability of the diode. The unit of responsivity is $V^{-1}$. To obtain large responsivity, large curvature in IV graph is required.

$$R = \frac{l''}{l'}, \text{ where } l'' = \frac{dI^2}{d^2V} \text{ and } l' = \frac{dI}{dV}$$ (2.3)

Responsivity ($R$) determines the rectified voltage, for the case when $v_{ac}$ is applied to the MIM diode, the rectified voltage $V_{rect}$ is given by the equation below[18].

$$V_{rect} = \frac{1}{4}(R)(v_{ac})^2$$ (2.4)

### 2.4 MIM Diode - Summary of Previously published work

In this section, overview of prominent work related to MIM diodes has been presented. After the initial work by Fisher[15], point-contact MIM diodes were made by pressing a thin metal wire against an oxidized sheet of metal[19]. These diodes offered advantage of achieving small junction areas without the need of fine lithography. Progress in lithography has allowed small area MIM diodes fabrication with more reliability. Additionally, the availability of different deposition techniques especially for thin insulator layer such as ALD has given the freedom to choose barrier and metal materials. It is worth mentioning here, that for the case when insulator layer is deposited instead of oxidation of the bottom metal, importance needs to be given to the roughness of the bottom electrode.

Table 1 below shows the summary of previously reported MIM diodes. It can be seen from the table, that most of the work on MIM diodes has been on DC characterization of diodes, some works where the applications have been shown, targets the high frequency optical rectennas and detectors, which are working in Terahertz range. MIM diodes with some type of RF
characterization have been shown in very few instances [10, 20-23]. Another notable observation is that except for few reported MIM diodes[20, 23-25], zero-bias resistance has been in the range of Mega ohms. Additionally most of the reported MIM diodes have been based on using crystalline metals as bottom electrodes, e.g copper, nickel, niobium and titanium. However, recently work has been reported in [17, 26-29] mentioning issue of high surface roughness, in the case of crystalline metals being used as bottom electrodes. The reported work mentions use of amorphous metal alloy to overcome bottom electrode surface roughness issue.

Based on the summary presented in this section, some of the reported works which are more relevant to the work being presented in this thesis, will be discussed in the next sub-section. These include: 1) MIM diodes with RF characterization, and 2) MIM diodes fabricated using amorphous alloy.
Table 1. Previously reported MIM diodes; Design, Resistance, Responsivity and RF Characterization

<table>
<thead>
<tr>
<th>MIM Design</th>
<th>Zero Bias Resistance (ohm)</th>
<th>Zero Bias Responsivity (1/V)</th>
<th>Maximum Responsivity (1/V)</th>
<th>Oxide Thickness (nm)</th>
<th>RF Characterization</th>
</tr>
</thead>
<tbody>
<tr>
<td>Hoofring 1989 [30]</td>
<td>Thin film Ni-NiO-Au (0.64 mm2)</td>
<td>-</td>
<td>2.8</td>
<td>5.5</td>
<td>2.2</td>
</tr>
<tr>
<td>I. Wilke, et. al 1994 [24]</td>
<td>Ni-NiO-Ni (0.0576 mm2)</td>
<td>100</td>
<td>-</td>
<td>1.6</td>
<td>4</td>
</tr>
<tr>
<td>M. Abdel-Rahman et al. 2004 [20]</td>
<td>Thin film Ni-NiO-Ni (0.075 mm2 and 0.0014 mm2)</td>
<td>180</td>
<td>-</td>
<td>2.75 &amp; 1.65</td>
<td>3.5</td>
</tr>
<tr>
<td>Esfandiari 2005 [31]</td>
<td>Thin film Ni-NiO-Pt (0.0025 mm2)</td>
<td>-</td>
<td>-3</td>
<td>-13</td>
<td>2</td>
</tr>
<tr>
<td>S. Krishnan et al. 2008 [32]</td>
<td>Thin film Ni-NiO-cr/Au (1 mm2)</td>
<td>500K</td>
<td>1</td>
<td>5</td>
<td>3</td>
</tr>
<tr>
<td>Choi et al. 2010 [33]</td>
<td>polysilicon-SiO2-polysilicon(60 mm2)</td>
<td>-</td>
<td>12</td>
<td>-31</td>
<td>1.38</td>
</tr>
<tr>
<td>Dagenais et al. 2010 [33]</td>
<td>Thin film polysilicon-SiO2-Au (0.35 mm2)</td>
<td>120 M</td>
<td>2.5</td>
<td>-14.5</td>
<td>60</td>
</tr>
<tr>
<td>Bean et al. 2011 [34]</td>
<td>Thin Al-AlOx-Pt (0.5652 mm2)</td>
<td>220 K</td>
<td>0.5</td>
<td>-2.3</td>
<td>0.6</td>
</tr>
<tr>
<td>K. Choi, et al. 2011 [22]</td>
<td>Ni/NiO/Ni (0.029 um2)</td>
<td>15.25 M</td>
<td>-0.14</td>
<td>4.44</td>
<td>4</td>
</tr>
<tr>
<td>Zhang et al. 2013 [35]</td>
<td>Ni-NiO-Cu (0.008 mm²)</td>
<td>1.2 M</td>
<td>-</td>
<td>7.3</td>
<td>2-12</td>
</tr>
<tr>
<td>Kinzel et al. 2013 [25]</td>
<td>Al-Al2O3-Pt (0.008 mm²)</td>
<td>124.6</td>
<td>1.24 x 10⁻³</td>
<td>0.03</td>
<td>2</td>
</tr>
<tr>
<td>Zhu et al. 2013 [36]</td>
<td>Graphene-Air-Graphene</td>
<td>-</td>
<td>0.12</td>
<td>0.24</td>
<td>-</td>
</tr>
<tr>
<td>NA</td>
<td>Cu/CuO/Cu</td>
<td>180 K</td>
<td>-</td>
<td>4.497</td>
<td>2</td>
</tr>
<tr>
<td>M. Abdel-Rahman, et al 2013 [38]</td>
<td>ZCAN/Al2O3/HfO2/Al</td>
<td>-</td>
<td>-</td>
<td>-</td>
<td>1-5</td>
</tr>
<tr>
<td>N.Alnardani, et al 2013 [28]</td>
<td>ZCAN/HfO2/Al2O3/Al</td>
<td>-</td>
<td>-</td>
<td>-</td>
<td>10</td>
</tr>
</tbody>
</table>
2.5 MIM Diodes with RF characterization

As discussed, work on MIM Diodes with RF characterization has been limited. In this section three reported works on RF characterization of MIM diodes will be discussed. The three referenced works cover DC characterization of MIM diodes followed by RF characterization[10, 22, 23].

2.5.1 MIM Diode using Field Enhancement with RF characterization

In the work reported by Choi [22], field enhancement technique in a Ni/NiO/Ni MIM Tunneling structure has been employed to improve diode performance. MIM Diodes implemented by field enhancement are referred as focused asymmetric MIM (FAMIM) diodes. The first step of fabrication process involved deposition of SiO$_2$ using PECVD followed by patterning of electrode using e-beam writer. Nickel Bottom electrode was then deposited using e-beam evaporator. NiO was formed using Reactive Ion Etching Tool (RIE) with oxygen plasma. Second electrode was formed using the same process of e-beam writer, and deposition using e-beam evaporator. In the referenced work other than the current-voltage characteristics dynamic response at 6.4 GHz has also been presented. MIM Diodes with notable performance showed zero bias resistance around 15.25 M and maximum sensitivity of 4.44 at 0.1V, with insulator thickness in the range of 2-4 nm. The sensitivity is good compared to the values reported in literature, however the zero-bias resistance value is very high, which is generally not good for integration with RF applications.

Three types of MIM Diodes were employed in the referenced work, as shown in figure 5. Type-A MIM is the conventional structure with a simple overlap structure of two metal lines. Tunnel barrier lowers as a result of a more abrupt junction at the interface of the second electrode, creating a work function difference even in the case of similar metals, which eventually results in forward tunneling current becoming larger than the reverse tunneling current. In type-B
MIM, a sharply pointed electrode facing a second planar electrode with a tunneling barrier was implemented. Asymmetric field was generated under bias in the tunneling barrier due to the pointed electrode. Higher electric field was generated under reverse bias due to lightning rod effect.

In type-C MIM both the electrodes are sharply pointed, in which case even if surface plasmons are excited they do not travel into the tunneling junction, instead the excited surface plasmons travel to the bias lines. So in the type-C MIM the near field enhancement does not occur, or it is very weak. Type-A and type-B MIMs show field enhancement and show benefit of GFE technique, while type-C diode serves purpose of a conventional MIM diode for comparison purpose.

Figure 5. SEM images of MIM diodes; Type-A and type-B diodes use enhancement technique while type-C diode is used as for reference[22]

Referenced work covers DC and RF rectification results of FAMIM Diodes. Rectifying performance at zero bias at 6.4 GHz with -12dBm RF power was measured. For measurement, RF source was connected to a commercial RF log-periodic antenna. Needle probes connecting the dc bias lines of MIM diode, were reported to be acting as receive antenna. The rectifying
performance was quantified by measuring DC current of MIM diode at different DC bias points.

However, RF testing has its limitations, and conceptually the theory and results presented are weak. The use of needle tips as antenna is far from satisfactory which means that it is not guaranteed whether the incident RF power is being coupled to the diode or not.

2.5.2 MIM Diodes: DC and RF Characterization of High Frequency ALD Enhanced Nanostructured Metal-Insulator-Metal Diodes

In the doctorate thesis work by Ajayi [10], MIM diodes have been investigated using different configurations. Insulators such as Al₂O₃, HfO₂ and TiO₂ were utilized using ALD, as single insulator (MIM), dual insulator (MIIM), and also as triple insulator (MIIIM). Platinum and Titanium, with work function of 5.3 eV and 4.1 eV respectively, were used as electrodes using e-beam evaporation. The referenced work also covers, limited RF characterization from 1 GHz till 65 GHz.

MIM diodes with junction areas, 10 µm x 10 µm, 20 µm x 20 µm and 30 µm x 30 µm were fabricated. Different parameters for characterizing MIM diodes DC performance have been presented. In addition to the already defined zero bias resistance, responsivity, and asymmetry, non-linearity has also been calculated. The non-linearity parameter is given by:

\[ Non\ -\ linearit = \frac{di}{dv} \]  

DC characteristics of Pt/HfO₂/Ti have been presented with 2 nm and 3 nm insulator thickness. For diodes with junction size 30 µm x 30 µm, zero-bias resistance of 1.8 KΩ and 1.1 MΩ was calculated for 2 nm and 3 nm insulator thickness respectively. While, for the same junction size, non-linearity of 3.2 at 0.8 V was observed for 2 nm thick insulator, and non-linearity of 4 was observed at 0.5 V for 3 nm thick insulator.
Pt/TiO$_2$/Ti diodes with thickness of 3 nm and 4 nm have also been presented. Zero-bias resistance of 29 Ω and 122 Ω was calculated for insulator thickness of 3 nm and 4 nm respectively, for diodes with junction size 30 µm x 30 µm. While, non-linearity of 1.1 at 0.3 V was observed for 3 nm thick insulator, and non-linearity of 1.4 was observed at 0.5V for 3 nm thick insulator.

The reason for much lower resistance for the case of TiO$_2$ has been attributed to the different electrical properties compared to the other group IV metal oxides. This is explained as follows: soft photons are formed from the Ti ions which result in material’s high permittivity, which eventually leads Ti-O bond to create carrier trap and high leakage paths. High leakage paths results in reduction of resistance. HfO$_2$ shows better performance than TiO$_2$ for non-linearity due to larger bandgap. The reported resistance values are comparable to the ones reported in other MIM diodes reported in literature. However, the measure of non-linearity used in the reported work is different from the one used in other MIM diodes reported in literature. This makes it difficult to compare non-linearity with other MIM diodes reported in literature.

In order to increase the nonlinearity, multiple insulators have been used simultaneously in the referenced work. These include HfO$_2$ and TiO$_2$ to form MIIM. This leads to gain in non-linearity; helping to achieve superior rectification efficiency. However, the gain in asymmetry and non-linearity (reported 5.5 for MIIM) due to multiple insulators is at the expense of higher resistance.

Work of more interest in the referenced thesis is on, RF characterization of MIM diode, since prior work on MIM diodes with RF characterization has been limited. An equivalent model for MIM diode as shown below was used for RF characterization in Advanced Design System (ADS) software. The circuit equivalent consists of voltage dependent and independent components; Rj(Vj) represents voltage dependent nonlinear junction resistance of the diode,
Rs represents the series resistance, and Cj represents the diode shunt junction capacitance. Additionally, CPW structures with MIM diodes of junction area 200 nm x 200 nm integrated were measured for s-parameters from 1 GHz to 65 GHz using network analyzer, as shown in figure 6. The measured s-parameters data was used in ADS software to fit on the equivalent circuit model. An ADS gradient based optimization technique was used to extract both voltage independent and dependent parameters. The obtained resistance was reported to be similar to the resistance obtained from DC characterization, which ideally is not the case as mentioned in [9] with increase in frequency( increase in photon energy) resistance decreases.

MIM diodes for which RF characterization has been reported by the author are Pt/Al2O3(3 nm)/Ti, Pt/HfO2(3 nm)/Ti, Pt/Al2O3(1.5 nm)-TiO2 (1.5 nm)/Ti and Pt/Al2O3(1.5nm)-HfO2(1.5 nm)-TiO2 (1.5 nm)/ Ti, shown in table 2. Resistance in the range of Giga Ohms shows inability of these diodes to be integrated with antenna and other RF applications.
<table>
<thead>
<tr>
<th>Extracted parameters at 0 V</th>
<th>Pt/Al₂O₃/Ti</th>
<th>Pt/HfO₂/Ti</th>
<th>Pt/Al₂O₃-TiO₂/Ti</th>
<th>Pt/Al₂O₃-HfO₂-TiO₂/Ti</th>
</tr>
</thead>
<tbody>
<tr>
<td>Extracted Rj (GΩ)</td>
<td>1.9</td>
<td>1.15</td>
<td>80.2</td>
<td>50.5</td>
</tr>
<tr>
<td>Extracted Cj (fF)</td>
<td>8</td>
<td>7</td>
<td>6</td>
<td>7</td>
</tr>
<tr>
<td>Lin (pH)</td>
<td>242.1</td>
<td>282</td>
<td>263</td>
<td>291</td>
</tr>
<tr>
<td>Lout (pH)</td>
<td>3.1</td>
<td>3.2</td>
<td>3</td>
<td>2.8</td>
</tr>
<tr>
<td>Cin (fF)</td>
<td>25.1</td>
<td>23</td>
<td>22</td>
<td>23</td>
</tr>
<tr>
<td>Cout (fF)</td>
<td>22.1</td>
<td>17.87</td>
<td>16</td>
<td>17</td>
</tr>
<tr>
<td>Rs (Ω)</td>
<td>50.96</td>
<td>73</td>
<td>95</td>
<td>84</td>
</tr>
</tbody>
</table>

However, the reported RF characterization is limited because the equivalent circuit model can lead to many different combinations of resistance and capacitance, and hence it is not a very good way to obtain resistance. Additionally, RF characterization needs to take in to consideration input impedance, which can be obtained from two-port s-parameters. This is missing in the reported RF characterization.

### 2.5.3 MIM Diode for Microwave Circuits

Work reported in [23] shows DC and RF performance for MIM diodes which were fabricated on flexible substrate- Polyetheretheretherketone (PEEK). Diodes with two areas: 9 µm² and 48 µm² were fabricated. DC characterization using I- V response has been shown. In addition to this RF measurements have also been shown. RF measurements included single port s-parameters measurement in the range of 1-20 GHz. S-parameters measurement was used in MIM diode equivalent model in ADS software to obtain diode resistance. Additionally, voltage detection in the range of 1-6 GHz and 10-18 GHz was shown using MIM diode with a bias voltage of 200 mV. The reported work also covered use of MIM diode as frequency multiplier and mixer.

Though the referenced work comes under the category of RF characterization, but there are limitations of the reported work. Firstly, the DC characterization of MIM diode shows I-V response only. Neither calculation of DC resistance nor measure of rectification ability has
been shown. The s-parameter measurement has been done using only a single port and the resistance was obtained using ADS software. This means that the obtained resistance does not actually represent measured RF impedance of MIM diode. Additionally zero-bias rectification using a MIM diode has not been shown since the measurement set-up in the work refers to voltage detection only and that also using a voltage bias of 200 mV. Even with the bias of 200 mV the detected voltage is in the range of 10 mV only. The referenced work shows some missing links for RF characterization of MIM diode which need to be addressed.

2.6 MIM Diodes based on Amorphous Alloys

MIM diode work has mostly been based on using crystalline metals. However, in the work by Nasir [17, 26-29], the large surface roughness problem by using crystalline metals for bottom electrode has been mentioned. The referenced work reports roughness of the crystalline metals to be larger than the thickness of the insulator used in MIM diode. This probable issue could cause non-uniform electric fields across a MIM device, making control of quantum mechanical tunneling problematic.

The authors of the referenced work propose the use of amorphous alloys instead of crystalline metals for overcoming the issue of bottom electrode roughness and field uniformity roadblocks. ZrCuAlNi (with atomic composition: 40% Zr, 35 % Cu, 15 % Al and 10 % Ni) has been used as an amorphous alloy. In the referenced work, ZrCuAlNi alloy was reported to show RMS roughness around 0.2 nm, while crystalline metal aluminum showed RMS roughness around 5 nm. AFM images showing the roughness measurements are shown in figure 7.
Figure 7. AFM of a)100nm ZrCuAlNi b) 150nm Al from Nasir’s paper [26]

MIM Diodes of approximately 1 mm$^2$ area were fabricated using 200nm of ZrCuAlNi as bottom electrode on SiO$_2$, followed by thin Al$_2$O$_3$ layer (2-10 nm) as insulator. Top electrode was made from either ZrCuAlNi alloy or Aluminum. In the case of ZrCuAlNi being used as top electrode, symmetric current density-applied electric density curve was observed due to equivalent barriers at metal/insulator interfaces. In comparison, the MIM diodes with Aluminum as top electrode show asymmetric barrier leading to asymmetric current density-applied electric field curve. The respective asymmetric and symmetric behavior of Al and ZrCuAlNi as top electrodes are shown in figure 8 below.
In the reported work, author mentions that amorphous alloys offer a solution to the issue of crystalline metal surface roughness. In addition to the fabricated MIM Diodes, the referenced work also covers the simulated current density-field curves. The fabricated results showed good match with the simulation. However it is also important to mention here, that though amorphous metals have been shown to be good alternative to crystalline metals in terms of lower surface roughness, lower conductivity of amorphous alloy compromises the electrical performance of MIM Diode. Conductivity of amorphous alloy has been reported to be two orders of magnitude lower than the crystalline metals[27]. More investigation into crystalline metal surface roughness is required before completely overruling the use of crystalline metals as bottom electrodes for MIM diodes.

2.7 Summary

All the referenced works, have some drawbacks which need to be highlighted, and addressed:

- Bottom electrode surface roughness for crystalline metals needs to be investigated, since there has been contradictory work. Plenty of MIM diodes have been reported using crystalline metals, however one recent work mentions the inability of using crystalline
metals as bottom electrodes due to surface roughness. The alternate option proposed is using amorphous alloys but that will present the problem of poor conductivity.

- RF Characterization of MIM Diodes has not been reported in detail. The partial work which has been presented has limitations, specifically the use of probes as antennas in [22] to couple RF energy to diodes is wrong. In the other RF characterization works by [10] and [23], resistance for MIM diode in RF range has been obtained using a simplified ADS model which in essence does not represent actual RF characterization. Additionally s-parameters measurement for [23] was done using a single port only. RF characterization using s-parameters to get input impedance and actual rectification of RF to DC at zero-bias using MIM diode has also not been showcased till date.

Based on the observations made above, this thesis work investigates the issue of surface roughness for crystalline metals, particularly in comparison with the amorphous alloys, and attempts to do a comprehensive RF characterization which could prove to be beneficial in the evolution of MIM diodes, and their use in RF applications such as rectenna.
3. SURFACE ROUGHNESS MEASUREMENTS AND FABRICATION OF MIM DIODES

MIM diodes have been under investigation for many decades but the commercialization of MIM diodes based electronics has been hindered due to the lack of sophisticated nano-fabrication processes. MIM diodes offer various challenges in fabrication, however the issue of bottom electrode surface roughness has been reported as one of the most crucial problem. Bottom electrode surface roughness is important, since the insulator sandwiched between the electrodes has a thickness of only few nanometers. Bottom electrodes with surface roughness larger than the insulator thickness could lead to unreliable and poor performance, in addition to low yield of MIM diodes. The resolution of surface roughness issue requires bottom electrodes which are smooth, and ideally surface roughness should be less than the thickness of the insulator.

As discussed in the previous chapter, most reported work on MIM diodes has been done using crystalline metals as bottom electrodes. However, there is an exception of one recent work which proposes the use of amorphous alloys as bottom electrodes. Amorphous alloy was used due to the reported lower surface roughness compared to crystalline metals [27].

To thoroughly investigate the issue of surface roughness, a complete study of crystalline metals, and amorphous alloy surface roughness, with various thicknesses, and deposited using various deposition methods are presented in this chapter. This is followed by the fabrication details of MIM diodes realized in this work.

3.1 Surface Roughness Analysis

Surface roughness analysis of crystalline metals and amorphous alloy is the first requirement for fabricating reliable MIM diodes. Surface roughness analysis is done for different crystalline metals and an amorphous alloy using Atomic Force Microscopy (AFM). For the purpose of
this study, only bottom metal electrode has been fabricated so that microscopy can be easily performed.

AFM, a type of Scanning Probe Microscope (SPM) probes the sample and makes measurement in three dimensions, x, y, and z. AFM measurement enables the presentation of three-dimensional image of a sample surface, with a resolution of 0.1 to 1.0 nm in the x-y plane, and 0.01 nm in z-direction. Contact mode AFM, which is one of the widely used scanning probe mode has been used for the surface roughness analysis presented in this work. Contact mode uses a sharp tip attached to a low spring cantilever which is scanned across the sample. Force of roughly $10^{-9}$ N is maintained on the cantilever which pushes the probe tip against the sample. The probe tip deflection relative to spatial variation is recorded and converted to image of the surface using Picoimage software[39].

Metals with various thicknesses, and deposition methods; sputtering, e-beam evaporation and ALD were studied for surface roughness analysis. The choice of metals used for the surface roughness study is discussed in the next section, followed by the 3-D images and results obtained for the surface roughness study using AFM.

### 3.2 Materials Selection

Surface roughness analysis and fabrication of MIM diodes started with taking into consideration the metals physical properties, such as resistivity, metal work function and options of metal deposition choices in KAUST nano-fabrication facilities. Metals with large work function difference were targeted for use in this thesis. Metal properties have been used from Smithell’s metal reference book [40] and are shown in table 3.
Table 3. Metal Properties: Work Function, Resistivity, Melting Point and Density[40]

<table>
<thead>
<tr>
<th>Metal</th>
<th>Work Function (eV)</th>
<th>Resistivity at room temperature (10^−8 Ωm)</th>
<th>Melting Point (°C)</th>
<th>Density (g cm³)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Aluminum (Al)</td>
<td>4.2</td>
<td>2.61</td>
<td>660</td>
<td>2.70</td>
</tr>
<tr>
<td>Chromium (Cr)</td>
<td>4.4</td>
<td>12.9</td>
<td>1907</td>
<td>7.15</td>
</tr>
<tr>
<td>Copper (Cu)</td>
<td>4.5</td>
<td>1.58</td>
<td>1084</td>
<td>8.96</td>
</tr>
<tr>
<td>Gold (Au)</td>
<td>4.8</td>
<td>2.01</td>
<td>1064</td>
<td>19.3</td>
</tr>
<tr>
<td>Iron (Fe)</td>
<td>4.4</td>
<td>9.8</td>
<td>1538</td>
<td>7.87</td>
</tr>
<tr>
<td>Nickel (Ni)</td>
<td>4.9</td>
<td>6.2</td>
<td>1455</td>
<td>8.90</td>
</tr>
<tr>
<td>Platinum (Pt)</td>
<td>5.3</td>
<td>10.4</td>
<td>1768</td>
<td>21.5</td>
</tr>
<tr>
<td>Silver (Ag)</td>
<td>4.7</td>
<td>1.47</td>
<td>961</td>
<td>10.5</td>
</tr>
<tr>
<td>Titanium (Ti)</td>
<td>4.1</td>
<td>39</td>
<td>1668</td>
<td>4.51</td>
</tr>
<tr>
<td>Tungsten (W)</td>
<td>4.5</td>
<td>5.3</td>
<td>3422</td>
<td>19.3</td>
</tr>
</tbody>
</table>

In addition to the electrical properties of metals, mechanical properties such as toughness, brittleness, ductility, malleability, and corrosion determine the choice of metals to be used for surface roughness and MIM diode fabrication. Platinum, copper and gold have a very high melting point, which is useful as the thin insulator has to be realized through Atomic Layer Deposition (ALD) which is done at high temperature (> 160 °C). For the case of low melting point metal, such as aluminum, there is high chance of crystallization and increase in surface roughness after insulator deposition. Based on the properties given; copper, gold and platinum crystalline metals were used as bottom electrodes for surface roughness analysis. Additionally ZrCuAlNi amorphous alloy was also used as a bottom electrode for surface roughness analysis. This would help in comparison between crystalline metals and amorphous alloy.
3.3 Surface Roughness study based on Metal Deposition methods

This section covers the results and discussion on the surface roughness study of crystalline metals and amorphous alloy, based on sputtering, e-beam evaporation and ALD deposition techniques. Copper, platinum, and gold were investigated for surface roughness issue. In addition to the mentioned crystalline metals, ZrCuAlNi amorphous alloy was also studied for surface roughness. For the surface roughness study only the bottom electrode surface roughness was investigated using AFM and complete MIM diodes were not fabricated, as shown in the stack up in figure 9. All depositions were done on 0.525 mm thick silicon wafer which had been thermally oxidized to a thickness of 300 nm. Additionally for crystalline metals; platinum, copper and gold, 10 nm of titanium adhesion layer was used before depositing the metal for which roughness was being studied. Special care was taken to make sure that the samples were clean. This involved depositions conducted in clean room facility, and use of wafer carriers to transfer the samples to AFM lab. AFM scan for all the samples was done on an area of 1 x 1 µm².

3.3.1 Sputtering- Surface Roughness Study

Physical Vapor Deposition (PVD) covers an extensive range of vapor-phase technologies. Generally it is used to describe variety of methods, in which thin solid films are deposited by
condensation of a vaporized form of solid material. In PVD, physical ejection of material as atoms or molecules and condensation and nucleation of the atoms on to a substrate is involved.

The first type of PVD used in this work is sputtering. Sputtering is the ejection of atoms by bombardment of the target by energetic particles, mostly argon ions, done under high vacuum. The collision between incident energetic particles and surface atoms results in the ejection of target atoms which are deposited on the substrate[41].

Copper, platinum, gold and amorphous alloy (ZrCuAlNi) were sputtered using ESC Sputtering machine without any intended substrate heating and studied for surface roughness using AFM.

3.3.1.1 Copper

Copper sputtering roughness study covered two thicknesses; 50 nm and 500 nm. This was required to see the variation of surface roughness based on the thickness of copper film. For both thicknesses, copper was sputtered with DC power of 400 W, argon gas flow rate of 25 SCCM and process pressure of 10 mTorr. Titanium adhesion layer with 10 nm thickness was deposited before copper depositions using same sputtering conditions as for copper deposition. AFM result in the form of 3-D rendering is shown for the case of 50 nm sputtered copper in figure 10.
The AFM result shows a Root – Mean - Square (RMS) roughness of 0.58 nm and a peak roughness of 2.92 nm, for the case of 50 nm copper thickness. The surface shows a smooth deposition with peak roughness close to RMS roughness. This result shows that copper can be used as bottom electrode for MIM diodes fabrication, for the case with insulator thickness above 1 nm.

For the case of 500 nm thick copper deposition, same parameters in sputtering tool were used except for increasing the deposition time. The AFM result in figure 11 shows a RMS roughness of 2.30 nm and a peak roughness of 10 nm. This is expected, as thicker layer would lead to increase in grain size which leads to increase in surface roughness[42]. It is worth mentioning here that the peak roughness of 10 nm probably refers to a foreign particle as evident from the AFM image, and therefore the conclusion for this case should be based on RMS surface roughness.
3.3.1.2 Platinum

Platinum sputtering roughness study was done with 50 nm deposition; sputtered using DC power of 400 W, argon gas flow rate of 25 SCCM and process pressure of 10 mTorr. Titanium adhesion layer with 10 nm thickness was deposited before platinum deposition using same sputtering conditions as for platinum deposition. The AFM result in figure 12 shows a RMS roughness of 0.535 nm and a peak roughness of 3.61 nm. The 3-D image shows that the contrast of the film is limited, meaning the film has a uniform deposition with little variation in roughness. The result shows peak roughness comparable to the RMS roughness, and similar to the roughness of 50 nm thick sputtered copper. In [27] for the case of sputtered platinum, peak roughness of 22 nm was reported, however the AFM scan area (10 x 10 μm²) was larger than that used in this work. However, the results presented in this thesis showcases that platinum with low peak roughness can be deposited using sputtering.
3.3.1.3 Gold

In this study 500 nm of gold was deposited using sputtering with same DC power, gas flow rate and pressure conditions, as for the previous two cases explained. And as for the platinum and copper sputtering, 10 nm thick titanium adhesion layer was deposited using the same sputtering conditions. The AFM result in figure 12 shows a RMS roughness of 2.65 nm and a peak roughness of 12.8 nm. This result though shows the same trend as observed for the case of 500 nm thick copper- thicker depositions lead to higher surface roughness, because of increase in grain size. However even with 500 nm thick crystalline metals, relatively smooth layers can be obtained, which is in contrast to the results reported in [27]. In [27] crystalline metal even with high melting point such as iridium have been reported with RMS roughness of 11 nm, and peak roughness of 120 nm.
Figure 13. AFM Micrograph of as-deposited gold film- 500 nm sputtering

3.3.1.4 ZrCuAlNi- Amorphous Alloy

The last analysis of surface roughness using sputtering was done for amorphous alloy ZrCuAlNi. This alloy was chosen because it has been used for surface roughness analysis and MIM diodes fabrication previously[17, 26-29]. The alloy with its reported work function of 4.8 eV offers a good choice as a bottom electrode for MIM diode, where, as explained previously electrodes with significant work function difference are required. The purpose of this work is to compare the roughness of amorphous alloy with roughness of crystalline metals (presented in previous sub-section) and conclude whether it is worth the loss in conductivity to utilize amorphous alloys for MIM Diodes. Amorphous alloys are known to be more resistive than crystalline metals by approximately two orders of magnitude[17]. Hence, ZrCuAlNi alloy could serve as a good measure for surface roughness comparison with crystalline metals.

ZrCuAlNi amorphous alloy with atomic composition: 40% Zr, 35% Cu, 15% Al and 10% Ni, was sputtered to a thickness of 50 nm. ZrCuAlNi alloy was sputtered with DC power of 75 W, argon gas flow rate of 30 SCCM and process pressure of 8 mTorr, with no intended
substrate heating. This involved optimizing the sputtering recipe to get a smooth deposition. AFM showed RMS roughness of 0.526 nm and peak roughness of 2.69 nm, with 3-D surface image shown in figure 14. The referenced work [17] for ZrCuAlNi showed RMS roughness of 0.3 nm and peak roughness of 3 nm for an area of 10 x 10 µm². The obtained surface roughness results though show very smooth deposition, however it is worth mentioning that the surface roughness result is similar to that obtained for 50 nm thick sputtered copper and platinum.

Figure 14. AFM Micrograph of as-deposited ZrCuAlNi film- 50 nm sputtering

3.3.2 E-beam Evaporation – Surface Roughness Study

The second type of PVD utilized in this thesis for metal deposition is e-beam evaporation. In this process, electron beam is directed towards the target (anode) which evaporates and deposits on the substrate, under high vacuum condition [41].

In this study 50 nm and 500 nm of copper was deposited using e-beam deposition with vacuum pressure 0.05 mTorr. Prior to copper deposition, 10 nm thick titanium adhesion layer was deposited using e-beam evaporation, with same settings as for copper deposition. For 50 nm copper thickness, AFM result shows a RMS roughness of 0.843 nm and a peak roughness of
3.31 nm. The deposition shows relatively smooth deposition, as visible from the 3-D image in figure 15.

![AFM Micrograph of as-deposited Copper film- 50 nm e-beam evaporation](image)

For 500 nm thick copper, AFM result in figure 16 shows a RMS roughness of 1.80 nm and peak roughness of 8.29 nm. E-beam evaporation in comparison to sputtering, has shown higher surface roughness in this study. The probable reason of this trend is crystallization, as temperature is higher in e-beam evaporation than in the case of sputtering.
3.3.3 ALD – Surface Roughness Study

Atomic layer deposition (ALD) is a chemical gas phase thin film deposition technique based on self-saturating surface reaction. Two or more precursor chemicals are introduced to the substrate surface separately. At the introduction of each precursor surface, a monolayer of material is formed. This prevents uncontrolled gas-phase reactions and helps in getting conformal and uniform material layers. Typically, each cycle leads to a monolayer of 1 Å. Film growth is insensitive to process parameters other than choice of the precursor chemicals and temperature. ALD is fast becoming a popular technique to deposit variety of insulators, especially for the case when thin insulator (few nm) is a requirement.

Titanium adhesion layer – 10 nm thick, was deposited using electron beam evaporation at a pressure of 0.05 mTorr on the silicon substrate which had been thermally oxidized. This was followed by 50nm deposition of Platinum using ALD at a temperature of 300 °C with oxygen used as reactant gas and Methylcyclopentadienyl Tri-methyl Platinum (MeCpPtMe$_3$) as Platinum precursor. This allowed smooth deposition with growth rate of 0.5 Å per cycle.
AFM result in figure 17 showed RMS roughness of 3.85 nm and peak roughness of 18 nm. This number shows roughness much higher than the platinum deposited through sputtering. Literature of surface analysis of ALD platinum film shows roughness around 4 nm\cite{43}, however it is believed that with further optimization even smoother films for metals using ALD can be obtained.

Figure 17. AFM Micrograph of as-deposited platinum film- 50 nm ALD

3.4 Summary of Surface Roughness Analysis

Based on the AFM surface roughness study, it can be concluded that with lower thickness (50 nm) one could achieve sub 1 nm roughness for crystalline metals using sputtering and e-beam evaporation. Also sputtering compared to e-beam evaporation and ALD was found to be better in terms of smoother metal deposition with controlled peak roughness. Surface roughness results have been summarized in the table 4; notably sputtered platinum and copper with 50 nm thickness showed least surface roughness which is comparable to the ZrCuAlNi amorphous alloy surface roughness. The result in this thesis shows that crystalline metals can achieve low
surface roughness (0.5 nm) which is comparable to the amorphous alloy surface roughness. Based on the surface roughness analysis Pt, Cu, and ZrCuAlNi were tried as bottom electrodes for MIM diode fabrication. The fabrication of MIM diode will be covered in the next subchapter.

In comparison, the work by Nasir [27] showed surface roughness on an area of 10 x 10 µm² of crystalline metals to be around 2 nm for platinum with peak roughness around 22 nm, while aluminum was reported to show roughness of 4 nm and peak roughness of 43 nm [27]. Also in the reported work by Nasir, it is worth noticing that surface roughness data was collected on different film thicknesses; 100 nm thick amorphous alloy and 150 nm thick aluminum metal, while for other metals exact thickness of the films was not reported. This meant it was impossible to conclude whether the surface roughness reported was due to crystalline metals or because of thicker crystalline films compared to amorphous alloy film thickness. This issue has been resolved in this thesis and a truer comparison has been presented.

Table 4. RMS Roughness measured using AFM for Crystalline Metals and Amorphous Alloy

<table>
<thead>
<tr>
<th>Crystalline Metal/ Amorphous Alloy</th>
<th>Deposition Method</th>
<th>Thickness (nm)</th>
<th>AFM Scan Area (µm²)</th>
<th>RMS Roughness</th>
<th>Peak Roughness</th>
</tr>
</thead>
<tbody>
<tr>
<td>Copper Sputtering</td>
<td>50</td>
<td>1</td>
<td>0.58</td>
<td>2.92</td>
<td></td>
</tr>
<tr>
<td>Copper Sputtering</td>
<td>500</td>
<td>1</td>
<td>2.30</td>
<td>10</td>
<td></td>
</tr>
<tr>
<td>Copper Evaporation</td>
<td>50</td>
<td>1</td>
<td>0.843</td>
<td>3.31</td>
<td></td>
</tr>
<tr>
<td>Copper Evaporation</td>
<td>500</td>
<td>1</td>
<td>1.80</td>
<td>8.29</td>
<td></td>
</tr>
<tr>
<td>Platinum ALD</td>
<td>50</td>
<td>1</td>
<td>3.85</td>
<td>18</td>
<td></td>
</tr>
<tr>
<td>Platinum Sputtering</td>
<td>50</td>
<td>1</td>
<td>0.535</td>
<td>3.61</td>
<td></td>
</tr>
<tr>
<td>Gold Sputtering</td>
<td>500</td>
<td>1</td>
<td>2.65</td>
<td>12.8</td>
<td></td>
</tr>
<tr>
<td>ZrCuAlNi Sputtering</td>
<td>50</td>
<td>1</td>
<td>0.526</td>
<td>2.69</td>
<td></td>
</tr>
</tbody>
</table>
3.5 **MIM Diode Fabrication**

Surface roughness study using AFM presented in the previous section formed the basis for the MIM diode fabrication. Bottom electrodes with 50 nm thickness showed low surface roughness, which can ensure that the thin oxide deposited on top of the bottom metal is not non-uniform due to surface roughness issue and that the two metal electrodes are separated by the thin oxide. This is important for stable MIM devices. Specifically, four MIM diodes fabricated based on different bottom electrode deposition methods are presented in this section.

A. 50 nm thick Pt deposited using ALD
B. 50 nm thick Pt deposited using sputtering
C. 50 nm thick ZrCuAlNi deposited using sputtering
D. 150 nm thick Cu deposited using e-beam evaporation

MIM diode fabrication A and B will compare the fabrications based on two deposition methods; ALD and sputtering. Sputtering though has shown lower surface roughness but the two fabrications will serve the purpose for comparison while keeping material systems the same. Amorphous alloy ZrCuAlNi is used as bottom electrode in MIM diode fabrication C, which also showed low surface roughness, and comparison with crystalline metal MIM diodes will be beneficial. For the MIM diode fabrication D, 150 nm thick copper was used as bottom electrode, to see the variation based on thicker and rougher crystalline metal compared to 50nm thickness.

Fundamental processes used in MIM diode fabrication include lithography, deposition, lift-off and etching, just like in any other micro or nano-fabrication process. Other supplementary processes that are important include mask fabrication, metrology analysis, and wafer cleaning. The chapter will start with mask design followed by the fabrication process details of MIM diodes.
3.5.1 Mask Design

MIM diodes were fabricated using UV lithography. The mask for UV lithography carried the design for the MIM diodes with an area 5” x 5”, and thickness of 0.09”. Mask design covered the considerations mentioned below and are also shown in the figures below.

a) MIM diodes with various areas were included in the mask design; 0.09 mm$^2$, 0.04 mm$^2$ and 0.01 mm$^2$ to study the effect of junction area.

b) CPW, a transmission structure used to carry RF signals, with ground and signal currents on the same layer designed for silicon substrate, matched for two widths: 0.1 mm and 0.2 mm was included in the mask.

c) MIM diodes design integrated with two-port RF feeding structure - CPW was also included. CPW with width of 0.1 mm and 0.2 mm were included in the structure, and junction area of MIM diode was 0.01 mm$^2$ and 0.4 mm$^2$ respectively. This would serve the purpose of complete RF characterization including s-parameter measurement and determining input impedance of MIM diodes.

d) MIM diodes integrated with RF feeding structure - CPW on one port and resistor/capacitor load on the rectified side. This was included to actually see rectification of RF power using MIM diode. CPW with width 0.1 mm and 0.2 mm were included in the structure, which relate to junction areas of 0.01 mm$^2$ and 0.4 mm$^2$ for the MIM diode respectively.

With the design considerations mentioned, the design of the mask was done using L-Edit software. This is a standard Computer Aided Design (CAD) software which is used in KAUST nanofabrication facility. L-Edit has an option of segmental design where small parts of mask can be modularized into cells. Another advantage of L-Edit is that all layers are designed in a single file, later each mask layer can be exported in to a variety of file formats. In this case four layer structure was designed in L-Edit; 1) Bottom electrode mask, 2) Oxide layer mask, 3) Top electrode mask, and 4) Pad layer for RF feed. L-Edit design file image is shown below.
Figure 18. L-Edit Overall Mask Design- 1) Bottom Electrode (Grey) 2) Oxide Layer (Fawn) 3) Top electrode (Blue) and 4) RF Pad layer (Red)

Figure 19. MIM diode mask a) MIM diodes for DC testing b) CPW with line width = 0.1 mm c) MIM diode integrated with CPW, and d) MIM with CPW and pads for load. Four layer Mask Design: 1) Bottom Electrode (Grey) 2) Oxide Layer (Fawn) 3) Top electrode (Blue) and 4) RF Pad layer (Red)
3.5.2 Fabrication Process

Mask design was followed by the MIM fabrication process steps. It is worth mentioning here that other than the bottom electrode deposition all steps were common for the four MIM diode fabrications. ZnO was used as insulator, titanium was used as top electrode and copper was used as RF pad layer for all fabrications. MIM diode fabrications which will be covered in the section have been discussed in the introduction of the sub-chapter 3.5 and also shown in table 5.

Table 5. MIM Diode Fabrication

<table>
<thead>
<tr>
<th>MIM Diode Fabrication</th>
<th>Bottom Electrode</th>
<th>Deposition</th>
<th>Bottom Electrode Thickness</th>
<th>Insulator</th>
<th>Insulator Thickness</th>
<th>Top Electrode</th>
<th>RF Pad Layer</th>
</tr>
</thead>
<tbody>
<tr>
<td>A</td>
<td>Platinum</td>
<td>ALD</td>
<td>50nm</td>
<td>ZnO</td>
<td>4 nm</td>
<td>Titanium</td>
<td>Copper</td>
</tr>
<tr>
<td>B</td>
<td>Platinum</td>
<td>Sputter</td>
<td>50nm</td>
<td>ZnO</td>
<td>4 nm</td>
<td>Titanium</td>
<td>Copper</td>
</tr>
<tr>
<td>C</td>
<td>ZrCuAlNi</td>
<td>Sputter</td>
<td>50nm</td>
<td>ZnO</td>
<td>4 nm</td>
<td>Titanium</td>
<td>Copper</td>
</tr>
<tr>
<td>D</td>
<td>Copper</td>
<td>E-beam evaporation</td>
<td>150nm</td>
<td>ZnO</td>
<td>4 nm</td>
<td>Titanium</td>
<td>Copper</td>
</tr>
</tbody>
</table>

ZnO insulator was selected based on dielectric constant, electron affinity, band gap and options available at KAUST nano-fab. Another important consideration is the method to deposit the thin oxide layer; out of all the choices, ALD was found to be the best choice due to the benefits of self-controlled reaction to achieve thickness of few nanometers. Material properties for selecting the insulator layer are also shown in the table below [44-47]. The condition for MIM diode has been mentioned before: work function for metals must be larger than the electron affinity of the insulator. ZnO with 2.077 eV electron affinity offers a good option. Electron affinity of ZnO is lower than the work functions of copper, platinum, titanium and ZrCuAlNi: 4.5 eV, 5.3 eV, 4.1 eV and 4.8 eV respectively. Titanium was used as top electrode for MIM diode fabrications because it offered work function difference with respect to the bottom electrode – in addition to the advantage of high melting point, which was also mentioned in the
material selection sub-chapter. Titanium is also easy to deposit using e-beam evaporation compared to other metal options.

Table 6. Insulator Properties: Dielectric constant, Band Gap and Electron affinity [44-47]

<table>
<thead>
<tr>
<th>Material</th>
<th>Dielectric constant</th>
<th>Band Gap (eV)</th>
<th>Electron affinity (eV)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Al$_2$O$_3$</td>
<td>3.4</td>
<td>8.8</td>
<td>1</td>
</tr>
<tr>
<td>HfO$_2$</td>
<td>4</td>
<td>6</td>
<td>2.5</td>
</tr>
<tr>
<td>SiO$_2$</td>
<td>2.25</td>
<td>9</td>
<td>0.9</td>
</tr>
<tr>
<td>Ta$_2$O$_5$</td>
<td>4.84</td>
<td>4.4</td>
<td>3.2</td>
</tr>
<tr>
<td>TiO$_2$</td>
<td>7.8</td>
<td>3.05</td>
<td>3.9</td>
</tr>
<tr>
<td>ZnO</td>
<td>3.68</td>
<td>3.4</td>
<td>2.077</td>
</tr>
<tr>
<td>ZrO$_2$</td>
<td>4.8</td>
<td>5.8</td>
<td>2.5</td>
</tr>
</tbody>
</table>

It needs to be clarified that the process for bottom electrode was slightly different for each fabrication and will be covered in a separate section after the common fabrication details are explained.

3.5.2.1 Thermal Oxidation

High resistivity silicon wafers (550 Ωcm) were cleaned, followed by thermal oxidation at high temperature (>950 °C), to get a 300 nm thick layer of silicon dioxide. Thermal oxidation was done following dry-wet-dry cycle.

It is important to mention the reason of using dry-wet-dry cycle. Dry Thermal oxidation (Si+O$_2$→SiO$_2$) is known to give better quality however at the same time it is a slow process. On the other side wet oxidation (Si+ 2H$_2$O→SiO$_2$+H$_2$) is a faster process but the quality of silicon dioxide formed is poor. So to start oxidation dry cycle is formed to achieve high quality of silicon dioxide for the contact with silicon, followed by wet oxidation to speed up silicon dioxide formation and in the end dry oxidation is used again to give a good top layer quality for good contact with depositions in other fabrication steps.
3.5.2.2 Lithography and Depositions

Lithography is used to transfer pattern on the surface of wafer, which had been thermally oxidized. In this fabrication optical lithography was used for fabrication followed by deposition. Optical lithography is used to create micro-sized patterns in a photoresist layer already spun on the wafer. Procedures involved in photolithography and deposition are mentioned below.

Resist coating using spinner: A small amount of photoresist, i.e organic polymer in liquid form was dispensed on the wafer using a nozzle that is then spun at to get a uniform layer of photoresist using the recipes shown in table 7. AZ 1512 with 1.4 μm thickness was used as a photoresist for processing bottom electrode; for the case of sputtering and e-beam evaporation, oxide layer and top electrode, while ECI 3027 with 4 μm was used for processing pad layer. Both AZ1512 and ECI3027 are positive resists meaning every opening in the mask that allows UV radiation to reach photoresist (exposed area) is dissolved in the developer. For the case of using ALD for platinum deposition of bottom electrode, AZ5214 was used as photoresist – converted to negative tone. For image reversal AZ 5214 was spun using the recipe given in table 7 below, this was followed by soft bake at 105° C and exposure in mask aligner with energy 70 mJ/cm². After the exposure image reversal was done by bake at 120° C and UV flood with a blank mask and energy 200 mJ/cm². The recipe has been summarized in table 7.
Table 7. Photoresist spin coating recipe and exposure energy

<table>
<thead>
<tr>
<th></th>
<th>Step</th>
<th>Speed/rpm</th>
<th>Ramp/ rpm/s</th>
<th>Time/s</th>
<th>Exposure/ mJ/cm²</th>
<th>Soft Bake</th>
</tr>
</thead>
<tbody>
<tr>
<td>AZ 1512 1.4 µm</td>
<td>1</td>
<td>800</td>
<td>1000</td>
<td>3</td>
<td>40</td>
<td>60 s at 100° C</td>
</tr>
<tr>
<td></td>
<td>2</td>
<td>1500</td>
<td>1500</td>
<td>3</td>
<td></td>
<td></td>
</tr>
<tr>
<td></td>
<td>3</td>
<td>3000</td>
<td>3000</td>
<td>30</td>
<td></td>
<td></td>
</tr>
<tr>
<td>ECI 3027 4 µm</td>
<td>1</td>
<td>700</td>
<td>1000</td>
<td>3</td>
<td>200</td>
<td>60 s at 100° C</td>
</tr>
<tr>
<td></td>
<td>2</td>
<td>1200</td>
<td>1500</td>
<td>3</td>
<td></td>
<td></td>
</tr>
<tr>
<td></td>
<td>3</td>
<td>1750</td>
<td>3000</td>
<td>30</td>
<td></td>
<td></td>
</tr>
<tr>
<td>AZ5214 1.6 µm</td>
<td>1</td>
<td>800</td>
<td>1000</td>
<td>3</td>
<td>70</td>
<td>120 s at 105° C</td>
</tr>
<tr>
<td></td>
<td>2</td>
<td>1500</td>
<td>1500</td>
<td>3</td>
<td>200 for Flood exposure</td>
<td>120 s at 120° C (reversal bake)</td>
</tr>
<tr>
<td></td>
<td>3</td>
<td>3000</td>
<td>3000</td>
<td>30</td>
<td></td>
<td></td>
</tr>
</tbody>
</table>

- Soft bake: In this step, wafer underwent soft bake according to the recipe in the table above. This is required to remove any residual solvent and also to aid in polymer cross linking.

- Alignment and exposure in UV lithography aligner: After the soft-bake, wafer undergoes alignment and exposure in the UV lithography aligner. Alignment is required when multiple layers are required; aligning one layer to the other. For this purpose, alignment marks in the mask of each layer are present. The most crucial step which follows the alignment is the exposure of the photoresist coated wafer through the mask which needs to be imprinted. EVG 6200 tool, which consists of a UV light bulb, with a signal either g-line (436 nm) or i-line (365 nm) was used for the exposure according to the recipes given in the table.

- Development: The exposed resist-coated wafer was developed by immersing the wafer in the resist remover known as developer. MIF AZ 726 developer which consists of 2.38 % Tetra Methyl- Ammonium Hydroxide dissolved in water was used. After development wafer actually shows the pattern of the mask it was exposed through. For each combination of photoresist and developer, an optimum development time is required; 20 s for AZ1512 and, 60 s for ECI 3027 and AZ5214.
- O2 Descum: This step was done using RIE etching tool to remove any photoresist remnants from the unwanted areas. O2 descum recipe has been mentioned in the table 9. With this step, the wafer is ready for actual deposition of the material for which the photoresist pattern was developed.

- Deposition: In this step, wafer went actual deposition of the material using ALD, e-beam evaporation and sputtering. For copper, platinum and gold 10 nm of titanium adhesion layer was used. General overview of process conditions used for the different depositions in the fabrication is given in table 8: MIM diode material deposition details below, this includes bottom electrode, oxide layer, top electrode, and pad layer.

### Table 8. MIM diode material deposition details

<table>
<thead>
<tr>
<th>Material Deposition</th>
<th>Method</th>
<th>Power (W)</th>
<th>Pressure (mtorr)</th>
<th>Flow Rate for Ar/Ar/Ar (scm)</th>
<th>Temperature (°C)</th>
<th>Deposition Time or Cycles</th>
<th>Thickness (nm)</th>
<th>Purpose</th>
</tr>
</thead>
<tbody>
<tr>
<td>Platinum</td>
<td>ALD</td>
<td>400</td>
<td>15</td>
<td>20 for Ar 80 for O2</td>
<td>300</td>
<td>1000 cycles</td>
<td>50</td>
<td>Bottom electrode</td>
</tr>
<tr>
<td>Platinum</td>
<td>Sputter</td>
<td>400 (DC)</td>
<td>10</td>
<td>25 for Ar 80 for O2</td>
<td>25</td>
<td>90 s</td>
<td>50</td>
<td>Bottom electrode</td>
</tr>
<tr>
<td>Copper</td>
<td>E-beam evaporation</td>
<td>NA</td>
<td>0.05</td>
<td>NA</td>
<td>25</td>
<td>20 min</td>
<td>150</td>
<td>Bottom electrode</td>
</tr>
<tr>
<td>Titanium</td>
<td>E-beam evaporation</td>
<td>NA</td>
<td>0.05</td>
<td>NA</td>
<td>25</td>
<td>20 min</td>
<td>100</td>
<td>Top electrode</td>
</tr>
<tr>
<td>ZrCuAlNi</td>
<td>Sputter</td>
<td>75 (DC)</td>
<td>8</td>
<td>30 for Ar</td>
<td>25</td>
<td>600 s</td>
<td>50</td>
<td>Bottom electrode</td>
</tr>
<tr>
<td>Copper</td>
<td>Sputter</td>
<td>400 (DC)</td>
<td>10</td>
<td>25 for Ar</td>
<td>25</td>
<td>500 s</td>
<td>500</td>
<td>RF Pad layer</td>
</tr>
<tr>
<td>Titanium</td>
<td>Sputter</td>
<td>400 (DC)</td>
<td>10</td>
<td>25 for Ar</td>
<td>25</td>
<td>60 s</td>
<td>10</td>
<td>Adhesion layer for bottom electrode</td>
</tr>
<tr>
<td>Zinc Oxide</td>
<td>ALD</td>
<td>500 (Forward)</td>
<td>15</td>
<td>20 for Ar 80 for O2</td>
<td>160</td>
<td>30 cycles</td>
<td>4</td>
<td>Insulator</td>
</tr>
</tbody>
</table>

- Lift-off: After deposition of the bottom electrode (except for the case when Pt was deposited using ALD), lift-off was done by immersing the wafer in acetone and using ultrasound sonicator to speed up the process. Visual inspection showed whether the lift-
off had been completed, following which the wafer was cleaned using Isopropyl Alcohol (IPA). This marked the end of the process for a single layer.

- **Etching:** RIE and RIE-ICP etching was used to etch ZnO and platinum according to the recipe given in the table 9.

After the completion of the first layer, i.e. bottom electrode, mentioned steps are repeated for subsequent layers. It is important to mention here that lift-off process was followed for bottom electrode of MIM diode fabrications B, C, and D. For MIM diode fabrication A - since ALD was used for bottom electrode - this meant platinum deposition was done before photoresist was spun on the wafer. After UV lithography, plasma etching was done to remove platinum from unwanted areas according to the etching recipe given in the table below. As mentioned before, this required use of negative photoresist. Image reversal was used to convert positive photoresist (AZ5214) into negative photoresist (allowing same mask for bottom electrode as used for lift-off process).

Table 9. MIM diode: ICP-RIE details for Descum and Etching

<table>
<thead>
<tr>
<th>Etching Type</th>
<th>Material/ Etched</th>
<th>Process</th>
<th>ICP Power (W)</th>
<th>RF Power (W)</th>
<th>Etching Time/ Cycles</th>
</tr>
</thead>
<tbody>
<tr>
<td>ICP-RIE Etching</td>
<td>Platinum</td>
<td>150</td>
<td>300</td>
<td>8 min</td>
<td></td>
</tr>
<tr>
<td>ICP-RIE Etching</td>
<td>Zinc Oxide</td>
<td>100</td>
<td>1000</td>
<td>20 s</td>
<td></td>
</tr>
<tr>
<td>ICP-RIE Etching</td>
<td>Descum</td>
<td>250</td>
<td>40</td>
<td>30 s</td>
<td></td>
</tr>
</tbody>
</table>

The second layer is the insulator ZnO – 4 nm thick. ZnO was deposited via ALD (O₂ plasma based recipe) using 150 W power and Argon pressure of 3mTorr with diethyl zinc (DEZ) as Zn precursor at 160 °C resulting in growth rate of 1.3 Å per cycle. DEZ reacts with O₂ (gas) to form ZnO. DEZ allows deposition of ZnO at low temperatures. As this deposition was done using ALD at a relatively high temperature of 160° C, this required process to be run without photoresist. It is important to mention that ALD was done using O₂ plasma based recipe rather than thermal recipe (H₂O based) to get resistive films. O₂ plasma being more reactive than H₂O,
oxidizes Zn more completely leading to a more stoichiometric film. This also leads to reduction in oxygen vacancies and in interstitial zinc, eventually resulting in more resistive film [48].

After ZnO deposition and photoresist spinning process, UV lithography process was followed again. However, this time alignment between two layers was required; oxide layer mask and the bottom electrode pattern on the wafer were aligned using the alignment marks. After the development of the second layer in 726 MIF developer, areas where ZnO was required had coating of photoresist while in other areas ZnO was exposed. The unwanted ZnO was etched using RIE etching. Material deposition and etching details are covered in the tables 8 and 9 above. For third and fourth layer same steps as first layer (using lift-off) were followed for UV lithography. Third layer, which is the top electrode was deposited using e-beam evaporation of titanium (150 nm thick). While the fourth layer that is the RF pad layer was deposited using sputtering of copper (500 nm thick). Step by step fabrication of each layer for MIM diode is summarized in the figure below. The microscope image below show the case of MIM diode A (Pt bottom electrode using ALD) with area 0.04 mm².
Figure 20. Step-by-step MIM Diode Fabrication 1) Silicon wafer 2) SiO2 oxidized on Silicon wafer 3) Bottom Electrode deposited 4) ZnO deposited 5) Top Electrode Deposited and 6) pad layer deposited for each layer

Figure 21. MIM diode fabrication A (0.04 mm² junction area)

3.6 MIM Diode Metrology

Transmission Electron Microscopy (TEM) results of fabricated MIM diodes are discussed in this section. The cross-section image was obtained using TEM housed in KAUST Imaging Lab. TEM was required to see the cross section of the fabricated MIM diode - notably thin ZnO
layer. TEM was performed on MIM diode fabrication A - Pt/ZnO/Ti stack - up. The first result of TEM figure 22, shows the result in bright field TEM mode. Figure 22 in low magnitude shows the general stack up. Thicknesses of silicon wafer, SiO₂, Ti adhesion layer, platinum bottom electrode and titanium top electrode were observed- and found to be as intended. The stack up was observed in addition to the platinum and carbon protection coating done during the preparation of the sample for TEM. However, it is difficult to see the ZnO layer, which was the case even with the high magnitude image in bright field mode. To counter the problem, high - angle annular dark field (HAADF) STEM (Scanning Tunneling Electron Microscopy) was used, as its image contrast is more sensitive to atomic number of elements, increasing the chance of imaging thin films.

![Cross-section image using bright field TEM for MIM diode A - Pt/ZnO/Ti stack up showing Si/SiO₂/Ti/Pt/ZnO/Ti stack up](image)

HAADF results are shown in figure 23. In this image, it can be observed that on platinum edge, some material with few nanometer thickness is present. With high magnitude and more focused electron beam in the junction area, ZnO layer was observed. Figures 24 and 25: High magnitude cross-section image, shows the junction area with focused beam in bright field STEM and
increased electron count, establishes the fact that ZnO layer is present with thickness around 4 nm. It has to be clarified that due to the possible unintended mis-orientation of the crystals of different layers could lead to variation in the observed thickness. ZnO and other layers were also seen by Energy Dispersive X-Ray (EDX) analysis, which shows observed peaks for Pt, Ti, Zn and O in figure 26.

Figure 23. Cross-section image using dark field STEM for MIM diode A- Pt/ZnO/Ti- showing Si/SiO2/Ti/Pt/ZnO/Ti stack up
Figure 24. Cross-section image using STEM for MIM diode A- Pt/ZnO/Ti- showing ZnO layer

Figure 25. Cross-section image using bright field STEM for MIM diode A- Pt/ZnO/Ti- showing ZnO layer
3.7 Summary

The previous sub-section covered fabrication details of the MIM diodes. Based on the surface roughness analysis and material properties, suitable insulator and metals were selected for fabrication of MIM diodes. Pt, Cu, and ZrCuAlNi were used as bottom electrodes, while ZnO was used as insulator, and Ti was used as top electrode, for all fabrications. Metal depositions were done using ALD, sputtering, and e-beam evaporation, while ZnO deposition was done using ALD. Thin layer of crystalline metals (Pt) and amorphous alloy (ZrCuAlNi) showed same surface roughness (sub 1 nm), leading to conclusion that crystalline metal is better for use as bottom electrode as they offer better conductivity compared to amorphous alloy. ALD technique offers an excellent uniformity and accurate thickness control of insulator layer. TEM on fabricated MIM diode showed the stack – up and ZnO layer- establishing fact that ZnO was isolating the two electrodes. Detailed TEM results were covered in the section; which included TEM using both bright and dark field mode.
Multiple junction area MIM diode structures were fabricated for DC characterization, as well as structures for RF characterization; which includes s-parameters, and RF rectification, were fabricated. With this in mind, the next chapter will cover both the DC and RF characterization results for MIM diodes fabricated.
4. DC AND RF CHARACTERIZATION OF MIM DIODES

In this chapter, DC and RF characterization of fabricated MIM diodes is presented. DC characterization of a MIM diode is the basic performance metric. DC characterization for MIM diode is conducted to principally obtain current (I) vs voltage (V) characteristics. Using the measured I-V characteristics, different parameters; resistance, first derivative of current with respect to voltage, second derivative of current with respect to voltage and sensitivity are obtained.

Firstly, the measurement setup for DC characterization setup will be discussed followed by I-V measurements and other mentioned performance parameters.

Similarly RF characterization of MIM diode will also be presented. For integration with other RF components such as antenna, it is important to measure the input impedance of the MIM diode. Similarly, it is important to assess the rectification ability of the diode for an incoming RF signal. RF characterization setup will be covered, followed by measurements. RF characterization measurements include s-parameters measurement, using which input impedance was obtained with the aid of MATLAB. Second part of RF characterization covers RF to DC rectification measurement using RF signal at 2.45 GHz.

4.1 DC Characterization

DC characterization of fabricated MIM diodes was performed using Keithley semiconductor parametric analyzer on a probe station. Semiconductor parametric analyzer was connected in series with the probe station. Voltage sweep settings were made on semiconductor parametric analyzer. I-V response for MIM diodes was obtained on the screen output of the semiconductor analyzer with both positive and negative polarities. The device characterization setup is shown in figure 27.
The DC measurement was performed on various fabricated MIM diodes, highlighted in the table 10 below. I-V response measurements were performed at room temperature, which were subsequently analyzed in MATLAB to obtain single derivative with respect to current, second derivative with respect to current, and sensitivity.

The important DC performance parameters obtained from I-V response for the MIM diodes fabricated in this work are given in table 11. As explained previously, resistance of MIM diode gives a measure of the impedance for which the MIM diode needs to be matched in the case of integration with RF applications. While responsivity gives a measure of rectification ability of the MIM diode.

Table 10. Summary of fabricated MIM diodes

<table>
<thead>
<tr>
<th>MIM Diode Fabrication</th>
<th>Structure</th>
<th>MIM Diode Junction Areas (mm²)</th>
</tr>
</thead>
<tbody>
<tr>
<td>A</td>
<td>Pt (ALD)/ZnO/Ti</td>
<td>0.09, 0.04, 0.01</td>
</tr>
<tr>
<td>B</td>
<td>Pt (Sputter)/ZnO/Ti</td>
<td>0.09, 0.04, 0.01</td>
</tr>
<tr>
<td>C</td>
<td>ZrCuAlNi(sputter)/ZnO/Ti</td>
<td>0.09, 0.04, 0.01</td>
</tr>
<tr>
<td>D</td>
<td>Cu (e-beam)/ZnO/Ti</td>
<td>0.09, 0.04, 0.01</td>
</tr>
</tbody>
</table>
Table 11. DC Performance parameters of revaluating MIM diodes

<table>
<thead>
<tr>
<th>Resistance</th>
<th>Responsivity</th>
</tr>
</thead>
<tbody>
<tr>
<td>( r_D = 1/(\frac{dI}{dV}) )</td>
<td>( R = (\frac{dI^2}{d^2V}) / (\frac{dI}{dV}) )</td>
</tr>
</tbody>
</table>

4.1.1 MIM Diode A- Pt (ALD)/ZnO/Ti - DC Characteristics

I-V characteristics of Pt (ALD)/ZnO/Ti MIM diodes (MIM Diode A), with various areas were obtained with a sweep voltage from -1 V to 1 V. The diodes were fabricated using ALD for 50 nm thick platinum bottom electrode, 4 nm thick ZnO insulator and 100 nm thick titanium layer as top electrode using e-beam evaporation.

The results are summarized in table 12. It can be observed that the results for the different areas show similarity, which could be attributed to the generally large areas which were designed for the junction areas. The responsivity for all the diodes is in the range of 0.5 to 1.5, with not much difference due to area change. Also from the table it can be observed that the mean values obtained from DC characterization are close to the values obtained for MIM diodes with maximum responsivity. The best performing fabricated diode shows a resistance of 1335 Ω and responsivity of 1.49 for MIM diode with an area of 0.09mm².

These numbers in comparison to the literature show that the resistance is reasonable, except for few cases where zero-bias resistance of less than 500 Ω is shown, which was highlighted in chapter-2. Zero-bias resistance mostly has been in the range of MΩ [22, 32, 33, 35, 38]. However it has to be mentioned here that literature does show responsivities that are higher than the ones obtained in the work being presented. Work in [22] shows MIM diodes with responsivity around 4.65, however the zero-bias resistance is 43.38 MΩ. Though it looks like that the responsivity values in this thesis are lower, but it needs to be highlighted that the values
depend on the interface roughness of bottom electrode and insulator, meaning the values being in the similar order is encouraging enough. In addition to the table, plots of I-V response, DC resistance, and responsivity plots for the diodes with best sensitivity are shown below. Current for the MIM diodes with increase in area increases. This is expected as larger area means there is larger area for tunneling of electrons, also leading to decrease in the resistance.

Table 12. MIM diode fabrication A – Pt (ALD)/ZnO/Ti- Summary of DC characterization results

<table>
<thead>
<tr>
<th>MIM Diode A-</th>
<th>Resistance Zero Bias Mean (ohm)</th>
<th>Responsivity @ 1V Mean (1/V)</th>
<th>Best responsivity @ 1V</th>
<th>Current @ 1V for best responsivity diode(mA)</th>
<th>Zero-bias resistance for diode with best responsivity</th>
</tr>
</thead>
<tbody>
<tr>
<td>Area(mm²)</td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>0.01</td>
<td>1503</td>
<td>0.661</td>
<td>1</td>
<td>0.55</td>
<td>2389</td>
</tr>
<tr>
<td>0.04</td>
<td>2005</td>
<td>0.875</td>
<td>1.13</td>
<td>0.91</td>
<td>1556</td>
</tr>
<tr>
<td>0.09</td>
<td>1858</td>
<td>0.949</td>
<td>1.49</td>
<td>1.41</td>
<td>1335</td>
</tr>
</tbody>
</table>
4.1.2 MIM Diode B- Pt (Sputter)/ZnO/Ti - DC Characteristics

In this part I-V response, resistance and responsivity is presented for MIM diodes fabricated using Platinum (sputtering) as bottom electrode. As explained in the fabrication chapter (fabrication referred as MIM diode- B), the MIM diodes bottom electrode was fabricated using platinum via sputtering, followed by 4 nm thick ZnO layer, and 100 nm thick titanium deposited via e-beam evaporation formed the top electrode. Different junction sizes; 0.01, 0.04 and 0.09 mm², have been summarized in table 13 and figure 29.

The resistance for the diodes has been in the range of 3 kΩ- 16 kΩ. The responsivity of the diodes is similar to the ones fabricated using platinum deposition via ALD. The best performing MIM diode showed responsivity of 1.45 and zero-bias resistance of 16 kΩ. Current, resistance and responsivity plots have been shown for the best performing MIM diode for each area.

Table 13. MIM diode fabrication B – Pt (Sputter)/ZnO/Ti- Summary of DC characterization results

<table>
<thead>
<tr>
<th>MIM Diode B- Area(mm²)</th>
<th>Resistance Zero Bias Mean (ohm)</th>
<th>Responsivity @ 1 V Mean (1/V)</th>
<th>Best responsivity @ 1 V</th>
<th>Current @ 1V for best responsivity diode(mA)</th>
<th>Zero-bias resistance for diode with best responsivity</th>
</tr>
</thead>
<tbody>
<tr>
<td>0.01</td>
<td>2213</td>
<td>0.654</td>
<td>0.877</td>
<td>0.397</td>
<td>3095</td>
</tr>
<tr>
<td>0.04</td>
<td>3196</td>
<td>0.694</td>
<td>0.841</td>
<td>0.144</td>
<td>8410</td>
</tr>
<tr>
<td>0.09</td>
<td>6920</td>
<td>0.886</td>
<td>1.45</td>
<td>0.115</td>
<td>16250</td>
</tr>
</tbody>
</table>
4.1.3 MIM Diode C- ZrCuAlNi (Sputter)/ZnO/Ti - DC Characteristics

Another batch of MIM diodes was investigated for I-V response, including resistance and responsivity derived from I-V response, shown in table 14 and figure 30. For this fabrication, 50 nm thick ZrCuAlNi was deposited as bottom electrode using sputtering, while insulator was still the same as for other fabrications- 4 nm thick ZnO, and 100 nm thick titanium deposited using e-beam evaporation was used as top-electrode. In this case the current for the diodes was
much lower compared to the fabrications in which crystalline metals was used. Additionally the resistance for the case of ZrCuAlNi is also much larger compared to other fabrications. This could be attributed to the lower conductivity of amorphous alloy. Lower conductivity of amorphous alloy has been referenced before also in this work, with conductivity values known to be two orders of magnitude lower compared to crystalline metals, as mentioned in [27].

The best fabricated diode shows responsivity of 2.53 with resistance around 36 KΩ. Higher responsivity in this case, might be attributed to the appropriate work function difference between the two electrodes (ZrCuAlNi is reported to have work function of 4.8 eV[27], while titanium has a work function of 4.1 eV, which has been referenced earlier also [40]). This work function could have led to higher non-linearity.

Table 14. MIM diode fabrication C – ZrCuAlNi (Sputter)/ZnO/Ti- Summary of DC characterization results

<table>
<thead>
<tr>
<th>MIM Diode C-Area(mm²)</th>
<th>Resistance Zero Bias Mean (ohm)</th>
<th>Responsivity @ 0.5 V Mean (1/V)</th>
<th>Best responsivity @ 0.5 V</th>
<th>Current @ 0.5 V for best responsivity diode(mA)</th>
<th>Zero-bias resistance for diode with best responsivity</th>
</tr>
</thead>
<tbody>
<tr>
<td>0.01</td>
<td>26755</td>
<td>2.18</td>
<td>2.53</td>
<td>0.0225</td>
<td>36846</td>
</tr>
<tr>
<td>0.04</td>
<td>24300</td>
<td>2.15</td>
<td>2.50</td>
<td>0.0144</td>
<td>49727</td>
</tr>
<tr>
<td>0.09</td>
<td>17420</td>
<td>2.14</td>
<td>2.35</td>
<td>0.0382</td>
<td>19643</td>
</tr>
</tbody>
</table>
4.1.4 MIM Diode D- Cu (e-beam)/ZnO/Ti - DC Characteristics

The last batch of MIM diodes investigated for DC performance were fabricated using copper as bottom electrode using e-beam evaporation. For this case bottom electrode with a thickness of 150 nm was used which is different from the standard of 50 nm bottom electrodes used for other fabrications. ZnO – 4 nm thick, and titanium- 100 nm thick were deposited using the
same methods as for other fabrications- ALD and e-beam evaporation respectively. The results which include, I-V response in addition to the resistance and responsivity are presented for the mentioned MIM diodes in table 16 and figure 31. MIM diodes fabricated with copper showed good responsivity. The best performing diode showed responsivity of 3.64 and resistance of 9 KΩ. Higher responsivity could be attributed to appropriate work function difference between copper (4.5 eV) and titanium (4.1 eV). The mentioned work function difference is more optimal compared to the case, where other bottom electrodes are used; platinum (5.3 eV) and ZrCuAlNi (4.8 eV). Though it is better to have a large work function difference between the two electrodes of MIM diode, but for the case when work function difference is too high, inability to cross the barrier could possibly lead to lower tunneling (lower responsivity).

In the initial stage of the thesis, MIM diodes fabrication for bottom electrodes was done using thicker metals (150 nm and 300 nm thick), which included copper, gold, and platinum. For the case of thicker metals very few devices were functional, additionally the functional devices showed variable performance for a single fabrication. This observation and the study of crystalline metals surface roughness, lead to the fabrication of MIM diodes using 50 nm thick crystalline metal as bottom electrode. The results established the initial conclusion drawn from the surface roughness study that crystalline metals with sub 1 nm surface roughness can show reliable MIM diodes. In comparison, thicker metals showed higher surface roughness and hence less reliable results. For this reason, a comparison of two fabrications in terms of number of functional devices has been presented (MIM diode fabrication B- Platinum 50 nm vs MIM Diode Fabrication D- Copper 150 nm) in table 15.

As discussed and shown earlier, thicker film will lead to higher surface roughness. Higher surface roughness will eventually lead to devices which are possibly shorted and unreliable in terms of electrical performance. For this fabrication, successful fabrication of MIM diodes was less than 15 %. Compared to this for the case, when 50 nm of platinum was deposited using
sputtering the percentage of successful diode fabrications was much higher, as shown in the

table 15 below.

Table 15. Successful MIM Diode Fabrications - Summary

<table>
<thead>
<tr>
<th>MIM diode fabrication material</th>
<th>Bottom Electrode Thickness</th>
<th>MIM Diodes fabricated</th>
<th>Functional MIM diodes</th>
<th>Percentage of successful fabrications</th>
</tr>
</thead>
<tbody>
<tr>
<td>Pt (Sputter)/ZnO/Ti</td>
<td>50 nm</td>
<td>44</td>
<td>34</td>
<td>77%</td>
</tr>
<tr>
<td>Cu (e-beam)/ZnO/Ti</td>
<td>150 nm</td>
<td>44</td>
<td>6</td>
<td>13%</td>
</tr>
</tbody>
</table>

Table 16. MIM diode fabrication D – Cu (e-beam evaporation)/ZnO/Ti- Summary of DC characterization results

<table>
<thead>
<tr>
<th>MIM Diode D-Area(mm²)</th>
<th>Best responsivity @ 0.3 V</th>
<th>Current @ 0.3 V for best responsivity diode(mA)</th>
<th>Zero-bias resistance for diode with best responsivity</th>
</tr>
</thead>
<tbody>
<tr>
<td>0.01</td>
<td>1.99</td>
<td>0.0760</td>
<td>4522</td>
</tr>
<tr>
<td>0.04</td>
<td>3.64</td>
<td>0.0390</td>
<td>9794</td>
</tr>
<tr>
<td>0.09</td>
<td>3.38</td>
<td>0.0148</td>
<td>25253</td>
</tr>
</tbody>
</table>
4.2 RF Characterization of MIM Diodes

One of the main contributions of this work is the comprehensive RF characterization of MIM diodes. The work includes RF performance based on s-parameters and input impedance, from frequency 500 MHz to 10 GHz. DC resistance and responsivity for MIM diodes have already been presented. Based on the DC characterization of MIM diodes, zero-bias responsivity (0.2 V⁻¹ responsivity at 0 V) was obtained with zero-bias resistance in the range of few kilo-ohms.
(1-3 KΩ). Zero-bias responsivity could be used for RF to DC rectification. RF performance measurement of MIM diodes is essential for their integration with other RF components, which was done for the case of MIM diode fabrication- B (50 nm Platinum using sputtering).

The RF measurement setup for RF characterization, covers two types of measurements: 1) S-parameter measurement of MIM diodes, and 2) RF to DC rectification measurement at 2.45 GHz.

For s-parameter measurement, Agilent PNA Network Analyzer was used. PNA was connected to the pads of the MIM diode using probes with 150 µm pitch size. The diodes were designed for frequencies in GHz range, and the setup also covered the two port measurement in the frequency range 500 MHz to 10 GHz. PNA was calibrated using the Short-Open- Load-Thru (SOLT) calibration standard on CSR04 standard. S-parameters of the devices were measured at zero-bias.

RF to DC rectification setup, shown as block diagram in figure 32 used Agilent RF signal generator, in conjunction with ZVE-3W- 83+ Power Amplifier, with Nano voltmeter for measurement of rectified voltage. For RF to DC rectification, measurement set up also involved Agilent Spectrum Analyzer to measure RF power.
4.2.1 RF Characterization using S-parameters and Input Impedance of MIM Diode

In this part, RF characteristics using s-parameters and input impedance for MIM diodes are presented. Platinum (sputtering) was used as bottom electrode, ZnO as insulator, titanium as top electrode and copper as RF pad layer for the MIM diode fabrication.

For s-parameters measurement two structures were measured, namely 1) CPW (Co Planar Waveguide) fabricated using copper pads, and 2) MIM diode integrated with CPW.

Initial measurements covered the s-parameters for standard CPW lines with dimensions: width of CPW transmission line= 0.1 mm, width of ground pads = 1 mm, length of ground pads= 10 mm, gap between transmission line and ground pad = 0.06 mm, as shown in figure 33. RF probes were placed at the beginning of the structure- extreme left and right, as shown in the figure.
S-parameters for the CPW line show excellent transmission with $S_{11}$ below -10 dB for the entire frequency range. Insertion loss which is given by $-20 \log S_{21}$ (in dB), is very small, evident from the results in figure 34, showing $S_{11}$, $S_{12}$, $S_{21}$ and $S_{22}$ in dB.

S-parameters were converted to input impedance using conversion to $z$-parameter matrix ($Z_{11}$, $Z_{12}$, $Z_{21}$ and $Z_{22}$) and using input impedance formulae for a two-port measurement (shown in equation 4.1) in MATLAB. $Z_0$ Refers to the system impedance, in this case Network Analyzer had an impedance of 50 $\Omega$. Real part of input impedance shows a matched CPW line, with values around 50 $\Omega$, while imaginary part is close to 0 (small positive value refers to inductive behavior of CPW line, which is expected).

$$Z_{in} = Z_{11} - \frac{(Z_{12}Z_{21})}{Z_{22} + Z_0}, \text{where } Z_0 = 50 \Omega$$  \hspace{1cm} (4.1)
As mentioned before, second part of the measurement included MIM diode integrated with CPW. CPW line for which results have been presented was integrated with a MIM diode of junction area $0.01\text{mm}^2$, labeled in figure 35. Bottom electrode was fabricated using 50 nm of platinum (grey color in figure), with ZnO 4nm thick insulator layer (fonce color in figure), and 50 nm titanium (blue color in figure) for the top electrode, and copper- 500 nm thick was used as pad layer (red color in figure). All the dimensions for widths and gaps were same as used for the CPW line measurement. S-parameters for MIM diode with a CPW, were measured using the setup mentioned before (RF probes were placed at the beginning of the structure-extreme left and right, as shown in the figure). S-parameters show mismatch, with $S_{11}$ around -5 dB, and high insertion loss with $S_{21}$ around -20 dB. The change in s-parameters is due to the inclusion of MIM diode structure in between CPW line, which results in mismatch.
The s-parameters measurement results shown in figure 36 are translated to input impedance using equation 4.1, also mentioned earlier. Figure 37 shows input impedance for five MIM diodes integrated with CPW (same design as shown in figure 35), showcasing the reliability with which results are repeated. From the input impedance figure, it can be observed that for smaller frequencies (500 MHz) - large real impedance (300 Ω to 400 Ω) is obtained, and for higher frequencies 3 to 10 GHz – real impedance around 30 Ω is obtained. It can also be observed from the input impedance that the trend of resistance follows the zero-bias DC resistance reported earlier - resistance decreases with the increase in frequency [9]. Imaginary impedance shows capacitive behavior, which is also expected because of the MIM diode junction. Capacitance for one of the cases in which MIM diode is integrated with CPW is shown in the figure 38 (separately for 500 MHz- 8 GHz and 8GHz- 10 GHz). Capacitance behavior as expected for the case of MIM diode is observed from the capacitance figure. The results from this input impedance calculation can be used for the case when MIM diode needs to be matched with an antenna.

Figure 35. MIM Diode integrated with CPW used for s-parameters measurement
Figure 36. MIM Diode integrated with CPW s-parameters measurement

Figure 37. MIM Diode integrated with CPW - multiple diodes real and imaginary impedance
4.2.2 RF Characterization using RF to DC rectification with a MIM Diode

In this part RF to DC rectification at 2.45 GHz has been shown for the MIM diode fabricated using the same materials as for s-parameters measurement MIM diode structure. For this purpose two set of measurements were done: 1) RF Power was incident on MIM diode integrated with CPW structure used for s-parameters measurement (shown in figure 35), and 2) RF to DC rectification using the structure shown in figure 40- RF power incident on CPW which is rectified using the MIM diode, and then fed to load (Resistor and Capacitor in parallel).

The first measurement shows that RF power is filtered by the MIM diode, with negligible RF power observed at the output in figure 39. RF input power at 2.45 GHz was swept using a
signal generator connected to CPW of the structure via a power amplifier. RF power at output is six orders of magnitude lower, which was measured using spectrum analyzer. The power levels at the output shows that fundamental frequency is low at the output side- meaning rectification is taking place. This is the intended goal of rectification, as ideally filtering of RF power at output means that rectification is taking place.

In the second measurement, RF to DC rectification concept using MIM diode has been shown, Actual structure used for this measurement is shown in figure 40 ( resistor 100 KΩ and capacitor of 100 pF was used at load side). The measurement set-up figure was shown before in figure 32. The measurements are carried out at 2.45 GHz, for varying RF power levels (corrected for mismatch) as shown in figure 41. The output DC voltage was measured across a load (resistor and capacitor) using a Nano-voltmeter. It can be seen from figure 41, that at zero bias, the device provides a maximum rectified voltage of 4.7 mV obtained with incident RF power of 0.4 W. Figure 41 also shows that voltage does not increase beyond 4.7 mV due to the saturation of the input power (limitation of power amplifier used). Although this voltage value is small, it has to be considered that the MIM diode device used here has a very small area of 0.01 mm². Also its non-linearity and subsequently the rectification ability is not the best. This needs to be further optimized. It is expected that with optimization of the device structure such as using different insulator layers (which is expected to enhance the non-linearity) the output DC voltage can be increased. Nonetheless, it is encouraging to see that at zero bias, rectification of RF signals is possible. As far as the knowledge of the authors is concerned, this is the first
demonstration of rectification of an RF signal using MIM diode at zero bias. This showcases the potential of using MIM diode as rectifier in RF applications such as rectenna design.

Figure 39. MIM diode- filtering RF Power at output - 2.45 GHz

Figure 40. MIM diode RF to DC measurement structure and probe station
4.3 Summary of DC and RF Characterization

Table 17 compares the work done in this thesis with the work from literature on MIM diodes, specific to DC and RF characterization.

The references in the table are for specific work which include RF characterization of MIM diode. The referenced work for RF characterization has limitations with no work showing RF impedance - calculation using the concept of input impedance from s-parameters measurement. Also no work has so far shown zero-bias RF to DC rectification. Some of the referenced works cover s-parameter measurements, however the resistances across RF range were obtained using equivalent circuit model in ADS. Also rectification has been shown using a voltage bias. The work in this thesis addresses the loop holes of RF characterization by obtaining true impedance across RF range and also showcasing RF to DC rectification without any bias.

It can also be observed that surface roughness of bottom electrode has not been investigated in most of the works. The exception is the work by Nasir [27], in which the issue of bottom electrode surface roughness has been analyzed, the referenced work proposes the use of amorphous alloys instead of crystalline metals, as mentioned before in this thesis. The work in this thesis has shown crystalline metals with sub 1 nm surface roughness- comparable with the
results of amorphous alloy, leading to conclusion that crystalline metals are suitable for use as bottom electrodes for MIM diodes.

### Table 17. Comparison of work of this thesis with other work from literature specific to RF characterization and surface roughness analysis

<table>
<thead>
<tr>
<th>Literature Work</th>
<th>Bottom Electrode Surface Roughness</th>
<th>Zero Bias DC Resistance (ohm)</th>
<th>Maximum Responsivity (1/V)</th>
<th>RF Characterization</th>
<th>RF S-Parameter measurement</th>
<th>RF to DC rectification measurement at ZERO-bias</th>
<th>Advantage</th>
<th>Disadvantage</th>
</tr>
</thead>
<tbody>
<tr>
<td>M. Abdel-Rahman et al. 2004 [20]</td>
<td>✗</td>
<td>180</td>
<td>1.65</td>
<td>92.5 GHz and 28 THz</td>
<td>✗</td>
<td>✗</td>
<td>Dual-band voltage detection shown</td>
<td>No rectification shown</td>
</tr>
<tr>
<td>S. Rockwell, et.al 2007 [21]</td>
<td>✗</td>
<td>211 K</td>
<td>13.4</td>
<td>60 GHz</td>
<td>✓</td>
<td>✗</td>
<td>Detector application with bias voltage showcased</td>
<td>No rectification shown</td>
</tr>
<tr>
<td>K. Choi, et.al 2011 [22]</td>
<td>✗</td>
<td>15.25 M</td>
<td>4.44</td>
<td>6.4 GHz</td>
<td>✗</td>
<td>✗</td>
<td>MIM diode field enhancement shown</td>
<td>RF characterization not complete</td>
</tr>
<tr>
<td>A.Kaur, et.al 2014 [23]</td>
<td>✗</td>
<td>110</td>
<td>✗</td>
<td>1-18 GHz</td>
<td>✓</td>
<td>✗</td>
<td>Voltage detection, frequency multiplication shown</td>
<td>Rectification not shown</td>
</tr>
<tr>
<td>O.A.Ajayi 2014 [10]</td>
<td>✗</td>
<td>1.8 K</td>
<td>✗</td>
<td>1-65 GHz</td>
<td>✓</td>
<td>✗</td>
<td>S-parameter measured across a broad RF range</td>
<td>Rectification not shown</td>
</tr>
<tr>
<td>Alimardani, et.al [27]</td>
<td>✓</td>
<td>✗</td>
<td>✗</td>
<td>✗</td>
<td>✓</td>
<td>✗</td>
<td>Focus on surface roughness study and use of amorphous alloys for MIM diode bottom electrode</td>
<td>RF characterization not shown</td>
</tr>
<tr>
<td><strong>THIS WORK</strong></td>
<td>✓</td>
<td>1.3 K</td>
<td>1.5</td>
<td>0.5-10 GHz</td>
<td>✓</td>
<td>✓</td>
<td><strong>Surface roughness study showing low roughness</strong></td>
<td><strong>Responsivity comparable only to the low values reported in literature.</strong></td>
</tr>
</tbody>
</table>


5. CONCLUDING REMARK AND FUTURE WORK

5.1 Conclusion

In this thesis, MIM diodes in RF applications have been investigated. Initial work targeted the issue of surface roughness for bottom electrode of MIM diode, this included investigation of surface roughness of crystalline metals and amorphous alloys. Surface roughness analysis via AFM was done for crystalline metals with various thicknesses - deposited using ALD, e-beam evaporation and sputtering. Additionally, surface roughness was done on amorphous alloy to compare with the results obtained for crystalline metals.

Based on the surface roughness analysis it was concluded that crystalline metals with sub 1nm surface roughness which is comparable with surface roughness of amorphous alloy can be obtained using traditional deposition methods; such as sputtering. This conclusion also helped in the MIM diodes fabrication conducted in this thesis.

MIM diodes were fabricated using crystalline metals as bottom electrode with different deposition methods. For the MIM diodes fabricated, DC characterization was conducted to measure performance. MIM diodes showed rectification ability at zero bias. MIM diode RF characterization results were also obtained. This was done using two methods: 1) S-parameters measurement and input impedance from 500 MHz to 10 GHz, and 2) RF to DC rectification at zero bias. Before this work MIM diodes RF characterization was very limited. Input impedance in RF range had not be obtained, neither was RF to DC rectification ability at zero-bias shown. The presented results of input impedance can be useful in the case when MIM diodes need to be matched for integration in RF applications, such as rectennas. The second part of RF characterization which showcased zero-bias RF to DC rectification also highlights the potential of using MIM diodes in rectennas to harvest and transfer RF energy in smart sensors and other applications.
5.2 Future Work

Future work in this area could explore the integration of MIM diodes with antennas to actually show case zero-bias RF rectification using ambient RF energy. Additionally multiple MIM diodes can be implemented in rectification configuration for RF applications to achieve better efficiency.

Another interesting possibility is to use the multiple insulator diodes from literature (MIIM and MIIM), and integrate them with antennas to achieve better efficiency then that reported in this thesis. However, the eventual goal of this research area should be to achieve MIM diodes fabrication using large-scale printing process (using printers) which could offer the advantage of roll to roll printing of diodes which are integrated with antennas, and are ready for RF energy harvesting. Printing should be targeted on flexible and low-cost substrates which could lead to commercialization of MIM diodes in RF application- using the advantage of zero-bias operation over semiconductor based diodes.
BIBLIOGRAPHY


[27] N. Alimardani, "Investigation of metal-insulator-metal (MIM) and nanolaminate barrier MIIM tunnel devices fabricated via atomic layer deposition," 2013.


