Fundamentals of High Power Impulse Magnetron Sputtering

Johan Böhlmark
**Cover:**

The cover is based on a picture taken through a view port of a vacuum chamber. The blue light is an Ar/Ti plasma created by a High Power Impulse Magnetron Sputtering (HIPIMS) discharge.

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Abstract
In plasma assisted thin film growth, control over the energy and direction of the incoming species is desired. If the growth species are ionized this can be achieved by the use of a substrate bias or a magnetic field. Ions may be accelerated by an applied potential, whereas neutral particles may not. Thin films grown by ionized physical vapor deposition (I-PVD) have lately shown promising results regarding film structure and adhesion. High power impulse magnetron sputtering (HIPIMS) is a relatively newly developed technique, which relies on the creation of a dense plasma in front of the sputtering target to produce a large fraction of ions of the sputtered material. In HIPIMS, high power pulses with a length of ~100 µs are applied to a conventional planar magnetron. The highly energetic nature of the discharge, which involves power densities of several kW/cm², creates a dense plasma in front of the target, which allows for a large fraction of the sputtered material to be ionized.

The work presented in this thesis involves plasma analysis using electrostatic probes, optical emission spectroscopy (OES), magnetic probes, energy resolved mass spectrometry, and other fundamental observation techniques. These techniques used together are powerful plasma analysis tools, and used together give a good overview of the plasma properties is achieved.

Electrostatic probe measurements reveal a dense doughnut shaped plasma expanding from the erosion zone of the magnetron. The peak plasma density during the active cycle of the discharge exceeds 10¹⁹ electrons/m³. The expanding plasma is reflected by the chamber wall back into the center part of the chamber, resulting in a second density peak several hundreds of µs after the pulse is turned off.

Optical emission spectroscopy (OES) measurements of the plasma indicate that the degree of ionization of sputtered Ti is very high, over 90 % in the peak of the pulse. Even at relatively low applied target power (~200 W/cm² peak power) the recorded spectrum is totally dominated by radiation from ions. The recorded HIPIMS spectra were compared to a spectrum taken from a DC magnetron discharge, showing a completely different appearance.

Magnetic field measurements performed with a coil type probe show significant deformation in the magnetic field of the magnetrons during the pulse. Spatially resolved measurements show evidence of a dense azimuthally E×B drifting current. Circulating currents mainly flow within 2
away cm from the target surface in an early part of the pulse, to later diffuse axially into the chamber and decrease in intensity. We record peak current densities of the E×B drift to be of the order of $10^5$ A/m$^2$.

A mass spectrometry (MS) study of the plasma reveals that the HIPIMS discharge contains a larger fraction of highly energetic ions as compared to the continuous DC discharge. Especially ions of the target material are more energetic. Time resolved studies show broad distributions of ion energies in the early stage of the discharge, which quickly narrows down after pulse switch-off. Ti ions with energies up to 100 eV are detected. The time average plasma contains mainly low energy Ar ions, but during the active phase of the discharge, the plasma is highly metallic. Shortly after pulse switch-on, the peak value of the Ti$^{1+}$/Ar$^{1+}$ ratio is over 2. The HIPIMS discharge also contains a significant amount of doubly charged ions.
Preface

The presented research work was conducted since September 2002 in the Plasma & Coatings Physics Division at IFM, Linköping University, Sweden. It focuses on explaining the fundamental aspects of the high power impulse magnetron discharge. This work has been conducted in parallel with work done by Dr Jones Alami and Dr Martina Lattemann under supervision of Professor Ulf Helmersson. All the experiments have been carried out at IFM, Linköping University, but I also had the pleasure to collaborate worldwide with numerous groups in plasma science and thin film growth. To name a few; the Alfven Institute at KTH Stockholm, Sheffield Hallam University in the UK, the University of Iceland, and the Large Plasma Laboratory at UCLA in California, US. The Alfven Institute, and the Large Plasma Laboratory has helped me with constructing and understanding some of my plasma measurements. The University of Iceland has been involved mostly in data evaluation. The collaboration with the Sheffield Hallam University has been almost continuous since the start, and involves most of my work. Thanks to my partners outstanding knowledge in the field of their research lots of new ideas and explanations arose, and I am very happy for these fruitful collaborations.

The aim of this work has been to better understand the HIPIMS discharge. A deep understanding of the features is necessary for a future development and possible industrialization of the process. This thesis provides a fraction of this understanding, and may found the basis for some future research in the field. During my time as a PhD student I have learnt and discovered many things, but it seems the more you learn, the more you realize how much we do not know.

The investigations presented in this thesis would also not have been possible without the financial support from the Swedish Foundation for Strategic Research, Swedish Science Council, and the European Commission.

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Papers included in the thesis

I. “Ionization of sputtered metals in high power pulsed magnetron sputtering”
J. Bohlmark, J. Alami, C. Christou, A. P. Ehiasarian, and U. Helmersson
J. Vac. Sci. Technol A 23(1) 18 (2005)

II. “Measurement of the magnetic field change in a pulsed high current magnetron discharge”

III. “Spatial electron density distribution in a high-power pulsed magnetron discharge”
J. Bohlmark, J. T. Gudmundsson, J. Alami, M. Lattemann, and U. Helmersson
IEEE transactions on plasma science 33(2) 346 (2005)

IV. “The ion energy distributions and ion flux composition from a high power impulse magnetron sputtering discharge”
Submitted to Thin Solid Films

V. “Investigation of high power impulse magnetron sputtering pretreated interfaces for adhesion enhancement of hard coatings on steel”
M. Lattemann, A. P. Ehiasarian, J. Bohlmark, P. Á. O. Persson, and U. Helmersson
Accepted for publication in Surface and Coatings Technol. (2005)

VI “Guiding the deposition flux in an ionized magnetron discharge”
J. Bohlmark, M. Östbye, M. Lattemann, H. Ljungrantz, T. Rosell, and U. Helmersson
Manuscript in final preparation
Contribution to the included papers

Papers I-IV contains predominately my own work, including data collection, evaluation, and writing. Paper I is based on previous work done by Dr. C. Christou on a different type of plasma. Dr. Christou also helped in evaluating the experiments, and is therefore included as co-author. Paper II was originally suggested by Dr M. VanZeeland, who also helped me designing the probes. A major part of the evaluation and background theory comes from Dr. N. Brenning and Dr. I. Axnäs at the Alfven Institute at KTH, Stockholm. Paper III is work on the plasma dynamics done in parallel with similar work done by Dr J. Alami. The massive amount of data that was collected during the early stages of this paper would not have been possible to acquire and evaluate without the data collection system and evaluation software. The complete plasma measurement setup was constructed by our technician Dr. L. Samuelsson in collaboration with Dr J. Alami and Dr J. T. Gudmundsson. Paper IV contains data collected with an energy resolved mass spectrometer. I made the measurements and evaluated the data. Dr. Y. Gonzalvo at Hiden Analytical UK performed some calculations regarding the instrument, and also helped with the adjustments of the instrument. Paper V is the outcome of a European HIPIMS project. My contribution to this paper is film growth, etching and an active part of the data evaluation. Paper VI is the result of a diploma work performed by M. Östbye in collaboration with Impact Coatings AB. I acted as supervisor for Östbye. Co-authors whose contribution are not specifically pointed out, have contributed with useful discussions and/or corrections. Naturally, significant contributions to all my papers have come from my supervisor Prof. U. Helmersson via discussions, supervision and corrections.
Other related publications (not included)

“The ion energy distributions in a high power impulse magnetron plasma”

“Ionization of sputtered Ti during high power pulsed magnetron sputtering”
J. Bohlmark, J. Alami, J.T. Gudmundsson and U. Helmersson

"Optical emission study of the ionization fractions in a high power pulsed magnetron plasma"
J. Böhlmark, A. P. Ehiasarian, P. E. Hovsepian, and U. Helmersson

“Ion-assisted physical vapor deposition for enhanced film properties on nonflat surfaces”

“Plasma parameters during high power pulsed magnetron sputtering”
J. Alami, J.T. Gudmundsson, J. Böhlmark, K.B. Gyfason, and U. Helmersson

“Influence of pressure and power on the composition and time evolution of plasmas in high power impulse magnetron sputtering”
A. P. Ehiasarian, R. New, P. E. Hovsepian, J. Böhlmark, J. Alami, and U. Helmersson,

“High power impulse magnetron sputtering (HIPIMS) pre-treatment for the deposition of hard coatings”

“Plasma dynamics in a highly ionized pulsed magnetron discharge”
J. Alami, J. T. Gudmundsson, J. Bohlmark, J. Birch, and U. Helmersson

“Structural analysis and growth of beta and bcc-Ta on inclined surfaces by highly ionized magnetron sputtering”
Submitted for publication

“Ionized Physical Vapor Deposition (IPVD): A review of technology and applications”
U. Helmersson, M. Lattemann, J. Bohlmark, A. P. Ehiasarian, and J. T. Gudmundsson,
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Last but certainly not least, my parents, Jan-Erik, and Kristina and my sister Helena, for all the support I have gotten on the long journey towards my PhD.

If I forgot someone who feels that he or she should be specifically pointed out, please contact me and I will buy you a dinner…
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1. Introduction

This chapter gives a short introduction to the field of thin films and how thin films are produced. The aims of this research are presented and an outline of this thesis is given.

1.1 What are thin films?

Thin films allow combining the bulk properties of materials with the surface properties of other. As the demand for high technology products increases, more advanced materials are needed. An efficient, and most often less expensive way to meet the increased demands is the use of thin films. Thin films are material layers with thickness ranging from a few atomic layers (~ nm) to a few micrometers. Thin films as science is quite new, but as art one of the oldest technologies. Decorative gold leafs from ancient Luxor dating from the Eighteenth Dynasty (about 1500 B.C.) measured 0.3 micrometer in thickness [1]. As a frame of reference for the reader, a human hair is about 75 micrometer in diameter. Today, thin films can be produced by virtually any material for a wide range of applications, e.g. optical, decorative, electrical, thermal, wear protective, and corrosion protective coatings. Thin films in some form are today used in practically any high-tech product, e.g. semiconductor based electronics, watches, glasses, CD’s, TV’s, medical equipment, etc. The growth of thin films can be controlled on an atomic scale, which provides tools for producing materials with outstanding properties. During thin film growth, individual atoms or ions are deposited onto a surface. By changing the deposition environment, e.g. temperature or the kinetic energy of the incoming species, the structure and, thus, the performance of the film can be strongly affected. For instance, thin films can be produced in crystallographic structures that have outstanding properties, but are not easily produced in bulk form. As an example, the formation of diamond structure from a bulk graphite sample requires a huge pressure, whereas the production of a thin diamond like film is a quite straightforward process.

There are various types of thin films on the market depending on the application. Tribological (wear protective) films are often produced from metal nitrides, oxides or carbides, such as AlₓOᵧ, TiN or TiC, but a variety of combinations are available [2]. Combining different
1. Introduction

Elements opens for an opportunity to tailor fit the film for the specific application. For tribological films, the benefits of a bulk sample and film material are combined. The bulk sample provides mechanical stability, and the film gives a wear protective inert surface with reduced friction. Optical and decorative films are used to reflect, transmit, or absorb light [3]. Examples of optical applications are, mirrors of various types, anti-reflection films on glasses or TV’s, heat flow controlling films on windows, or interference-based films for coloring. A common decorative film is naturally Au that is expensive in bulk production, but cheap as a thin film. The most common decorative films are metal oxides and nitrides in various combinations. Today, films in some form are used in all modern electrical equipment, either to act as conductor, insulator, or semiconductor.

1.2 How to produce thin films?

There are various ways to produce thin films; electroplating, chemical vapor deposition, heat evaporation, laser evaporation, plasma assisted evaporation, and a number of combinations of these methods. See for example reference 4. Electroplating is the deposition of a metallic coating onto an object by putting a negative charge onto the object and immersing it into a solution, which contains a salt of the metal to be deposited [5]. The metallic ions of the salt carry a positive charge and are attracted to the part. When they reach it, the negatively charged part provides the electrons to "reduce" the positively charged ions to metallic form.

Chemical vapor deposition (CVD) is a process where volatile gases are introduced to react and form a compound of materials to be deposited. Chemical vapor deposition has been, and still is within many areas, the most commercially practiced method because it did not require high vacuum or unusual levels of electrical power [4]. On the other hand, it sometimes relies on or creates hazardous gases and requires high temperatures, which for some applications is a major drawback. CVD is only possible when the reactants exist in gas form, which is not always the case.

Material may also be evaporated from a surface by heating an object. Every material has a temperature dependent vapor pressure. The vapor pressure is strongly increased if the object is heated, and the evaporated material can be used for depositions. Adding energy via electrical energy or a pulsed laser beam can heat the object.
Plasma assisted methods relies on the creation of a plasma in the vicinity of the deposition source. There are several common plasma assisted deposition techniques and combinations of them. The two most common are called arc evaporation and magnetron sputtering. Arc evaporation processes are high-current low DC voltage gas discharges. A very luminous small cathode spot forms that passes extremely high current densities. This causes erosion of the cathode by melting and vaporization as well as ejection of solid and molten particles. Magnetron sputtering is a technique where an external magnetic field is applied to an object to confine a plasma. Ions from the plasma are used to bombard the surface and evaporate material. Usually the plasma is sustained by applying a negative voltage to the object and current is drawn through the plasma. There are a number of ways to apply the voltage to the cathode depending on the nature of the discharge: DC, Pulsed DC, radio frequency (usually 13.56 MHz), bipolar pulses of various frequencies, and high power pulses. Applying high power pulses to a magnetron is a relatively new method to produce thin films, and is called High Power Impulse Magnetron Sputtering (HIPIMS). The technique is also known as High Power Pulsed Magnetron Sputtering (HPPMS). The terms plasma, magnetron sputtering, and HIPIMS are described in more detail in the following chapters.

1.3 Aims of this work

The general aim of this work is to explain the nature of the relatively newly developed method for producing thin films: High Power Impulse Magnetron Sputtering (HIPIMS). Many features of the method are not, or only partially, understood, and a deeper understanding is necessary for further development. Today, the technique is mostly used in academia and more fundamental knowledge about the nature of the process is desired for a full-scale industrial application. A few important parts of this required knowledge is provided in this thesis. The main objective of this thesis is to increase the knowledge of process parameters and plasma properties and its effect on thin film growth. This may lead to a better understanding of the process and an improvement in film quality. More knowledge of the process helps in the design of new deposition systems.

Work presented in this thesis is concentrated on the process and the plasma properties. In HIPIMS, many questions about the process remains to be answered, such as:

- What are the ionization fractions of the sputtered material?
1. Introduction

- What is the composition of the plasma? The high power applied to the cathode temporarily introduces much metal into the plasma. How much?
- What is the plasma dynamics?
- What is the energy of different species in the plasma?

Questions that will be discussed in this thesis are the ionization fractions of sputtered material and how the high-energy nature of the discharge affects the plasma dynamics and magnetic properties of the plasma. The ionization fraction of sputtered material is of great importance when understanding the correlation between plasma properties and film structure. The magnetic properties of the discharge strongly affect the current path and the impedance of the plasma, which in turn determines the design of new deposition system and choice of power supplies. The ion energy distributions from the HIPIMS discharge are presented. The ion energy is important knowledge in thin film growth since it is strongly connected to the microstructure of the films. Data collected with various plasma analysis techniques are presented; Langmuir probe measurements, optical emission spectroscopy, magnetic probes, mass spectrometry, and other fundamental observations. These techniques used together are powerful plasma analysis tools, and used together give a good overview of the plasma properties can be achieved.

1.4 Outline of the thesis

The thesis is structured as follows. In chapter 2 the term plasma is introduced and how it can be used to produce thin films. Chapter 3 contains basic plasma physics, necessary to understand the included papers. In chapter 4 the basics of the used analysis techniques are presented. In chapter 5 the term magnetron is introduced and explained. Chapter 6 addresses the term Ionized Physical Vapor Deposition. Chapter 7 introduces the term High Power Impulse Magnetron Sputtering. Chapter 8 contains my personal view of the future of High Power Impulse Magnetron Sputtering. In Chapter 9 the contribution to the field is presented. At the end of the thesis 6 papers are attached.
2. Plasma in thin film growth

This chapter introduces the concept of plasma, and how it can be used to produce thin films.

2.1 What is a plasma?
Irving Langmuir first introduced the term plasma in 1929 [6] to describe the behavior of ionized gases in high current vacuum tubes. Plasma is often referred to as the "Fourth State of Matter", the other three being solid, liquid and gas. Plasma is a distinct state of matter containing a significant number of electrically charged particles, a number sufficient to affect its electrical properties and behavior. In an ordinary gas each atom contains an equal number of positive and negative charges where the positive charges in the nucleus are surrounded by the of negatively charged electrons, so that each atom is electrically "neutral". A gas becomes plasma when the addition of heat or other energy causes a significant number of atoms to release some of their electrons. The remaining parts of those atoms are left with a positive charge, and the detached negative electrons are free to move about. When enough atoms are ionized to significantly affect the electrical characteristics of the gas, it is a plasma. The transformation between gas and plasma is depending on the material and gas pressure, but a typical characteristic of a plasma is the ability to screen out an internal electric field. Commonly used examples of well-known plasmas are the aurora borealis (or the northern light), neon signs, fluorescing lights and the solar corona.

Plasma science underlies numerous important technological applications and devices as well as our understanding of much of the universe around us. They provide the foundation for present applications such as plasma processing of semiconductors and thin films, sterilization of medical products, lamps, lasers, high power microwave sources, and pulsed power switches [7]. They also provide the foundation for important potential applications such as the generation of electrical energy from fusion as well as for pollution control and removal of hazardous chemicals [8]. Plasma is believed to make up more than 99 % of the visible universe, including the sun, the stars, galaxies, and the vast majority of the solar system. Plasma science includes a variety of science disciplines ranging from plasma physics to aspects of chemistry, atomic and molecular physics, and material science. Its broad nature also characterizes its plasma physics component, which includes ionized gases that range from weakly ionized to highly ionized,
2. Plasma in thin film growth

from collisional to collisionless, and from cold to hot. These terms characterize various plasmas ranging from relatively high-pressure gases with a small fraction of the atoms ionized and relatively low charged-particle temperatures, e.g., plasmas used in computer-chip processing and light sources, to very low-density gases with a large fraction of the gas atoms ionized and very high-temperature as such found in fusion plasmas. Fusion plasmas are often considered to be the energy source of the future [9]. Energy will be released by the same reaction that keeps all stars alive, fusion of light elements into heavier. This source of energy is believed to replace conventional nuclear energy in the long-term future. These unique behaviors cause plasmas to be useful in a large and growing number of important applications in our lives.

![Image](image.png)

**Figure 2.1. A neon plasma. Figure courtesy of the Basic Plasma Science Facility at UCLA [10].**

### 2.2 Plasma used for thin film production

Today plasmas are used in a number of ways in the field of thin film production and surface modification. Consider an object inserted into a plasma. If the object is subjected to a negative electrical potential compared to the plasma body, the positively charged ions in the plasma will be accelerated towards the object. At the object surface a number of processes and combinations of them can occur, see figure 2.2.
2. Plasma in thin film growth

Figure 2.2. Effects of bombarding ions or neutral atoms onto a surface. (a) Adsorption, (b) displacement (momentum transfer), (c) removal of surface atom (sputtering or etching), (d) ion implantation.

In (a) the arriving ion (or neutral) is adsorbed onto the surface, and a chemical or physical bonding is created. The incoming specie may also transfer some of its momentum to one of the surface atoms (b), and cause it to move around on the surface (displacement). If the energy of the incoming ion is sufficiently high it may start to remove some of the surface atoms (sputtering, shown in (c)), and if the energy is increased even further, the incoming ion can be implanted (d). In addition, there can also be a number of combinations of these processes. Adsorption is typically a low energy process, which is dominant around a few tens of eV, or less. Sputtering is initiated when the energy of the incoming atom or ion is of the order of 100 eV, while implantation occurs at ~ 1 keV [11]. Naturally, there are no distinct borderlines between the different processes, but in reality they overlap each other. Also, these processes are strongly dependent on the chemical and physical properties of the involved atoms and ions. These processes are important in many plasma assisted thin film growth techniques and found the basis for most plasma-based applications in the field of thin film growth and surface modification.
2. Plasma in thin film growth

The process where plasma is used to remove material from the solid to be deposited onto a substrate, is often referred to as physical vapor deposition (PVD) [4]. Where the word “physical” refers to the use of sputtering, evaporation, ablation etc. Compare to chemical vapor deposition (CVD), which involves chemical reactions of gases. The removal of material by the use of a plasma can be done in two ways, sputtering or by the use of an electric arc.

Sputtering is the process where individual ions are impinging onto a surface. When an ion impact establishes a train of collision events in the target, leading to the ejection of matrix atoms, we speak of sputtering. Sputtering was first reported in 1852 by W. R. Grove [12]. Since sputtering is the result of momentum transfer it has been aptly likened to “atomic pool” where the ion breaks up the close-packed rack of atoms, scattering some backward, to leave the surface. The principle processes in sputtering are illustrated in figure 2.3.

![Figure 2.3. The principle of sputtering. Three energy regimes have been identified. (a) Single knock-on (low energy), (b) linear cascade, (c) spike (high energy). From P. Sigmund [13].](image)

The exact processes occurring at the target surface is depending on the energy of the incoming ion. Three different regimes have been identified, (a) Single knock-on (low energy), (b) linear cascade, and (c) spike (high energy) [13]. The sputtered atoms then become part of the plasma, as neutral atoms or ions (if they are ionized). The sputtered atoms are then available for deposition. Sputtering is typically a high voltage, low current discharge (hundreds of volts and tens of mAcm$^{-2}$).

The other type of plasma-based thin film production technique is the use of a cathodic arc [14-16]. Arcs are typically low voltage high current discharges (tens of Volts and hundreds of
Amperes). The discharge can occur in high vacuum, but it should be realized that the term “vacuum arc” is not physically correct, since in perfect vacuum no arc can exist. The initiation of a vacuum arc requires two electrodes that first touch and then separates a small distance. A very small cathode spot is then formed, which carries the discharge. The discharge spot is typically $10^{-8}$ to $10^{-4}$ m$^2$ in diameter [4], and carries extremely high current densities ($10^8$ to $10^{12}$ Am$^{-2}$ [4,17]). This causes erosion of the cathode spot by melting and vaporization as well as ejection of molten and solid particles. The local high power density creates a highly ionized plasma of the cathode material, which then sustain the discharge. The principle of an arc discharge is shown in figure 2.4.

![Figure 2.4. The principle of a cathodic arc. From P. C. Johnson [18].](image)

Today, the most common plasma-based thin film growth techniques are sputtering and cathodic arc evaporation, and various combinations and versions of them. For sputtering, the most common method is so called magnetron sputtering, which is addressed in more detail in the following chapters. Sputtering alone typically produces a low-density, weakly ionized plasma, whereas cathodic arc produces a dense highly ionized plasma, but a major drawback are the microdroplets, which reduce the quality of the films when not filtered out.
2. Plasma in thin film growth
3. Basic plasma physics

This chapter introduces some fundamental physics of sputtering plasmas. The basic physics needed to understand the operation of a magnetron is introduced.

3.1 Plasma ignition
To ignite a plasma, a discharge gas is introduced. The gas is often inert, to avoid unwanted reactions with the target material, but can also contain a fraction of a reactive gas if a compound film is desired. The most common gas used in sputtering processes is Ar, mixed with N\textsubscript{2} or O\textsubscript{2}. An electric field is then applied between the sputtering source (cathode) and an anode. Often, the chamber wall is used as anode in the system. Even at room temperature, the gas will contain a small fraction of ions and some free electrons (from background radiation). The ions and electrons are then accelerated towards the cathode and the anode respectively. The electrons then generate new ions and electrons via collisions with the gas atoms. In addition, new electrons are also generated at the cathode surface. When the ions reach a close distance from the cathode surface (a few Angstroms), electrons can tunnel from the cathode, and the ions are neutralized. When an ion is neutralized, the energy corresponding to the ionization energy of the ion is released. This energy can then be given to a surface electron, via an Auger process. If the ionization potential of the ion is greater than the electron work function, new electrons can be emitted into the gas. These electrons are so-called secondary electrons. Electrons are then accelerated by the electric field, and generate new ions and free electrons. By the addition and acceleration of new electrons, an “avalanche” of ionizations has started. If the applied power is sufficiently high, a plasma is created. This process is called plasma breakdown. The plasma then adjusts itself regarding temperature, density, and distribution in space until it reaches a balance between charge losses and supplied energy.

3.2 Energy and temperature
A gas at thermal equilibrium has particles of all velocities, and the most probable distribution of those velocities is the Maxwellian distribution. The one-dimensional Maxwellian distribution is given by
3. Basic plasma physics

\[ f(u) = A \exp \left( -\frac{mu^2}{2kT} \right) \]  

(1)

where \( f(u) \) is the number of particles with velocity between \( u \) and \( u+du \), \( mu^2/2 \) is the kinetic energy, and \( k \) is Boltzmann’s constant [19]. The constant \( A \) is related to the density \( n \) by

\[ A = n \left( \frac{m}{2\pi kT} \right)^{0.5} \]  

(2)

The average kinetic energy (\( E_{av} \)) of a Maxwellian distribution is \( kT/2 \) per degree of freedom. Since \( T \) and \( E_{av} \) are so closely related, it is common in plasma physics to give temperature in units of energy [20]. To avoid confusion on the number of dimensions involved, it is not \( E_{av} \), but the average corresponding to \( kT \) that is used to denote the temperature. For example if we have \( kT = 1 \text{ eV} = 1.602 \times 10^{-19} \text{ J} \), we have

\[ T = \frac{1.602 \times 10^{-19}}{1.38 \times 10^{-23}} \approx 11600 \text{ K} \]  

(3)

Thus, the conversion factor is \( 1 \text{ eV} \approx 11600 \text{ K} \). In many processing plasmas several temperatures can exist at the same time. Often the ions and the electrons have Maxwellian distributions with different temperatures. In most cases, the energy to the plasma is supplied via energetic electrons. The moment transfer efficiency is relatively low, due to the large differences in mass. Then each species can have its own thermal equilibrium, but the lifetime of the plasma species is not long enough for the two temperatures to equalize. In the presence of a magnetic field, the temperature can also be different in different directions.

Furthermore, a common misunderstanding is that a high temperature is directly connected to lots of heat. However, most plasmas are created in a low pressure environment with very little heat capacity. The electron temperature inside a common neon sign is several tens of thousands of degrees, but still does not melt the glass. This is because the electrons can transfer very little energy to the glass, due to the low pressure and their low mass.
3. Basic plasma physics

3.3 Ionization processes

The ionization in a sputtering plasma relies on several different phenomena. The three most important responsible for the generation of ions in an inert gas plasma are electron impact ionization of neutral atoms, electron impact ionization of an excited atom, and Penning ionization by collision with an electronically excited atom according to [21]

\[
\text{Atom} + e^- \rightarrow \text{Ion} + 2e^- \\
\text{Atom}^* + e^- \rightarrow \text{Ion} + 2e^- \\
\text{Atom} + \text{Atom}^* \rightarrow \text{Ion} + e^- + \text{Atom} \quad \text{(Penning)}
\]

For high-density plasmas \((n_e > 10^{17} \text{ m}^{-3})\) the electron impact ionization of neutral atoms is the dominating process [21] and will therefore be the only subject for discussion in this thesis. The mean ionization distance from electron impact ionization can be determined according to [21].

\[
\lambda_{\text{ie}} = \frac{v_M}{n_e K_i(T_e)}
\]

(4)

where \(v_M\) is the velocity of the sputtered atom, \(n_e\) is the density of electrons, and \(K_i(T_e)\) is the ionization rate constant according to

\[
K_i(T_e) = K_0 e^{-\frac{E_0}{kT_e}}
\]

(5)

where \(K_0\) and \(E_0\) are material dependent and available in literature for some materials [22-27]. Atoms sputtered from the surface of the cathode typically follow the Thompson distribution with average energies of the order of a couple of eV, corresponding to velocities of about \(10^3\) m/s [28]. We can now estimate the average distance an atom travels before it suffers an ionizing collision by using equation 4 and 5. An example calculated for Cu and C is illustrated in figures 3.1 and 3.2. From figures 3.1 and 3.2, it is realized that the mean ionization distance for a sputtered particle is strongly material dependent. In order to reduce the expected ionization distance for Cu so that it matches the dimensions of the highly energetic plasma (~cm) [29,30], we require plasma densities of the order of \(10^{19} \text{ m}^{-3}\) and electron temperature of around 3 eV. For C these values are even higher. The difference is due to the higher ionization potential and smaller cross section of the carbon atom.
To achieve an effective ionization of the deposition material in sputtering processes, it is, therefore, necessary to apply enough electrical power to reach electron densities of the order of $10^{19}$ m$^{-3}$, while the electron temperature should be at least a few eV. In reality this corresponds to a power of the order of kW cm$^{-2}$ on the target surface. Usually this amount of power cannot be applied constantly, due to insufficient cathode cooling, but has to be applied in pulses with a
low duty factor. The duty factor is defined as the ratio between pulse on time and the total cycle time.

### 3.4 Debye shielding

A fundamental characteristic of a plasma is its ability to shield out an externally applied electric field. If an electric field were applied to a plasma, by inserting two electrodes. The two electrodes attract charge carriers of the opposite charge, and a “cloud” of electrons and ions will surround each electrode.

![Figure 3.3. The principle of Debye shielding.](image)

For a very cold plasma, with no thermal motion of the charge carriers, the electric field of the electrodes would be totally shielded. No electric field would be present inside the plasma body. If the temperature is finite, however, some of the ions and electrons at the edge of the cloud could escape the attractive force of the electrode. In this case, some of the electric field will penetrate into the plasma. The region where the electric field penetrates the plasma is called a sheath. The thickness of a sheath is of the order of the Debye length $\lambda_D$. The Debye length is defined as

$$\lambda_D = \left(\frac{\varepsilon_0 kT_e}{ne^2}\right)^{0.5}$$ (6)
3. Basic plasma physics

where \( T_e \) is the electron temperature, \( n \) is the plasma density [20]. Expression 6 can in a simplified form be written as \( \lambda_D = 69(T_e/n)^{0.5} \) m, where \( T_e \) is given in Kelvin. Sheaths appear where a plasma meets a solid such as a chamber wall, electrode, substrate, or probe. The thickness of a sheath depends on the potential difference between the solid surface and the plasma potential [31]. The Debye length is an important parameter in processing plasmas since it describes a plasma’s ability to penetrate into holes, and the behavior of the plasma around inserted objects.

### 3.5 Single particle motion

Consider a charged particle with charge \( q \) moving in a magnetic and electric field denoted \( B \) and \( E \) respectively. The equation of motion for this particle is then

\[
\frac{m\,dv}{dt} = q(E + v \times B) \tag{7}
\]

We choose \( E \) to lie in the x-z plane so that \( E_y = 0 \) and \( B \) is assumed to be only in the z-direction, resulting in

\[
\frac{dv_z}{dt} = \frac{q}{m} E_z \tag{8}
\]

which is a straightforward acceleration along \( B \). The transverse components of equation 7 are

\[
\frac{dv_x}{dt} = \frac{q}{m} E_x \pm \omega_c v_y \tag{9}
\]

\[
\frac{dv_y}{dt} = 0 \pm \omega_c v_x \tag{10}
\]

which by the use of differentiation can be solved as

\[
v_x = v_{\perp} e^{i\omega_c t} \tag{11}
\]

\[
v_y = \pm iv_{\perp} e^{i\omega_c t} - \frac{E_x}{B} \tag{12}
\]

\( v_{\perp} \) is the velocity component perpendicular to \( B \) and \( \omega_c \) is the so-called cyclotron frequency (\( \omega_c = qB/m \)). Equation 8, 11, and 12 describes an oscillating motion around a drifting center. The radius of each oscillation is often referred to as the Larmor radius \( r_L \) and the “position” in which the particle oscillates around is called the guiding center. The Larmor radius \( r_L \) is defined as [20]

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Figure 3.4 shows the expected trajectory of a particle moving in a crossed magnetic and electric field.

![Image of particle drift in crossed magnetic and electric fields]

Figure 3.4. Particle drifts in crossed magnetic and electric fields. From F. Chen [20].

The drift of the guiding center of the particle is denoted $v_{gc}$. The velocity of the guiding center can be calculated according to [20]

$$v_{gc} = \frac{E \times B}{B^2}$$

(14)

It is important to notice that the direction of $v_{gc}$ is independent of $q$, $m$, and $v$ in the case of uniform $B$ and $E$ fields. Ions and electron drift due to the $E$ field in the same direction, but oscillating around their guiding centers in different direction. However, it should be noted that since ions have much greater mass, they exhibit a much greater Larmor radius, in many cases even bigger than the dimensions of the plasma.
3. Basic plasma physics
4. Plasma analysis

This chapter introduces the fundamentals of plasma diagnostics. Four different plasma-probing techniques are addressed: electrostatic probes, magneto dynamic probes, optical emission spectroscopy, and mass spectrometry.

4.1 Why plasma analysis?
A plasma can be characterized by a number of properties such as number of ionized species per volume (density), temperature of ions and electrons, degree of ionization, ion and neutral composition, and dynamics. Plasma analysis is important in any plasma assisted thin film deposition technique, since the processes occurring during the material transport through the plasma directly affects the properties of the arriving flux regarding incident energy and direction, and ionization. This in turn strongly affects the properties of the films [32,33]. The experimental methods best suited, depends on which feature that one wants information about. In this work 4 different major plasma probing techniques were used; electrostatic probes, magneto dynamic probes, optical emission spectroscopy (OES), and mass spectrometry. These techniques combined are very powerful, and can give a complete picture of the plasma parameters. The typical features of each technique are described in more detail in the following sections.

4.2 Main principle of electrostatic probes
Electrostatic probes are often referred to as Langmuir probes. The name comes from the American scientist Irving Langmuir working with low-pressure discharges. He was awarded the Nobel Prize in Chemistry 1932 for his discoveries. The technique relies on the insertion of one or several electrodes into the plasma. The electrode is then biased with various potentials and the current drawn from the plasma body is measured. The current drawn from the plasma body is mainly depending on the applied bias, the density of the plasma, and the temperature of ions and electrons. In the type of plasma discussed in this thesis, the current drawn by a Langmuir probe is mainly electron based, since the electrons have much higher mobility than ions. Thus, Langmuir probe measurements can give information about the plasma density and electron
4. Plasma analysis

A plasma confined in a vacuum chamber constantly loses some of its electrons and ions to the grounded walls. Assume that the plasma body initially is at ground potential. Since electrons are smaller and lighter than the ions, more electrons compared to ions will be lost via diffusion to the chamber walls. This will charge up the plasma body and a potential sheath will develop between the plasma and the chamber wall. The sheath will repel some of the electrons. As a result, the plasma body charges up until the loss of electrons and ions is the same. The potential at which the loss of ions is the same as the loss of electrons, is referred to as the plasma potential ($V_p$). Usually the plasma potential is of the order of a few volts (positive) in the type of plasma discussed in this thesis. In the absence of magnetic fields inside the bulk plasma, the plasma potential is usually constant throughout the whole plasma. When a magnetic field is present, the situation might be a bit different. In this case, the plasma potential does not need to be constant through the whole plasma, and may also locally be negative [29].

Now consider a Langmuir probe inserted into the plasma. If the probe is left electrically insulated, no charge carriers can escape, and the probe is charged up. Again, since electrons are more mobile, more electrons than ions will reach the probe surface and the probe will charge up negatively. The potential at equilibrium is reached is called the floating potential ($V_f$). The floating and plasma potential are important parameters in coating plasmas, since they indirectly affect how the films are formed. If the probe is connected to a power source, such as a battery or a variable power supply, a current will flow through the probe. If a large negative potential is applied to the probe compared to the plasma potential, the probe will attract the positively charged ions. The large potential barrier between the plasma and the probe will repel all

Figure 4.1. A single tip Langmuir probe.
4. Plasma analysis

electrons. Ions that reach the probe surface will be neutralized with an electron from the probe. In this process secondary electrons may also be ejected. This result in that a current will flow through the plasma and the probe, usually referred to as ion current, although it is only carried by ions inside the plasma, and at the probe surface. As the potential applied to the probe is decreased, some highly energetic electrons can overcome the potential barrier between the plasma and the probe. When the probe potential has dropped to a level where as many electrons reach the probe from thermal motion, as ions are attracted, the probe is at the floating potential.

If the probe potential is made even more positive, more and more electrons start to reach the probe, and the total current changes direction, so called electron current. The electron current is electron based throughout the whole circuit. When the probe potential is further increased, and reached the plasma potential, all electrons can reach the probe. Increasing the potential even further, the probe now attracts all electrons, and the current is steadily increasing. Due to the higher mobility of electrons, the electron current is usually 1-3 orders of magnitudes higher than the ion current. The shape of the current as function of applied probe voltage is often referred to as probe I-V curve. A schematic of a probe I-V curve can be seen in figure 4.2.

![Figure 4.2. An example of a Langmuir probe I-V characteristics.](image-url)
4. Plasma analysis

From the Langmuir probe characteristics, the electron temperature (or in fact the whole energy distribution function) and the plasma density can be estimated. A number of theoretical approaches to evaluate I-V traces exist, as the one shown in figure 4.2 [see for example ref. 20, 34,35]. Druyvesteyn first developed the theory used in this thesis during the 1930’s [36]. The theory developed by Druyvesteyn allows for an estimation of the electron energy distribution function (EEDF) according to

\[
f_e = \frac{2m}{e^2A_{pr}} \left( \frac{2eV}{m} \right)^{0.5} \frac{d^2I}{dV^2}
\]  

(1)

where m is the electron mass, e is the elementary charge, A_{pr} is the area of the probe, and V is the difference between the plasma potential and the probe potential. The plasma density can then be extracted from equation 1, by integrating over all energies up to the plasma potential. The theory does not take into account that an ion current is also present, but the error generated by this assumption is expected to be minor.

4.3 Magneto-dynamic probes

Magneto-dynamic probes are used to determine how currents are flowing inside a plasma. The current flowing in a plasma is due to the relative motion of the ions and electrons. Assuming that all ions are positively charged (which is common, but not always the case), the current density, j, can be defined as

\[
j = -(u_e - u_i)n_e
\]  

(2)

where \( u \) is the drift velocity of electrons and ions, respectively, and n is the electron density. Thus, currents are present in the bulk plasma if the relative motion of ions and electrons is different. This is often the case, especially in the presence of magnetic fields, since the properties of electrons and ions are so different. The ions are less mobile and heavier than the electrons, and therefore much less affected by magnetic fields, or other perturbations. The currents flowing in a plasma generates magnetic fields, which in turn can be detected. Typically, a plasma is considered to be of diamagnetic nature, meaning that currents tend to flow in way that generates magnetic fields that opposes an externally applied field. The relation between a magnetic vector field and current is
4. Plasma analysis

\[ j\mu_0 = \nabla \times B \] (3)

which allows for calculation of the current density with same spatial resolution as the magnetic vector field [see for example ref 37]. The most common ways to detect currents in a plasma is by using a Hall probe or a B-dot probe. The Hall probe relies on the Hall effect [37], and can be used to measure both DC and AC components of the current. The B-dot probe can only be used to record the AC component of the field, but is the more commonly used in pulsed plasmas. The B-dot probe is usually a quite simple device, which is basically a small coil. The magnetic field change generated by a pulsed plasma can then be detected by the coil according to

\[ U(t) = NA \frac{dB(t)}{dt} \] (4)

where \( N \) is the number of turns in the coil, \( A \) is the cross section area, and \( U \) is the induced voltage by the magnetic field. In practice, often two coils are used with the same area but opposite winding direction. Pulsed plasmas often contain high frequency electric field components, which can penetrate through the probe insulation and disturb the measurement. This problem can be overcome by using double winding. The electrostatic charging by the plasma is the same for the two coils, but the magnetically induced voltage has opposite sign. By taking the difference in the recorded voltage between the two windings, the magnetically induced component can be extracted. The B-dot probe is an important experimental device to extract information about the plasma dynamics, the transport of charged particles in pulsed plasmas and other dynamic phenomena in plasmas.

4.4 Optical emission spectroscopy (OES)

Spectroscopy is the study of spectra, i.e. characteristic wavelengths or colors. In optical emission spectroscopy (OES), the light emitted by atoms and ions in the wavelength range from about 100 nm to 900 nm are recorded. This range includes the ultraviolet, and visible light (from violet at 380 nm to red at 760 nm), and the near infra-red range. Light is electromagnetic radiation generated by the acceleration of charged particles or by transitions of electrons in atoms and ions. In a plasma, the free plasma electrons creates excited states, which recombine
4. Plasma analysis

and sends out photons. The excited states are usually created by electron excitation, but can also be created by a photon.

![Image of electron excitation]

**Figure 4.3. The generation of light gives a fingerprint of the species in the plasma.**

The photons sent out from a plasma can then give a fingerprint of the species in the plasma, since every atom and ion has different electron energy levels. OES is a very powerful, but also very complicated analysis technique, where several plasma properties can be extracted. In the simplest approach, a qualitative study of the species in the plasma can be performed. The light is recorded and divided into wavelength components by a spectrometer. Each wavelength component then corresponds to an atomic energy level according to

\[ E = \frac{hc}{\lambda} \]

where \( h \) is Plank’s constant, \( c \) is the speed of light, and \( \lambda \) is the wavelength. The energy levels of various atoms and molecules are quite well documented [38-41], so an identification of the emitting species is quite straightforward. A quantification is, however, more difficult to obtain. The recorded intensity of an individual emission line is dependent on several things such as excitation mechanism, atomic transition probability, re-absorption of the emitted photons, and spectrometer sensitivity for the different lines. In a plasma, the main excitation mechanism is excitation by one of the energetic free plasma electrons. The relative intensity of various emission lines is an indirect mapping of the distribution of excited states of different ions and
4. Plasma analysis

...atoms. The distribution of excited states is then in turn mainly generated by electron collisions. Thus one can, by studying the emission spectrum of individual species, get information about the energy distribution of the plasma electrons [42]. With knowledge of the distribution of excited states, and the transition probabilities, quantitative estimations are also possible with OES [43-45]. It is also possible to estimate the temperature of ions and neutrals, using the Doppler shift. Highly energetic ions and neutrals will create broader emission peaks. This however requires a high-resolution spectrometer.

In principle, there are two main types of spectrometers; prism based and grating based. In prism spectrometers, the light is passed through a slit, and divided into wavelength components via a prism. The principle is shown in figure 4.4.

![Figure 4.4. The principle of prism based spectrometers. After K. Krane [46].](image)

The other type of spectrometers is grating based. The light is projected onto a grating, and a camera records the reflected light. The reflected light from a grating will form a diffraction pattern, and can via a computer algorithm be transferred to a spectrum.

4.5 Mass spectrometry

Mass spectrometry of a plasma is the extraction and detection of individual plasma species, ions or neutrals. An extraction probe is inserted into the plasma, into which gas atoms and ions from the plasma may enter. If neutral atoms are to be analyzed, they first have to be ionized in the instrument. The ions are then passed through a series of filters and electrostatic lenses, and then
4. Plasma analysis

detected. Mass spectrometry can also be energy resolved. The principle of mass spectrometry is illustrated in figure 4.5.

![Figure 4.5. The main principle of plasma mass spectrometry.](image)

The whole system is usually separately pumped in order to increase the mean free path for the ions inside the device. The extraction part is simply a small hole (~ 50 – 300 µm diameter) where ions and neutrals can pass through. The front can be electrically floating biased or grounded. Inside the extraction orifice, an ionization source is situated. This is usually a heated filament that emits electrons. The next two parts (energy and mass filter) of a spectrometer is depending on the type of mass spectrometer. There are two main types of spectrometers; Time-of-Flight (TOF), and spectrometers based on electrostatic filters.

A TOF mass spectrometer uses the differences in transit time through a drift region to separate ions of different masses. It operates in a pulsed mode so that ions have to be produced or extracted in pulses. An electric field accelerates all ions into a field-free drift region with a kinetic energy of \( qV \), where \( q \) is the ion charge and \( V \) is the applied voltage. Since the ion kinetic energy is \( \frac{1}{2}mv^2 \), lighter ions have a higher velocity than heavier ions and reach the detector at the end of the drift region earlier. Since the flight time is also depending on the kinetic energy of ions entering the device, there is also information available about the ion energy distribution by studying the peak broadening.

Mass spectrometers based on electrostatic filters divides the energy and the mass filtering into two parts. The energy filter is generally a quite simple device, where only ions of certain energy
may pass through, for example a so-called Bessel box. A schematic of a Bessel box is shown in figure 4.6. Ions with too high or low energy (B and C) will not travel through the filter.

![Figure 4.6. The main principle of a Bessel box.](image)

The mass filter is a bit more complicated. The most common mass filter is a so-called quadrupole. A quadrupole mass filter consists of four parallel metal rods arranged as in figure 4.7. Two opposite rods have an applied potential of \( V + A \sin(\omega t) \) and the other two rods have a potential of \( -(V + A \sin(\omega t)) \), where \( V \) is a dc voltage and \( A \sin(\omega t) \) is an AC voltage. The applied voltages affect the trajectory of ions traveling down the flight path centered between the four rods. For given dc and ac voltages, only ions of a certain mass-to-charge ratio pass through the quadrupole filter and all other ions are thrown out of their original path. A mass spectrum is obtained by monitoring the ions passing through the quadrupole filter as the voltages on the rods are varied. There are two methods: varying \( \omega \) and holding \( V \) and \( A \) constant, or varying \( V \) and \( A \) \( (V/A) \) fixed for a constant \( \omega \).
Figure 4.7. The principle of a quadrupole mass filter.
5. The magnetron

This chapter introduces the functionality of a planar magnetron. The basis principles are explained.

5.1 Background

In a basic sputtering process, a target plate is bombarded by energetic ions generated in a glow discharge plasma situated in a close vicinity. The rate of material leaving the target is depending on the number of bombarding ions. It is therefore desired to increase the plasma density in front of the sputtering source. Basic sputtering without any plasma confinement has the disadvantages of low deposition rates and low ionization efficiencies of the plasma. These limitations have lead to the development of magnetron sputtering. The magnetron was first introduced in the early 70s [47-49], and has rapidly developed to the point where it has become the number one choice for the deposition of a wide range of industrial coatings. The driving force behind the development of magnetron sputtering has been the increasing demand of high-quality functional thin films in widespread application areas. Magnetron sputtering now makes a significant impact in a large number of application areas including hard, wear-resistant coatings, decorative coatings, and coatings with specific optical or electrical characteristics. The basic sputtering process was known, and used for many years despite its limitations. However, it was the development of the magnetrons in various forms, which really arouse the importance of sputtering. Magnetron sputtering today exists in a number of forms regarding geometry and electrical power, all depending on the application. As in any plasma based deposition process it is possible to grow high quality films at low temperatures using magnetron sputtering since the ions from the plasma can be used for films densification and formation of metastable phases.

5.2 Basic principle of a planar magnetron

Magnetrons make use of the fact that a magnetic field configured parallel to the target surface can constrain the motion of secondary electrons ejected by the bombarding ions, to a close vicinity of the target surface. An array of permanent magnets is placed behind the sputtering source. The magnets are placed in such a way that one pole is positioned at the central axis of the target, and the second pole is placed in a ring around the outer edge of the target. A schematic of the magnetron is shown in figure 5.1.
This configuration creates crossed E and B fields, where electrons drift perpendicular to both E and B according to \( v_H = \frac{E}{B} \). If the magnets are arranged in such a way that they create closed drift region, electrons are trapped, and relies on collisions to escape. By trapping the electrons in this way, the probability for ionization is increased by orders of magnitudes. Ions are also subjected to the same force, but due to their larger mass, the Larmor radius often exceeds the dimensions of the plasma. Although in general one says that the ions are not directly confined, they are so indirectly by trapping the electrons, to keep the quasi neutrality of the plasma. The trapping of electrons and ions creates a dense plasma, which in turn leads to an increased ion bombardment of the target, giving higher sputtering rates and, therefore, higher deposition rates at the substrate. The electron confinement also allows for a magnetron to be operated at much lower voltages compared to basic sputtering (~ 500 V instead of 2-3 kV) and be used at lower pressures (typically mTorr region). This is an advantage since less material is lost to the chamber wall and back to the target through scattering in the discharge gas. The shape of the magnetic field efficiently erodes the target where the magnetic field lines are parallel to the target surface, a so-called race-track is created. A picture of a sputtered circular target is shown in figure 5.2.
The magnetic field configuration of magnetrons can be divided in three different categories; intermediate (balanced), and unbalanced type I and II. The differences in design between the different categories are only slight. However, the difference in performance regarding film growth between the different types can be significant. During film growth it is often desired to have an effective ion bombardment of the film. This can be achieved by applying a bias onto the substrate. In most cases it is preferred to have a high ion current containing low energy ions, rather than high energy, since this can generate stress and defects in the film [50]. It is therefore desired to design the magnetic field in such a way that it allows for some of the plasma to escape from the close vicinity of the cathode, and reach the substrate. This is the reason for the different magnetron designs. A schematic of the three different designs are illustrated in figure 5.3.
5. The magnetron

5.3 Magnetron operation

Magnetrons can be operated in a number of ways depending on the application [51]. In the simplest case, a direct current (DC) is applied to a single (conducting) target. Compound films can be grown in several ways. If the compound is conducting, a coating may be produced from a target of the same composition as the desired film. It is also possible to use multiple magnetrons of different target materials. Another alternative is to use a pure metal target in combination with a reactive gas. Reactions between the sputtered material and the reactive gas can now take place at the film surface and a compound film can be grown. In this way TiAlN can be reactively grown by using a single TiAl target, or by using separate Ti and Al targets, in a N$_2$ environment. If the desired coating is not electrically conducting, DC sputtering from a compound target is not possible (for example Al$_2$O$_3$, which is a common coating material in industry). If a DC voltage is applied to such a target, the surface is charged up, and the sputtering process will immediately stop. This problem can be overcome in two ways; by using a metal target in a reactive environment or by the use of a quickly oscillating voltage. If one chooses the reactive alternative, one must keep a very good control over the partial pressure of the reactive gas. If too much gas is supplied to the discharge, the target surface will be “poisoned”, and the sputtering stops, or gets very inefficient (low rate, or a lot of arcing). On the
5. The magnetron

other hand, if one does not supply enough gas, the compound will not form with the right stoichiometry. The partial pressure must, therefore, be controlled so that it keeps the pressure at a level where the target is kept relatively “clean” by the sputtering, but there is enough gas to form the desired stoichiometry. This can be a quite complicated and unstable process. The other alternative is to use an alternating voltage of some kind (pulses or other type of oscillating voltage). The two most common ones are rf frequency sputtering (usually 13.56 MHz) or bipolar pulsed sputtering. In rf sputtering most materials can be used as cathode, but the deposition rate is usually very low. In bipolar pulsed sputtering, the voltage is reversed for a short time. This attracts some of the electrons from the plasma, and neutralizes the charge that has accumulated on the target surface. Since the electrons have so high mobility, it is usually only necessary to have a reversed bias supplied for 10 – 20 % of the time.
5. The magnetron

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6. Ionized PVD

This chapter introduces the concept of Ionized Physical Vapor Deposition (IPVD). The basics and features of various techniques are mentioned.

6.1 Background
State of the art sputtering processes allow for the deposition of materials for a large number of applications. There is a constantly increasing demand for the development of coatings with tailored and enhanced properties such as high hardness, wear and corrosion resistance, low friction, and specific electrical or optical behavior. For high-quality coatings there is a lot of room for improvements using versatile PVD processes. An important feature of plasma based PVD processes is the availability of bombardment of plasma ions during film growth [see for example ref 52 and 53]. Bombarding ions transfer momentum to the surface, which in many cases is a great advantage during low temperature film growth. An optimized ion bombardment has beneficial effects on the microstructure, which in turn often leads to improved mechanical and chemical properties of the coating. The increasing requirements on coatings and the process techniques drive the development of enhanced deposition techniques, which allows better control of the flux of material and further decrease in deposition temperature. Common sputtering processes have ions available for momentum transfer, but mainly ions of the discharge gas whereas ions of the sputtered material are relatively rare. For many applications it is desired to have also ions of the sputtering material, since this allows for an effective ion bombardment and control over energy and direction. It can also be a powerful tool in other surface modification processes such as etching and implantation.

Over the last years various techniques have been developed that ionizes the material flux. When the flux of ions is higher than the flux of neutrals of the sputtered material at the substrate, the process is referred to as IPVD [21]. The development of IPVD techniques was initially mainly driven by the need to deposit metal layers and diffusion barriers into trenches and vias of high aspect ratio IC structures [54-56], but has during the last years found a number of areas where beneficial effects were observed.
6. Ionized PVD

6.2 Advantages of IPVD

The main advantage by using IPVD is the versatility. One gets an “extra knob” to turn to vary the direction and energy of the deposition flux. The energy and direction of incoming ions are important parameters in thin film growth [see for example ref 33].

In trench filling it is desired to have a collimated flux towards the opening of the hole, and effective re-sputtering at the corners of the opening of the trench. This is possible if a large fraction of the deposition flux is ionized. A figure 6.1 show the filling of trench with an ionized flux of Cu. $\beta$ is defined is the ionized flux fraction. It is seen that for increasing ionized flux, the trench is filled better. The control over direction can be utilized in any deposition process where the substrate is complex shaped.

![Figure 6.1. The metallization of a trench with IPVD. From Lu and Kushner [57].](image)

Another key feature of thin films is that in general a dense microstructure is desired. This is effectively achieved with a highly ionized flux of deposition material and a substrate bias. The highly ionized flux gives an effective film densification during film growth, with a dense, less columnar microstructure. The dense structure may reduce the friction of the coating, increase the mechanical performance, and increase the resistance against corrosion. Figure 6.2 shows two films (TiC/CaO) grown under the same conditions. In (a), the degree of ionization is low,
and a columnar structure is achieved. In (b) the film is grown under effective ion bombardment, and a denser, less columnar structure is achieved.

Figure 6.2. A densification effect by the use of IPVD. (a) Without additional plasma ionization, (b) with additional plasma ionization. From Kulish et al [58].

Håkansson et al studied the microstructure of TiN films grown by various physical vapor deposition techniques [59]. They found that the most pronounced difference in film structure was caused by an effective ion bombardment. Films grown with conventional magnetron sputtering exhibited intercolumnar porosity, but films prepared with additional ion bombardment appeared denser.
6. Ionized PVD

6.3 Existing IPVD methods

Today there exist several techniques that effectively ionize the deposition material. The most common IPVD method today is the cathodic arc discharge, mentioned in section 2.2. In an arc discharge the flux of material is usually highly ionized. The cathodic arc however has the disadvantage of the microdroplets, and for many applications (i.e. electronic applications) these microdroplets are a major drawback [60]. Therefore an alternative technique has been developed; filtered arc. In filtered arc a magnetic filter is used to remove the microdroplets from the deposition flux. A schematic of a filtered arc setup is shown in figure 6.3.

![Schematic diagram of a cathode arc source with a 90°-duct filter. From Karpov et al [61].](image)

The magnetic filter only traps the ionized part of the plasma, and guides to the substrate. The uncharged and heavy microdroplets are not affected by the field, and does not generally reach the substrate.

There are also magnetron-based alternatives available for IPVD, involving conventional magnetron sputtering with external ionization of the deposition flux or an additional plasma confinement. One alternative is to use an inductively coupled plasma (ICP) source in the region between the cathode and the substrate. It is basically a conventional magnetron setup with an additional coil. The inductive coil is then driven at 13.56 MHz using a rf power supply. A schematic of an ICP sputtering setup is seen in figure 6.4.
6. Ionized PVD

The plasma electrons are accelerated by the oscillating rf field, and creates a denser plasma inside the coil. The dense plasma in turn ionizes some of the deposition material as it passes through this region.

Another approach to increase the ionization fraction is to use a supplementary electron cyclotron resonance (ECR) discharge. ECR discharges are typically operated at microwave frequencies (few GHz) with a strong magnetic field. Here electrons are heated by a resonance between $\omega$ and the electron cyclotron frequency. Due to this cyclotron resonance, the gyrating electrons start to rotate in phase with the wave.

The hollow cathode magnetron is another common approach to generate high-density plasma for IPVD. In hollow cathode magnetron sputtering an intense glow discharge is formed in a cup shaped cathode or between two parallel plates. The geometry then confines the discharge both physically and electrostatically. The electrons captured in the geometry are between two “electrostatic mirrors”, and therefore the probability for ionization increases drastically for a sputtered neutral. A schematic of a hollow cathode is illustrated in figure 6.5.
The methods mentioned above are all today used in academia and industry. All techniques however suffer from some drawbacks. The arc evaporation process has the problem of the microdroplets. The other IPVD techniques suffers from complex geometry, and are therefore not easily applied to an existing large-scale industrial magnetron sputtering process, since it involves complicated reconstruction of existing deposition systems. The additional equipment also raises the cost of the deposition system. This has lead to the development of a new IPVD technique; High Power Impulse Magnetron Sputtering (HIPIMS). HIPIMS is the main topic of this thesis, and is discussed in more detail in the following chapter.
This chapter is about High Power Impulse Magnetron Sputtering (HIPIMS). The basic principle is discussed and the features of the discharge are addressed.

### 7.1 Main principle

HIPIMS stands for High Power Impulse Magnetron Sputtering. The technique is sometimes also mentioned as High Power Pulsed Magnetron Sputtering (HPPMS). HIPIMS was first reported in 1999 by Kouznetsov et al., [64-70] who also patented the technique together with the company Chemfilt R&D (now called Chemfilt Ionsputtering AB). The basis of the technique is to increase the plasma density in front of a sputtering source, and, thereby, decreasing the mean ionization distance for the sputtered particles. The increase in plasma density is simply achieved by applying a high electrical power. As described in chapter 3, the electrical power applied to the target surface has to be high enough so electron densities of the order of \(10^{19} - 10^{20} \text{ m}^{-3}\) are reached in the vicinity of the sputtering source. In reality this corresponds to electrical power of the order of kWcm\(^{-2}\) on the target surface, which is too much to be ran in constant mode with conventional target cooling. The solution to this problem is to apply the high power in pulses with a low duty factor. The duty factor is the ratio of the pulse on time and the cycle time. The target cooling is limited by the average power rather than the peak power, and by applying the power in pulses the average power can be kept at where the cooling is sufficient. An image of the HIPIMS discharge can be seen in figure 7.1. Note that the discharge is uniformly distributed over the target surface. This is necessary to avoid the formation of microdroplets.
7.2 Electrical parameters and power supply

The peak power that must be delivered to the target is typically of the order of kW cm\(^{-2}\), meaning that the power supply must be able to deliver a peak electrical power in the range 10 kW-5 MW, depending on the target size (2" up to large-scale industrial systems). Figure 7.2 shows voltage and current traces taken from a HIPIMS discharge. The discharge operates on a 150 mm Ti cathode. The traces correspond to a peak power of 330 kW or 440 W average power. The pulse is then repeated with such a frequency that the average power is kept low. In the work presented in this thesis 50 Hz was used.
A common way to define the strength of the pulse is to define the pulse energy. The pulse energy can be obtained in several ways. One is to measure the voltage and current (as in figure 7.2) and multiply the two traces. The result is the discharge power as function of time. The integration of this curve then gives the pulse energy. Alternatively one can measure the voltage or current separately, and with knowledge of the capacitance of the capacitor bank, the stored energy can be obtained. A typical value of the pulse energy is \( \sim 50\text{mJcm}^{-2} \).

The electrical pulses are generally generated by discharging a capacitor bank via a semiconductor-based switch. The capacitors are charged by a power supply, and discharged in a repetitive manner. The size of the capacitor bank is typically 1-50 \( \mu \text{F} \), depending on the size of the target. A schematic of a HIPIMS power supply is shown in figure 7.2.
Often, there is also an inductance on the output. The size of the inductance is typically 20-50 \( \mu \text{H} \) [66,71,72]. The inductance prevents that the current increase is too steep, and damages the circuitry (for example the capacitors or the switch). Some power supplies also has the possibility to detect and suppress a transition to an arc discharge. These power supplies actively measures the voltage or the current on the output, and if it deviates too much from the normal operation it opens the switch, and the discharge is stopped. Versatile HIPIMS power supplies are today commercially available via Chemfilt Ionsputtering AB, with variable frequency and pulse width.

![Figure 7.3. Sinex 2.0 AS-14. A newly developed HIPIMS power supply from Chemfilt Ionsputtering AB.](image)

### 7.3 Plasma properties

The high power applied to the cathode generates a doughnut shaped plasma in front of the sputtering source. Langmuir probe measurements reveals that plasma densities of the order of \( 10^{19} \text{ m}^{-3} \) are obtained [73-75]. Figure 7.4 shows spatially resolved measurements of the plasma density in front of the sputtering source. It visualizes the plasma density during the plasma expansion in a plane parallel to the target, at a distance of 5 cm. This is visualized in more detail in paper III.
The electron temperature close to the target is more difficult to estimate. It might even not be physically correct to talk about a temperature in the close region of the target. Electrons ejected from the target surface are accelerated over the sheath region. They enter the bulk plasma region with more or less the same energy as the applied cathode potential (~ 100’s of eV). Electron energy distributions are then developed via various scattering processes. The concept of temperature is defined from a fully developed Maxwellian distribution. If a Maxwellian distribution is not developed, the concept of temperature is not physically correct, and the average electron energy is a better measure of how energetic the electrons are. However, at some distance from the target, the electrons have suffered a sufficient number of collisions that a Maxwellian-like distribution starts to develop. In this region, the magnetic field strength has dropped to a level where probe measurements are allowed. Gudmundsson et al [74,75] have measured the electron energy distribution, resolved in time and space. They found that the electron temperature is only 0.5 - 2 eV. However, it is expected that the average electron energy is much higher (1-2 orders of magnitude), in a close location to the target surface. Figure 7.5 shows the electron temperature at 10 cm from the target, according to Gudmundsson et al. Mozgrin et al [76] reports average electron temperatures of about 3-8 eV in a close vicinity of the target for similar conditions.
The highly energetic nature of the plasma (plasma kinetic energy density = nkT is large) allows a large fraction of the sputtered material to be ionized. The plasma density supplies for a short collision time, and the relatively high electron energy close to the target during the discharge allows ionization of sputtered material. More information of the plasma properties can be found in the attached papers.

**7.4 Advantages of HIPIMS**

As in any IPVD technique, the main advantage is the control over energy and direction of the deposition material. In some sense, HIPIMS can be seen as a method that combines the advantages of conventional magnetron sputtering and arc evaporation. It produces a highly ionized droplet free plasma. As mentioned previously, today there exists a number of plasma based processes that effectively ionize the deposition material. The main advantage of HIPIMS compared to these methods is the simplicity. Any existing magnetron-based deposition system can directly, or with little reconstruction, be turned into an IPVD system. This is an important feature for large-scale industrial applications. In principle all one needs is a HIPIMS power supply. The highly ionized conditions in HIPIMS can be used in a number of areas to improve the properties and performance of thin films.

The plasma conditions in HIPIMS can be used for growth of films using a bombardment of ions of the target material itself, which often results in dense, droplet free films. Dekoven et al [77] found that C films grown by HIPIMS had significantly higher density (> 35 %) compared to films grown by conventional magnetron sputtering. Sproul et al [78] demonstrated the
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possibility to produce oxide films with control over the optical parameters. Alami *et al* [79] showed that Ti-Si-C films grown by HIPIMS exhibited an enhanced microstructure. Films grown with HIPIMS on tilted surfaces showed improved quality [80]. Figure 7.6 shows two Ta films grown under similar conditions on 90° tilted Si Substrates in respect to the target surface. The film shown in (a) is a film grown by HIPIMS while (b) is grown with conventional sputtering. The film produced by HIPIMS exhibited a dense microstructure, with less columnar structure. No tilting of the columns was observed.

Figure 7.6 A comparison between two Ta films grown by HIPIMS and conventional magnetron sputtering. From Alami *et al* [80].

Ehiasarian *et al* [81-83] showed that CrN films prepared by HIPIMS pretreatment and HIPIMS deposition have been shown to have excellent adhesion, high density, and high corrosion and wear protection capabilities. Figure 7.7 shows the wear coefficient for various deposition techniques, and figure 7.8 shows the friction coefficient. The coatings produced by HIPIMS showed the lowest wear coefficient value, and a relatively low friction.
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Figure 7.7. The wear coefficient of various deposition techniques. From Ehiasarian et al [83].

Figure 7.8. The friction coefficient for CrN/NbN and Cr coatings deposited by Arc bond sputtering and HIPIMS. From Ehiasarian et al [83].

Furthermore Kouznetsov et al showed that HIPIMS could be a good alternative for filling of narrow trenches. Figure 7.9 shows a Cu filled trench produced with HIPIMS.
The highly ionized conditions in HIPIMS are also well suited for material pre-treatment. In conventional pretreatment, the substrate is bombarded with high-energy gas ions, usually Ar ions. The bombarding ions sputter the surface and remove the natural oxide layer that exists on most materials. In HIPIMS, a large fraction of the plasma ions, are from the target material (see paper IV). These ions can be used in surface pretreatment, for implantation. During the etching process, a part of the bombarding metal ions are implanted into the interface region, and creates a gradual change in composition between the substrate and the film. This is believed to have beneficial effects on the adhesion of the film. See paper V and references [81-83]. Figure 7.10 shows a STEM-EDS scan across the interface region for a CrN film grown onto a steel substrate. Implanted metal can be observed.

Figure 7.10. An STEM-EDS scan across the interface region of HIPIMS pre-treated films. From Ehiasarian et al [81].
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7.6 Drawbacks and problems

There are two main problems with the current version of HIPIMS, reduction of deposition rate and the transition to an arc discharge.

The deposition rate for the HIPIMS discharge is expected to be in the range 30-80 % compared to a conventional magnetron discharge, with the same average power. Bugaev et al [84] reported factor a reduction with a factor 2 for Cu and Ti sputtering. For reactive sputtering from a Ti target (TiO₂ films) a reduction of 4-7 times has been observed [85]. One explanation for the reduction in deposition rate is that some of the material that is ionized is attracted back to the cathode. In the presence of a magnetic field, as in a magnetron discharge, not all potential drop occurs over the sheath region, but a fraction of the applied voltage will penetrate the bulk plasma and create a plasma potential gradient. If a sputtered atom is ionized within this region it needs to have enough kinetic energy to overcome this potential in order to reach the substrate. If not, the sputtered atom is drawn back to the target, and causes so-called self-sputtering. In this case the ion is no longer available for deposition, and the rate drops. A plot of the deposition rate for HIPIMS compared to conventional sputtering versus the ratio between the self-sputtering yield (figure 7.11), and the Ar yield (that is the dominant mechanism in conventional sputtering) is evidence that self-sputtering is an important mechanism in HIPIMS.

![Figure 7.11. The relative deposition rate for HIPIMS versus self sputter yield. From Helmersson et al [62].](image-url)
Christie has also demonstrated this phenomenon with a parameter model, which fits well with experimental observations [86].

The other problem with HIPIMS is the arcing tendency on the cathode surface. There are mainly two types of arcs. Heavy arcs and light arcs. The light arc is minor problem, since it does not produce large number of microdroplets, as the heavy arc does. The light arc can be seen as a little spark on the target surface that does not extend further into the bulk plasma. The heavy arc can however be a serious problem. Heavy arcs can sometimes be observed as extended plasma columns going from the cathode into the bulk plasma. The arc locally heats the target, and microdroplets can be ejected. Figure 7.12 and 7.13 show a heavy arc, and the heating of the spots.

Figure 7.12. An arc occurring at the target surface. *
The origin of arcs is not fully investigated, but it is has been noticed that the arcing frequency can be related to the target purity, and surface morphology. Dielectric particles on the target surface may be the source of arcing, since they can be charged up to a high voltage, and then be discharged. The local discharge may strongly heat the spot, and initiate the arc. It has also been seen that the arcing frequency is strongly depending on the target material. Materials with relatively high melting point, such as Ti, Ta, and Cr has a very low tendency to arc, while materials with relatively low melting point, like Cu and Al, have a higher tendency to arc. Choosing a modest power level, or shorter pulse length can reduce the arcing frequency. It is also important to choose a pure target material, and sometimes pre-sputtering with conventional DC current can be necessary. Another alternative to reduce the arcing problem is to use a power supply with arc suppression [87].
8. Outlook

In this chapter I want to give my own personal view of the future of HIPIMS. Want remains to be understood? What problems should be solved, and where can the technique best be applied?

8.1 What remains to be understood?

Although HIPIMS has not yet been fully accepted by the industry, my personal believe is that it will be in the future, at least for some specific applications. There will always be a demand for increased performance of all high technology products, which in turn increases the requirements regarding film quality and thin film processing. Here HIPIMS can play a role. The plasma properties of HIPIMS in combination with the simplicity should be well suited for some applications. It is now up to the researchers in the field to find these. HIPIMS is still a relatively new technique and many things still remain to be understood. The technique is quite unique in the sense that it in a relatively easy way produces a highly ionized droplet free flux of deposition material, and can be applied to existing large scale deposition systems with no, or very little reconstruction [88]. In order to further increase the interest for the technique, more research should be conducted in a number of areas.

- Deposition rate. The main reason that the technique has not already been introduced in industry is the reduction in deposition rate. Work should therefore be focused on increasing the rate. This work may be focused on finding the optimum type of pulse or magnetic field design, and may also involve work on designing new power supplies.
- Arcing. More work should also be performed on the arcing phenomenon. What can be done to reduce the arcing frequency?
- What is the fraction of ionized material as function of the process parameters? Investigating the ionized flux fraction is a quite difficult task, where the experimental techniques often are associated with large uncertainties.
- The self-sputtering phenomenon. It is evident that self-sputtering occurs in HIPIMS, but very little work has been done to investigate this in detail.
- Film growth and surface engineering in general. Not enough work has been performed to show the beneficial effects of the technique on direct applications.
8. Outlook

- The charge transport phenomenon. It has been seen that the confinement of the magnetron is not as efficient as classical theory predicts. Why?
- Plasma chemistry and reactive film growth. What happens in to reactivity of the plasma species in the HIPIMS discharge? This is important knowledge that can be used in reactive film growth with HIPIMS.
- Design of new magnetrons. Can the HIPIMS discharge be optimized regarding rate and ionization by the design of new magnetrons?

8.2 Application areas

HIPIMS is currently taking the first steps towards industrial applications. The unique features of the discharge should be well suited for a number of application areas.

- Surface pre-treatment. The highly ionized and metallic plasma is well suited for combined etching and ion implantation. These might involve:
  - Adhesion enhancer for coatings on various tools (etching)
  - Adhesion layer for coatings on various substrates

- Film growth. The current version of HIPIMS, with the reduced deposition rate, should be applied to products where the increase in production cost is small in comparison to the gain in film quality. These can be areas where really thin films are desired or for coatings on expensive products, for example high-tech electronics. A few suggested application areas are listed below.
  - Metallization in general
  - Interconnects in IC structures
  - Protective overcoats on hard disks and read-write heads (C)
  - Deposition onto complex shaped objects
  - Coatings on medical products
  - Conducting & transparent electrodes in flat panel displays, touch panel displays, solar cells, etc. (ITO)
  - Al coatings in numerous applications
  - Optical coatings: anti-reflective and anti-static coatings, optical filters (SiO₂, TiO₂, SnO₂, ZnO, Al₂O₃, MgO, Nb₂O₅)
  - Wear- & corrosion-resistant coatings: cutting tools, tribological applications, etc. (CrNₓ or TiN)
  - Ag mirrors
  - Coatings on plastics
One of the reasons that not many researchers in the field do today use HIPIMS is that flexible power supplies has not been commercially available. Recently Chemfilt Ionsputtering AB have developed a new more flexible power supply, which will open the field for new users. This is an important development for the future of HIPIMS. To further increase the number of users and open for new areas to be explored, it is important that HIPIMS power supplies with tailor fit properties are available to the market. To meet the specific demands of the end products, HIPIMS may in the future exist in a number of forms regarding discharge size, number of targets, cathode geometry, power level, pulse type, and more.
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9. Contribution to the field

This chapter contains a summary of the work presented in the papers. What new knowledge came out if this work?

Paper I is about the ionization fraction of sputtered Ti. Optical emission spectroscopy (OES) was used to estimate the composition of the metal part of the plasma. The optical emission from the plasma was dominated by emission from metal ions. The distribution of metal ionized states clearly differed from the distribution of excited states, and we suggest the presence of a hot dense plasma surrounded by a cooler plasma. Sputtered material was ionized close to the target and transported into a cooler plasma region where the emission was also recorded. A Maxwell-Boltzmann distribution of excited states was assumed and the emission from the plasma was quantified and showed singly ionized ion to neutral ratios of over 90%. Even at relatively low applied pulse power, a high degree of ionization was observed. OES analysis of the discharge in a DC magnetron discharge was also made, which demonstrated much lower ionization. The results are evidence that HIPIMS has an effective ionization mechanism.

Paper II contains measurements of the magneto-dynamic properties of the discharge. Spatially resolved magnetic field measurements are presented and the magnetic disturbance is quantified for different process parameters. The magnetic field is severely deformed by the discharge and we record changes of several mT, depending on the spatial location of the measurement. The shape of the deformation reveals the presence of azimuthally drifting electrons close to the target surface. We note that the axial (discharge) current density is much too high compared to the azimuthal current density to be explained by classical collision terms, and an anomalous charge transport mechanism is needed. These results are important for the fundamental understanding of the HIPIMS discharge.

In paper III we present the spatial electron density distribution as function of time in a high power pulsed magnetron discharge. A Langmuir probe was positioned in various positions below the target and the electron density was mapped out. We recorded peak electron densities exceeding $10^{16}$ m$^{-3}$ in a close vicinity of the target. The dynamics of the discharge showed a dense plasma expanding from the “race-track” axially into the vacuum chamber, to be followed
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by a second density increase, hundreds of μs after the pulse was switched off. We also record electrons trapped in a magnetic bottle where the magnetron magnetic field is zero, formed due to the unbalanced magnetron. These results are fundamental in order to understand the plasma dynamics and the ionization mechanism.

In paper IV the energy distribution of sputtered and ionized metal atoms as well as ions from the sputtering gas are presented. Time resolved, and time averaged ion energy distributions were recorded with an energy resolving quadrupole mass spectrometer. The ion energy distributions recorded for the HIPIMS discharge are broader with maximum detected energy of 100 eV and contain a larger fraction of highly energetic ions (about 50 % with $E_i > 20$ eV) as compared to a conventional DC magnetron sputtering discharge. The composition of the ion flux was also determined, and reveals a high metal fraction. During the most intense moment of the discharge, the ionic flux consisted of approximately 50 % Ti$^{1+}$, 24 % Ti$^{2+}$, 23 % Ar$^{1+}$, and 3 % Ar$^{2+}$ ions. The plasma composition and plasma kinetics are absolute central knowledge in any plasma-based thin film deposition technique.

In paper V we demonstrate the possibility to use the HIPIMS discharge for surface etching and ion implantation. A surface pretreatment by HIPIMS followed by reactive unbalanced DC magnetron sputtering deposition was performed using a Cr target. When applying a negative substrate bias $U_b$ the adhesion was enhanced due to sputter cleaning of the surface and metal ion intermixing in the interface region. This intermixing, resulting in a gradual change of the composition, is considered to enhance the adhesion of the hard coatings on steel substrates. The adhesion was found to depend on the substrate bias as well as on the target power and, for low substrate bias, on the duration of the pretreatment. The influence of the target peak voltage, the substrate bias as well as pretreatment time on the constitution and morphology of the interface after the pretreatment is discussed applying analytical transmission electron microscopy.

Paper VI contains a study of the ability to control the deposition flux using an external magnetic field. An external magnetic field was created with a current carrying coil placed in front of the target. To measure the distribution of deposition material samples were placed in an array surrounding the target and the depositions were made with and without the external magnetic field. The distribution is significantly changed when the magnetic field is present. An increase of 80 % in deposition rate is observed for the sample placed in the central position (right in front
of the target center) and the deposition rate is strongly decreased on samples placed to the side of the target. The measurements were also performed on a conventional DC magnetron discharge, but no major effect of the magnetic field was observed in that case. The results show the potential to guide the deposition material by the use of a magnetic field, and are another indication of the effective ionization of the sputtered material.
9. Contribution to the field

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