MAGNETIC FIELD OPTIMIZATION FOR HIGH POWER IMPULSE MAGNETRON SPUTTERING

BY
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Abstract
High Power Pulsed Magnetron Sputtering (HPPMS) or High Power Impulse Magnetron Sputtering (HiPIMS) is a promising Physical Vapor Deposition technique with several advantages over DC Magnetron Sputtering (dcMS). HiPIMS has gained a lot of interest in the recent years from the coating industries. The films that are deposited by HiPIMS technique are of superior quality and their properties can be tailored for various applications. The main challenge that obstructs its broader implementation in industry and its use by researchers is its lower deposition rates compared to dcMS.

Magnetic field profile on the magnetron target surface defines plasma properties and potential distribution in the space above the target region. In this work, the magnetic field profile on the top of the target surface is modified to allow more ions to escape from the electric potential trap contributing to the increase in deposition rates. The “ε” magnet pack which was developed based on the idea of modifying the magnetic field configuration demonstrated increased deposition rates in HiPIMS compared to conventional magnet pack arrangement.

In order to keep the deposition rates high as in “ε” magnet pack and improve coating uniformity on substrates, a cylindrically symmetric “TriPack” magnet pack was developed based on the design solutions from “ε” magnet pack. The “TriPack” magnet pack gives higher deposition rates in HiPIMS compared to conventional magnet pack with superior uniformity. A gated ICCD camera was used to investigate the moving localized “ionization zones” in the TriPack. Langmuir probe and ion fraction measurements were also carried out to understand the behavior of high current pulsed discharge in this new magnetic field configuration. Particle flux and critical current
density models were developed to explain the reason behind increase in HiPIMS deposition rates and absence of ionization zones in this new magnetic configuration.
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Contents

Chapter 1 Introduction ......................................................................................................... 1

1.1 Motivation .................................................................................................................. 1

1.2 Thesis Statement ......................................................................................................... 2

1.3 Magnetron Sputtering ............................................................................................... 2

1.4 Ionized Physical Vapor Deposition ........................................................................... 5

1.5 High Power Impulse Magnetron Sputtering ............................................................... 6

1.5.1 HiPIMS Deposited Films ..................................................................................... 10

1.5.2 HiPIMS Deposition Rates ................................................................................... 12

1.5.2.1 Return Effect .................................................................................................. 13

1.5.2.2 Yield Effect .................................................................................................. 14

1.5.2.3 Ion Species Effect ......................................................................................... 15

1.5.2.4 Magnetic Unbalancing and Guiding Effect ...................................................... 16

1.5.3 HiPIMS Ionization Zones/Plasma Spokes ............................................................. 17

1.6 Modulated Pulse Power (MPP) Sputtering ............................................................... 22

1.7 Past Research on HiPIMS Relevant to this Work ....................................................... 23

1.8 Summary .................................................................................................................... 25

Chapter 2 Experimental Set-Up ........................................................................................ 27

2.1 Sputtering High-purity Atomic Deposition Experiment (SHADE) Chamber .......... 27

2.2 Magnetic Field Design Simulation Software ............................................................ 29
4.1.2 TriPack Magnetic Field Configuration .......................................................... 75

4.2 TriPack V300 Testing ..................................................................................... 78
  4.2.1 TriPack V300 Volt-Ampere Characteristics ............................................. 79
  4.2.2 TriPack V300 Deposition Rates .............................................................. 81
  4.2.3 Conclusion from TriPack V300 Deposition Rate Measurements .......... 85

4.3 Experimental Study of Plasma Dynamics in TriPack V300 ......................... 86
  4.3.1 ICCD Camera Diagnostics ....................................................................... 86
    4.3.1.1 ICCD Study of Ignition on All the Three Race Tracks ...................... 86
    4.3.1.2 ICCD Study of Ignition on One Race Track at a Time ....................... 94
    4.3.1.3 Influence of Race Track Width on “ionization zones” ......................... 100
  4.3.2 Optical Plasma Diagnostics ..................................................................... 109
  4.3.3 Ionization Fraction Measurement from TriPack V300 ......................... 110
  4.3.4 Substrate Uniformity Test from TriPack V300 ...................................... 113
  4.3.5 Study of Film Morphology from TriPack V300 ..................................... 114

4.4 Summary ....................................................................................................... 118

Chapter 5 Target Erosion Profile Model .............................................................. 119
  5.1 Target Erosion Model .................................................................................. 119
  5.2 Simulation Results for Conventional Magnet Pack Configuration .......... 125
  5.3 Simulation Results for TriPack V300 Magnet Pack Configuration .......... 126
  5.4 Conclusion ................................................................................................. 128
Chapter 6 Discussion on TriPack V300 Deposition Rates ........................................ 129

6.1 Reasons behind Higher Deposition Rates in TriPack V300 ......................... 130

6.1.1 Magnetic Field Gradients ........................................................................... 131
6.1.2 Diffusion across Magnetic Field ................................................................ 134
6.1.3 Particle Flux Transport Model .................................................................... 141

6.2 Absence of Ionization Zones in TriPack V300 HiPIMS .............................. 147

6.2.1 Critical Current Density Model ................................................................. 148
6.2.2 Reasons for the absence of “ionization zones” in TriPack V300 ............... 150

6.2.2.1 Gas Refill Process .................................................................................. 150
6.2.2.2 Electric Field Shear ................................................................................. 151
6.2.2.3 Local Effect ............................................................................................. 152
6.2.2.4 Sheath and Pre-sheath Spatial Distribution .............................................. 152

Chapter 7 Conclusions and Future Work ............................................................. 154

7.1 Conclusions ..................................................................................................... 154
7.2 Future Work ..................................................................................................... 157

References ........................................................................................................... 160
Chapter 1 Introduction

1.1 Motivation

The demand for high quality, high performance coatings have increased rapidly due to their application in diverse industries. Magnetron sputtering is the most popular and widely used thin film coating technique to deposit high quality industrial coatings[1]. The flexibility of magnetron sputtering process to deposit metals, insulators, ceramics, alloys, etc., makes it the most sought after among all other PVD techniques. In the recent years, the advancement in magnetron sputtering technology has made a huge impact especially in areas like low friction, wear-resistant, corrosion resistant, decorative, optical and hard coatings[2]. Development of Unbalanced Magnetrons (UM), Closed-Field Unbalanced Magnetron Sputtering (CFUBMS), Pulsed Magnetron Sputtering (PMS) and variable field strength magnetrons have revolutionized the capabilities of magnetron sputtering technology [1].

HiPIMS is a relatively new and promising pulsed magnetron sputtering technology which requires substantial development in order to be used commercially. The films that are deposited using HiPIMS technique are denser, smoother and have better adhesion to the substrate compared to the films that are deposited by dcMS[3]. Despite producing high quality films, HiPIMS technique suffers from low deposition rates. Deposition rates determine the throughput of the process. In the commercial coating industry, priority is given to higher throughput rather than the quality of the coatings because as such dcMS process produces reasonable film quality. Further reach of HiPIMS technology largely depends on whether it can match or even produce higher deposition rates than dcMS.
The motivation for this work comes from the need to address the deposition rates issue in HiPIMS.

### 1.2 Thesis Statement

The goal of this work is to find an optimized magnetic field configuration for HiPIMS to increase their deposition rates and to understand the influence of magnetic field configurations on HiPIMS discharges.

### 1.3 Magnetron Sputtering

Magnetron sputtering was developed to overcome the limitations of diode sputtering where the mean free path of electrons was long enough to cause substrate heating effects but not the ions necessary for sputtering[4]. Magnetron sputtering is an enhanced sputtering process where permanent magnets are placed behind the target to confine the electrons to the target vicinity there by increasing the ionization efficiency. Magnetron sputtering process is used to deposit hard, wear-resistant, low friction and corrosion resistant coatings for various industrial applications[1]. In magnetron sputtering process, the sputtered species (neutral and ions) reach the substrate with kinetic energies in the order of 2-30eV which results in high surface mobility in condensing particles that leads to smooth and conformal growth of films[5].

In a magnetron sputtering set-up, magnetic field is applied parallel to the target and the electric field is applied perpendicular to the target so the electrons experience, ExB drift. The trapping of the secondary electrons from the target in the closed ExB drift path increases the ionizing electron-atom collision probabilities. This increases the ion
bombardment to the target leading to higher deposition rates of the sputtered atoms at lower pressures[1]. Figure 1.1(a) is the schematic of a typical planar magnetron sputtering system.

Planar magnetrons are more popular than the cylindrical magnetrons because it is easier to manufacture a planar target than a cylindrical target of any material. The permanent magnets are arranged in the planar magnetron in such a way that ExB drift path (Figure 1.1(b)) returns back to the starting point[6] and this arrangement of permanent magnets is called a magnetic circuit or a magnet pack. The electron motion in the ExB direction gives rise to a circular hall current above the target surface which is greater than the discharge current by a factor of seven if the discharge gas in argon [7]. This factor is determined by the operating gas and pressure [8]. In a magnetron sputtering plasma, the average electron energies are in between 1-10eV and the electron/ion densities are around $10^{11}$cm$^{-3}$[4].

Figure 1.1: (a) Schematic of a typical planar magnetron showing the permanent magnets behind the target to create magnetic field parallel to the target, which along with the perpendicular electric field makes the electron drift in the ExB direction[4], (b) A diagram depicting the ExB drift of electrons on the top of the target surface [9].

In planar magnetrons, maximum ionization occurs at certain portions of the target where the magnetic field is parallel to the target and this region has the highest probability of electrons being located. This region is called the ‘race track’ and it sputters away much
faster than other regions of the magnetron target. Typically, majority of the ionizations takes place at about half the target radius and this is the radius where is sputtering is more dominant[10]. The strength of the magnetic field and cathode potential determines the shape of the ‘race track’/erosion pattern. An increase in the magnetic field strength makes the erosion region narrower whereas an increase in the target voltage makes it broader[11, 12].

Magnetic field configuration is an important aspect in design of a magnetron sputtering system. The magnetic field configuration of a magnetron sputter gun affects the plasma parameters which in turn affect the deposition rates [13-15]. When the magnetic field strength is decreased, the electron temperature decreases with an increase in electron density. The deposition rate increases with a decrease in the magnetic field strength due to the increase in applied magnetron power in order to maintain constant current operation. But the total energy of the deposition flux is reduced with a decrease in magnetic field strength[14]. To sum up, the magnetic field strength in a magnetron sputter gun determines the efficiency of electron confinement which in turn dictates the ionization efficiency.

In Direct Current Magnetron Sputtering (dcMS) discharges, a direct current (DC) voltage is applied to the cathode. The dcMS discharges consist of more inert gas ions than sputtered ions as the degree of ionization in the plasma is very low[16]. The sputtered neutrals have a cosine velocity distribution[17]. In order to increase the fraction of ions in the sputtered flux, Ionized Physical Vapor Deposition technique is used in place of dcMS.
1.4 Ionized Physical Vapor Deposition

Ionized Physical Vapor Deposition (iPVD) is a class of Physical Vapor Deposition (PVD) technique in which >50% of the deposition flux may be ionized[18]. Having more ionized sputter species is beneficial because they help in adatom mobility through the moment transfer to the growing film thereby giving raise to high quality coatings[19]. The target metal ions can be accelerated to the substrate by applying a DC bias on the substrate. Using specific substrate bias, the energy of these incoming metal ions can be tailored to match the typical surface and molecular binding energies which leads to denser films[20] with improved adhesion[21]. Since the ion flux is directional, they can be used to deposit diffusion barriers and seed layers on the side and bottom of high aspect ratio trenches and vias in the microelectronics industry[19]. In iPVD technique, magnetron sputtering is combined with dense secondary discharges like inductively coupled plasma (ICP) discharges or electron cyclotron resonance (ECR) discharges [22-24]. The sputtered metal vapor from magnetron sputtering is ionized by the secondary dense plasma and the metal ion flux is collimated by the plasma sheath. The secondary dense plasma for ionizing the sputtered metal atoms is created using gases like Argon that typically have higher ionization potential than the sputtered metal atoms[25]. Figure 1.2 is the schematic of a typical iPVD chamber that consists of a magnetron sputtering set-up with an ICP coil to create secondary plasma.
Some of the main advantages of this technique are that the deposition temperature can be reduced[26] and the deposition flux can be guided to specific areas of the substrate[27]. The secondary plasma discharge set-up makes iPVD systems more complicated. In order to achieve dense plasma, dcMS discharges have to be operated at high power densities and higher pressure but their maximum power is limited by thermal load on the target due to impinging ions[28]. This problem can be solved by applying short high power pulses to the magnetron target. With the increasing demand for better film quality, there is a constant need for improving the current iPVD techniques. High Power Impulse Magnetron Sputtering (HiPIMS) is an emerging class of iPVD technique that has the potential to cater the demands of the coating industries.

1.5 High Power Impulse Magnetron Sputtering

High Pulsed Power Magnetron Sputtering (HPPMS) or High Power Impulse Magnetron Sputtering (HiPIMS) is a type of iPVD technique where short high-peak-power (up to $3\text{ kW/cm}^2$) pulses are applied to the magnetron sputter target at low duty cycles (0.5-
The typical duration of these high power pulses are hundreds of microseconds and they are applied to the target at frequencies ranging from a few Hz to several kHz. In such discharges, the peak power densities during the pulse can be on the order of several tens of kilowatts per square inch whereas the average power densities are comparable to or equal to Direct Current Magnetron Sputtering (dcMS) discharges [29] to avoid melting or overheating the sputtering target. This results in plasma electron densities as high as $10^{19}$ m$^{-3}$ above the target surface during the short high-power pulses [30, 31]. These high electron densities near the target enhance ionization of sputtered materials [32]. The now-ionized sputtered material is accelerated back to the target resulting in an increase in the sputtering rate by having the discharge go into a self-sputtering mode [33]. Some sputtered ions escape and provide superior deposited film quality on the substrate. The films that are grown using HiPIMS technique are denser, smoother and have better adhesion to the substrate than the films deposited by direct current magnetron sputtering (dcMS) [3].

Figure 1.3 is a plot of peak power density at the magnetron target versus duty cycle for different pulsed magnetron discharges. The HiPIMS discharge range starts from target power densities above 0.5kW/cm$^2$ and Modulated Pulse Power (MPP) sputtering discharges range from 0.05kW/cm$^2$ to 0.5kW/cm$^2$. Time evolution of voltage and current in a typical HiPIMS discharge is shown Figure 1.4. The shape of the HiPIMS pulse is determined by the discharge and the electronics of the power supply. The discharge, in turn depends on the gas pressure and gas type[34]. The evolution of the HiPIMS current pulse during the discharge depends on the target material, pulsing frequency, operating gas and magnetic field strength [34-36].
Over the last decade, there have been several publications on different facets of HiPIMS. The peak electron densities during a HiPIMS pulse has been observed to be $10^{18} - 10^{19}$ m$^{-3}$ [30, 37, 38] which is three orders of magnitude compared to dcMS discharges where the
electrons densities is only around $10^{16} \text{m}^{-3}$[16]. The electron density in HiPIMS discharges increases linearly with increasing discharge current[39] and they largely depend on the target material[40]. The peak electron density travels with a fixed velocity away from the target [41] and the electron energy distribution function (EEDF) is Maxwellian-like during the HiPIMS pulse[42].

The degree of ionization in HiPIMS plasma is much higher than dcMS discharges. HiPIMS metal ionization fraction have been reported to be 70% for Cu[32], 99% for Ti[43], 30% for Cr [44] and 9.5% for Al[45]. The metal ionization fraction depends on the applied power, pulse time, pulse frequency and the distance from the target[46]. Optical emission spectroscopy and mass spectroscopy observations of HiPIMS discharges show that the discharge is initially argon dominated but it develops in to metal dominated discharge during the active phase of the discharge[47]. In addition to singly charged ions, multiply charged ions have also been observed. The singly charged metal ions cannot create the secondary electrons necessary to maintain metal self-sputtering as the first ionization energies of many metals are not sufficient enough to overcome the work function of the target material. Hence, these multiply charged metal ions are critical in transitioning the HiPIMS discharge from argon ion sputtering regime to a self-sputtering regime[33, 35]. Table 1.1 shows the typical plasma parameters for HiPIMS and dcMS discharges.
<table>
<thead>
<tr>
<th>Parameter</th>
<th>HiPIMS</th>
<th>dcMS</th>
</tr>
</thead>
<tbody>
<tr>
<td>Peak Power Density</td>
<td>$3\times10^4$ Wcm$^{-2}$</td>
<td>1 Wcm$^{-2}$</td>
</tr>
<tr>
<td>Maximum Average Output Power</td>
<td>20 kW</td>
<td>20 kW</td>
</tr>
<tr>
<td>Current Density</td>
<td>1-1000 Acm$^{-2}$</td>
<td>0.01-0.1 Acm$^{-2}$</td>
</tr>
<tr>
<td>Discharge Voltage</td>
<td>500-1000 V</td>
<td>500 V</td>
</tr>
<tr>
<td>Process Gas Pressure</td>
<td>$10^{-3}$-$10^{-2}$ Torr</td>
<td>$10^{-3}$-$10^{-2}$ Torr</td>
</tr>
<tr>
<td>Magnetic Field Strength</td>
<td>0.010-0.100 T</td>
<td>0.010-0.100 T</td>
</tr>
<tr>
<td>Electron Density</td>
<td>$10^{18}$-$10^{19}$ m$^{-3}$</td>
<td>$10^{16}$ m$^{-3}$</td>
</tr>
<tr>
<td>Electron Temperature</td>
<td>1-5 eV</td>
<td>1-7 eV</td>
</tr>
<tr>
<td>Degree of Metal Ionization in the</td>
<td>30-100 %</td>
<td>&lt;5 %</td>
</tr>
<tr>
<td>discharge</td>
<td></td>
<td></td>
</tr>
<tr>
<td>Ion Energy (average for metal ions)</td>
<td>20-100 eV</td>
<td>5 eV</td>
</tr>
</tbody>
</table>

### 1.5.1 HiPIMS Deposited Films

Higher fraction of ionized sputtered species during HiPIMS discharges allows tailoring and improving the properties of the growing film[31]. Increase in the ion flux to the substrate reduces intracolumnar as well as intercolumnar porosity due to enhanced surface mobility caused by the incoming ions[49]. It has been reported by Eriksson et al. that in the case of ion assisted film growth, where ion to metal flux is higher results in increased ad-atom mobility and smoothening of the surface[50]. Density of film coatings is a key parameter in diffusion barriers, thermal barriers, corrosion and wear resistant coatings[51]. The films that are deposited by HiPIMS technique exhibit ultra-dense microstructure and a smooth surface compared to films that are grown by dcMS techniques[52].
It can be observed from Figure 1.5 (a) and (c) that, Ta film deposited using HiPIMS technique are dense, featureless and have columnar structure normal to Si/Ta interface. Also, the films that are deposited using HiPIMS technique have increased film density, enhanced adhesion[21], and improved surface roughness[53]. Using HiPIMS technique, deposition on substrates with complex shapes and high aspect ratio trenches can be achieved[52]. It is possible to tailor phases[54], modify electrical and optical properties of thin films by HiPIMS technique[31]. Also, the deposition material can be guided to desired areas of the substrate in HiPIMS[27]. Hysteresis free reactive sputtering can be
achieved in HiPIMS discharges[55]. Deposition on heat-sensitive substrates like polymers has always been a problem with dcMS technique. HiPIMS generates lower thermal flux per unit deposition rate[56], hence deposition on heat-sensitive substrates can be achieved without any issues.

1.5.2 HiPIMS Deposition Rates

Despite having many important film properties, HiPIMS deposition rates are lower than dcMS discharges for the same average power[34]. Deposition rates are usually measured in nm/min, Å/s or Å/min[57]. The deposition rates depend on sputtering rates. The sputtering rates in turn depend on the ion current density at the target and the sputter yield, which is determined by the target material, the bombarding species, and the bombarding ion energy[34]. Deposition rates are determined by the power density, the target material, size of the erosion area (race track area), target-to-substrate distance, and the discharge pressure[34]. The deposition rates in dcMS are found to be directly proportional to the power applied to the target[58]. In HiPIMS regime, the absolute deposition rate increases almost linearly with increasing applied power, while the deposition rate per unit power decreases[59]. V. Kouznetsov et al. reported deposition rates of HiPIMS to be 80% lower than dcMS[32]. Figure 1.6 is a plot of deposition rates for dcMS and HiPIMS discharges for different target materials for the same average power. The left axis represents the deposition rate in nm/min, the right axis represents the ratio of HiPIMS deposition rate over dcMS sputtering rate and the bottom axis represent the different target materials. It can be observed from this plot that deposition rates in HiPIMS are consistently lower than dcMS. For example, in the case of aluminum, the HiPIMS deposition rates are only 50% of dcMS deposition rates.
The reduction in the deposition rates was not pronounced for materials with low sputtering yield[60]. It was reported that the decrease in deposition rates becomes more pronounced with increased pulse length[61]. Possible reason for this observation is that the shorter pulse length does not allow the gas rarefaction, self-sputtering and other processes to develop[34]. The deposition rate in the self-sputtering mode is found to be lower than when argon sputtering dominates[62]. For shorter pulses, Argon ions are the dominant sputtering species, hence there is little self-sputtering taking place which can keep the effective sputtering rates high[63]. There are several reasons for the lower deposition rates in HiPIMS. Some of the major ones are discussed in the coming sections.

### 1.5.2.1 Return Effect

There are several reasons for lower deposition rates, the return effect is considered to be the most important and prominent compared to other effects. In HiPIMS discharges, large fractions of sputtered atoms are ionized due to the dense plasma in front of the target.
Some of these newly formed ions return to the target contributing to currents and cause self-sputtering[57]. The sputtered material is ionized close to the target and the negative potential applied on the target can extend far into the plasma as an extended pre-sheath. Hence, many of the metal ions will be attracted back to the target surface by the cathode potential[64]. Figure 1.7 is a schematic representation of the fluxes involved in the deposition by HiPIMS technique under conditions when the plasma is dominated by metal sputtered from the target. $\alpha$ is the ionization probability, $\beta$ is the return probability and $\gamma$ is the sputter yield. The values of $\alpha$ and $\beta$ depend on the target material, HiPIMS system and discharge parameters.

![Figure 1.7: Schematic representation of the fluxes involved in the deposition by HiPIMS under conditions when the plasma is dominated by metal sputtered from the target. $\alpha$ represents the ionization probability, $\beta$ represents the return probability and $\gamma$ represents the sputter yield[57].](image)

1.5.2.2 Yield Effect

Increase in operating voltage requires the average current to drop down in order to maintain the same average power. The deposition rates of dcMS and HiPIMS discharges
at equal average power could only be the same if the sputtering yields were exactly proportional to the voltage\cite{65}. Unfortunately, according to sputtering theory, the yield has non-linear dependence to the voltage. HiPIMS pulses use higher voltages compared to dcMS discharges, hence there is a greater reduction in the HiPIMS deposition rate. This is because sputtering yield does not scale linearly with the energy of the incoming ions\cite{57}.

\subsection*{1.5.2.3 Ion Species Effect}

Sputter yield is mostly determined by the target material. It can be observed from Figure 1.8 that, the Argon ion sputtering and self-ion sputtering are off by factor of 10-15\%\cite{57}. When HiPIMS discharge transitions from argon sputtering to self-sputtering mode, this effect becomes prominent. Hence, during self-sputtering mode, the deposition rates go down. The first ionization energy of argon is 15.76eV and copper is 7.73eV (6.82eV for titanium) which means singly charged metal ions has less than twice the work function of the metal so they cannot result in any kinetic or potential emission of electrons. Therefore, change in sputtering ion, changes the secondary electron emission yield. Change in sputtering ion, changes the secondary electron emission yield. This becomes critical because the singly charged metal ions have less than twice the work function of the target material\cite{33} whereas singly charged noble gas ions have energies greater than or equal to the work function\cite{66}. Effect of this reduction in deposition rates is limited because, the secondary electrons appear only in the discharge current which only a small percentage of the total discharge current\cite{57}.
1.5.2.4 Magnetic Unbalancing and Guiding Effect

In case of unbalanced magnetrons, the fields extend far beyond the target helping the plasma to escape and assist in film growth. HiPIMS discharges are characterized by high hall or drift currents which affect the magnetic field distribution which in turn affects the local plasma density, ionization probability, return probability and the plasma transport of material from target to the substrate\cite{57}. Applying external magnetic fields can redirect the plasma flow and lead to enhanced localized deposition rates\cite{27}.

In addition to all these effects it has been found that significant fraction of ions of the sputtered material is transported sideways\cite{67}. Overall reduction in HiPIMS deposition is a combination of all the above mentioned factors\cite{68}. All these named effects are interconnected in HiPIMS discharges and have a strong correlation with the plasma parameters. The individual contribution from each effect is hard to quantify as it depend on the magnetic field configuration on the target surface, HPPMS pulsing power supply regime, geometry, etc.
1.5.3 HiPIMS Ionization Zones/Plasma Spokes

HiPIMS plasma is characterized by a high discharge current which generates plasma waves and instabilities that induces plasma flares[69], azimuthally symmetric particle jets[70] and self-organized patterns[71]. Plasma instability like the drifting ionization/”hot” zones/Plasma spokes on HiPIMS plasma have been observed many groups [72-75] around the world.

A. Kozyrev et al. first observed plasma inhomogeneities in the azimuthal direction on a 90mm titanium magnetron in High-Current Pulsed Magnetron Discharges (HPMD). At higher current, bunches form and the number of bunches increased with increasing currents (Figure 1.9). These bunches were found to rotate at a linear velocity of ~1cm/µs in electron hall current direction (ExB). Formation of these bunches were caused by the necessity to transfer the high density electron current across the magnetic field[72].

![Figure 1.9](image)

Figure 1.9: Images from high-speed optical frame camera at discharge currents of (a) 200A, (b) 400A and (c) 600A on a 90mm Titanium magnetron[72].

A. P. Ehiasarian et al. had reported plasma instabilities in HiPIMS discharges that include regions of bright and dark plasma arranged periodically along the race track region of a 50mm titanium target (Figure 1.10). The dense plasma structures move anticlockwise in the ExB direction with linear velocities of 10-20km/s. These drift wave instabilities under certain experimental conditions, saturate and form azimuthally periodic structures rotating between 100-200kHz[73].
A. Anders et al.[76] found localized hot ionization zones (Figure 1.11 (a)) drifting along the race track in the ExB direction with a velocity that is 10\% of the electrons ExB drift velocity. It can be observed from Figure 1.11 (b), that there are four distinct ionization zones and the zones move in the counter clockwise (ExB) direction which can be seen from the streak camera images. The azimuthal velocity of these ionization zones was calculated to be around 10^4 m/s from the ICCD images. The azimuthal velocity of these ionization zones depends on the gas type and the target material. Table 1.2 shows the observed azimuthal velocities for different target materials and gases, the plus-minus indicates the range of velocities measured within several measurements. An azimuthal electric field is associated with each ionization zone that breaks the magnetic confinement of electrons via the ExB drift component which is actually in the axial direction. It has been proposed that the dense plasma/ionization regions have high stopping power for the highly energetic drifting electrons which in turn leads to a dense plasma. These fluctuations tend to grow at the expense of less ionization events in the downstream, therefore the zones takes triangular shape with the densest part of plasma along the magnetic field line[76].
Figure 1.11: (a) Localized ionization zones (spots of intense plasma) drifting during HiPIMS plasma on a 76mm Nb target[76], (b) Top: ICCD Image at 42.6μs from the start of the HiPIMS pulse, Bottom: Corresponding streak camera image of the ICCD gated camera snapshot[76].

<table>
<thead>
<tr>
<th>Target name</th>
<th>Atomic mass (amu)</th>
<th>Surface binding energy (eV)</th>
<th>Gas name</th>
<th>Atomic mass (amu)</th>
<th>Observed azimuthal velocities (m/s)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Al</td>
<td>30.0</td>
<td>3.19</td>
<td>Ar</td>
<td>39.9</td>
<td>8100 ± 300</td>
</tr>
<tr>
<td>Al</td>
<td>30.0</td>
<td>3.19</td>
<td>Kr</td>
<td>83.8</td>
<td>5400 ± 300</td>
</tr>
<tr>
<td>Cu</td>
<td>63.5</td>
<td>3.48</td>
<td>Ar</td>
<td>39.9</td>
<td>4200 ± 400</td>
</tr>
<tr>
<td>Cu</td>
<td>63.5</td>
<td>3.48</td>
<td>Kr</td>
<td>83.8</td>
<td>4200 ± 150</td>
</tr>
<tr>
<td>Nb</td>
<td>92.9</td>
<td>5.93</td>
<td>Ar</td>
<td>39.9</td>
<td>7000 ± 2000</td>
</tr>
<tr>
<td>Nb</td>
<td>92.9</td>
<td>5.93</td>
<td>Kr</td>
<td>83.8</td>
<td>5700 ± 700</td>
</tr>
<tr>
<td>W</td>
<td>183.4</td>
<td>8.7</td>
<td>Ar</td>
<td>39.9</td>
<td>6250 ± 300</td>
</tr>
<tr>
<td>W</td>
<td>183.4</td>
<td>8.7</td>
<td>Kr</td>
<td>83.8</td>
<td>4000 ± 300</td>
</tr>
</tbody>
</table>

Table 1.2 Observed velocities of the ionization zone for different target and different gases[76].

Dr. Boeuf’s PIC-MCC (Particle-In-Cell Monte Carlo Collisions) simulations show the formation of plasma non-uniformities perpendicular to the magnetic field leading to anomalous transport across magnetic fields in Hall thrusters and cylindrical magnetrons. Formation of non-uniformities in the azimuthal direction cannot be described by 1D simulations, so 2D simulations were employed in this work. It was revealed from the 2D simulations that the electron sheath next to the anode was unstable and it transformed in
to rotating electron vortices (Figure 1.12 (a)) at lower pressures whose time of evolution is much longer than the rotational period. At higher pressures (above 1mTorr), the plasma properties are not azimuthally symmetric and there is an abrupt azimuthal drop or spoke that defines separation between two plasma regions. The potential drop in the front of these spokes causes the ions to accelerate azimuthally which can be observed in Figure 1.12 (b). All these simulation results correspond to flute mode where plasma is assumed to be uniform across the magnetic field. Lower plasma densities were used in these simulations. These simulation results do not say how it can be applicable in case of higher plasma densities. As stated by Dr. Boeuf, a lot of work is needed to quantify these instabilities on average electron transit time from anode to cathode and to compare this predicted 2D anomalous electron transport simulations with 1D collisional transport models[77].

It was earlier though that the ionization zones propagate between $10^3 \text{m/s}$ to $10^4 \text{m/s}$ in the ExB direction[73]. But Yang et al.[78] has observed that these ionization zones
actually move in the ExB direction with velocities of $10^3 - 10^4$ m/s in case of higher discharge currents and in the case of lower discharge currents, they move with velocities of $10^3$ m/s in the –ExB direction (Figure 1.13 (a)). The number of ionization zones is determined by the pressure and discharge current. It is hypothesized that at higher currents, large fraction of neutral atoms within the ionization zone are ionized thereby reducing the neutral density. This is followed by a reduction in the local ion densities as ions want to move away following the local electric field. The new drifting electrons experience depletion of neutrals, therefore they have to drift further in the ExB direction before encountering sufficient neutrals. Hence, these ionization zones propagate in the ExB direction in case of higher currents. When the discharge currents are low, there are adequate neutrals available all the time as they are never depleted because of the ionization, therefore the electrons approach this zone from the –ExB direction and deposit it on the –ExB side so the zones now propagate in the –ExB direction[78].

Figure 1.13: (a) Images showing the direction change of ionization zone at argon pressure of 0.8Pa on a gold target[78], (b) ICCD Images of HiPIMS discharges on different target at different discharge currents[79].

It has been shown that these localized ionization zones disappeared in the case of high discharge currents for high yield targets (Figure 1.13(b)). The disappearance of the ionization zones was proposed to be due to an increased supply of neutral atoms from the target that cools the electrons, which in turn lowers the ionization rate, which means the
electrons don’t have to drift further to find high neutral density regions. They did not observe this disappearance of ionization zones in case of Nb and Ti targets[79].

Andersson et al.[80] has shown that the discharge inhomogeneities/ionization zones smoothen out at higher currents during gasless HiPIMS of copper which is shown in Figure 1.14. It has been suggested that the presence of adequate neutrals aid in smoothing of the inhomogeneities.

![Image of ICCD Images of HiPIMS discharges on copper current at different discharge currents][80]

**Figure 1.14:** ICCD Images of HiPIMS discharges on copper current at different discharge currents[80].

### 1.6 Modulated Pulse Power (MPP) Sputtering

Modulated Pulse Power (MPP) sputtering is another type of iPVD technique that is based on the HPPMS, which can achieve dense plasma and high ionization of the sputtered material[81]. The main difference between the HPPMS and MPP techniques lies in the magnitude, duration and shape of the high power pulses[82]. In HPPMS, a short single high power pulse in the order of 50-150 µs with a duty cycle of 1-10% is applied to the target[83]. Whereas in MPP, pulse length in the order of 500-3000 µs is with a duty cycle in the order of 10-28% is applied to the target. The MPP technique consists of long macro
pulses with smaller micro pulses that can be adjusted arbitrarily. MPP sputtering gives the freedom to control the macro and micro pulse duration, frequency and discharge voltage which gives this technique much more flexibility compared to HiPIMS. The discharge parameters can be tailored according to the specific needs of sputtering. Figure 1.15 shows the time evolution of voltage, current and power of a typical MPP discharge. Like HPPMS, the films that are deposited using MPP sputtering have denser microstructure with finer grains[84].

![Figure 1.15: Time evolution of voltage, current and power during a typical MPP discharge [81].](image)

### 1.7 Past Research on HiPIMS Relevant to this Work

There were a few attempts in the past to investigate the influence of magnetic field on HiPIMS parameters. It has been found that in case of reduced magnetic field strength, the ion flux towards the substrate is increased due to the reduction in the metal ions return effect in front of the target[85, 86]. The most recent research publication by J. Capek et al.[85] shows that for high magnetic field strength, the back-attraction of the target ions towards the target is the dominant effect, while for low magnetic field strength the ion
back-attraction, the sub-linear dependence of the sputtering yield on the ion energy, and the variation in material transport effects are all important. Also, A. Mishra et al [87] reported that with a reduction of magnetic field by 33% at the target surface, the deposition rate of titanium increased by a factor of 6. But in this case, due to lower magnetic field, the voltage had to be increased to get the same currents, therefore the overall power was also increased.

J. Bohlmark et al.[27] created an external magnetic field using a current carrying coil in front of the target which acted as a focusing lens for the deposition material. The spatial distribution of material significantly changed when the plasma is exposed to an external magnetic field. The deposition rate was increased with about 80% for a sample placed on the center axis whereas for a conventional dcMS only a minor effect of the magnetic field was observed.

L. Meng et al [88] developed a time dependent HiPIMS ionization region model to predict the temporal densities of different species like electrons, metal atoms, metal ions, argon ions and argon atoms.

Figure 1.16: (a) Electron density and densities of Ar+ and Cu+ during a HiPIMS discharge, (b) Ionization fraction of Cu, Ar and the fraction of Cu+ ions in the total ion flux[88].

Figure 1.16 (a) shows how the electron densities and densities of Ar+ and Cu+ during a HiPIMS discharge and Figure 1.16 (b) show the time evolution of Ionization fraction of
Cu, Ar and the fraction of Cu+ ions in the total ion flux. This time-dependent model not only describes the development of ion species but also predicts the transition to self-sputtering in HiPIMS.

Initial results from magnetic field optimization for HiPIMS on a 14” (36cm) copper target at Center for Plasma Materials Interaction (CPMI) in University of Illinois, show that spiral magnet pack design was able to sustain high impulse current, attain downstream plasma with superior uniformity, and yield better target utilization without the assistance of magnet rotation in HiPIMS[89]

1.8 Summary

Past studies on various aspects of HiPIMS clearly indicates the importance of the following aspects of a magnetron: magnetic field magnitude and the magnetic field profile on the magnetron target surface [27, 86, 90], plasma impedance [91], plasma instabilities [67] and power supply pulsing parameters [56, 92].

In spite of all the recent studies on ionization zones in HiPIMS, there are no concrete theoretical explanations that can describe how and why these ionization zones forms, why they propagate in certain direction under certain conditions, how they stabilize and how these ionization zones are related to deposition rates.

Once the HPPMS discharge gets to a state with a high fraction of ionized sputtered material, the electric field distribution inside the plasma prevents ions from escaping the plasma region. The plasma electric potential during the discharge controls the movement of ions, this means the electric potential distribution in plasma during the discharge has to be controlled or at least modified to increase the ion flow away from this trap. There are
publications [93] that describe the relationship between magnetic field topology and electric potential distribution inside the plasma. Several attempts to investigate the influence of magnetic field on HPPMS parameters were done previously and can be summarized as follows: in case of reduced magnetic field strength the ion flux towards the substrate is increased due to the reduction in the metal ions return effect in front of the target [85, 86, 94].

There are several HiPIMS publications that discuss the reasons for lower deposition rates but do not describe any solution [3, 57, 65]. Magnetic field profile on the magnetron target surface defines plasma properties and potential distribution in the space above the target region. The magnetic field influence on the behavior of high current pulsed discharges is not yet completely understood. From all the previous experimental work, it can be concluded that the shape and magnitude of the magnetic field above the cathode has the major contribution to the deposition rates. Hence it is very important to investigate the magnetic field influence on HiPIMS discharges as it can lead to higher deposition rates by mere changing of magnetic field pattern on the target surface. This work aims at optimizing magnetic field configuration for HiPIMS to increase the deposition rates and understand the influence of magnetic field configurations on HiPIMS discharges.
Chapter 2 Experimental Set-Up

In this chapter, the experimental set-up, power supplies, magnetic field design software and various diagnostic tools that were used in this work will be described in detail.

2.1 Sputtering High-purity Atomic Deposition Experiment (SHADE) Chamber

SHADE chamber is a dual magnetron setup for depositing thin films under an ultra-high vacuum (UHV) environment. The SHADE chamber (Figure 2.1 and Figure 2.2 (a and b)) is equipped with a load lock for sample transfer and a rotatable substrate holder for increasing the uniformity of deposition, the substrate holder can be biased if required.

The stainless steel vacuum chamber is pumped by oil free vacuum pumps to a base pressure of $1 \times 10^{-7}$ Torr. Gas flow into the chamber is controlled by mass flow controllers (MFC). For in-situ measurements of deposited film thickness, the SHADE chamber was equipped with an Inficon DLAE47 Quartz Crystal Microbalance (QCM). For all the deposition rate experiments, the QCM was positioned equidistant between the two

![Figure 2.1: Schematic diagram of the SHADE chamber.](image-url)
magnetrons. To increase the accuracy of the QCM, two QCMs (one for control and the other one for actual measurements) were used simultaneously at the same location. Control QCM is masked with a stainless steel shim stock to subtract the noise due to thermal drift.

A pair of Kurt J. Lesker’s 4” TORUS magnetron sputter sources was installed in the SHADE chamber for this work. In this manner two different magnetic configurations can literally be tested side-by-side keeping all other parameters identical. All the different power supplies used in this work is discussed in the latter sections in detail.

![Figure 2.2: (a) Photograph of the SHADE chamber with the dual magnetron set-up, (b) Photograph of the SHADE chamber with pumping systems and various feedthrough.](image)

The discharge voltage during magnetron operation was measured using a 100:1 Tektronix voltage probe and the discharge current was measured using LEM LF 1005-S Hall Effect current monitor. Aluminum (Al), titanium (Ti), copper (Cu) and carbon (C) targets were used in this work.
2.2 Magnetic Field Design Simulation Software

COMSOL Multiphysics finite element analysis software was used to simulate the magnetic field profile above the target surface in this work. The magnetic and electric field modules of COMSOL Multiphysics was used to calculate the magnetic flux densities and surface magnetic field, $B_{//} (((B_x^2 + B_y^2), XY plane is parallel to the target surface))$ for a given arrangement of magnets. The distribution of $B_{//}$ above the target surface helps in predicting the racetrack region corresponding to a certain arrangement of magnets in a magnet pack. All models have one-to-one scale. The Charged Particle Tracing (CPT) module of COMSOL Multiphysics was used to simulate the electron trajectories above the target surface for different magnet packs. In the CPT module, the electrons were injected into the race-track from the target surface with energy of 0.1eV. The average electron temperatures in HiPIMS discharges are in the order of 0.3-0.4 eV at 20mTorr[42]. In the CPT module, electrons are ejected from the target surface with energy of 0.1 eV which is in the range of the average electron energy. More importantly, in the model, the cathode is biased to -600V, so the energy at which they are launched is less relevant because they are greatly accelerated by the -600V target voltage to over the average electron temperatures, making the 0.1eV assumption conservative. These electron trajectory simulations take into account electron-electron cumblic interactions and the electron-field interactions that arise from the static electric and magnetic fields. This model does not make any assumptions on the total electron densities. This model is intended to show the basic trajectories of certain number of electrons on the top of the race track. The electron-field interactions in this model take in to account only the
interaction of electrons with the cathode potential and magnetic field due to the permanent magnets and not the field generated by hall current. This approach allows predicting the general behaviour of electrons in the trap. This approach is a crude method for a HiPIMS discharge but still gives some information on the electron trajectories.

The electron trajectories, magnetic fields, and electric fields were simulated inside a sphere of 25 inch diameter to avoid edge effects on boundaries in the model. The outer wall (the edge of the whole model) boundary condition for magnetic field was set to zero surface currents and also zero potential for electric field simulations. The magnets were modeled as recommended by the COMSOL software manual. In the model, the cathode was biased to – 600V and the plasma sheath was artificially set to 1 mm. In this artificial sheath region on the top of the target surface, 85% of the voltage was dropped so it mimics a more realistic sheath drop. The time step for the model was controlled by the COMSOL solver and was as small as 0.01 ns. All electrons were released homogeneously from cathode surface at time step zero. In order to obtain more data points for trajectory tracing, the total number of electrons released from the target surface was set to 1000.

2.3 Conventional 4” Magnetron Gun

Kurt J. Lesker’s 4” TORUS magnetron sputter sources Figure 2.3(a) comes with a magnet pack which consists of a central cylindrical magnet surrounded by another concentric cylindrical magnet that are oppositely poled (Figure 2.3(b)). In this conventional arch shaped magnetic field configuration, the arch of the magnetic field lines starts from the centre of the magnetron target and continues to the outer edge. This type of magnet pack will be referred to as a “conventional magnet pack”. Figure 2.4(a)
shows the shape and magnitude of $B_{//}$ field on the XY plane of the target surface simulated using COMSOL Multiphysics. X and Y axis represents the physical dimension in inches of the target. Figure 2.4(b) shows the streamline plot of magnetic field lines in XZ plane, $B_x$ and $B_z$ (magnetic field component along x and z directions) of the conventional magnet pack that was simulated in COMSOL Multiphysics. Figure 2.5 is a photograph of target erosion/“race track” pattern due to the conventional magnet pack arrangement on a 4” aluminum target.

![Figure 2.3: (a) Photograph Kurt J Lesker Company’s 4” TORUS, (b) Arrangement of magnets in the conventional magnet pack.](image)

![Figure 2.4: (a) $B_{//}$ on the target surface of the conventional magnet pack, the black line AA’ shows the location of the cut section (b) Streamline plot of $B_x$ and $B_z$ components in the XZ plane cut section along AA’, the red lines indicate the $B_x$ and $B_z$ components.](image)
In order to simulate the electron trajectory in this conventional magnet pack, COMSOL Multiphysics’s CPT module was used. The details of these simulations are well described in Section 2.2. The results from these simulations indicate that, at time $t = 0$, electrons start to gyrate along the magnetic field and drift in the ExB direction as shown in Figure 2.6(a, b and c). Figure 2.6(b) shows the full electron trajectory in ExB direction at 1µs. It can be seen from Figure 2.6(c) that the electrons are trapped efficiently above the target surface. This process is well described in A. Anders et al. [95] and various books. The different colors in the electron trajectory plots represent the electron velocities. While their values are not relevant for this work, red is $3.5 \times 10^6$ m/s going to blue at $0.5 \times 10^6$ m/s. One can see that in the conventional magnet pack, the electron trap is highly efficient which in turn makes the HPPMS ion recycling process also very efficient.
2.4 Power Supplies

2.4.1 Advanced Energy Pinnacle Plus

The Advanced Energy Pinnacle Plus power supply is rated for single output 10kW average power. This power supply can be operated in DC as well as pulsed-DC mode.
Figure 2.7 is the photograph of the Advanced Energy Pinnacle Plus power supply and Table 2.1 shows the operating parameters of this power supply. In this work, this power supply was used in dcMS sputtering experiments and also as a DC input power source for Starfire Impulse power supply which will be discussed in the next section of this chapter.

### 2.4.2 Starfire Impulse HiPIMS Power Supply

Starfire Impulse power supply is a 2kW pulsed power module for HiPIMS applications. It needs an external DC input for operation. In this work, the output DC voltage from Advanced Energy Pinnacle Plus was fed in to Starfire Impulse power supply. The capacitors inside Starfire Impulse power supply can be charged up to 1000V. The output
peak voltage is 1000V and the output peak current can go up to 200A. Figure 2.8 is the photograph of the Starfire Impulse power supply used in this work.

![Starfire Impulse Power Supply](image)

**Figure 2.8: Photograph of the Starfire Impulse supply used in this work.**

<table>
<thead>
<tr>
<th>Parameters</th>
<th>Range</th>
</tr>
</thead>
<tbody>
<tr>
<td>Average power</td>
<td>0-2kW</td>
</tr>
<tr>
<td>Voltage</td>
<td>325-1000V</td>
</tr>
<tr>
<td>Current</td>
<td>Up to 200A</td>
</tr>
<tr>
<td>Pulse length</td>
<td>5µs-1ms</td>
</tr>
<tr>
<td>Frequency</td>
<td>1Hz-10kHz</td>
</tr>
</tbody>
</table>

**Table 2.2 Pulse parameters range of Starfire Impulse power Supply.**

The Starfire Impulse power supply used in this work was the alpha version with very limited features. The newer versions of this power supply have superior arc detection and suppression technology, real-time discharge voltage and current monitoring capabilities, master/slave module timing for substrate bias and cathode synchronization for co-deposition. Figure 2.9 shows the Voltage-Current waveforms from Starfire Impulse power supply on 4” conventional TORUS at 2mTorr and the pulsing parameters range for this power supply is given in Table 2.2.
Figure 2.9: Voltage-Current waveforms from Starfire Impulse supply with a conventional magnet pack and titanium at 2mTorr.

2.4.3 Huettinger HiPIMS Power Supply

Huettinger TruPlasma Highpulse 4002 DC generator was used to power the 4’’ magnetron sputter guns in this work. The power supply unit consists of a DC charging unit, high impulse generator unit and impedance matching circuit. Figure 2.10 is the photograph of the Huettinger HiPIMS power supply in this work. The pulsing parameters range for this power supply is given in Table 2.3.

<table>
<thead>
<tr>
<th>Parameters</th>
<th>Range</th>
</tr>
</thead>
<tbody>
<tr>
<td>Average power</td>
<td>0-10kW</td>
</tr>
<tr>
<td>Voltage</td>
<td>Up to 2000V</td>
</tr>
<tr>
<td>Current</td>
<td>Up to 1kA peak</td>
</tr>
<tr>
<td>Pulse length</td>
<td>1-200μs</td>
</tr>
<tr>
<td>Frequency</td>
<td>2-500Hz</td>
</tr>
</tbody>
</table>
The DC charging/current supply unit of this power supply contains a converter which charges the capacitor bank in the pulse generator unit. The capacitor bank consists of four separate sections, hence the output from the DC current supply unit is split into four separate channels and each channel charges a specific section of the capacitor bank. The pulse generator unit has capacitor banks, semiconductor switches and freewheeling diodes. This unit communicates with the main control circuit in the DC current supply unit. The impedance matching circuit shapes the output current pulse in order to match the cathode specification if necessary. Figure 2.11 shows the Voltage-Current waveforms from Huettinger HiPIMS power supply on 4” conventional TORUS at 10mTorr.
2.4.4 Zpulser Cyprium IV Modulated Pulsed Power (MPP) Supply

Zpulser MPP supply allows achieving longer pulse lengths compared to certain HiPIMS power supplies. They generate long macro pulse (up to 1ms) which is composed of a train of 20-30μs micropulses. The “ON” and “OFF” times of the macro and micro pulses along with the frequencies can be varied according to the process requirements.

Figure 2.11: Voltage-Current waveforms from Huettinger HiPIMS power supply with a conventional magnet pack and aluminum target at 10mTorr.

Figure 2.12: (a) Photograph of the Zpulser MPP power supply used in this work, (b) Voltage-Current waveforms from Zpulser MPP power supply with a conventional magnet pack and aluminum target at 10mTorr.
Table 2.4 Pulse parameters range of Huettinger HiPIMS power Supply.

<table>
<thead>
<tr>
<th>Parameters</th>
<th>Range</th>
</tr>
</thead>
<tbody>
<tr>
<td>Average power</td>
<td>0-10 kW</td>
</tr>
<tr>
<td>Peak power</td>
<td>0-147 kW</td>
</tr>
<tr>
<td>Voltage</td>
<td>350-1000 V</td>
</tr>
<tr>
<td>Current</td>
<td>10-550 A</td>
</tr>
<tr>
<td>Pulse length</td>
<td>500 µs-1 ms</td>
</tr>
<tr>
<td>Frequency</td>
<td>1-400 Hz</td>
</tr>
<tr>
<td>Duty cycle</td>
<td>Up to 28%</td>
</tr>
</tbody>
</table>

Figure 2.12(a) is the photograph of the Zpulser MPP power supply power supply in this work and Figure 2.12(b) is the Voltage-Current waveforms from this power supply on 4” conventional TORUS at 10 mTorr with an aluminum target. The pulsing parameters range for this power supply is given in Table 2.4. In the Cyprium IV model, the pulse voltage is quickly decreased in each micro-pulse “OFF” period. The idea behind this feature is that if the voltage can quickly fall to zero, the ions of the sputtered species that were generated during the pulse “ON” time can be extracted more efficiently from the cathode side and transported to the substrate, thereby aiding in increasing the deposition rates. It should be noted that in this power supply, the output voltage for each micro-pulse cannot be controlled directly, they vary with the pulse “ON” and pulse “OFF” timings. The voltage will increase as pulse “ON” increases and the voltage will decrease as the pulse “OFF” time decreases[96].

39
2.5 Plasma Diagnostic Tools

A number of plasma diagnostic tools were used in this experiment and each of them is described in detail in this section.

2.5.1 Quartz Crystal Microbalance (QCM)

QCM is a simple, ultrasensitive\cite{97}, cost effective, high resolution mass sensing technique that is based on piezoelectric effect\cite{98}. QCM consists of a thin disk of single crystal quartz with metal electrodes deposited on each side of the disk. The QCM crystal is made to oscillate at its resonant frequency by connecting to an external oscillator. Whenever there is a mass change in the crystal, the electrodes induces a frequency shift which is related to the mass change\cite{99}. The quartz crystal oscillates at its resonance frequency which a function of the thickness and the atomics mass of the material deposited on to it. QCM are capable of measuring thickness of less than a single atomic layer with great accuracy. QCM was used to monitor deposition rates in this work. Figure 2.13 is the photograph of the QCM that was used in this work.

![Figure 2.13](image)

Figure 2.13: Photograph of the QCM set-up used in this work.

Dual QCM set-up was used to increase the accuracy of the deposition rates. The QCM set-up was water cooled and one of the QCM was masked to subtract the noise from thermal drift. The signal cables from the QCM were well shielded to eliminate electromagnetic noise during pulsed plasma operation. The quartz crystals were replaced
periodically to increase the accuracy in measuring deposition rates from various target material.

2.5.2 Triple Langmuir Probe (TLP)

Electron density and temperatures can be determined by Langmuir probes and optical techniques. In pulsed plasmas, the electron density and temperature changes at a fast time scale. Single Langmuir probes requires the voltage sweep to be faster time scale than plasma parameter change, hence they cannot be used to determine the time evolution of electron density and temperature in pulsed plasmas. On the other hand, optical techniques require sophisticated amplification devices and good time resolution to determine pulsed plasma parameters[100] and a collisional radiative model to interpret the results. Triple probe or TLP is a simple, easy to construct, less time consuming choice to determine the plasma parameters in rapidly changing plasma without having to sweep the voltage as in single Langmuir probes. The construction and design criterion for triple probes are well described in L. Meng et al.[101]. TLP is a well-studied [102-104] technique that consists of three tungsten wires/probes which are inserted in to the plasma. One of the probes is made to float and other two probes are biased using a battery pack as shown in Figure 2.14(a). The three probe tips are constructed in such a way that they are spatially close together to reduce error in measurements. According to the schematic shown in Figure 2.14(a), the probe tip 1 is biased positive (\(V_1\)) with respect to the floating potential (\(V_f\)), probe tip 2 is floating (\(V_f\)) and the probe tip 3 is biased negative (\(V_3\)) to with respect to the floating potential. Figure 2.14(b) shows the probe voltages with respect to the single Langmuir probe IV (Current-Voltage) trace.
TLP technique assumes Maxwellian distribution for electrons, no interaction between probe tips, negligible contribution from magnetic field, thin and collisionless sheath for current collection[100]. The electron temperatures and densities can be calculated from the following equations[106]

\[
\frac{1}{2} = 1 - \frac{\exp \left(-\frac{eV_{12}}{kT_e}\right)}{\exp \left(-\frac{eV_{13}}{kT_e}\right)} \\
\]

\[i_{ion}^{sat} = \frac{i_3}{1 - \exp \left(-\frac{eV_{23}}{kT_e}\right)} \\
\]

\[n_e(t) = \frac{i_{ion}^{sat}(t)}{0.61eA_x} \sqrt{\frac{M}{T_e(t)}}
\]

where \(e\) is the electronic charge, \(A\) is the area of the probe tip in m\(^2\), \(V_{12}\) is the voltage difference between tips 1 and 2, \(V_{13}\) is the voltage difference between tips 1 and 3, \(M\) is the mass of the ion in kg and \(I_{sat}\) is the ion saturation current which can be measured from the current collected by the probe tip 3.

The battery voltage is usually set between 28-50V to avoid arcing between the probes and also the TLP theory requires the applied battery voltage \((V_{13})\) to be several times the
electron temperature[106]. The value of the resistor in the TLP circuit is varied according to the plasma conditions to get a reasonable voltage drop. Tektronix P5200 (50:1 attenuation) differential probes were used to measure the voltage drop across the probes in the TLP circuit. Common mode rejection in high frequency was achieved by using differential probes and battery as opposed to using power supply[100]. The differential probes and oscilloscope were powered with an uninterrupted power supply (UPS) to avoid the interference due to ground loops. The TLP was mounted on a linear motion feedthrough so the position can be varied if needed.

The TLP collects only the high energy portion of electrons, which can lead to overestimation of electron temperature as the TLP theory is based on Maxwellian electron energy distribution. Several researchers have observed Druyvesteyn, bi-Maxwellian and Maxwellian electron distributions in HiPIMS plasma [37, 106] due to high plasma density that leads to frequent ionization and cumbolic collisions. Due to huge high-energy electron population during the initial stage of the discharge, there can be a substantial error in determining electron temperatures during this time. The electron energy distribution function during the latter part pulse is found to be Maxwellian-like[42], hence the TLP data for the first 10-25μs of the pulses is not relevant for this work and only the latter portion of the pulses are considered. Figure 2.15(a) shows the time evolution of electron density and temperature from the 4” conventional TORUS with an aluminum target at 5mTorr, 700V, 150μs, 100Hz HiPIMS pulsing conditions.
2.5.3 Gridded Energy Analyzer (GEA)-QCM Set-Up

K.M.Green et al.[107] had demonstrated ionization fraction measurement using a GEA-QCM set-up in ionized magnetron sputtering systems. In this work, GEA-QCM set-up was used to measure the metal ion fraction in DC and HiPIMS plasmas. The construction and design criterion is well described in [107-109]. Figure 2.16(a) is the schematic of the GEA-QCM set-up that was used in this work. The GEA set-up consists of three stainless steel meshes with transparencies chosen according to L.Meng et al. [108]. The meshes are separated by ceramic rings to avoid any connection between them. The transparency of top mesh is chosen in such a way that it prevents plasma penetration in to the set-up. Usually the top mesh is made to float to avoid any disturbance to the plasma. Since the top mesh floats, it prevents the electrons from penetrating in to the set-up. The middle grid which is called the electron repeller grid is always set to -50V to repel the high energy electrons and accelerate the ions. The bias on the bottom grid or the ion discriminator grid is scanned from 70V to -130V to repel or allow ions. When the bottom grid is biased negative (-90V), the QCM flux consists of metal atoms (M⁰) and metal ions (M⁺), where as if the bottom grid is biased positive (+90V), the QCM flux consist of only...
M\(^0\). By knowing the difference in the deposition flux at the QCM when the bottom grid is biased positive or negative, the ion flux can be estimated. Metal ion fraction in the total deposition flux is the ion flux at QCM divided by the total flux at the QCM. In this analysis, we assume that the QCM flux consist only of metal species and not the inert gas species. By collecting the current from the bottom grid, the argon ion (Ar\(^+\)) to M\(^+\) ratio can be determined. The deposition flux that reaches the bottom of the diagnostic can be determined empirically by introducing the geometric factor (G) which is a function of pressure\([107]\). The G factor is applied only for neutrals because the ions are accelerated to near normal velocity distribution due to the sheath, hence the G factor for ions is 1. The shadowing effect of GEA-QCM set-up used in this work was considered by introducing the G factor that was calculated based on K.M.Green at al.\([107]\). The G factor for our set-up was calculated to be 0.71 from equation 2.4. The limits of the integrals were calculated based on the target to set-up distance and the set-up dimensions. Equations 2.5-2.8 were used to calculate the metal ion fraction. Tg is the transparency of the grids in the set-up.

\[
\frac{\int_{0}^{25.47^\circ} \sin 2\theta \, d\theta}{\int_{0}^{31.30^\circ} \sin 2\theta \, d\theta} = 0.71
\]

2.4

\[
\varphi_{i\omega n_{QCM}} = \varphi_{tot_{QCM}} - \varphi_{neutral_{QCM}}
\]

2.5

\[
\varphi_{neutral_{QCM}} = \varphi_{neutral_{plasma}}(G)(T_g^3)
\]

2.6

\[
\varphi_{ion_{QCM}} = \varphi_{ion_{plasma}}(T_g^3)
\]

2.7

\[
IF = \frac{\varphi_{ion_{plasma}}}{\varphi_{ion_{plasma}} + \varphi_{neutral_{plasma}}} = \frac{\varphi_{tot_{QCM}} - \varphi_{neutral_{QCM}}}{\frac{\varphi_{tot_{QCM}} - \varphi_{neutral_{QCM}} + \varphi_{neutral_{QCM}}}{G}}
\]

2.8
The noise from the plasma and other electrical equipment were eliminated by using shielded QCM and grid bias cables. The interference due to microwaves during magnetron operation was discarded by placing a microwave window/enclosure made out of SS 316 on top of the GEA-QCM set-up. The enclosure was then wrapped with aluminum foil and copper tape to form a faraday shield to block out electric fields during pulsed plasma operation. Figure 2.16(b) is the photograph of the complete GEA-QCM assembly with the Faraday shield and Figure 2.16(c) shows the location of this set-up with respect to the magnetron target. This set-up was consistently placed at 4” away from the magnetron target for all the experiments.
2.5.4 Intensified Charge-Coupled Device (ICCD) Camera

ICCD camera consists of an intensifier and a CCD camera. The intensifier helps to detect up to a single photon with very fast shutter speeds. The intensifier unit of the ICCD camera collects the photons and converts them to electrons, which is then multiplied and reconverted to photons for CCD to detect. Princeton Instruments PI MAX-4 gated ICCD camera with an f=70mm Nikon lens was used for this work. This camera contains a 1 Megapixel (1024x1024) array of detectors and each of it is a square having a 12.8μm edge length. A custom external trigger circuit was built to trigger the camera based on the start of every HiPIMS voltage pulse. The ICCD camera was triggered once per pulse and the trigger time was every 20μs from the start of the consecutive HiPIMS voltage pulse. For example, for a 500μs HiPIMS voltage pulse, the ICCD camera was triggered every 20, 40, 60…480μs from the start of consecutive HiPIMS voltage pulse. Illustration of how the camera was triggered is shown in Figure 2.17. The gate width is varied between 3, 10 and 100ns. Figure 2.18 is the schematic of the ICCD camera set-up.
Figure 2.17: Illustration of how the ICCD camera was triggered with respect to the HiPIMS voltage pulse using the external trigger circuit.

Figure 2.18: Schematic of the ICCD camera set-up.

Figure 2.19: (a) ICCD image at 100ns gate width, 70μs gate delay from the conventional pack, 0.125” aluminum target at 20mTorr, 500V, 75μs, 300Hz HiPIMS pulsing parameters, (b) Voltage-Current waveform corresponding to the ICCD image on (a).

Figure 2.19(a) is the ICCD image taken at 100ns gate width, 70μs from the start of the HiPIMS pulse from the conventional pack, 0.125” thick aluminum target at 20mTorr, 500V, 75μs, 300Hz at 500W average power and Figure 2.19(b) is the Voltage-Current
waveform corresponding to the ICCD imaging condition shown in Figure 2.19(a). Ringing in the voltage and current wave form is the artifact of the monitoring setup.

2.5.5 Optical Emission Spectroscopy (OES)

Ocean optics Plascalc-2000-UV-VIS-NIR spectrometer along with a lens system was used in this work to obtain the optical emission spectrum of the magnetron discharges. The quartz view port in front of the magnetron was covered with a Mylar sheet to avoid deposition on to the quart window. A special lens was placed outside the chamber to focus the light collected from the window to the spectrometer unit via optical fiber cable. This spectrometer unit was connected to a computer, where the live spectrum was obtained. The OES measurements obtained using this method cannot be used for quantitative analysis as the optical transmission through the Mylar sheet changes with the amount of deposition on it. This diagnostic was used in this work to give qualitative information on HiPIMS discharges from different magnet packs. Figure 2.20 shows the schematic of the OES system used in this work and Figure 2.21 is the optical emission spectra obtained during HiPIMS operation of aluminum with conventional pack at 10mTorr and 500W average power.
Figure 2.20: Schematic of the OES set-up.

Figure 2.21: Spectra from OES for conventional pack with Huettinger HiPIMS power supply at 10mTorr and 500W average power. The line at ~396nm corresponds to optical emission from aluminum atom and the lines from 700-850nm correspond to optical emission from argon atom.
Chapter 3 Epsilon Magnet Pack Design and Testing

Increasing the deposition rates in HiPIMS through optimization of magnetic field configuration of the magnetrons started with a series of experiments at Center for Plasma-Material Interaction (CPMI) on a 14” magnetron with an adjustable magnet pack. It involved testing the idea of increasing the deposition rates by producing a magnetic field which would allow ions to leave the ionization region and move towards the substrate without being recycled.

3.1 Initial Results from 14” Galaxy Magnetron

MRC Galaxy 14” planar magnetron at CPMI has an adjustable magnet pack (Figure 3.1 (a), using which, magnetic field configurations of desired race track pattern, strengths and degree of unbalancing can be obtained[89]. A series of magnetic field configurations were tested and it was found that the “spiral design” (Figure 3.1 (b) and (c)) magnetic field configuration was able to generate a high pulse current, achieve a downstream plasma with superior uniformity, and yield a better target utilization even without the assistance of magnet rotation[89].

![Figure 3.1: (a) Galaxy’s adjustable magnet pack, (b) Arrangements of magnets in the 14” magnet pack, (c) B/ on the target surface simulated in Comsol Multiphysics[105].](image)

51
3.2 Spiral Design for 4’’ TORUS

Kurt J Lesker Company’s 4’’ TORUS is one industry’s best selling magnetron sputtering guns. Hence, the 14’’ spiral design was scaled proportionally in size to fit into Kurt J. Lesker Company’s 4’’ TORUS magnetron sputter source so superior film quality can be achieved through HiPIMS without compromising on the deposition rates.

The 4’’ spiral magnet pack is the scaled down version of the 14’’ spiral pack described in H. Yu et al. [89]. The spiral design magnet pack assembly was manufactured by Dexter Magnetic Technologies, Inc. Figure 3.2(a) is the simulated $B_// \parallel$ of the 4’’ spiral design, Figure 3.2(b) is the streamline plot of $B_x$ and $B_z$ components.

Experiments on the spiral magnet pack with a 0.25’’ thick aluminum target showed that the discharge current density of this magnet pack saturates at about 18mA/cm$^2$ as shown in Figure 3.3. The discharge does not get in to the high current mode even at voltages above 1500V and also the discharge cannot be ignited at pressures lower than 40mTorr. It
is obvious that the “spiral” pack is not operating in the magnetron mode [110] when scaled down to a smaller size, and also the HiPIMS high current mode cannot be achieved. The reason for this current limit is electron leak from the racetrack. Figure 3.3 (c) is photograph of the plasma during spiral magnet pack in dcMS operation.

Figure 3.3: (a) IV characteristic of the Spiral magnet pack at 40mTorr. The target current density is obtained by dividing the discharge current by the total ‘race track’ area, (b) Target erosion pattern on a 0.25” aluminum target from the spiral pack, (c) Plasma during spiral pack in dcMS operation.
The electron trajectories in this magnet pack were simulated in COMSOL Multiphysics as described in the earlier chapter. It can be observed from Figure 3.4(b) that the magnetic field lines near the target surface are open, unlike in a conventional magnet pack. It can be seen from Figure 3.4(b) and (c) that the sharp magnetic field gradients (greater than 50G/cm) and low magnetic field regions (below 200G) lead to a situation, where no more than 5% of all electrons are efficiently recycled inside the race-track.

Although the spiral design on the 14” magnet pack gave a high discharge current as discussed in H.Yu et al. [89], this design does not work for the 4” sputter guns. A possible explanation behind the working of the 14” spiral pack is that the length of the 14” target racetrack is long enough such that a secondary electron has at least one collision with neutrals before it leaks out of the racetrack. This is not true in the 4”
design. Furthermore, the importance of the potential distribution, and balance between ion leak and electron trapping was not anticipated in H.Yu et al. [89]. Higher gradients in the field along the pack, and higher radii of curvature at the bends also provide more opportunities for the electron to leak.

To summarize, the magnetic field simulation and experimental results from the direct scaling of 14” spiral pack down to 4”, provide several design guidelines:

- Sharp turn on the electrons path will cause intense leak. Recent studies show that high density plasma spikes [71, 76] that are observed in HPPMS discharges might not be able to make it through a turn radius of more than 4mm.

- The various magnet pack designs simulated in COMSOL, show that gradients of magnetic field along race track path should not be steeper than 50G/cm to ensure sufficient electron confinement

In order to overcome intense electron leak problem, a new “ε” design was developed based on the above guidelines.

### 3.3 Epsilon (ε) Design for 4” TORUS

The surface magnetic field above target in the “ε” magnet pack configuration is shown in Figure 3.5(a) and Figure 3.5(b) shows the $B_x$ and $B_z$ components in the XZ plane cut section along AA’. The racetrack consists of an outer circular area with a uniform magnetic field and an inner “Epsilon” shaped area with lower magnetic flux density, less sharp $B_{||}$ gradients than the spiral pack but similar sharp turns as in the spiral design. The magnetic field on the outer racetrack is about 400G and the inner race track is between
275G and 350G. The outer racetrack provides stable ignition at lower pressures and constant electron feed into the inner racetrack. The inner racetrack has a specially designed magnetic field line profile which sustains an electron leak from the central portion. The lower $B_{\|}$ in the inner race track and open field lines provide higher ion flux along the axis of the magnetron in the direction away from the target. Although “ε” pack has regions where electrons leak, it can achieve a sustainable high current pulsed discharge regime and set-up a potential distribution which allows more ions to escape in comparison to the conventional magnet pack. It should be noted that the fields in the conventional magnet pack are very similar in magnitude. The highest values of magnetic fields are 400G and over the bulk of the race track, the values are 275 to 350 Gauss.

Figure 3.5: (a) $B_{\|}$ on the target surface of the “ε” magnet pack, the black line AA’ shows the location of the cut section (b) Streamline plot of $B_x$ and $B_z$ components in the XZ plane cut section along AA’, the red lines indicate the $B_x$ and $B_z$ components.
Confinement and electron loss paths of this magnet pack can be observed in Figure 3.6 (b) and (c). This pack sustains discharges with volt-ampere characteristics that follow a well-known magnetron mode $I \propto V^n$ relationship, where “$n$” is the performance index of the electron trap[110] as shown in Figure 3.7.
Figure 3.7 is the plot of discharge voltage versus current density. Current density here represents the ratio of the peak discharge current divided by the total race track area specific to the magnet pack. Conventional pack and “ε” pack follow the magnetron mode whereas the 4” spiral pack doesn’t follow this mode.

The deposition rates from the “ε” pack were measured using a QCM. To verify the QCM deposition rate measurement accuracy during HPPMS discharge operation, a half masked test wafer was placed in the actual QCM location. The deposited film thickness on the test wafer was measured using a DEKTAK 3030 surface profilometer and it was compared to the QCM thickness readings. The difference in the measurement between the two techniques was found to be less than 2%. Aluminum targets were used in the “ε” pack experiments. For the “ε” magnet pack testing, a graded-thickness aluminum target was used. The outer rim of the graded aluminum target was 0.125” thick and the inner portion was 0.09” thick. This design was chosen to sustain a stable HiPIMS discharge.
with the pre-manufactured “ε” pack magnet assembly. Slightly stronger magnets in the inner “ε” portion of the epsilon pack would eliminate the need to use a graded target. On the conventional magnet pack, 0.125” thick aluminum targets were used.

Figure 3.8: Summary of QCM deposition rates for both the magnet packs with different power supplies at 10mTorr measured at the QCM.

In Figure 3.8, solid legends represent conventional pack and hollow legends represent “ε” pack. It can be seen that the “ε” pack Huettinger HiPIMS power supply gives higher deposition rates than MPP Zpulser power supply and also it gives twice higher deposition rates than conventional pack HiPIMS discharges for the same average power. In all these experiments, Huettinger HiPIMS power supply was operated at 200Hz, 1.5% duty cycle, Zpulser MPP supply was operated at 100 Hz, 10% duty cycle and AE pulsed DC power supply was operated at 5 kHz, 97.5% duty cycle. Due to asymmetry of the “ε” magnet pack, the deposition rates were measured for different orientations of the pack. This was
done by rotating the magnetron gun every 90 degrees along the central axis and recording the deposition rates from the QCM. The deposition rates from the four different orientations were found to be the same, within 1% or less. Deposition rates beyond 500W are not presented for the conventional pack with Huettinger HiPIMS power supply due to the melting of the target. It can also be seen from Figure 3.8 that the “ε” pack Huettinger HiPIMS deposition rates reach up to almost 80% of conventional DC sputtering at an average power of 400W.

![Figure 3.9: (a) Schematic of the 4” silicon substrate location with respect to the target, (b) Location of the uniformity test on the 4” silicon substrate, (c) Plasma during “ε” pack in operation.](image)

In order to test the uniformity of deposition, 4” silicon wafers were placed 4” coaxially away from the target as shown in Figure 3.9 (a). The thickness of the deposited films was
measured using X-ray reflectivity (XRR) technique. A PANalytical MRD X’pert x-ray diffraction system was used for this purpose. The deposited film thickness was measured for “ε” pack Huettinger HiPIMS, conventional pack HiPIMS and conventional pack DC at 10mTorr and 500W average power at the locations shown in Figure 3.9(b). The overall uniformity of the “ε” pack was similar to that of the conventional pack which can be observed from Table 3.1. Table 3.1 shows the depositions rates measured using XRR technique at five different locations on the 4” wafer as shown in Figure 3.9(b). Table 3.2 is the average deposition rates from “ε” and conventional magnet pack for Huettinger HiPIMS and DC power supplies at 10mTorr and 500W average power from different locations on the 4” wafer during uniformity experiments. It can be observed from Table 3.2, that the average deposition rates for all positions with “ε” pack Huettinger HiPIMS was 8.8±0.4 Å/s, with conventional pack HiPIMS was 4.1±0.2 Å/s and with conventional pack DC was 12.1±0.6 Å/s. The “ε” pack Huettinger HiPIMS gives about twice higher deposition rate than conventional pack with HiPIMS discharges even when the substrate is placed line of sight to the target. These data points agree well with the data obtained by QCM earlier.

Table 3.1: Deposition rates from “ε” and conventional magnet pack for Huettinger HiPIMS and DC power supplies at 10mTorr and 500W average power at locations indicated in Figure 3.9(b) during wafer uniformity experiments.

<table>
<thead>
<tr>
<th>Position</th>
<th>DC Deposition Rates (Å/s)</th>
<th>HiPIMS Deposition Rates (Å/s)</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>Conventional</td>
<td>“ε”</td>
</tr>
<tr>
<td>1</td>
<td>12.5±0.6</td>
<td>--</td>
</tr>
<tr>
<td>2</td>
<td>12.3±0.6</td>
<td>--</td>
</tr>
<tr>
<td>3</td>
<td>11.6±0.6</td>
<td>--</td>
</tr>
<tr>
<td>4</td>
<td>--</td>
<td>--</td>
</tr>
<tr>
<td>5</td>
<td>--</td>
<td>--</td>
</tr>
</tbody>
</table>
Table 3.2: Average deposition rates from “ε” and conventional magnet pack for Huettinger HiPIMS and DC power supplies at 10mTorr and 500W average power during wafer uniformity experiments.

<table>
<thead>
<tr>
<th>Magnet Pack</th>
<th>DC Deposition Rate (Å/s)</th>
<th>HiPIMS Deposition Rate (Å/s)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Conventional</td>
<td>12.1±0.5</td>
<td>4.1±0.2</td>
</tr>
<tr>
<td>“ε”</td>
<td></td>
<td>8.8±0.5</td>
</tr>
</tbody>
</table>

The total racetrack length for the conventional pack is 9.35” and 18.7” is for the “ε” pack (Figure 3.10 (c)). However, this difference in race track length does not explain the higher deposition of the “ε” pack. The total erosion area for the conventional pack is 10.2 in² and 9.1 in² for the “ε” pack since the conventional pack has a very broad erosion groove. The peak current at an average power of 500W with the conventional pack was ~95A and ~78A with the “ε” pack. Therefore the current density was 1400mA/cm² for the conventional pack and 1300mA/cm² for the “ε” pack. These values are very close. Since the current density and currents are similar, the reason for the significantly higher deposition rate must be due to confinement and return of sputtered material, not the generation of sputtered material.
Since the race track area was approximately the same for both magnet packs, one would expect the similar peak currents but it can be observed from Figure 3.11 (a,b,c and d) that in the case of “ε” pack, the peak current is lower than that of the conventional pack with Huettinger HiPIMS and Zpulser MPP supplies. These IV Oscillogram (Figure 3.11) trends clearly points to plasma mechanics behind deposition rate increase. Peak currents are lower because not all of the ionized metal atoms are recycled back to the target. It can be observed from Figure 3.11 (c and d) that Zpulser MPP generates 1ms long pulse (macropulse) that is composed of a train of 20-30µs long micropulses.
Figure 3.11: VI Oscillograms from (a) Conventional pack Huettinger HiPIMS power supply (b) “ε” pack Huettinger HiPIMS power supply, (c) Conventional pack Zpulser MPP supply, (d) “ε” pack Zpulser MPP supply, (e) Conventional pack AE pulsed DC power supply, (f) “ε” pack AE pulsed DC power supply.
Figure 3.12: (a) SEM image of conventional pack aluminum film deposited using DC power supply, (b) SEM Image of conventional pack aluminum film deposited using Huettiger HiPIMS power supply and (c) SEM Image of “ε” pack aluminum film deposited using Huettiger HiPIMS power supply.
The deposited aluminum films were characterized using Hitachi S-4800 SEM (Scanning Electron Microscope). Films grown using “ε” magnet pack with Huettinger HiPIMS power supply under the same conditions as conventional pack show much smaller grain structure and can be observed from Figure 3.12 (b) and (c).

To summarize,

1. $B_{\parallel}$ in the middle of the race track for conventional and “ε” magnet packs are nearly identical. It is about 400G to 300G for “ε” and about 300G for the conventional pack.

2. The peak current in the “ε” pack is only 10% lower than the peak current in the conventional pack during a single HPPMS discharge.

3. The IV Oscillograms traces do not show any anomalous differences in the case of Huettinger HiPIMS power supply (Figure 3.11 (a) and (b)). But a different trend is observed in Zpulser power supply which is seen Figure 3.11 (c and d).

4. The film microstructure with Huettinger HiPIMS power supply is very different for “ε” and conventional pack. Films grown using “ε” magnet pack under the same conditions as conventional pack (pressure and gas) show much smaller grain structure which can be observed from Figure 3.12 (b) and (c).

These experimental observations point to the measured increase in deposition rate being due to differences in the plasma transport properties of “ε” and conventional magnetic field configurations. Since the race track area was approximately the same for both magnet packs, one would expect the similar peak currents but it can be observed from Figure 3.11 (a, b, c and d) that in the case of “ε” pack, the peak current is lower than that of the conventional pack although the voltage is slightly higher. This means
approximately the same amount of sputtered material is supplied to the plasma region in case of “ε” pack. However fewer ions are returned to the target. The difference between the Huettinger HiPIMS results and the Zpulser MPP results are consistent with the following observation. There is a change in slope at 20µs in Figure 3.11 (a) and (b). It is believed that this is the point where metal self-sputtering becomes dominant and the HiPIMS mode is realized[105]. At this point the current is 50A, but then rises to nearly 100 A. Each of the independent MPP pulses lasts only 20- 25µs, and follows the same initial slope and shape as the first 20 µs of the HiPIMS pulse. Indeed they only reach 50A. Since the peak current is not as high in the MPP discharges as it is in the HiPIMS discharges, not as much material is sputtered. This can explain the lower deposition rates for MPP. Since similar amounts of material are supplied to the plasma region but more material is delivered to the substrate, it must be the case that more material is transported across the field towards the substrate. The efficiency of the ionized aluminum trap is poorer. The difference in the microstructure of the films grown using “ε” pack and conventional pack (Figure 3.12 (b) and (c)) clearly indicates that the deposition flux consisted of more ions, though it is not a solid proof, this is the most probable explanation. Based on the assumption that the material flux consist more of ions, than even in conventional pack HPPMS discharge we can assume that more ions escape from the plasma region. This higher ion flux has to be compensated by electrons following ions otherwise trapped by magnetic field electrons would suppress flux dramatically. The higher electron flux can be only due to magnetic field topology which implements ‘‘opened field lines’’ allowing electrons to escape plasma region. On the other hand the trap has much sharper magnetic field gradients along ‘‘z’’ direction which also may
impact the increased ion flux toward the substrate. It should be noted that the difference in performance between the conventional and “ε” pack is not due to lowering the magnetic field magnitudes. It differs from an unbalanced magnetron configuration where there is intentional confinement of the electrons, but at larger distances from the surface of the target, the electric field in the pre-sheath is smaller.

In order to understand the reason behind the higher deposition rates, ICCD camera diagnostic was used to investigate the HiPIMS plasma in the “ε” pack and conventional pack. Figure 3.13 and Figure 3.14 are the ICCD images taken at different pressures (10mTorr and 20mTorr argon) and different average powers (250W and 500W) with 100ns gate width and 70µs from the start of the HiPIMS voltage pulse. The scale bar on these images shows the relative intensities, where blue color represents the lowest intensity and red color represents the highest intensity. All these measurements were done through a quartz window port that was covered with a Mylar sheet. Since the optical transmission of the Mylar sheet changes with the deposition, the actual intensities do not have any significance. Hence, the images are represented with relative intensities. Although these ICCD images cannot be used for any quantitative analysis, they can give a lot of information on the formation of ‘ionization zones’/plasma spokes in the discharge. It can be observed from Figure 3.13 that there is distinct plasma spoke formation in the case of conventional magnet pack but in the “ε” pack, the HiPIMS plasma looks homogenous at 100ns gate width. With the increase of HiPIMS average power (500W), it can be observed that the more than two ionization zones appear in the conventional pack and the number of zones increases with the pressure whereas, in the
case of “ε” pack, the plasma looks uniform with no zone formation like the conventional pack (Figure 3.14).

Figure 3.13: (a) ICCD images of zones from conventional pack, aluminum target at 10mTorr, 250W HiPIMS average power taken at 100ns gate width and 70μs from the start of the HiPIMS pulse, (b) ICCD images of zones from “ε” pack, aluminum target at 10mTorr, 250W HiPIMS average power taken at 100ns gate width and 70μs from the start of the HiPIMS pulse, (c) ICCD images of zones from conventional pack, aluminum target at 20mTorr, 250W HiPIMS average power taken at 100ns gate width and 70μs from the start of the HiPIMS pulse, (d) ICCD images of zones from “ε” pack, aluminum target at 20mTorr, 250W HiPIMS average power taken at 100ns gate width and 70μs from the start of the HiPIMS pulse.

Figure 3.14: (a) ICCD images of zones from conventional pack, aluminum target at 10mTorr, 500W HiPIMS average power taken at 100ns gate width and 70μs from the start of the HiPIMS pulse, (b) ICCD images of zones from “ε” pack, aluminum target at 10mTorr, 500W HiPIMS average power taken at 100ns gate width and 70μs from the start of the HiPIMS pulse, (c) ICCD images of zones from conventional pack, aluminum target at 20mTorr, 500W HiPIMS average power taken at 100ns gate width and 70μs from the start of the HiPIMS pulse, (d) ICCD images of zones from “ε” pack, aluminum target at 20mTorr, 500W HiPIMS average power taken at 100ns gate width and 70μs from the start of the HiPIMS pulse.
Figure 3.15: (a) ICCD images of zones from conventional pack, aluminum target at 10mTorr, 250W HiPIMS average power taken at 10ns gate width and 70μs from the start of the HiPIMS pulse, (b) ICCD images of zones from “ε” pack, aluminum target at 10mTorr, 250W HiPIMS average power taken at 10ns gate width and 70μs from the start of the HiPIMS pulse, (c) ICCD images of zones from conventional pack, aluminum target at 20mTorr, 250W HiPIMS average power taken at 10ns gate width and 70μs from the start of the HiPIMS pulse, (d) ICCD images of zones from “ε” pack, aluminum target at 20mTorr, 250W HiPIMS average power taken at 10ns gate width and 70μs from the start of the HiPIMS pulse.

Figure 3.16: (a) ICCD images of zones from conventional pack, aluminum target at 10mTorr, 500W HiPIMS average power taken at 10ns gate width and 70μs from the start of the HiPIMS pulse, (b) ICCD images of zones from “ε” pack, aluminum target at 10mTorr, 500W HiPIMS average power taken at 10ns gate width and 70μs from the start of the HiPIMS pulse, (c) ICCD images of zones from conventional pack, aluminum target at 20mTorr, 500W HiPIMS average power taken at 10ns gate width and 70μs from the start of the HiPIMS pulse, (d) ICCD images of zones from “ε” pack, aluminum target at 20mTorr, 500W HiPIMS average power taken at 10ns gate width and 70μs from the start of the HiPIMS pulse.
The inner epsilon region of the “ε” pack looks dim in all the images because, the optical signal collected by the ICCD camera from that region is very low compared to the outer ring. This means, the plasma intensity in that region is low. Since, no ionization zones were detected in the “ε” pack, there were concerns if the zones were moving faster and that 100ns gate width was not enough to detect them. Hence, the imaging (Figure 3.15 and Figure 3.16) were done at a gate width of 10ns. Even at a gate width of 10ns, no ‘ionization zones’ were observed in the “ε” pack even though the peak current in this pack is only 10% lower than the peak current in the conventional pack during a single HiPIMS pulse indicating a lot of hidden physics behind this observation of homogenous plasma without any appearance of ‘ionization zones’ in the “ε” pack.

Time resolved electron density and temperatures measurements for the “ε” pack was carried out using a TLP. The TLP was mounted on a motion feedthrough, so the probe can be moved in “z” and “θ” directions in front of the target. Probe measurements were taken for different “z” and “θ” directions on the “ε” pack with zPulser MPP power supply at an average power of 500W. The “θ” direction measurements were then mapped in (X, Y) coordinates for visualization (Figure 3.17). Figure 3.17 (b and c) are the plots of peak electron densities and temperatures at different locations (“θ”) on the target corresponding to different lengths (“z”) from the target. Z=90mm represents the measurements at the substrate level as the substrate is usually placed at 100mm from the target. Figure 3.17 (d) shows the time evolution of electron density, temperature and discharge current at 5mTorr, 500W average power taken at (89, 65), which corresponds to the co-ordinates shown in
Figure 3.16(a). The peak electron density and temperature at Z=40mm from the target is around $1-1.4 \times 10^{18} \text{m}^{-3}$ and 3-5eV respectively.

Figure 3.17: (a) Rough projection of “[]” direction measurements on the target for visualization, (b) Electron density at different locations for different “z” @ 15mTorr, 500W average power using Zpulser power supply, (c) Electron temperature at different locations for different “z” @ 15mTorr, 500W using Zpulser power supply, (d) Time evolution of ne, Te and discharge current at z=40mm, 15mTorr, 500W average power using Zpulser power supply at co-ordinates (89,65) corresponding to Figure 3.16(a).
3.4 Summary

The “ε” magnet pack gives higher deposition rates in HiPIMS compared to conventional pack HiPIMS. The increased deposition rates in “ε” magnet pack can be observed in MPP discharges too. The ε” magnet pack follows the magnetron mode of discharge unlike the “Spiral” magnet pack which could not be operated in high current mode. The “Spiral” magnet pack that worked in the 14” magnetron did not work on the 4” magnetron gun indicating that direct scaling of magnets does not work in certain magnetic field configurations. The total racetrack length of the “ε” magnet pack is higher than the conventional pack but the total erosion area is smaller than the conventional pack indicating that the higher deposition rates are not due to increasing in the erosion area but rather due to difference in plasma dynamics. The films that were deposited from “ε” magnet pack in HiPIMS discharge have much smaller grains compared to the films deposited using conventional pack DC and HiPIMS discharges. Although the “ε” magnet pack gives higher deposition rates and better film quality, the substrate uniformity is relatively poor compared to the conventional magnet pack. In order to keep the deposition rates high but at the same time improve the deposition uniformity without the need for substrate rotation, a cylindrically symmetric “TriPack” magnet pack was developed based on the magnetic field design solutions from “ε” magnet pack. The design and testing of the “TriPack” is discussed in detail in the next chapter.
Chapter 4 TriPack Magnet Pack Design and Testing

The magnetic fields in the “ε” magnet pack discussed in the previous chapter are asymmetric. Therefore, a new cylindrically symmetric TriPack magnet pack was developed to create better substrate uniformity. In this chapter the design and testing of TriPack magnet pack is discussed in detail.

4.1 TriPack Design

The TriPack design consists for three counter rotating race tracks (inner, middle and outer) meaning, the drift currents run in opposite directions in the three race tracks 4.2(a)). The inner and outer racetracks have tangential field directed (red color) in opposite direction to the middle race track (blue color). The blue color in 4.2(b) represents magnetic field opposite in direction to red color.

4.1.1 New Cooling Well Design

The TriPack magnet pack could not be placed inside the conventional magnetron gun due to space constraints. Hence, a new high power cooling well (Figure 4.1(b)) was designed to accommodate the TriPack magnet pack in the magnetron body. The conventional Kurt J Lesker Company’s TORUS cooling well (Figure 4.1(a and c) consists of three slots for water inlet, water outlet and power cable. Hence, the conventional magnet pack consisted of three small circular openings to fit inside the magnetron. With the new cooling well design, the TriPack magnet pack can be accommodated without any slots because the water is fed from the sides as shown in Figure 4.1(b and d).
Main idea behind not having slots is to avoid the space constraint, so that there more room for magnet placement. With the new cooling well design, the average power of operation can be increased from 100W/sq in to 200W/sq in. The conventional magnet pack can also be accommodated inside the high power cooling well to operate at higher powers.

4.1.2 TriPack Magnetic Field Configuration

4.2(a) is the magnetic field parallel to the TriPack target surface and 4.2(b) shows the $B_x$ and $B_z$ components for the TriPack. It can be observed from the streamline plot (4.2(b)) that this pack has magnetic zero at about 0.4” from the target surface between the inner and the outer track. How the magnetic zero affects the performance of this pack is the subject of section 6.1.
Figure 4.2: (a) B/ on the target surface of the TriPack, (b) Streamline plot of Bx and Bz components for the TriPack, (c) 2D axisymmetric electron trajectory from the TriPack at t=1000ns.

Figure 4.2(c) is the 2D axisymmetric view of the electron trajectories in the TriPack at 1000ns. It can be observed that the central region has electrons escaping from the arc trajectory which can lead to increased deposition rates.

The TriPack magnet pack was built by Starfire Industries with cylindrical magnets. Due to the dead space between the magnets in the assembly, the measured fields on the target surface was much lower that what was predicted by modeling. This lead to ignition problem with the inner and middle race tracks.

In order to overcome this problem, specially designed targets were used for TriPack testing. The targets were 0.25” thick and were machined to accommodate four concentric rings made out of magnetic material (soft iron). These rings served as magnetic field conduits that helped in obtaining the higher magnetic field values above the target surface so discharge on all race tracks can be achieved without any problem. Since these rings
were only 0.125” tall and were embedded in to the back of the target surface (Figure 4.3(b)) that is attached to the cooling well side, no iron was exposed to plasma. Also, the target was machined to look like a pre-eroded target to obtain the required magnetic field magnitudes on the target surface. Figure 4.3(a) is a 2D axisymmetric illustration of the modified TriPack target, where the grey coloured rectangles represent the iron pieces. The coloured iso-lines show the total magnitude of the magnetic field in Gauss. This should not be misinterpreted with the surface magnetic field plot shown in Figure 4.2(b) where only the radial component of magnetic field is presented. The TriPack with the modified targets will be referred to as TriPack V300 in this thesis. It is called V300 because this version contains 300 cylindrical magnets. With all these modifications, the peak radial magnetic field magnitudes on each trace were 615Gauss, 500Gauss and 410Gauss for inner, middle and outer race tracks respectively.
Figure 4.3: (a) 2D axisymmetric illustration of the modified TriPack V300 target. The colored iso-lines show the total magnitude of magnetic field in Gauss, (b) Titanium TriPack V300 target with magnetic inserts, (c) Pre-eroded target with magnetic insert slots.

4.2 TriPack V300 Testing

The new design of the magnetic field implemented in the TriPack V300 provided stable magnetron discharge ignition and operation at pressures greater than 1 mTorr. The HiPIMS discharge volt-ampere (V-I) characteristic for the TriPack follows the conventional magnetron trend. The X axis on Figure 4.4(a) represents the current density, which is the single pulse peak current divided by the target erosion area, and the Y axis represent the discharge voltage. This plot was constructed from the voltage and current traces obtained from the Huettinger power supply on a copper target, at 10mTorr argon pressure. Hollow red colour legends correspond to the TriPack V300 for an aluminium target with weaker magnetic fields. The weaker fields were obtained by removing magnetic rings from the target. The magnetic field was reduced by more than 200 Gauss
when the magnetic rings were removed, but the shape of magnetic field lines remained the same. This was confirmed from modelling in COMSOL Multiphysics. In the TriPack V300 design, argon gas was directly fed in to the race track region so the local gas density is higher near the target surface (Figure 4.4(c)) than rest of the chamber to avoid ignition issues in the pack. The indicated pressure in these experiments corresponds to the chamber pressure. Figure 4.4(b) is the photograph of plasma during TriPack V300 during HiPIMS discharge with a titanium target.

4.2.1 TriPack V300 Volt-Ampere Characteristics

![Volt-Ampere characteristics and plasma photographs](image)

Figure 4.4: (a) Volt-Ampere characteristics of the conventional pack and TriPack V300 at 10mTorr, (b) Plasma during TriPack V300 with a titanium target in HiPIMS operation.
The performance of the TriPack V300 was also tested using Starfire Impulse HiPIMS power supply. This power supply provides more “freedom” for parameter optimization during HiPIMS discharges. Its operation frequency ranges from 1 Hz to 10 kHz, can reach pulse lengths up to 1ms, and can provide up to 2 kW average powers, which is more than enough to sustain HiPIMS discharges on smaller magnetrons. Figure 4.5(a and b) are the voltage and current traces obtained from the conventional and TriPack, respectively using the Starfire Impulse power supply for an aluminium target at 13mTorr and 500W average power. In the case of conventional HiPIMS operation, the current slowly reaches 150A (Figure 4.5(a)) at the end of the pulse for pulsing parameters of 850V (discharge voltage), 220Hz (frequency) and 30µs pulse (pulse time). The current in the TriPack V300 steeply increases to only about 12A for pulsing parameters of 850V, 300Hz and pulse time of 200µs. There is a distinct difference in the shape of the discharge current trace between both the magnet packs. Such a dramatic difference can be attributed to a difference in the plasma dynamics between in this new magnetic field profile and conventional magnetic field profile.

The most dramatic difference is seen in the peak discharge currents. Although the TriPack V300 peak current is only 12A, the peak electron density and temperature were measured to be 1-6x10^{18}/m^3 and 4 (±3) eV respectively at 1” away from the titanium target surface. The electron temperatures and densities were measured using a Triple Langmuir Probe (TLP) technique and these electron densities and temperatures are typical for HiPIMS discharge.
4.2.2 TriPack V300 Deposition Rates

The deposition rates from TriPack V300 and convectional magnet pack was measured using a dual water cooled QCM set-up as described in the diagnostics section. The deposition rates from the conventional pack and TriPack were obtained at 13mTorr and 500W average power with Advanced Energy DC, Huettinger HiPIMS and Starfire Impulse HiPIMS power supplies. The pulsing parameters (discharge voltage, pulse time and frequency) were different for different target materials, magnet packs and HiPIMS power supplies (Huettinger and Starfire Impulse) but, the average power was always kept at 500W. Three different target materials, namely titanium, carbon and aluminium, were compared for deposition rates. The results from the deposition rate experiments are summarized in Figure 4.6.

The Y axis in Figure 4.6 represents the deposition rates normalized to conventional pack DC deposition rates at 500W for each material, and the X axis represents the different target materials. In the case of titanium, TriPack V300 HiPIMS deposition rates were higher than the conventional pack DC deposition rates. Titanium deposition rate measurement can be difficult because the titanium targets can get hot during long term
operation. There are several publications[111] that comment about this effect. The deposition rate experiments from the conventional and TriPack V300 magnet packs were performed with a cold titanium target to avoid all hot target effects. In order to operate with the cold titanium target, the deposition rate measurements were done for only 15 seconds with a cooling time of about 30 seconds between measurements. A series of deposition rate measurements were done for 15 seconds and compared to the average deposition rate over several experiments. The deposition rates remained the same for all the experiments. With carbon target, the TriPack V300 HiPIMS deposition rates were about the same as the conventional DC deposition rates and in the case of an aluminium target, TriPack V300 HiPIMS deposition rates were lower than conventional DC deposition rates but higher than conventional HiPIMS deposition rates. The effects of changing pulsing parameters in HiPIMS are more distinct in the TriPack V300 than in conventional pack. In Figure 4.6, the colour gradient represents the magnitude of the deposition rate variation as a function of varying pulsing parameters. It should be noted that titanium and carbon are very sensitive to pulsing parameters. Among all the parameters, only the influence of pulsing parameters on the deposition rates of copper target are shown in Figure 4.7. Copper was chosen because it gives the highest deposition rates in comparison with most other materials, and does not have the “hot target” problem seen with titanium targets. The QCM measurements were verified using mass change experiments, where the deposition rates were calculated from mass change of 2” silicon wafer before and after 1 hour deposition experiments. It can be observed from Table 4.1 that in HiPIMS discharge, TriPack V300 with an aluminium target at an average power of 250W gives higher deposition rates than conventional pack HiPIMS.
Figure 4.6: Deposition rates from conventional pack and the TriPack V300 with titanium, carbon and aluminium targets at 13mTorr normalized to the DC deposition rates with the conventional pack. All deposition rates on titanium were measured from a “cold” titanium target. The gradients on the bar plot represent the range of deposition rates that can be obtained by varying pulsing parameters, but keeping the average power at 500W. The black error bars represent the error in acquiring the QCM data during our experiments. DC deposition rates with the conventional pack were ~1Å/s for titanium, ~0.1 Å/s for carbon and ~12 Å/s for aluminium at 4” away from the target.

Table 4.1: Aluminum deposition rates measured from mass change of 2” silicon wafer before and after 1 hour deposition experiments.

<table>
<thead>
<tr>
<th>Power supply type</th>
<th>Conventional Pack Deposition rate (Å/s)</th>
<th>Tri Pack V300 Deposition rate (Å/s)</th>
</tr>
</thead>
<tbody>
<tr>
<td>DC</td>
<td>9.1±1.4</td>
<td></td>
</tr>
<tr>
<td>Starfire “Impulse” for HiPIMS</td>
<td>4.0±0.6</td>
<td>7.1±1.1</td>
</tr>
</tbody>
</table>

Higher deposition rates can be achieved with moderate frequency and pulse length.
Figure 4.7: Variation of copper HiPIMS deposition rates by pulsing parameters change in TriPack V300 at 10mTorr. The presented deposition rates around 500W were taken exactly at 500W but the data points have been offset on the power axis to display the error bars more clearly.

The data presented in Figure 4.7 makes it clear that HiPIMS discharge physics is very sophisticated. The deposition rate increased twofold simply by increasing the pulse time by a factor of 2 and decreasing the pulsing frequency, while the discharge voltage was kept constant. The pure argon pressure during these experiments was kept at 10mTorr. It should be noted that the deposition rates around 500W were taken exactly at 500W as expressed in Figure 4.7. The data points have an offset on the power axis to display the error bars more clearly. The general trend is that the lowest deposition rates were observed with a low repetition rate and longer pulses, and higher deposition rates were observed with high repetition rates and very short pulses. Highest deposition rate was observed at 650V, 150µs, 250Hz pulsing condition which is with a longer pulse and moderate frequency and it doesn’t follow the general trend. This clearly points to more sophisticated gas dynamics in the TriPack V300.
The reason for the observed change in the deposition rates when applying different pulsing parameters at the same average power may be due to the change in the local gas density during the discharge. The deposition rates did not change with the change in pulsing parameters for the conventional pack HiPIMS case, which clearly indicates that this pulsing effect on deposition rates is due to the unique TriPack V300 magnetic field configuration.

4.2.3 Conclusion from TriPack V300 Deposition Rate Measurements

To summarize, TriPack V300 gives higher deposition rates in HiPIMS than conventional pack HiPIMS. There have been several publications in the past that show that lowering the magnetic field strength leads to higher deposition rates, due to reduction in the metal ion “return effect”\[87\]. This is not the case for the TriPack V300 with the modified target, where the radial component of the magnetic field on the surface of the target is the more than the conventional pack. The race track area of the conventional pack is only 6 in\(^2\), whereas the race track area of the TriPack V300 is \(~8\) in\(^2\). The TriPack V300 race track area is 25% larger, but has much lower discharge current when compared with the conventional pack. Both these observations combined do not support the simple explanation that higher race-track area gives higher deposition rates. The observed difference in deposition rates, erosion area, voltage-current traces and pulsing parameters influence on deposition rates of TriPack V300 from conventional magnet pack indicates that the plasma dynamics of this magnet pack is very different from the conventional magnet pack. Hence, in order to understand the discharge dynamics in this pack various plasma diagnostics were performed and a comprehensive theory is presented in chapter 6.
4.3 Experimental Study of Plasma Dynamics in TriPack V300

4.3.1 ICCD Camera Diagnostics

The plasma instabilities (EXB) in the TriPack V300 were observed using an ICCD camera. Details on how the ICCD camera trigger is discussed in the diagnostics section. Table 4.2 shows the list of experimental conditions during ICCD camera operation.

<table>
<thead>
<tr>
<th>Voltage</th>
<th>Average power (W)</th>
<th>Pulse time (µs)</th>
<th>Trigger delay (µs)</th>
</tr>
</thead>
<tbody>
<tr>
<td>650</td>
<td>250</td>
<td>50,200,250,500</td>
<td>Every 20</td>
</tr>
<tr>
<td></td>
<td>500</td>
<td></td>
<td></td>
</tr>
<tr>
<td>950</td>
<td>250</td>
<td>50,200,250,500</td>
<td>Every 20</td>
</tr>
<tr>
<td></td>
<td>500</td>
<td></td>
<td></td>
</tr>
</tbody>
</table>

It can be seen from Table 4.2, that the TriPack V300 was operated at different voltages (650V and 950V), powers (250W and 500W) and pulse lengths (50,200,250 and 500µs) with a titanium target. For a certain voltage and pulse length, frequencies were regulated to get a specific average power at 20mTorr argon pressure. The ICCD images were taken every 20µs from consecutive HiPIMS voltage pulses. The scale bar on the ICCD images shows the relative intensities and for all images shown in this chapter, the gate width/exposure time was fixed at 10ns.

4.3.1.1 ICCD Study of Ignition on All the Three Race Tracks

Figure 4.8, Figure 4.9, Figure 4.10 & Figure 4.11 show the ICCD images from TriPack V300 HiPIMS discharges for different pulse widths taken at different time snaps for a specific voltage (650V) and average power (250W). In the 50µs HiPIMS pulse case
(Figure 4.8), the discharge is more intense in the inner and middle inner track compared to the outer race track at 20\(\mu\)s. At 40\(\mu\)s, the outer race track gets brighter but the intensity is much lower compared to the inner and middle race tracks. The peak current for this case was about 32A. No “ionization zones”/plasma spokes are observed. The discharge looks homogeneous on all the three race tracks.

![Figure 4.8 ICCD images of TriPack V300 titanium HiPIMS discharge with times snaps for 50\(\mu\)s, 650V, 250W and 20mTorr process conditions. For a 50\(\mu\)s HiPIMS pulse, pictures were taken at 20\(\mu\)s and 40\(\mu\)s as shown in this figure. The exposure time of these images are 10ns.](image)

For a 200\(\mu\)s HiPIMS pulse (Figure 4.9), initially the discharge on the outer race track is much less intense compared to the inner and middle race tracks. As the discharge progresses, the inner and middle race tracks get much more intense and eventually fade away during the end of the pulse whereas, the outer race track gets intense as the discharge progress but does not fade away during the end of the pulse. The peak current in this case was around 35A. The 250\(\mu\)s pulse width (Figure 4.10) case follows the same trend as the 200\(\mu\)s HiPIMS pulse.
Figure 4.9: ICCD images of TriPack V300 titanium HiPIMS discharge with times snaps for 200μs, 650V, 250W and 20mTorr process conditions. For a 200μs HiPIMS pulse, pictures were taken at 20, 40......180μs as shown in this figure. The exposure time of these images are 10ns.

Figure 4.10: ICCD images of TriPack V300 titanium HiPIMS discharge with times snaps for 250μs, 650V, 250W and 20mTorr process conditions. For a 250μs HiPIMS pulse, pictures were taken at 20, 40......240μs as shown in this figure. The exposure time of these images are 10ns.

The ICCD images shown in Figure 4.11 correspond to the parameters shown in Figure 4.12. Figure 4.12(a) shows the side view of the pre-eroded TriPack V300 target with the
slots for magnetic inserts, Figure 4.12(b) shows the Streamline plot of $B_x$ and $B_z$ components on the pre-eroded target surface, Figure 4.12(c) is the photograph of the TriPack V300 pre-eroded target, and Figure 4.12(d) is the VI oscillogram that corresponds to the ICCD images shown in Figure 4.11.

In the 500μs case, initially there is an intense discharge in the inner race track compared to the middle race track and the outer race track looks very faint. As the discharge progresses, the relative intensities change substantially. At 100μs, the middle and outer tracks looks more intense than the inner race track. As time progress, the inner and middle tracks fade away while the outer track remains bright till the end of the discharge. The plasma around the race tracks remains homogeneous without any “ionization zones”/plasma spokes and the peak current was around 35A during this discharge. The white circle in the 420μs case of Figure 4.11 indicates the location of gas inlet to this magnet pack.

Figure 4.11: ICCD images of TriPack V300 titanium HiPIMS discharge with times snaps for 500μs, 650V, 250W and 20mTorr process conditions. For a 500μs HiPIMS pulse, pictures were taken at 20, 40.....480μs as shown in this figure. The exposure time of these images are 10ns.
Figure 4.12: (a) Side view of the TriPack V300 titanium target with the peak radial magnetic field values on the three race tracks, (b) Streamline plot of Bx and Bz components on the target surface simulated in COMSOL Multiphysics, (c) Photograph of the pre-eroded titanium target, (d) VI oscillogram corresponding to Figure 4.11.

Figure 4.13: (a) Intensity obtained from each race track as a function of time, (b) VI trace with the intensities corresponding to ICCD images shown in Figure 4.11.
Figure 4.13(a) shows the time evolution of the intensities from each race track for titanium target during HiPIMS discharge at 650V, 500μs, 250W and 20mTorr. It can be seen that the peak inner and middle race track intensities are about the same, but the outer race track intensity is much smaller than the other two. The peak intensities are achieved in all the race tracks at around 60μs. The intensities from the inner and middle race tracks decays much more steeply than the outer track and during the latter part of the pulse, the intensity of the outer race track is higher than the other two. Figure 4.13 (b) shows the VI traces with the intensities from each race track. The time evolution if the intensities from each track follow the trend of the discharge current.

To understand the effect of higher powers on the TriPack V300, the average discharge power was increased to 500W at 950V and 500μs on a titanium target. Figure 4.14 shows details of this experiment.
Figure 4.14: (a) Side view of the TriPack V300 titanium target with the peak radial magnetic field values on the three race tracks, (b) Streamline plot of Bx and Bz components on the target surface simulated in COMSOL Multiphysics, (c) Photograph of the pre-eroded titanium target, (d) VI oscillogram corresponding to Figure 4.15.

It can be seen from Figure 4.14(b) that the voltage pulse drops from 950V to almost 800V at the end of the discharge. This drooping in voltage is due insufficient capacitance in the power supply to maintain the set voltage. Figure 4.15 shows the ICCD images for the conditions described in Figure 4.14. For concise understanding, not all images from each time snaps are presented. Only the important ones are presented. In the higher power (500W) case, the plasma in the middle race track is much more intense that the other two racks at 20µ from the start of the HiPIMS voltage pulse. The inner race track never gets bright throughout the discharge although the radial magnetic field on that race track is much higher than the other two. The outer race track gets brighter but the inner and middle race tracks fades away during latter part (300-480µs) of the discharge. Even at higher powers, the plasma looks homogeneous without any “ionization zones”. The time
evolution of the intensities (Figure 4.16(a)) from this case shows that the peak intensities from the inner and middle race track are much higher that the outer track, the intensities on all the race tracks reaches peak value at 60µs. This trend is very similar to the 250W case discussed previously. The intensity from the outer race track does not fall steeply as in the inner and middle cases. During the end of the pulse, the outer race track intensity is higher than the other two. It should be observed that in the 500W case, the individual intensities from each race track are about twice higher than the 250W case. The time evolution of the intensities from each race track in the 500W follows the discharge current trend like the 250W case.

Figure 4.15: ICCD images of TriPack V300 titanium HiPIMS discharge with times snaps for 500µs, 950V, 500W and 20mTorr process conditions. The exposure time of these images are 10ns.
4.3.1.2 ICCD Study of Ignition on One Race Track at a Time

In order to understand if there is any plasma interaction between the race tracks, discharge was ignited on only one race track at a time. This was achieved by inserting thick aluminum spacers on specific race tracks to avoid discharges on them. To achieve discharge on the inner rack, aluminum spacers were placed on the middle and outer race tracks. The details of this experiment are shown in Figure 4.17 and ICCD images shown in Figure 4.18. It can be observed from these ICCD images that the discharge was homogeneous without any “ionization zones’/plasma spokes. The intensity remained more or less the same throughout the discharge.
Figure 4.17: (a) Side view of the TriPack V300 titanium target with the magnetic insert slots and aluminium spacers on the middle and outer race track, (b) Streamline plot of Bx and Bz components on the target surface simulated in COMSOL Multiphysics, (c) Photograph of the pre-eroded titanium target with aluminium spacers on the middle and outer race tracks, (d) VI oscillogram corresponding to Figure 4.18.

Figure 4.18: ICCD images of TriPack V300 titanium HiPIMS discharge on the inner race track alone with times snaps. The discharge corresponds to 500μs, 650V, 250W and 20mTorr process conditions. The exposure time of these images are 10ns.

Figure 4.17(d) shows the VI trace for this experiment and it shows very small currents with negative amplitude. The negative amplitude does not mean anything because the discharge current from this experiment is much smaller than the minimum detection limit of the discharge current monitor which is rated to measure up to 1500A. The peak current
during the discharge is around 0.5A. This does not mean absence of HiPIMS discharge because the measured electron densities and temperatures ($N_e \sim 1-3 \times 10^{18}$, $T_e \sim 3-5$ eV) are comparable to the values that are obtained from typical HiPIMS discharges. Although the peak radial peak field magnitude on this race track is much higher than the other two race tracks, the discharge currents are much smaller compared to the other two.

HiPIMS discharge on the middle race track was achieved by placing aluminum spacers on the inner and outer race tracks. The aluminum spacers were spot welded to avoid spacers dropping down during the discharge. Figure 4.19 show the experimental conditions corresponding to the ICCD images in Figure 4.20. At 20$\mu$s from the start of the HiPIMS discharge, the plasma on the middle race track faint which then become very bright at 100$\mu$s. The peak current during this discharge is around 15A. The discharge on the middle race track was homogeneous with no “ionization zones”/plasma spokes.

Figure 4.19: (a) Side view of the TriPack V300 titanium target with the magnetic insert slots and aluminium spacers on the inner and outer race track, (b) Streamline plot of $B_x$ and $B_z$ components on the target surface simulated in COMSOL
In the outer race track experiments, aluminum spacers were inserted in to the inner and middle race tracks as shown in Figure 4.21(b) to obtain discharge only in the outer race track area. The experimental conditions are described in Figure 4.21 and the corresponding ICCD images are shown in Figure 4.22. The plasma on the outer rack is homogenous throughout the discharge. The peak current during the discharge is around 15A.
Figure 4.21: (a) Side view of the TriPack V300 titanium target with the magnetic insert slots and aluminium spacers on the inner and middle race track, (b) Streamline plot of Bx and Bz components on the target surface simulated in COMSOL Multiphysics, (c) Photograph of the pre-eroded titanium target with aluminium spacers on the inner and middle race tracks, (d) VI oscillogram corresponding to Figure 4.22.

Figure 4.22: ICCD images of TriPack V300 titanium HiPIMS discharge on the outer race track alone with times snaps. The discharge corresponds to 500μs, 650V, 250W and 20mTorr process conditions. The exposure time of these images are 10ns.

Figure 4.23 is the time evolution of currents from individual race tracks, currents from simulations operation of all the three race tracks and algebraic sum of currents from the
individual race tracks at different powers (250W and 500W). It should be noted that in this plot, the algebraic sum of the individual currents corresponds to 750W rather than 250W. In Figure 4.23(a and b), the currents from simultaneous operation of all the three race tracks flows the overall trend of the algebraic sum of the currents.

![TriPack V300](image1)

Figure 4.23: (a) Time evolution of current from simultaneous operation of all three race tracks, individual race tracks, and the algebraic sum from the individual race tracks at 250W, (b) Time evolution of current from simultaneous operation of all three race tracks, individual race tracks, and the algebraic sum from the individual race tracks at 500W.

Some of the major observations from the above ICCD camera experiments involving discharge on individual race tracks and simulataneous operation of all the race tracks are:

1. The plasma looks homogeneous without the absence of “ionization zone”/plasma spokes in all the cases.

2. The current obtained from the inner race was much smaller than the middle and outer race tracks although the magnitude of the radial magnetic field of the inner rack is higher than the other two race tracks.
3. The inner race track area is ~0.6in², which 43% of the middle race track area (~1.4in²) and only 10% of the outer race track area (6.0in²). Such low discharge currents in the inner race track case can be due to smaller race track area. On the other hand, the middle race track area is only 23% of the outer race track area but the peak currents from the middle race track and outer race track are the same (15A). The difference in magnitude of the radial magnetic field between them is around 90Gauss (Middle: 500Gauss, Outer: 410Gauss).

**4.3.1.3 Influence of Race Track Width on “ionization zones”**

All the ICCD observations from study of TriPack V300 leads to a question if the “ionization zone”/plasma spokes formation were related to the race track widths (race track width dictates the race track area) because the race track widths (race track area) in the TriPack V300 are not the same for all the three race tracks. In order to investigate the effect of race track widths on the “ionization zone”/plasma spokes, a series of ICCD experiments were conducted where, the race track widths of the target (aluminum in this case) on the conventional magnet pack were varied but the magnitude of their radial magnetic field was kept constant at 650Gauss. This was achieved by carefully placing iron spacers between the magnetron target (Figure 4.24) to shape the magnetic field lines and their magnitude on the top of the target. The dimensions of the target and the iron spacers are critical in achieving the right magnetic field values. Hence, the set-up was simulated in COMSOL Multiphysics to get the accurate dimensions of the target and the iron spacers.
Four different race track widths (0.22”, 0.44”, 0.66” at $B_r=650\text{Gauss}$ and 1.10” at $B_r=450\text{Gauss}$) were studied and aluminum was chosen as the target material due to its ease of machining. In all these experiments, the ICCD camera was triggered every 20µs from the start of the consecutive pulses. Not all ICCD images from different time snaps are shown, only the important ones are shown.

Figure 4.25 shows the details of the Narrow 1 configuration with the corresponding VI trace. The HiPIMS operating parameters and the argon pressure was kept constant at 20mTorr to be consistent with the TriPack V300 experiments. It can be observed from Figure 4.26 that for the Narrow 1 (0.22” wide race track) case, at 20µs from the start of the discharge, the plasma looks more or less homogeneous and at 100µs, plasma contraction can be seen. The “ionization zones”/plasma spokes starts to appear at about 160µs and lasts till the end of the pulse (480µs). The peak discharge current during this experiment is ~ 35A.
Figure 4.25: (a) Schematic of Narrow 1 configuration that corresponds to a race track width of 0.22” at Br=650 Gauss, (b) VI oscillogram corresponding to Figure 4.26, (c) Photograph of the aluminium target placed between iron spacers.

Figure 4.26: ICCD images from Narrow 1 configuration during HiPIMS discharge with times snaps. The discharge corresponds to 500μs, 650V, 250W and 20mTorr process conditions. The exposure time of these images are 10ns.

The race track width was 0.44” for the Narrow 2 case. The details of this experiment and the corresponding ICCD images are shown in Figure 4.27 and Figure 4.28 respectively. In this case, at 20μs from the start of the discharge, the plasma is very intense and homogenous and at 140μs, distinct plasma spokes can be observed and they last till the end of the pulse (480μs). The peak HiPIMS discharge current during this experiment is ~85A.
Figure 4.27: (a) Schematic of Narrow 2 configuration that corresponds to a race track width of 0.44” at Br=650Gauss, (b) VI oscillogram corresponding to Figure 4.28, (c) Photograph of the aluminium target placed between iron spacers.

Figure 4.28: ICCD images from Narrow 2 configuration during HiPIMS discharge with times snaps. The discharge corresponds to 500μs, 650V, 250W and 20mTorr process conditions. The exposure time of these images are 10ns.

For the Narrow 3 case, the race tract width was 0.66’’ and details are shown in Figure 4.29. The corresponding ICCD images (Figure 4.30) indicate that initially at 20μs, the plasma is homogeneous and distinct plasma spokes starts to appear at 40μs from the start of the discharge. The number of spokes decreases as time progresses and they eventually
fade away at the end of the pulse (480µs). The peak current during this discharge is around 78A. The peak discharge current in this case is expected to be more than the Narrow 2 case due to the increase in the race track area but discharge current was lower than the previous case by 7A.

Figure 4.29: (a) Schematic of Narrow 3 configuration that corresponds to a race track width of 0.66" at Br=650Gauss, (b) VI oscillogram corresponding to Figure 4.30, (c) Photograph of the aluminium target placed between iron spacers.

Figure 4.30: ICCD images from Narrow 3 configuration during HiPIMS discharge with times snaps. The discharge corresponds to 500µs, 650V, 250W and 20mTorr process conditions. The exposure time of these images are 10ns.
In the regular case, the race track width was 1.1” but the magnitude of the radial magnetic field is 450Gauss. Therefore, this case has wider track but lower radial magnetic field compared to the previous cases. This case is called Regular because this set-up is the same as conventional magnet pack set-up with a 0.25” thick aluminum target. The details of this experiment are shown in Figure 4.31. In this experiment, the pulse width was set at 250µs instead of 500µs because of the process constraints in achieving the same average power (250W) with a fixed voltage (650V). It can be observed from the corresponding ICCD images (Figure 4.32) that initially at 20µs from the start of the discharge, the plasma looks intense and homogeneous. At 140µs, distinct plasma spokes appears and the number of spokes is reduced as the time progresses. The plasma spokes lasts until the end of the pulse (250µs). The peak HiPIMS current during this case is ~138A.
Some of the main observations from varying race track width experiments are as follows:

1. Narrower the race track width is, later the plasma spokes are formed

2. Lower the magnitude of the radial magnetic field ($B_r$), later the plasma spokes are formed.
3. The peak discharge current in the Narrow 3 case is lower than the Narrow 2 case in spite of the wider race track.

All these observations can aid in building a consistent plasma spoke model for HiPIMS discharges. VI traces during HiPIMS discharge contains a lot of information about the discharge process. Figure 4.33 shows the time evolution of discharge currents during titanium HiPIMS discharge from simultaneous operation of all three race tracks, individual race tracks, and the algebraic sum from the individual race tracks at different powers (250W, 500W and 750W). The discharge voltage was fixed at 1000V in all the cases, but pulse time and frequency were varied in each case to obtain the same average power on all the three race tracks individually at 20mTorr. In the 250W case (Figure 4.33(a)), the algebraic sum of the currents from the individual race tracks operated at 250W corresponds to 750W case, hence all the three race tracks were simultaneously operated at 750W average power for comparison. The algebraic sum of currents and the discharge current during all three race tracks in operation in the 250W (Figure 4.33(a)) case follows the same trend but in the 500W (Figure 4.33(b)) and 750W (Figure 4.33(c)) case, the trends look very different from each other indicating evidence of “cross talk” between race tracks at higher powers. It was possible to achieve high power (1500W and 2250W) operation in the TriPack due to the new cooling well that allows high power operation. These plots are very crude comparison and cannot be sued for any quantitative analysis as the pulse widths and frequencies were different in certain cases.
Figure 4.33: (a) Time evolution of current from simultaneous operation of all three race tracks, individual race tracks, and the algebraic sum from the individual race tracks with a titanium target at (a) 250W average power, (b) 500W average power and (c) 750W average power. In all the cases, the voltage was fixed at 1000V but pulse time and frequency were varied in each case to obtain the same average power on all the three race tracks individually at 20mTorr.

Figure 4.34(a) shows the time evolution of electron density and temperature during HiPIMS discharge (700V, 100µs, 250W at 20mTorr) on TriPack V300 with a titanium target at 3” away from the target. Figure 4.34(b) is the VI oscillogram corresponding to the electron density and temperature measurements shown in (a). It can be observed from Figure 4.34(a) that the peak density is ~5x10^{19} m^{-3} and peak electron temperature is ~4eV at 3” away from the titanium target with TriPack V300 in operation at 20mTorr and 250W average power.
4.3.2 Optical Plasma Diagnostics

The TriPack V300 was further investigated using a spectrometer. The spectrometer set-up used in this experiment is discussed in detail in Chapter 2. The measured intensities are expressed in arbitrary units because the absolute values don’t have any significance due to the problems with calibration. These spectrums cannot be used for quantitative analysis but qualitative information can be obtained by comparing relative intensities from a single spectrum. Figure 4.35 shows the optical emission spectrum from conventional pack DC and HiPIMS discharges. It can be observed from Figure 4.35(a) and (b), that the discharge in HiPIMS contains more aluminium than argon just by comparing the relative intensity of aluminium at ~397nm and argon at 700-850nm. Figure 4.36 is the optical emission spectrum from TriPack DC and HiPIMS discharges. The relative intensities from aluminium peak at 397nm compared to the argon peaks from 650-900nm in TriPack V300 HiPIMS (4.36(b) and TriPack V300 DC (4.36(a)) is very different. Since the intensities from some of the peaks in the spectrum are saturated, it is hard to get any quantitative analysis out of it but from the relative intensities of Al I and Ar I peaks, it can be observed that TriPack V300 gets in to HiPIMS conditions. The
spectrometer that was used in the OES experiments was not sensitive enough to detect the ions of the target species.

Figure 4.35: (a) Optical spectrum during conventional pack DC in operation at 10mTorr and 500W average power, (b) Optical spectrum during conventional pack HiPIMS in operation at 10mTorr and 500W average power.

Figure 4.36: (a) Optical spectrum during TriPack V300 DC in operation at 10mTorr and 500W average power, (b) Optical spectrum during TriPack V300 HiPIMS in operation at 10mTorr and 500W average power.

4.3.3 Ionization Fraction Measurement from TriPack V300

GEA-QCM set-up that was described in the diagnostics section was used to measure the metal ion fraction at the substrate. The error in the QCM measurement was estimated to be ~0.01A/s from various QCM calibration experiments. Figure 4.37 shows the deposition rates measured at the QCM versus the ion discriminator grid voltage for the
conventional pack at 1mTorr with a copper target. The GEA-QCM set-up was placed line of sight at 4” away (substrate level) from the target. Conventional pack DC and HiPIMS discharges were compared during this experiment. It can be seen from Figure 4.37 that the measured deposition rates at the QCM from HiPIMS and DC experiments look almost the same because, the difference in deposition rates are below the QCM’s measurement error (0.01A/s) which means, the ion fraction is below 5% and the exact number cannot be determined. Hence, the QCM-GEA diagnostic is not ideal for low power operation where the deposition rates are very low.

![Figure 4.37: Deposition rate at the QCM versus ion discriminator grid voltage from the GEA set-up at 500W average power.](image)

In order to estimate the ion fraction in the deposited flux from the TriPack V300 and conventional pack, two circular stainless steel coupons with known masses were installed side by side as shown in Figure 4.38. Both the coupons were separated by a ceramic break and a bias cable was attached to one of the coupons for biasing it positive during deposition to repel off the ions. These coupons were mounted on a rotatable feed-through so the whole experiment can be done without breaking the vacuum. The target material was copper in these experiments and the target to coupon distance was 4”. A grounded
mesh was placed in front of the copper target to avoid the change in plasma potential due to coupon bias. In all these experiments, Starfire Impulse power supply was used for HiPIMS discharges. Copper films were grown on the stainless steel coupons at +35V and floating potential. The positive voltage repels all the ions, so the deposition flux on the biased coupon consists of only copper neutrals. The coupon that was at floating potential receives both ion and neutral copper flux. By comparing the relative change in the masses before and after deposition on the two coupons, the ion fraction can be determined.

The results from the ion fraction experiments are shown in Table 4.3 and these experiments were performed at 10mTorr, 500W average power and 4” away from the target. The weighing balance that was used to measure the stainless steel coupons in these can resolve accurately above 1 mg. Therefore, the observed 1 mg change in the biased and un-biased cases of conventional DC and HiPIMS experiments cannot be used for ion fraction calculations as it is below the accuracy of the weighing balance. This means, the ion fraction in these cases would be less than 5% and the exact number is below the
detection limit of this set-up. It can be observed from Table 4.3 that in the case of TriPack HiPIMS, the copper ion fraction is ~16% which almost thrice the ion fraction of conventional packs DC and HiPIMS cases.

Table 4.3: Results from copper ion fraction experiments.

<table>
<thead>
<tr>
<th>Magnet Pack</th>
<th>Type of Discharge</th>
<th>Average Power (W)</th>
<th>Mass change in biased coupon(mg)</th>
<th>Mass change in un-biased coupon(mg)</th>
<th>Ion Fraction (%)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Conventional DC</td>
<td>500</td>
<td>20</td>
<td>19</td>
<td>&lt;5±2</td>
<td></td>
</tr>
<tr>
<td>Conventional HiPIMS</td>
<td>500</td>
<td>16</td>
<td>15</td>
<td>&lt;5±2</td>
<td></td>
</tr>
<tr>
<td>TriPack V300 HiPIMS</td>
<td>500</td>
<td>16</td>
<td>20</td>
<td>16±3</td>
<td></td>
</tr>
</tbody>
</table>

4.3.4 Substrate Uniformity Test from TriPack V300

The deposition uniformity of TriPack V300 was tested by depositing HiPIMS copper films on 4” silicon substrate that is placed line of sight at 4” away from the target as shown in Figure 4.38(a). The deposition was carried out at 10mTorr with an average power of 500W. Starfire Impulse power supply was used for HiPIMS discharge in this experiment. The thickness of the deposited copper film was measured using XRR technique in the locations shown in Figure 4.38(b). Since TriPack V300 is symmetric, locations 1, 2 and 3 were only measured. The deposition rates at each location were determined by dividing the thickness of deposition with the total deposition time. Table 4.4 shows the deposition rate normalized to the maximum deposition rate (location 1) at different locations. It is evident from Table 4.4 that TriPack V300 has better substrate uniformity compared to the “ε” magnet pack. The edge uniformity of TriPack V300 is even superior to conventional pack uniformity.
Table 4.4: Normalized deposition rates from TriPack, “ε” pack and conventional magnet pack for HiPIMS and DC discharges at 10mTorr and 500W average power at locations indicated in Figure 4.38(b) during wafer uniformity experiments.

<table>
<thead>
<tr>
<th>Position</th>
<th>DC Conventional</th>
<th>DC “ε”</th>
<th>HiPIMS Conventional</th>
<th>HiPIMS “ε”</th>
<th>TriPack V300</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>1.00</td>
<td>--</td>
<td>1.00</td>
<td>1.00</td>
<td>1.00</td>
</tr>
<tr>
<td>2</td>
<td>0.98</td>
<td>--</td>
<td>1.00</td>
<td>0.88</td>
<td>0.93</td>
</tr>
<tr>
<td>3</td>
<td>0.93</td>
<td>--</td>
<td>0.90</td>
<td>0.88</td>
<td>0.92</td>
</tr>
<tr>
<td>4</td>
<td>--</td>
<td>--</td>
<td>--</td>
<td>0.95</td>
<td></td>
</tr>
<tr>
<td>5</td>
<td>--</td>
<td>--</td>
<td>--</td>
<td>0.90</td>
<td></td>
</tr>
</tbody>
</table>

Figure 4.39: (a) Schematic of the 4” silicon substrate location with respect to the target, (b) Location of the uniformity test on the 4” silicon substrate.

4.3.5 Study of Film Morphology from TriPack V300

In order to understand the surface morphology of the films deposited using TriPack V300, copper thin films were deposited on silicon substrate using conventional and TriPack V300 with DC and HiPIMS at 10mTorr and 500W average power. The target to substrate distance in these experiments is 4”. Figure 4.39 to Figure 4.42 shows the top down SEM images of copper films deposited using conventional and TriPack V300 at different magnifications. It is evident from Figure 4.42(a) that the copper thin film deposited using
conventional dcMS consist of highly porous large grains with globular microstructures. In the case of conventional pack HiPIMS (Figure 4.42(b)), the copper grains are smaller and less porous compared to dcMS deposited copper film, indicating the presence of few copper ions. It is very clear from Figure 4.42(c) that the copper film deposited using TriPack V300 with HiPIMS consists of very fine globular nanostructure indicating the presence of high ion flux with low energies. In all these experiments, the substrate was not biased. Hence, the ion energies of the incoming ion flux were not altered. Increase in ion flux to the substrate causes repeated nucleation that suppresses the columnar structure and transforms the films from a polycrystalline to globular nanocrystalline microstructure[29]. It can also be observed from the SEM images that the copper film that was deposited using TriPack V300 HiPIMS has lower surface roughness and higher density compared to films deposited using conventional pack DC and HiPIMS. These observations are further supported by the higher ion fraction observed during TriPack V300 HiPIMS operation.
Figure 4.40: (a) Top down 10k zoom SEM image of conventional pack copper film deposited with dcMS at 10mTorr and 500W average power, (b) Top down 10k zoom SEM image of conventional pack copper film deposited with HiPIMS at 10mTorr and 500W average power (c) Top down 10k zoom SEM image of TriPack V300 copper film deposited with HiPIMS at 10mTorr and 500W average power.

Figure 4.41: (a) Top down 50k zoom SEM image of conventional pack copper film deposited with dcMS at 10mTorr and 500W average power, (b) Top down 50k zoom SEM image of conventional pack copper film deposited with HiPIMS at 10mTorr and 500W average power (c) Top down 50k zoom SEM image of TriPack V300 copper film deposited with HiPIMS at 10mTorr and 500W average power.
Figure 4.42: (a) Top down 100k zoom SEM image of conventional pack copper film deposited with dcMS at 10mTorr and 500W average power, (b) Top down 100k zoom SEM image of conventional pack copper film deposited with HiPIMS at 10mTorr and 500W average power (c) Top down 100k zoom SEM image of TriPack V300 copper film deposited with HiPIMS at 10mTorr and 500W average power.

Figure 4.43: (a) Top down 200k zoom SEM image of conventional pack copper film deposited with dcMS at 10mTorr and 500W average power, (b) Top down 200k zoom SEM image of conventional pack copper film deposited with HiPIMS at 10mTorr and 500W average power (c) Top down 200k zoom SEM image of TriPack V300 copper film deposited with HiPIMS at 10mTorr and 500W average power.
4.4 Summary

The results from TriPack V300 testing are summarized in Table 4.5.

Table 4.5: Summary of TriPack V300 results.

<table>
<thead>
<tr>
<th>Parameter</th>
<th>Characteristics</th>
</tr>
</thead>
<tbody>
<tr>
<td>Volt-Ampere Characteristics</td>
<td>TriPack V300 follows magnetron mode of discharge.</td>
</tr>
<tr>
<td>Deposition Rates</td>
<td>TriPack V300 gives higher deposition rates in HiPIMS than conventional HiPIMS</td>
</tr>
<tr>
<td>Current Density</td>
<td>HiPIMS current density in TriPack V300 is much lower than conventional magnet pack</td>
</tr>
<tr>
<td>Erosion Area</td>
<td>Erosion area of TriPack V300 is ~25% more than conventional magnet pack</td>
</tr>
<tr>
<td>ExB plasma instabilities</td>
<td>The “ionization zones” were not observed in TriPack V300. The plasma was homogeneous on all the three race tracks whereas in conventional pack, “ionization zones” can be observed. The “ionization zones” were not observed even with discharge on one race track at the same time in the TriPack V300.</td>
</tr>
<tr>
<td>Substrate Uniformity</td>
<td>TriPack V300 gives better substrate uniformity than “ε” magnet pack.</td>
</tr>
<tr>
<td>Ion Fraction</td>
<td>TriPack V300 gives higher ion fraction in HiPIMS than conventional pack for the same average power.</td>
</tr>
<tr>
<td>Film Morphology</td>
<td>The films deposited using HiPIMS from TriPack V300 has much small grain size compared to conventional pack DC and HiPIMS.</td>
</tr>
</tbody>
</table>

The reason behind higher deposition rates and the absence of ionization zones are elaborately discussed in Chapter 6. Based on the plasma diagnostics of the TriPack V300, it is evident that the plasma dynamics in this magnet pack is complex and a thorough analysis is presented in sections 6.1.2, 6.1.3 and 6.2.1 for better understanding.
Chapter 5 Target Erosion Profile Model

Target utilization is an important aspect of commercial magnetron sputtering systems. One of the main disadvantages of magnetron sputtering is its poor target utilization in spite of producing high quality films. The cost of target material has steadily increased over the years in order to keep with the increasing demand for high quality films. The current target utilization of magnetron sputtering systems are under 30% [10]. Hence, magnetron designs have to be optimized for better target utilization to keep the costs low.

In this thesis, target erosion profile is calculated in COMSOL Multiphysics based on I.Y.Burmakinskii et al.[112] model with a few modifications. Burmakinskii-Rogov’s model is based on these few major assumptions:

1. Magnetic field configuration is the main factor that determines the target erosion parameter.
2. Electric field lines are directed vertically towards the cathode.
3. The secondary electron co-efficient does not exceed 0.1 electron per ion.

5.1 Target Erosion Model

This target erosion model was developed by Burmakinskii and Rogov. In this work, their model is modified to fit the experimental results. Burmakinskii-Rogov’s model is described in detail in this section.

Figure 5.1 is the schematic showing the magnetron sputtering target with the magnetic system. In the region $S$ shown in Figure 5.1,

\[
\left| \frac{\partial E_z}{\partial z} \right| R_L \ll |E_z|, \quad \left| \frac{\partial B_y}{\partial z} \right| R_L \ll |B_y| \tag{5.1}
\]
where $R_L$ is the larmor radius, $E_z$ is the z component of the electric field, $B_y$ is the y component of the magnetic field.

At an arbitrary point in the discharge region, the number of ionization events per unit volume per unit time in the vicinity of this point is

$$N_i(y, z) \approx n_e(y, z)v_i(y, z)$$  \hspace{1cm} 5.2

![Figure 5.1: Arrangement of cathode and the magnetic system: (1) cathode, (2) central magnet, (3) peripheral magnet, (4) magnetic core, (5) magnetic field lines, and (6) discharge region[112].](image)

where $n_e(y, z)$ is the density of ionizing electrons at that arbitrary point and $v_i(y, z)$ is the ionization frequency. The ion flux density $J_i(y)$ at the cathode at a point with the y co-ordinate is

$$J_i(y) \propto \int_{z_{cat}}^{z^*} n_e(y, z)v_i(y, z)dz$$  \hspace{1cm} 5.3

The erosion depth $h(y)$ at a point with the y co-ordinate is given by

$$h(y) \propto f_i(y)\langle S_y(y) \rangle \propto \langle S_y(y) \rangle \int_{z_{cat}}^{z^*} n_e(y, z)v_i(y, z)dz$$  \hspace{1cm} 5.4
\(<S_y(y)>\) is the energy averaged material sputtering co-efficient and it is given by [113]

\[
(S_y(y)) = \frac{\frac{e}{M_i} \int_0^\infty f_i(\varepsilon, y)S_y(\varepsilon) d\varepsilon}{J_i(y)}
\]  

5.5

e is the ion charge equal in magnitude to the electron charge, \(M_i\) is the ion mass, and \(f_i(\varepsilon, y)\) is the ion energy distribution function at the cathode.

\[
J_i(y) = \frac{e}{M_i} \int_0^\infty f_i(\varepsilon, y) d\varepsilon
\]  

5.6

The erosion depth \(h(y)\) is given by

\[
h(y) \propto \int_{Z_{cat}}^{z^*} n_e(y, z) v_i(y, z) dz
\]  

5.7

Applying mean-value theorem to the above equation yields the following expression.

\[
h(y) \propto \{v_i(y)\} \int_{Z_{cat}}^{z^*} n_e(y, z) dz
\]  

5.8

In the above equation, \(v_i(y, z)\) is averaged over \(z\) co-ordinate. The upper limit of the integral is set to \(\infty\) because the plasma density decreases rapidly with the distance from the cathode.

\[
\int_{Z_{cat}}^{z^*} n_e(y, z) dz \rightarrow \int_{Z_{cat}}^{\infty} n_e(y, z) dz = [nl]_e
\]  

5.9

The absolute value of the constant \([nl]_e\) is not that important because it would only be a normalization factor. Therefore,

\[
h(y) \propto \{v_i(y)\}
\]  

5.10

The ionization frequency is a function of total kinetic energy \(W_e\) (velocity \(V_e\)) of electrons and the density of the working gas.
\[
h(y) \propto \{\sigma_i(W_e(y))V_e(y)\} \approx F(\{V_e(y)\})
\]

\(F(\{V_e(y)\})\) is a function of the average electron velocity. The average electron velocity is the resultant of the parallel and transverse components.

\[
{V_e}^2 = \{V_{\parallel}\}^2 + \{V_{\perp}\}^2
\]

The transverse component of the electron velocity is given by

\[
V_{\perp} = \frac{E \times B}{B^2} \approx \frac{E_x B_y}{B^2}
\]

The average transverse component is defined as

\[
\{V_{\perp}(y)\} \approx \frac{\int_{z_{cat}}^{z} E_x(y, z) B_y(y, z) dz}{\int_{z_{cat}}^{z} B^2(y, z) dz} \approx \frac{\int_{z_{cat}}^{z} E_y(y, z) B_y(y, z) dz}{\int_{z_{cat}}^{z} B^2(y, z) dz} \approx \left[ E_z \right] \frac{\int_{z_{cat}}^{z} B_y(y, z) dz}{\int_{z_{cat}}^{z} B^2(y, z) dz}
\]

\[
\{V_{\perp}(y)\} \approx \left[ E_z \right] \frac{\int_{z_{cat}}^{\infty} B_y(y, z) dz}{\int_{z_{cat}}^{\infty} B^2(y, z) dz}
\]

By setting the upper limit as \(\infty\) in the above equation yields,

\[
\left[ E_z \right] = \frac{\int_{z_{cat}}^{\infty} E_x(y, z) B_y(y, z) dz}{\int_{z_{cat}}^{\infty} B_y(y, z) dz}
\]

\[
= \frac{\left( -\varphi(y, z) B_y(y, z) \right)_{z_{cat}}^{\infty} + \int_{z_{cat}}^{\infty} \varphi(y, z) \frac{\partial B_y(y, z)}{\partial z} dz}{\int_{z_{cat}}^{\infty} B_y(y, z) dz}
\]

\(\varphi(y, z)\) is the potential at the point with co-ordinates \(y\) and \(z\).

\(B_y(z) = B_{cat} \exp\left\{-z/\lambda_B\right\}\)

\(B_{\perp}(z)\) is the magnetic field component parallel to the cathode and \(\lambda_B\) is a constant that depends on the relative positions, dimensions of the magnets and the ration of their magnetization.

\(\varphi(y, z_{cat}) = U_d; \ \varphi(y, \infty) \to 0\)
where $B_{cat}$ is the magnetic induction on the cathode surface and $U_d$ is the discharge voltage.

Equations 5.18 and 5.19 are satisfied. Therefore, equation 5.16 becomes

$$\left[E_z\right] = \frac{U_d B_{cat} + \langle \varphi \rangle \int_{z_{cat}}^{0} dB_y}{\int_{z_{cat}}^{0} B_{cat} \exp\{-z/\lambda_\beta\} dz} \approx \frac{U_d B_{cat} - \langle \varphi \rangle B_{cat}}{\lambda_\beta B_{cat}}$$

Since $\langle \varphi \rangle \approx U_d/2$,

$$\left[E_z\right] \approx \frac{U_d}{2\lambda_\beta}$$

$$\{V_\perp(y)\} \approx \frac{U_d}{2\lambda_\beta} \int_{z_{cat}}^{\infty} B_y(y, z) dz$$

The total electron energy is the sum of parallel and transverse components of energy.

$$W_e = W_\parallel + W_\perp$$

The transverse energy component can be approximated as

$$[W_\perp(y)] \approx \{V_\perp(y)\}^2 m_e$$

The parallel energy component is slightly greater than the ionization energy of the working gas. Therefore,

$$W_\parallel(y, z) = W_\parallel^0 \geq eJ_p$$

From equations 5.22, 5.24 and 5.25, the average electron energy in section $y$ is

$$[W_e(y)] = W_\parallel^0 + [W_\perp(y)]$$

The ionization cross-section of the working gas can be calculated from the formula [113]
\[ \sigma_i(W_e) = 2.66\pi a_0^2 \frac{J_n}{J_p} g \left( \frac{W_e}{J_p} - 1 \right) \frac{(W_e/J_p - 1)^2}{(W_e/J_p)^2} \ln \left( \frac{W_e}{J_p} \right) \]  

\[ J_p \text{ is the ionization energy which is } 15.8\text{eV for argon, } J_n \text{ is a constant which is taken to be } 13.8\text{eV, } \pi a_0^2 \text{ is } 8.8 \times 10^{-21} \text{ m}^2, g \text{ is the number of electrons in the upper level which } 8 \text{ for argon and } W_e \text{ is in electronvolts.} \]

The electron velocity in terms of total kinetic energy is

\[ V_e(y) \approx \sqrt{\frac{2[W_e(y)]}{m_e}} \]  

From equations 5.26, 5.27 and 5.28, the erosion depth of the cathode is found to be

\[ h(y) \approx h_{\text{norm}} \sigma_i([W_i(y)]) V_e(y) \]  

\[ h_{\text{norm}} \text{ is the normalization co-efficient which can optimized to get the appropriate target thickness.} \]

\[ \frac{B(z_{\text{cat}})}{z_{\text{cat}}} \ll 1 \]  

Numerical calculations involving known magnetic fields, the upper integration limit \( z_{\text{cal}} \) is taken from equation 5.30, 5.31 or 5.32.

\[ \int_{z_{\text{cat}}}^{z_{\text{cal}}} B^2(y, z) dz \approx \int_{z_{\text{cat}}}^{\infty} B^2(y, z) dz \]  

\[ \int_{z_{\text{cat}}}^{z_{\text{cal}}} |B_y(y, z)| dz \approx \int_{z_{\text{cat}}}^{\infty} |B_y(y, z)| (dz) \]  

The constant \( \lambda_B \) is determined by calculating the magnetic field for a given magnetic system. The target erosion profile can be obtained by solving for \( h(y) \).
5.2 Simulation Results for Conventional Magnet Pack Configuration

All the above equations from I.Y.Burmakinskii’s model is implemented in COMSOL Multiphysics, so the target erosion profile for any magnet pack arrangement can be obtained during the design phase of the magnet packs.

Target erosion profile from a partially eroded 4” conventional magnetron (conventional pack) target (0.25” thick) was measured a high resolution 3D scanner to create a 3D model of the target. The 3D model was then used to obtain 2D axisymmetric target erosion profile. Aluminum target was used for this purpose.

The Burmakinskii-Rogov’s model results from the 4” conventional magnetron (0.25” thick target) configuration was compared with the actual measured erosion profile. Due to the axis symmetry of the magnet pack, the target erosion profile was simulated for only one side of the target. In order to match the simulation results to the experimental values, the empirical formula for the ionization cross-section of the working gas described in Burmakinskii-Rogov’s model has to be modified as

\[ \sigma_i(W_e) = 2.66 \pi a_0^2 \frac{J_n}{J_p} g \left( \frac{W_e}{J_p} - 1 \right) \left( \frac{W_e}{J_p} \right)^2 \sqrt{\ln \left( \frac{W_e}{J_p} \right)} \]

The normalization co-efficient, \( h_{norm} \) was optimized to get appropriate target thickness. The logarithmic term is replaced with the square root of the logarithmic term as shown in equation 5.33. The reason behind such a good fit between the modified ionization cross-section equation and the experimental results needs further investigation. Introduction of square root to the logarithm term is a very conservative modification as it only decreases the effective ionization cross-section area.
Figure 5.2 shows the measured target erosion profile, simulated target erosion profiles from the original and improved models for conventional magnet pack. The x-axis represents the target radius in inch (4” diameter) and y-axis represents the normalized target thickness. The experimental and the improved model results match very well. The percentage target utilization by area in this case can be calculated by integrating the area under the curve and dividing it by the cross-sectional area. The percentage utilization is found to be ~30% in the conventional magnetron case.

\[ h(y) \]

\[ \text{Normalized Thickness} \]

\[ \text{Length (inch)} \]

\[ \text{Experimental} \]

\[ \text{Improved Model} \]

\[ \text{Original Model} \]

Figure 5.2: Comparison between experimental target erosion profile and simulated target erosion profile on a 4” conventional magnetron.

5.3 Simulation Results for TriPack V300 Magnet Pack Configuration

The target erosion profile \( h(y) \) is simulated based on the modified ionization cross-section formula (Equation 5.33). Figure 5.3 shows the target erosion profile from the TriPack
V300. In this case, the percentage target utilization by area is ~33%. It can be seen from the target erosion profile that inner and middle race tracks are deeper and narrower than the outer race track. This is because the TriPack V300 magnet pack was optimized for deposition rates and not for target utilization. The black circles on the graph indicate the location of the maximum erosion profile measured from TriPack V300. The simulation results agree well with the experimental data.

Figure 5.3: Simulated target erosion profile from TriPack V300. The black circles on the graph indicate the location of the maximum erosion measured from TriPack V300.
5.4 Conclusion

The ionization cross-section equation of the working gas in Burmakinskii-Rogov’s model was modified to match the simulation results with the experimental measurements. This modified equation gives ~95% accurate erosion profile while the prediction from the original equation is only ~50% accurate. The reason behind such good fit between the modified theory and the experimental results needs to be thoroughly investigated as it involves detailed theoretical and experimental efforts. The target utilization of TriPack V300 and conventional magnet are very similar. Since this modified model can be implemented in COMSOL Multiphysics, the target erosion profile can be calculated instantly for any magnetic field configurations.
Chapter 6  Discussion on TriPack V300 Deposition Rates

The magnetic field profile on the target surface is a critical parameter in the design of magnetron sputtering sources as they define the plasma properties and potential distribution in the space above the target region. The change in plasma parameters can lead to higher deposition rates. A detailed analysis of the magnetic field configurations in the conventional pack, “ε” pack and TriPack V300 along with the reasons behind higher deposition rates and absence of “ionization zones” will be discussed in this chapter.

Past research on magnetic field strengths of magnetrons suggests that lower magnetic field leads to wider race track and higher target utilization whereas stronger magnetic field provides better electron confinement\[114\]. Figure 6.1 is the plot of ionization efficiency versus tangential magnetic field strength obtained using Monte Carlo simulation of electron transport. It is evident from the plot that the ionization efficiency increases only up to a certain level with the increasing tangential magnetic field before saturating at higher fields. Higher fields leads poor target utilization, hence there is a fine trade-off between target utilization and ionization efficiency. Also, lowering of magnetic field leads to higher deposition rates in HiPIMS due lower metal ion “return effect” \[85\].

In the “ε” pack and TriPack V300 design, tangential magnetic field values are almost equal to or greater than the conventional magnet pack. Hence, the observed higher deposition rates are not due to lowering of magnetic field. The “ε” pack and TriPack V300 design is optimized for proper magnetic field values to achieve better target utilization and ionization efficiency.
6.1 Reasons behind Higher Deposition Rates in TriPack V300

Some of the basic observations from TriPack V300 deposition rate experiments are

1. TriPack V300 gives higher deposition rate in HiPIMS than conventional pack HiPIMS.

2. The HiPIMS deposition rates in TriPack V300 can be varied by changing the pulsing parameters.

3. In the case of titanium, the deposition rate of TriPack V300 in HiPIMS was higher than even conventional DC case.

4. Current density of TriPack V300 is much lower than the current density of conventional pack for the same average power in HiPIMS.

5. The erosion area of TriPack V300 was \( \sim 25\% \) more that the erosion area of the conventional magnet pack.
The fundamental reason behind these observations is the change in magnetic field configuration of TriPack V300. Therefore, various features of the magnetic field configurations are analyzed for better understanding.

### 6.1.1 Magnetic Field Gradients

The gradient of the magnetic field component that is parallel to the target (tangential field/radial magnetic field $B_r$, in case of cylindrical symmetry) is an important factor in determining the plasma parameters in front of the target surface. For better understanding, the gradient (along “z” direction) of tangential magnetic fields on three different locations (Figure 6.2(a and b)) on the top of the conventional pack and TriPack V300 target surfaces are plotted against the distance from the target surface (Figure 6.4(a & b)). TriPack V300 consists of three race tracks, where the inner and outer racetracks have tangential field directed (red color) in opposite direction to the middle race track (blue color). In case of TriPack V300, location 1 corresponds to inner race track, location 2 corresponds to middle race track and location 3 corresponds to outer race track.

![Figure 6.2: (a) Magnitude of the tangential magnetic field distribution above 0.25” target surface in the conventional magnet pack with $B_r$ and $B_z$ components, Color legend is in Gauss. Numbered lines correspond to locations on Figure 6.3 (a). Since this magnet pack is cylindrically symmetric, 2D axisymmetric simulation results are presented, (b) Magnitude of the tangential magnetic field distribution above 0.25” target surface in the TriPack V300 magnet pack with $B_r$ and $B_z$ components, Color legend is in Gauss. Since this magnet pack is cylindrically symmetric, 2D axisymmetric simulation results are presented. Numbered lines correspond to locations on Figure 6.3 (b).](image-url)
Figure 6.3: (a) Magnitude of tangential magnetic field at various distances from the conventional target surface on the three locations corresponding to Figure 6.2 (a), (b) Magnitude of tangential magnetic field at various distances from the TriPack V300 target surface on the three locations corresponding to Figure 6.2 (b).

Figure 6.4: (a) Gradient of tangential magnetic field at various distances from the conventional target surface on the three locations corresponding to Figure 6.2 (a), (b) Gradient of tangential magnetic field at various distances from the conventional target surface on the three locations corresponding to Figure 6.2 (b).

Figure 6.3(a) is the plot of tangential magnetic field on the top of the conventional magnetron target surface on the three locations and Figure 6.3(b) is the tangential magnetic field on the top of TriPack V300 on the inner, middle and outer race tracks. It can be observed from Figure 6.4(a) that the conventional magnet pack has tangential magnetic field gradients of several hundred Gauss per inch in the region above the target. Although the gradients are low in the region (location 2) above the race track, the magnitude of the tangential magnetic field is ~430Gauss (Figure 6.3(a)). Whereas in the case of TriPack V300, the tangential magnetic field gradients from all the three race tracks in TriPack V300 are much higher and falls sharply (Figure 6.4(b)) compared to the gradients from the conventional pack (Figure 6.4(a)). The steep gradients in TriPack
V300 provides faster plasma leak into regions with lower magnetic field. The electrons escape from this region due to smaller tangential magnetic field. The low magnetic field region contains plasma electron that are no longer trapped above the target surface. These electrons follow the open magnetic field lines and ions now follow these electrons (that are no longer trapped) resulting in higher deposition rates. This argument is further supported by the measured ionization fraction in the TriPack V300 compared to conventional pack in HiPIMS.

Since the magnetic field from the “ε” magnet pack is asymmetric, the magnetic field gradients (Figure 6.5(b)) from six different locations on the right side of Figure 6.5(a) are plotted at different distances from the target surface. Figure 6.5(a) is a plot of tangential magnetic distribution on “ε” magnet pack. It can be seen from Figure 6.5(a) that the outer shell (location 6) has a shape of strongly unbalanced magnetron configuration and the inner racetracks have tangential field directed in opposite directions. The blue color in Figure 6.5(a) represents magnetic field opposite in direction to red color. The tangential field gradients along the six locations are shown in Figure 6.5(b).

Figure 6.5: (a) Magnitude of the tangential magnetic field distribution above 0.125” target surface in the “ε” magnet pack with Bx and Bz components. Color legend is in Gauss. Numbered lines correspond to locations on Figure 6.5(b), (b) Gradient of tangential magnetic field above the target at different locations.
It can be noted from Figure 6.5(b) that there is very small or no magnetic field gradient in the regions with lower tangential magnetic field and the tangential magnetic field gradient in the outer shell is very sharp. The plasma dynamics in such an asymmetric pack is complicated and the higher deposition rates in this pack could be due to combination of sharp tangential magnetic field gradients along the race track as well as along the “z” direction (Figure 6.5(b)) which enhances electron leakage that in turn facilitates increased ion flow from the trap thereby increasing the deposition rates on the substrate. The TriPack V300 design is based on “ε” pack and therefore, they have similar diffusion co-efficient which is explained in detail in the following section.

6.1.2 Diffusion across Magnetic Field

The Debye length and sheath thickness for TriPack V300 and conventional pack HiPIMS plasma can be calculated from equations 6.1 and 6.2.

\[ \lambda_D = \sqrt{\frac{\varepsilon_0 kT_e}{n_e e^2}} \quad 6.1 \]

\[ S_{\text{child}} = \frac{\sqrt{2}}{3} \lambda_D \left( \frac{2eV_0}{kT_e} \right)^{\frac{3}{4}} \quad 6.2 \]

where \( T_e \) is the electron temperature and \( V_0 \) is the discharge voltage. The child sheath formula cannot be applied in the case of HiPIMS due to the presence of magnetic field and non-Maxwellian electrons but can be used for order of magnitude estimate[115]. For an electron temperature of 5eV and discharge voltage of 600V, the Debye length is \(~5.2 \times 10^{-6} m\) and the sheath thickness is roughly estimated to be \(~10^{-3} m\). Therefore, the pre-sheath thickness can be assumed to be \(~1-2cm\). By taking into account the acceleration of ions in the pre-sheath, ion energy is roughly assumed to be \(~2.5eV\). The working
pressure for both magnet packs is 10mTorr. Hence, the neutral density can be estimated from equation 6.3[116].

\[
n_n = 3.13 \times 10^{13} p(\text{mTorr}) \text{cm}^{-3}
\]

For the working gas pressure of 10mTorr, \(n_n=3.3\times10^{14}\ \text{cm}^{-3}\). During the HiPIMS process, the sputtered atoms collide with the working gas which leads to heating and expansion of the working gas. This effect is known as gas rarefaction and the background gas temperature increases from ~300K to 600K during this process in the HiPIMS discharge [117]. At 600K and 10mTorr, the neutral density is estimated to be ~ \(n_n=1.65\times10^{14}\ \text{cm}^{-3}\).

Particles (electrons and ions) can move across the magnetic field along the gradients when there are collisions. In order to roughly estimate the diffusion co-efficient across the magnetic fields, the following assumptions are made

1. The working pressure for both packs is 10mTorr.
2. The neutral density taking in to account the gas rarefaction process is \(n_n=1.65\times10^{14}\ \text{cm}^{-3}\).
3. The collision frequency is driven by ion-neutral, electron-neutral, ion-ion, electron-electron, and electron-ion collisions. The electron density \((n_e)\) measured from the TLP experiments is \(\sim 10^{13}\ \text{cm}^{-3}\) and the neutral gas density is \(1.65\times10^{14}\ \text{cm}^{-3}\), which means the largest contributing collision process will be electron-neutral and ion-neutral collision and they will dominate the diffusion co-efficient. Hence, only these two processes will be considered in diffusion co-efficient calculations.
4. The electron energy is assumed to be 5eV (TLP measurements) and the energy of the ions are assumed to be \(\sim 2.5\text{eV}\) taking in to account the acceleration in the plasma pre-sheath.
The diffusion co-efficient across the magnetic field is calculated from equation 6.4[116].

\[ D_{\text{perp}} = \frac{r_L^2}{\tau} \]  \hspace{1cm} 6.4

Here, \( r_L \) is the Larmor radius and \( \tau \) is the mean time between collisions. The Larmor radius can be calculated from equation 6.5.

\[ r_L = \frac{mv}{Bq} \]  \hspace{1cm} 6.5

\[ \tau^{-1} = n_n \sigma v \]  \hspace{1cm} 6.6

\[ D_{\text{perp}} = \frac{\lambda v_{\text{avg}}}{2} \]  \hspace{1cm} 6.7

\[ \lambda = \frac{0.061 \, m}{p[mTorr]} \]  \hspace{1cm} 6.8

m is the mass of the particle, \( v \) is the velocity component perpendicular to the direction of magnetic field, B is the magnetic field, q is the electronic charge, \( \sigma \) is the collision cross-section, \( \lambda \) is the mean free path [118] and \( v_{\text{avg}} \) is the average speed. If the magnetic field \( B \to 0 \), then \( r_L \to \infty \). When \( r_L \geq \lambda \) (mean free path), the diffusion is no longer controlled by magnetic field, therefore the formula for diffusion co-efficient across the magnetic field becomes,

\[ D_{\text{perp}} = \frac{\lambda^2}{\tau} \]  \hspace{1cm} 6.9

From the above equations, the electron diffusion across the magnetic field due to electron-neutral (electron-argon) collision process is calculated along the three locations shown in Figure 6.2(a and b) for conventional and TriPack V300. It would be more accurate to consider electron-metal collisions but due to unavailability of collision cross-sections data, electron-argon collision process is considered.
Figure 6.6: (a) Electron diffusion co-efficient across the magnet field at various distances from the target surface on the three locations corresponding to Figure 6.2 of conventional pack, (b) Electron diffusion co-efficient across the magnet field at various distances from the target surface on the three locations corresponding to Figure 6.6 of TriPack V300, (c) Comparison of electron diffusion co-efficient from conventional pack location 2 and TriPack V300 middle race track.
Figure 6.6(a) is a plot of electron diffusion co-efficient across the magnetic field corresponding to the three locations on the conventional magnet pack, Figure 6.6(b) is a plot of electron diffusion co-efficient across the magnetic field corresponding to the three locations on the TriPack V300 and Figure 6.6(c) is the comparison plot of electron diffusion co-efficient across the magnetic field for conventional pack location 2 and TriPack V300 middle race track.

It can be observed from Figure 6.6(a and b) that the electron diffusion co-efficient ($D_{\text{perp}}$) across the magnetic field at different distances from the target surface on the three locations of the TriPack V300 rises very steeply than the conventional magnet pack. In reality, the flat portion on these curves is not really a hard straight line as shown in the Figure 6.6-6.9 and this region still has some magnetic field influence on the particles. Since the location of the middle race track of the TriPack V300 and location 2 of the conventional magnet pack are about the same on the target surface, their electron $D_{\text{perp}}$ across the magnetic field are compared in Figure 6.6(c). It can be seen from Figure 6.6(c) that $D_{\text{perp}}$ in the middle race track of TriPack V300 increases much more steeply and reaches a value 652.8 m$^2$/s (diffusion co-efficient when $r_L = \lambda$) at 0.6” from the target surface whereas the conventional pack reaches the same value only at 1.7” from the target surface. This clearly indicates that the plasma diffuses along the steep magnetic field in the TriPack V300. Figure 6.7 is the comparison of ambipolar diffusion co-efficient from TriPack V300 middle race track with and without taking gas rarefaction into account. The calculated ambipolar diffusion co-efficient considering the gas rarefaction process is twice higher compared to the ambipolar diffusion co-efficient that is calculated without taking into account the gas rarefaction process.
The ambipolar diffusion co-efficient across the magnetic field can be calculated from the following equation [119]

\[
D_{ambi} = \frac{2D_{perp}^{ions}D_{perp}^{elect}}{D_{perp}^{ions} + D_{perp}^{elect}}
\]

\[6.10\]

\(D_{perp}^{elect}\) is the diffusion co-efficient of electrons across the magnetic field and it was calculated earlier for Figure 6.6. \(D_{perp}^{ions}\) is the diffusion co-efficient of ions across the magnetic field and it can be calculated from equation 6.9. For calculating \(D_{perp}^{ions}\), ion-neutral (argon ion-argon) cross section was used.
Figure 6.8: Comparison of ambipolar diffusion coefficient from conventional pack location 2 and TriPack V300 middle race track.

Figure 6.9: Comparison of average plasma diffusion speed from conventional pack location 2 and TriPack V300 middle race track.
Figure 6.8 is the comparison plot of ambipolar diffusion co-efficient ($D_{ambi}$) versus distance from the target surface along location 2 of conventional pack and middle race track of TriPack V300. At around 0.5” from the target surface, $D_{ambi}$ on the conventional magnet pack (location 2) is 3.3 m$^2$/sec and the $D_{ambi}$ on the middle race track of TriPack V300 is 5.5 m$^2$/sec. This means the ambipolar diffusion co-efficient in the case of TriPack V300 middle race track is 1.6 times more than the conventional magnet pack location 2 at 0.5” from the target surface. This clearly points to much higher plasma diffusion from TriPack V300 compared to the conventional pack. Figure 6.9 is the average plasma diffusion speed for TriPack V300 and conventional magnet pack calculated from equation 6.7. It is evident from this plot that average plasma diffusion speed of the conventional pack is only ~60% of TriPack V300 at 0.5” from the target surface.

To conclude, the observed increase in the deposition rates in TriPack V300 is the result of sophisticated plasma dynamics arising due to the unique magnetic field configuration in TriPack V300.

6.1.3 Particle Flux Transport Model

A. Anders et al. [57] has shown that the fluxes involved in the deposition by HiPIMS technique under conditions when the plasma is dominated by metal sputtered from the target can be described as shown in Figure 6.10(a). In Figure 6.10(a), $\alpha$ is the ionization probability in the plasma, $\beta$ is the probability of ions to return to the target and $\gamma$ is the sputter yield. The value of $\alpha$ and $\beta$ depend on the target material, HiPIMS system and discharge parameters.
Based on Figure 6.10(a), $\alpha$ and $\beta$ can be calculated from the following equations [57],

$$\phi_{a,\text{sub}} = (1 - \alpha)\phi_{a,\text{sputtered}}$$  \hspace{1cm} 6.11

$$\phi_{i,\text{sub}} = \alpha(1 - \beta)\phi_{a,\text{sputtered}}$$  \hspace{1cm} 6.12

where $\phi_{a,\text{sub}}$ is the atom flux to the substrate, $\phi_{i,\text{sub}}$ is the ion flux to the substrate and $\phi_{a,\text{sputtered}}$ is the atom flux from the target. For the conventional magnet pack with an aluminum target, $\alpha$ and $\beta$ can be calculated based on the deposition rates and discharge current during 10mTorr HiPIMS operation. The peak discharge current in the case of conventional pack is 150A, therefore $9.4 \times 10^{20}$ ions/sec reach the target. By multiplying the sputter yield (1.267) corresponding to the discharge voltage of 850V with the number of ions/sec, the number of atoms/sec that comes out of the target can be estimated. In the conventional magnet pack case, $1.2 \times 10^{21}$ atoms/sec comes out of the target. The conventional pack erosion area is calculated to be $\sim 38.70 \text{cm}^2$. Therefore, the target neutral flux $\phi_{a,\text{sputtered}}^{\text{con}}$ is $3.1 \times 10^{19}$ atoms/cm$^2$/sec. The measured aluminum deposition rate at the substrate for 500W HiPIMS average power is 6Å/s. Based on the deposition rate and density of aluminum, the total flux at the substrate is calculated to be $3.6 \times 10^{15}$
atoms/cm$^2$sec. From the ion fraction experiments, the ion flux to the substrate in the case of conventional pack is ~2%. Therefore, the ion flux to the substrate $\phi_{i,sub}^{con}$ is $7.2 \times 10^{13}$ atoms/cm$^2$sec. The $\alpha$ and $\beta$ values can be calculated from the following equations which takes the geometric solid angle in to account.

\[
\phi_{a,sub}^{con} = (1 - \alpha)\phi_{a,sputtered}^{con} \Omega 
\]

\[
\phi_{i,sub}^{con} = \alpha (1 - \beta)\phi_{a,sputtered}^{con} \Omega 
\]

The geometric solid angle calculated based on the QCM area and the throw distance is $8 \times 10^{-4}$ rad. The neutral flux to the substrate $\phi_{a,sub}^{con}$ is $\cdot98 \times 3.6 \times 10^{15}$ atoms/cm$^2$sec. Therefore, $\alpha = 0.858$ and $\beta = 0.997$.

In the case of TriPack V300, the peak current is 12A, therefore $7.5 \times 10^{19}$ ions/sec reach the target. Based on sputter yield and number of ions/sec, $9.5 \times 10^{19}$ atoms/sec comes out of the target. The TriPack erosion area is $\sim 51.61$cm$^2$. Therefore, the target neutral flux $\phi_{a,sputtered}^{tri}$ is $1.8 \times 10^{18}$ atoms/cm$^2$sec. The measured aluminum deposition rate at the substrate for 500W HiPIMS average power is $9\AA$/s. Therefore, the total flux at the substrate is $5.4 \times 10^{15}$ atoms/cm$^2$sec. From the ion fraction experiments, the ion flux to the substrate in the case of TriPack V300 is $\sim 16\%$. Therefore, the ion flux to the substrate $\phi_{i,sub}^{tri}$ is $8.6 \times 10^{14}$ atoms/cm$^2$sec. The $\alpha$ and $\beta$ values are calculated from the following equations taking the geometric solid angle in to account.

\[
\phi_{a,sub}^{tri} = (1 - \alpha)\phi_{a,sputtered}^{tri} \Omega 
\]

\[
\phi_{i,sub}^{tri} = \alpha (1 - \beta)\phi_{a,sputtered}^{tri} \Omega 
\]

Using the same geometric solid angle of $8 \times 10^{-4}$ rad and the neutral flux to the substrate $\phi_{a,sub}^{con} = \cdot84 \times 5.4 \times 10^{15}$ atoms/cm2sec, the ionization probability $\alpha$ is calculated to be
-2.5 which means the flux model proposed by A. Anders et al. [57] does not work in case of TriPack V300 because \(0 \leq \alpha \leq 1\) condition is not satisfied.

Since the magnetic field profile of TriPack V300 is very different from the conventional pack, a modified flux model as shown in Figure 6.10(b) is used. In Figure 6.10(b), \(\delta\) is the fraction of neutral atoms that are ionized en route to the target. \((1 - \alpha)\) corresponds to the neutrals that are not ionized by the highly confined plasma, \((1 - \alpha)\)*(1 - \(\delta\)) corresponds to the actual neutrals reaching the substrate. Based on this modified flux model, \(\alpha\), \(\beta\) and \(\delta\) can be calculated from the following equations.

\[
\phi_{\text{a,sub}}^{\text{tri}} = (1 - \alpha)(1 - \delta)\phi_{\text{a,spattered}}^{\text{tri}}\Omega P
\]

\[
\phi_{i,\text{sub}}^{\text{tri}} = [\alpha(1 - \beta) + \delta(1 - \alpha)]\phi_{\text{a,spattered}}^{\text{tri}}\Omega P
\]

Where \(\Omega\) is the geometrical solid angle and \(P\) is the peaking factor for flux from TriPack V300. The peaking factor takes in to account the non-uniform flux distribution from this pack. By substituting the values of \(\phi_{\text{a,sub}}^{\text{tri}}, \phi_{i,\text{sub}}^{\text{tri}}, \phi_{\text{a,spattered}}, \Omega\) in above equations, the following equations are obtained.

\[
3.125 = (1 - \alpha)(1 - \delta)P
\]

\[
.59 = [\alpha(1 - \beta) + \delta(1 - \alpha)]P
\]

Based on the diffusion speed comparison of both magnet packs from section 6.1.2, average plasma diffusion speed of TriPack V300 is 1.6 times the average plasma diffusion speed of conventional magnet pack. Hence, we assume that

\[
(1 - \beta_{\text{tri}}) = 1.6(1 - \beta_{\text{con}})
\]
From equation 6.21, $\beta_{tri} = .995$. By dividing equation 6.19 by equation 6.20, $\delta$ is calculated to be 0.158. Substituting $\delta = 0.158$ in equation 6.19, the following equation is obtained.

$$3.7 = (1 - \alpha)P$$

6.22

The value of $P$ can be estimated from the following equation.

$$P \leq \frac{\phi^{tri}_{total,sub}}{\phi^{tri}_{a,sputtered} * \Omega}$$

6.23

where, $\phi^{tri}_{total,sub}$ is the total flux arriving at the substrate in the TriPack V300 case. Based on equation 6.23, $P \leq 3.75$. By substituting $P_{max} = 3.75$ in 6.22, $\alpha$ is found to be 0.013.

Table 6.1 is the flux parameter comparison of TriPack V300 and conventional pack.

<table>
<thead>
<tr>
<th>Parameter</th>
<th>Conventional Pack</th>
<th>TriPack V300</th>
</tr>
</thead>
<tbody>
<tr>
<td>$\alpha$</td>
<td>$0.858 \pm 0.086$</td>
<td>$0.013 \pm 0.004$</td>
</tr>
<tr>
<td>$\beta$</td>
<td>0.997 +0.003 -0.199</td>
<td>0.995 +0.005 -0.199</td>
</tr>
<tr>
<td>$\delta$</td>
<td>0</td>
<td>0.158 +0.032</td>
</tr>
</tbody>
</table>

Table 6.1 Flux parameter comparison of conventional and TriPack V300.
In the case of conventional magnet pack, $\alpha = 0.858$, which means the ionization in the highly confined plasma region is very high. Almost all the ions that are generated in this highly confined plasma region come back to the target because of its high $\beta$ (0.997) value and this is also evident from the ~2% ion fraction measured at the substrate. Since all the ions are returned back to the target, $\delta=0$ in this case. The high value of discharge current (150A) is due to the huge contribution from the metal ion return effect and higher ionization probably in the highly confined plasma region.

In the case of TriPack V300, $\beta = 0.995$ which means all the ions that are produced in the narrow highly confined plasma region (Figure 6.11) comes back to the target. The low value of $\alpha$ indicates lower ionization in the highly confined plasma region, which is evident from low discharge current (12A) measured in this case. This does not mean that TriPack V300 is not operating in the HiPIMS mode. The measured electron density in this case is $\sim 5 \times 10^{18}$ m$^{-3}$, which is a prominent feature of HiPIMS discharges. The unconfined plasma region in the case of TriPack V300 extends farther and this region helps to ionize the neutrals from the target en route to the substrate leading to higher ion flux at the substrate. The value of $\delta$ (probability of neutrals that are ionized in the
unconfined plasma region) was calculated to be 0.158. Some of the neutrals from the target are ionized in the unconfined plasma region of the TriPack V300 and reach the substrate and this is evident from the 16% ion fraction measured at the substrate.

Hall thruster’s ExB configuration is very similar to magnetron sputtering configuration. In Hall thrusters, axial electric and radial magnetic fields are applied in an annular channel set-up. The electrons in this region experience closed ExB drift similar to magnetron sputtering devices. Due to the lower electron mobility across the magnetic field, the axial electric field is maintained so the electrons ionize the propellant gas and in such a condition, the electric field accelerates ions in the quasineutral plasma [120]. The TriPack V300 HiPIMS operation shows a strong resemblance to Hall thrusters. The peaking factor (P) of this magnet pack is 3.75 which indicates that the flux distribution from this pack is non-uniform. Moreover, the deposition flux consists of more ions compared to the conventional magnet pack. In the TriPack V300, the counter rotating race tracks can give raise to Hall thruster-like effect where the ions can be accelerated towards the substrate.

6.2 Absence of Ionization Zones in TriPack V300 HiPIMS

Some of the most important observations from the ICCD experiments are as follows

1. TriPack V300 in HiPIMS discharges doesn’t show the presence of “ionization zones” like the conventional magnet pack.

2. The HiPIMS plasma from TriPack V300 is homogeneous in all the three race tracks.

3. “Ionization zones” were not detected even when discharge was ignited on only one race track at a time.
The magnetic field change due to ExB drift currents in TriPack V300 was modeled in COMSOL Multiphysics by assuming the region on the top of the race track as a current carrying wire. The drift current is assumed to be roughly ~6x the discharge current[7]. In the case of TriPack V300, there was no change in the magnetic field profile due to the ExB drift currents.

It is very clear from all the above observations that the physics of HiPIMS is very complex in nature because the plasma evolves in time and volume during the sputtering process. This means the composition of the plasma, electron temperatures, ambient magnetic field and the spatial electric potential change over time. Due to the transient nature of the HiPIMS plasma, there are multiple coupled effects behind the absence of “ionization zones” in the TriPack V300 and it requires more detailed computational and experimental efforts to estimate the contribution from each of these coupled effects. Few of the possible reasons for the absence of “ionization zones” are discussed in this section.

Also, a model to predict the critical current density needed to create ionization zones is presented below.

### 6.2.1 Critical Current Density Model

From the literature, ionization appears if all the gas is ionized in a given location. Therefore,

\[
\frac{dn_{\text{ionization of gas}}}{dt} = n_e n_g (\sigma v)_{\text{ionization of gas by electrons}}
\]

where \(n_{\text{ionization of gas}}\) is density of ionized gas in the ionization zone. The rate at which the gas is ionized depends on the electron density \(n_e\), neutral gas density \(n_g\), electron-
atom ionization cross-section ($\sigma$) and the average velocity of the electrons ($v$). The density of the ionized gas can be written as

$$n_{ionization\ of\ gas} = n_e n_g \tau_{ionization\ of\ gas\ by\ electrons}$$  \hspace{1cm} 6.25

where $\tau$ is the neutral gas refill time which is the time for new gas to diffuse into ionization zone. Critical condition for “ionization zones” to form is when

$$n_{ionization\ of\ gas} = n_g.$$  

So,

$$1 = n_e \tau_{ionization\ of\ gas\ by\ electrons}$$  \hspace{1cm} 6.26

The electron density can be written (equation 6.27) in terms of current density ($J$), sputter yield ($Y$), ionization probability in the highly confined plasma ($\alpha$), gas refill time ($\tau$), $V$ is the speed of sputtered atoms and $\langle \sigma v \rangle_{ionization\ of\ gas\ by\ electrons}$.

$$n_e = n_{sputtered\ atom} * \alpha = J Y \alpha \frac{1}{V}$$  \hspace{1cm} 6.27

Substituting equation 6.26 in equation 6.27 leads to equation 6.28.

$$1 = J Y \alpha \frac{1}{V} \tau_{ionization\ of\ gas\ by\ electrons}$$  \hspace{1cm} 6.28

Equation 6.28 is the condition for “ionization zones” to occur. The equation for determining the critical current density for “ionization zones” to appear can be written as

$$J_{critical} = \frac{V}{Y \alpha \tau_{ionization\ of\ gas\ by\ electrons}}$$  \hspace{1cm} 6.29

By substituting the appropriate values on the RHS of equation 6.29, critical current densities for conventional pack and TriPack V300 are calculated to be $J_{critical}^{con} = 0.050 A/cm^2$ and $J_{critical}^{tri} = 8.7 A/cm^2$. Table 6.2 shows the comparison of experimental target density and simulated critical current density for both the magnet packs. The
critical density for “ionization zones” to occur in TriPack is much higher than the conventional pack because of its lower $\alpha$ and smaller $\tau$ compared to conventional pack. The ratio of the critical densities of the two magnet packs is

$$\frac{f_{\text{critical}}^{\text{con}}}{f_{\text{critical}}^{\text{tri}}} = 0.006$$

6.30

The value of critical density in the TriPack V300 is much higher compared to conventional pack. From Table 6.2, the experimental target current density of TriPack V300 is more than a magnitude below the threshold to form “ionization zones”. Hence, no ionization zones were observed during TriPack V300 operation. On the other hand, the current density of the conventional pack is around 75 times bigger than the critical density required for the ionization zones to be formed. Therefore ionization zones formation should be observed and that is in agreement with the experimental observation in section 4.3.1.

<table>
<thead>
<tr>
<th>Magnet Pack</th>
<th>Experimental target current density (A/cm$^2$)</th>
<th>Theoretical critical current density (A/cm$^2$)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Conventional</td>
<td>3.9±0.4</td>
<td>0.050±0.005</td>
</tr>
<tr>
<td>TriPack V300</td>
<td>0.23±0.02</td>
<td>8.7±2.6</td>
</tr>
</tbody>
</table>

**6.2.2 Reasons for the absence of “ionization zones” in TriPack V300**

**6.2.2.1 Gas Refill Process**

During TriPack V300 HiPIMS operation, the intensity from the individual race tracks changes over time. It should be noted that the light intensity from each race track were independent of each other and the individual intensities varies independent of each other at different times. This clearly indicates the influence of gas refill process in the race
tracks. The dynamics of gas refill process in these race tracks and the plasma escape time in this pack can give rise to a regime where there is always enough neutrals to ionize. This can lead to a situation without “ionization zones”. The average plasma diffusion speed in the case of TriPack V300 middle race track is 1810 m/s at 0.5” from the target surface whereas in the case of conventional pack it is only 1110 m/s. The plasma escape time is a function of the magnetic field profile which in case of the TriPack V300 is very different from the conventional magnet pack.

### 6.2.2.2 Electric Field Shear

The electric field shear that are observed in large fusion devices is responsible for plasma stabilization on boundary between the plasma and a wall[121]. The electric field shear may be responsible for HiPIMS plasma stabilization in the case of TriPack V300. Previous studies on HiPIMS suggests that for a copper target at higher currents, the discharge shifts to a regime without “ionization zones”[79, 80]. The TriPack V300 was not operated at high currents but the magnetic field configuration was very different from the conventional magnetic field configuration. Hence, from our observations, plasma stabilization can occur at smaller discharge currents depending on the magnetic field configuration. The three race tracks in TriPack V300 are much narrower than the conventional pack but the magnitude of the radial magnetic field is much higher. The gradient of electric field above the target surface on both the magnet packs is very different and this shear in TriPack V300 can lead to stabilization in HiPIMS.
6.2.2.3 Local Effect

The “ionization zones” disappear for certain target materials at certain conditions. There have been conflicting observations on transition to zone free HiPIMS on different target materials at different discharge currents[79, 122]. The HiPIMS current density itself is a function of sheath-presheath thickness, cathode material, discharge voltage, working gas pressure, and magnetic field topology. Therefore, the transition to zone-free HiPIMS are different for different target materials at different operating conditions due to the huge role played by the specifics of the magnetron set-up like the magnetic field strength, degree of field balance, etc [79]. The stabilization of “ionization zones” in titanium TriPack V300 could be due to the combination of magnetic field topology, argon pressure and target materials which are all local effects. The physics of plasma-surface interaction between the HiPIMS plasma made from the target material and the surface of the target itself can leads to other effects that were not observed during TriPack V300 experiments.

6.2.2.4 Sheath and Pre-sheath Spatial Distribution

The structure of the pre-sheath and the thickness of the sheath are complicated by the crossed electric and magnetic fields[7]. The rotating “ionization zones” in magnetrons are caused by the spatial extent of the potential distribution. The sheath and pre-sheath evolve their size and magnitude during single HiPIMS pulse. In HiPIMS, the sputtered material is ionized close to the target and the negative potential applied on the target can extend far into the plasma as an extended pre-sheath. In some unbalanced magnetron cases, 10–20% of the total applied voltage drops in the magnetic pre-sheath, which can extend to even 40 mm from the target surface [115]. The transition from zone-free mode
to stable homogeneous plasma mode can be caused through an increase in discharge current or magnetic field modification. The magnetic field configuration in turn affects the plasma sheath and pre-sheath potential distribution, which then can lead to stabilization of the “ionization zones”.

As stated earlier, combination of all the above mentioned coupled effects leads to zone free HiPIMS in case of TriPack V300. Understanding the origin and propagation of these instabilities not only helps in understanding the physics of HiPIMS but also have a broader impact in the field of Hall thrusters[123, 124]and arc discharges.
Chapter 7 Conclusions and Future Work

7.1 Conclusions

The demand for high quality, high performance coatings have increased rapidly due to their application in a wide variety of industries. Microelectronics, automotive, tooling, and medical devices industries, etc. suffers due to the high costs incurred on improving the performance of the coating process. In the recent years, the advancement in magnetron sputtering technology has made huge impact especially in areas like low friction, wear-resistant, corrosion resistant and hard coatings. Over the last few years, HiPIMS technology has revealed its tremendous potential in producing high quality, high performance coatings that have not yet been achieved by conventional magnetron sputtering technology. Commercial implementation of this technology has always been a problem due to its low deposition rates and high equipment cost.

In this dissertation, an optimized magnetic field design has been developed for HiPIMS discharges that are capable of giving higher deposition rates than conventional magnetic field configuration. The “Spiral” magnetic field design from the 14” Galaxy magnetron did not work for 4” magnetron gun. The current density from 4” “Spiral” magnet pack saturates at 18mA/cm² and it then decreases with increase in the discharge voltage. The “Spiral” pack doesn’t not allow HiPIMS mode of operation. The electron trajectory simulation from COMSOL Multiphysics indicated that “Spiral” pack had very poor electron recycling as a result of electron loss due to open field lines and sharp gradients in this pack. All these observations indicate that scaling magnetic packs proportionally doesn’t necessarily work for all designs.
The “ε” magnet pack design that was developed as a part of the magnetic field optimization effort was able to achieve higher deposition rates in HiPIMS compared to conventional magnetic field design. The “ε” magnet pack design consists of outer circular area and an inner “ε” shaped area. The “ε” magnet pack design combines advantages of conventional magnetron magnet pack discharge stability with better film characteristics of HiPIMS discharges without compromising on the deposition rates. At an average power of 500W, the deposition rate of “ε” magnet pack with an aluminum target at 10mTorr was 8.8±0.4Å/s whereas the deposition rates with the conventional pack HiPIMS was 4.1±0.2 Å/s and with conventional pack DC was 12.1±0.6 Å/s for the same experimental conditions (average power, target material and pressure). It is inferred from the “ε” pack experiments that proper selection of magnetic field strength and configuration is critical to achieve a sustainable high-current pulsed-discharge mode. The “ε” magnet pack gives higher deposition rates in HiPIMS but their substrate uniformity can be improved further.

The TriPack V300 magnet pack design that was developed based on the design solutions from “ε” magnet pack design consists of three circular race-tracks (inner, middle and the outer). The inner and the outer racetracks have tangential field directed in opposite direction to the middle race track. In the case of titanium at 13mTorr and 500W average power, TriPack V300 gave higher deposition rates in HiPIMS than conventional DC and HiPIMS. With a carbon target (at 500w,13mTorr), TriPack V300 gave higher deposition rates in HiPIMS than conventional HiPIMS and the TriPack V300 HiPIMS deposition rates were comparable to conventional DC and with an aluminum target (at 500w,13mTorr), TriPack V300 HiPIMS gave higher deposition rates than conventional
HiPIMS. A very important observation from the TriPack experiments is that the deposition rates can varied by just changing the pulsing parameters (Voltage, pulse time, frequency) for the same average power in the TriPack V300. This effect was not noticeable in the case of conventional magnet pack. In the case of copper, the deposition rate in HiPIMS discharge can be increased twofold by increasing the pulse time by a factor of 2 and decreasing the pulsing frequency but keeping the discharge voltage constant. Generally, lowest deposition rates were obtained with a low repetition rate and longer pulses, and higher deposition rates were obtained with high repetition rates and very short pulses. The films that were deposited using TriPack V300 magnet pack had much smaller grains compared to conventional pack DC and HiPIMS films. TriPack V300 was able to attain better substrate uniformity that “ɛ” magnet pack.

ICCD observation of TriPack V300 showed that the plasma was homogenous throughout all the three race tracks without any “ionization zones”. The “ionization zones” were not detected even when only one race track was ignited at a time. To understand the dependence of “ionization zones” on race track widths, the race track widths were varied but the radial magnetic field magnitude was kept the same on the conventional magnet pack. It was observed from these experiments that (1) narrower the race track width for the same radial magnetic field magnitude, later the “ionization zones” are formed, (2) lower the magnitude of radial magnetic field, later the “ionization zones” are formed, (3) steeper the gradients of radial magnetic field in the “Z” direction, later the “ionization zones” are formed. An erosion model that can predict the target erosion in magnetron sputtering systems was implemented in COMSOL Multiphysics. It was found that TriPack V300 gives around the same target utilization as the conventional magnet pack.
7.2 Future Work

The main focus of the future work on TriPack V300 will be to find the figure of merit of the HiPIMS films deposited with this new magnetic field configuration. This will include X-Ray Diffraction (XRD) analysis of the HiPIMS deposited films to study the crystal structure, understand the role of metal re-ionization from the material analysis, cross-section TEM analysis of the HiPIMS thin films to understand the lattice structure variation due to the impinging ions and also measure the deposition rate as function of time and correlate it to the ICCD and probe observations.

Commercial implementation of TriPack V300 is very difficult as it requires magnetic inserts and specially eroded targets for operation. In order to overcome the problem of lower fields, TriPack V4 was designed with larger magnets and also the magnetic elements (Nickel) were embedded in to the cooling well of the magnetron gun to increase the radial magnetic field as opposed to having magnetic inserts embedded in to the target. Hence, TriPack V4 will work only with this specific magnetic cooling well design.

Figure 7.1(a) shows the magnetic field parallel to TriPack V4 target surface and Figure 7.1(b) shows the streamline plot of $B_x$ and $B_z$ components from TriPack V4. Like the TriPack V300, TriPack V4 also has three race tracks with the inner and outer race tracks tangential magnetic field directed opposite in direction to the middle race track. Figure 7.1(c) is the 2D axisymmetric electron trajectory at 1000ns from TriPack V4 computed in COMSOL Multiphysics.
Future work on TriPack V4 would include the following tasks:

1. Deposition rate measurements from TriPack V4 for different target materials in dcMS and HiPIMS discharges. Measurement of deposition rates includes the measurement along the sides of the magnetron gun to see how much material is transported sideways.

2. Perform plasma diagnostics (Langmuir probe, optical spectroscopy, ion fraction and ICCD camera) on TriPack V4 to understand the behavior of high current pulsed discharges in the magnet pack.

3. Material characterization (stress, SEM, XRD, etc.) of the films deposited using this magnet pack.

4. Inject gas on to each race track and study the ionization zones to understand the influence of gas rarefaction.
5. Develop a plasma model to explain the absence of ionization zone in TriPack V300.

6. Obtain electric potential profile in the direction perpendicular to the wall as a function of space and time, to determine the role E-field shear stabilization at increasing levels of plasma current by Langmuir and emissive probe arrays.

7. Measure sheath and pre-sheath spatial dimension with probe array to understand the stabilization of HiPIMS discharges.
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