MASTER

The potential of ellipsometry in unraveling the plasma sheath potential

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The Potential of Ellipsometry in unraveling the Plasma Sheath Potential

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Abstract

The electric field in the sheath of a low pressure plasma plays an important role in many plasma material processing methods. To further optimize these methods - and even more importantly to understand fundamental plasma processes - a good understanding of this electric field profile is crucial. However, determining the absolute values and the profile of the sheath electric field experimentally has appeared to be extremely difficult and therefore all available methods lack spatial resolution or are invasive. For instance laser-based techniques have poor spatial resolution and their usage is limited to only those regions where the electric field is relatively high. Probe techniques disturb the electric field and are therefore not feasible to use in the plasma sheath.

In this report, a novel approach to electric field measurements in the plasma sheath based on plasma levitated micro-particles is proposed. The first steps that are taken to realize this idea are described here. In the framework of this project a rotating compensator ellipsometer has been developed, including the microcontroller system and the associated source code.

To verify the feasibility of the proposed method and the correct functioning of the built diagnostics, the following essential milestones have been achieved.

The correct working of the developed ellipsometer has been verified by an investigation of different oxidized Si wafers. The results obtained by the self-built ellipsometer and those obtained by a commercial ellipsometer are compared. The results are in good agreement thanks to the significant effort which is put in to the calibration of the in-house built ellipsometer.

To convert the measured ellipsometric angles into physical parameters a model based on Mie theory is derived. Calculations are performed to create a lookup table for the ellipsometric angles \( \Psi \) and \( \Delta \) for different particle radii.

The first experiments on confined clouds of particles are conducted and the first ellipsometric angles are determined. Analyzing the obtained data has not been possible yet as the model does not take into account the size distribution of the particles in the cloud. Insufficient light is detected during single particle experiments hence a better and more stable light source will be installed to increase the signal.

A crucial part of the proposed diagnostics is the imaging setup. This setup is fully functional and will be used to determine the position of the plasma levitated dust particle during the electric field measurements. Two test measurements to determine the change of position of a dust particle due to mass loss in a reactive oxygen plasma are performed successfully.

The work described above demonstrates the feasibility of the proposed diagnostics to develop into a mature measurement technique with superior specifications. To that end, the next steps to further improve the ellipsometry setup and challenges have been defined and elaborated towards measurements on a single particle and the proverbial Holy Grail for a plasma sheath physicist: determining the electric field profile at the boundary of a plasma.
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Preface

The results of my master graduation project in the group Elementary Processes in Gas Discharges (EPG) of Prof.dr.ir. Gerrit Kroesen at Eindhoven University of Technology are presented in this report. I started working in the group under supervision of Ir. Leroy Schepers and Dr.ir. Job Beckers approximately a year ago.

In this period I received the freedom to start working on a novel method to measure electric fields in plasmas. From the beginning it was clear that it would take multiple years to pursue the idea, so the proposed method has not been fully implemented yet. However, a new setup has been developed and built to pursue the idea and the first measurements are performed successfully. I have committed to a PhD project within EPG around the time of the start of the construction of the setup. Within that framework I will be able to continue working on this topic and complete the project in order to develop a mature and novel diagnostics tool that can unravel fundamental plasma physics in the space charge region of a wide range of types of plasmas. I am looking forward to start my PhD and I am expecting fruitful years.

Bart Platier
Eindhoven, 17 May 2015
Chapter 1
Introduction

Near a surface at the edge of a quasi-neutral plasma a positive space charge region, the plasma sheath, is formed due to the difference in mobility between electrons and ions. The presence of the space charge leads to the formation of an electric field through Poisson’s equation and consequently to a potential drop at the edges of a plasma. These electric fields are used to accelerate positive ions towards the surface in applications like deposition, etching and sputtering. Although experimental [1–7] and theoretical [8–10] work has been performed, currently no accurate method to determine the electric field profile is available for a broad range of low pressure plasma setups.

The physics of the plasma sheath becomes even more complex for plasmas in electronegative gases, like oxygen and etchants (e.g. CF$_4$ and SF$_6$), as they contain negative ions which influence and contribute to the charge distribution. These etchants are widely used in for instance the semiconductor industry, a sector with little margin for error, and therefore it is crucial to have good understanding of the used production processes.

The electric field plays an important role in levitating particles in both dusty and complex plasmas due to collection of charged plasma species on the particles. These particles - nm to µm in size - have a surface charge once immersed in plasma and are therefore able to counter gravity in the electric field of the sheath. Several studies are performed to explain the charging processes [11–14], however this topic is not fully elucidated.

Beckers [6] introduced a technique based on measuring the equilibrium position of a dust particle for different gravity conditions (Figure 1.1a) to obtain the product of the electric field and charge of a micro particle. A model, which takes into account only the electric and gravitational force, was used to separate the electric field and the particle charge (Figure 1.1b). The disadvantages of this method are the need to change the gravitational conditions and the use of a model which includes and excludes certain plasma sheath processes.

Theory of inventive problem solving (TRIZ) tools are used by the author in this investigation to develop a new method to measure the electric field in the sheath spatially resolved without affecting the plasma. One of the most promising ideas is levitating a micrometer-sized particle with piezo electric properties in the sheath. The particle will deform as a function of the local electric field. This deformation can be measured by means of ellipsometry and the obtained information concerning the deformation can be used to calculate the electric field. These measurements can be performed under micro or hypergravity conditions to nonintrusively and spatially resolved probe the electric field strength. However, using particles with different masses is a more pragmatic way
(a) Photograph of plasma-confined micro particle for three gravity conditions.

(b) Electric field and particle charge profiles.

Figure 1.1: Beckers et al. \cite{6} controlled the position of the particle by changing the gravity conditions (1.1a) which is used in concert with a plasma physical sheath model to obtain the electric field and particle charge profile (1.1b).

to probe the sheath, since levitation position depends on particle mass. The first steps to realize this idea are described in this thesis.

1.1 Goals

The main objective is to measure the electric field strength in the sheath of an O$_2$ plasma nonintrusively and spatially resolved. As developing the measuring technique proposed here including developing and constructing a setup, testing, performing measurements and verification with models would take multiple years, the following sub goals are defined for this MSc project:

- develop a method to ‘directly’ measure the electric field strength
- decide which type of ellipsometer to build
- develop electronics and source code for the ellipsometer
- build and calibrate the ellipsometer
- verify the working of the apparatus
- study dust particles with the ellipsometer

1.2 Outline

An introduction to plasma, with a focus on electronegativity and plasma levitation of dust particles, is given in Chapter 2. The interaction of light with a dust particle and how this can be used for the characterization of the dust particle is described in Chapter 3. The experiments and the developed experimental configuration are presented in Chapter 4. Chapter 5 gives an overview of the performed calibration of the ellipsometer, which was specifically built in the framework of this project. The results of the experiments are presented in Chapter 6 and these will be discussed in Chapter 7. The conclusions concerning this investigation are presented in Chapter 8. Detailed information on components of the setup can be found in the Appendices A-C.
Chapter 2

Levitating Particles in a Plasma

In this chapter a brief introduction to plasmas, the fourth state of matter, is given. Besides the basic plasma parameters, this chapter also covers the aspects of levitating a dust particle in the edge - the sheath - of a plasma and the possibility of a plasma being electronegative as is the case in for instance plasmas in an $O_2$ gas.

2.1 Radio-Frequency Plasma

An ionized gas, which contains freely moving ions and electrons, is called a plasma. One of the most common methods to generate a plasma is by applying a voltage over two electrodes. This type of plasma is referred to as a capacitively coupled plasma (CCP). Charge carriers between the electrodes are accelerated by the applied electric field. The kinetic energy gained in the field is used for further ionization of the gas. CCPs can be driven by a constant or an alternating voltage over the electrodes. In the case of a sinusoidal voltage with a frequency in the range of 1 - 500 MHz the plasma is referred to as a radio-frequency (RF) plasma. In this work a low pressure (0.1 - 10 Pa) plasma in the RF (13.56 MHz) regime is applied.

2.1.1 Relevant Plasma Parameters

A few plasma parameters are rather intuitive to describe physical plasma processes and these are described below.

The mass of an electron $m_e$ is much smaller than the mass of an ion $m_i$. Therefore the electrons have a much higher mobility and are therefore able to follow alternating electric fields at much higher frequencies. The plasma frequency is a parameter which indicates the frequency of an electric field at which a specific particle is able to follow the field. The plasma frequency of electrons and of ions $f_{e,i}$ is given by

$$f_{e,i} = \frac{1}{2\pi} \sqrt{\frac{n_{e,i}Z_{e,i}e^2}{m_{e,i}\varepsilon_0}}, \quad (2.1)$$

where the subscripted letter $e$ or $i$ indicates electrons and ions respectively, $n_{e,i}$ is the number density, $Z_{e,i}$ the charge number, $m_{e,i}$ the mass of one particle, $e$ the electron charge and $\varepsilon_0$ the dielectric constant in vacuum.

For a plasma in an Argon gas with $n_e=n_i=10^{15}$ m$^{-3}$ and $z=1$, $f_e$ and $f_i$ are $2.84 \cdot 10^8$ Hz and $1.05 \cdot 10^6$ Hz respectively. The driving frequency of 13.56 MHz is much lower than the plasma frequency of the electrons $f_e$ and much higher than the plasma frequency of
the ions $f_i$. Therefore the electrons are able to follow this driving frequency, while the ions can only follow time averaged electric fields. Due to the difference in interaction with the electric field the temperature of the electrons $T_e$ is higher than the temperature of the ions $T_i$ for a low pressure and low power CCP.

The bulk of a plasma is quasi-neutral, which means that the charge of plasma overall is neutral. However deviations from neutrality can occur on small length scales. A characteristic length scale in which these deviations still can occur is the Debye length $\lambda_D$, which is given by

$$\frac{1}{\lambda_D^2} = \frac{1}{\lambda_{De}^2} + \frac{1}{\lambda_{Di}^2}, \quad (2.2)$$

where $\lambda_{De}$ and $\lambda_{Di}$ are respectively the electron and ion Debye length. These lengths are given by

$$\lambda_{De,i} = \sqrt{\frac{\epsilon_0 k T_{e,i}}{e^2 n_{e,i}}}, \quad (2.3)$$

where $k$ is the Boltzmann constant.

The mean free path $\lambda_{mf}$ is the average distance between consecutive collisions. The $\lambda_{mf,1}$ of particle 1 to collide with particle of type 2 is given by

$$\lambda_{mf,1} = \frac{1}{n_2 \sigma_{12}}, \quad (2.4)$$

where $n_2$ is the number density of particle 2 and $\sigma_{12}$ the collision cross section for particle 1 with particle 2.

### 2.1.2 Plasma Sheath

Most laboratory plasmas are spatially confined by for example the reactor wall and the electrodes. Due to the interaction of the plasma with these surfaces a positive space-charge region is formed near these surfaces. This region is called the plasma sheath and is developed directly after igniting the plasma. First, the walls become negatively charged due to light electrons which are able to reach the wall earlier than the heavy ions which are left behind building up a space charge and consequently an electric field. Due to this electric field electrons are repelled and positive ions are attracted by the surface until an equilibrium between electron and ion currents to the wall is reached. The stationary layer above a surface in which these electric fields are present is typical a few Debye lengths thick. Ion densities in this region are in the same order (slightly less) as in the bulk, however electrons are depleted to a significant extend.

To sustain the plasma sheath, the ions entering this region must have a minimum velocity which is in the order of the speed of sound. This minimum velocity of the ions is referred to as the Bohm velocity. An intermediate layer is present between the bulk of the plasma and the sheath, the presheath, where the ions are accelerated by a small electric field.

A tentative overview of the time-averaged electron and ion densities, and the electric field throughout the different regions is shown in Figure 2.1.

The plasma sheath is not a steady state phenomena in a RF discharge and variations of the thickness and potential do occur within a cycle of the driving frequency. However it is possible to give a description of the plasma sheath time-averaged over multiple periods of the driving frequency as is done below for the collisionless and collisional sheath.
Figure 2.1: Tentative schematic of typical time-averaged electron and ion densities, and electric field profile in the different plasma regions. (Adapted from [15])
Collisionless Sheath

By assuming that the $\lambda_{mfp,i}$ is much larger than the thickness of the sheath $\xi$, the ionization and recombination processes can be neglected in the sheath and no energy will be lost by collisions. Therefore the particle flux must be constant for all vertical positions $z$ in the sheath for the case of a horizontal surface

$$u_{i,e,sh} n_{i,e,sh} = u_{i,e} (z) n_{i,e} (z),$$

where $u_{i,e}$ is the directed velocity and subscript $sh$ indicates that the quantity is evaluated at the presheath-sheath boundary.

An expression can be found for the potential $\phi$ in the sheath by making the following additional assumptions: quasi-neutrality at the sheath edge, electrons obeying the Boltzman distribution and cold ions

$$\frac{d^2 \phi (z)}{dz^2} = -\frac{e}{\epsilon_0} n_{i,sh} \left( \left\{ 1 - \frac{2e\phi (z)}{m_i u_{i,sh}^2} \right\}^{-1/2} - e^{\phi (z)} \right).$$

(2.6)

The sheath thickness $\xi_{\text{collisionless}}$ in the collisionless case can be approximated by the following equation \[16\]

$$\xi_{\text{collisionless}} = \sqrt{\frac{100}{243} \lambda D (\frac{2\phi_0}{T_e})^{3/4}},$$

(2.7)

where $\phi_0$ is the sheath potential at the sheath edge.

Collisional Sheath

The sheath is collision dominated when $\lambda_{mfp,i}$ is much smaller than $\xi$. In this type of sheaths, the ions collide often with gas neutrals and therefore they experience a drag force. The local ion velocity $u_i$ can be described by

$$u_i (z) \frac{du_i (z)}{dz} = -\frac{e}{m_i} \frac{d\phi (z)}{dz} - n_n \sigma_{in} u_i^2 (z),$$

(2.8)

where $n_n$ is the particle density of neutral particles and $\sigma_{in}$ the ion-neutral collision cross section.

For a collisional sheath the thickness $\xi_{\text{collisional}}$ can be approximated by the following equation \[17\]:

$$\xi_{\text{collisional}} = 1.155 \left( \frac{e^3 \phi_0^3 \lambda_{mfp}}{u_{i,sh}^2 m_i k^2 T_e^2 \lambda D} \right)^{1/5}.$$

(2.9)

2.2 Electronegativity of O$_2$ Plasmas

There are several definitions and scales for electronegativity, however what they all have in common is that the higher the electronegativity number the more electrons are attracted by an atom or a group of atoms.

An oxygen plasma contains many different species, including the negative ions O$^-$, O$_2^-$ and O$_3^-$ as O$_2$ is an electronegative gas. These negative ions contribute to and change the spatial distribution of charged particles and therefore also influence the...
sheath thickness, potential and electric field profile. Negative ion densities are reported in an oxygen plasma over 18 times higher than the electron density [18].

Vender [19] investigated experimentally as well as theoretically the charged-species profiles in oxygen RF plasmas and showed that $\text{O}^-$ is the most dominant negative ion. One of the number density profiles of negative ions in an oxygen plasma measured by Vender is shown in Figure 2.2. The much lower number density of electrons is indicated by the dashed line.

2.3 Dusty Plasma

Besides the neutrals, the (positive and negative) ions and the free electrons, a plasma can also contain macroscopic particles of solid or liquid material which are called dust particles. The range of dimensions of these particles can be as low as a nanometer up to several $\mu$m. The charging of and the forces on these particles are described below.

2.3.1 Charging of Dust Particles

The process which charges the reactor wall, as discussed in the previous section, also causes the dust particles having a surface charge once immersed in plasma and is called charge carrier collection. Although there are many more charging processes [20], only a few will be treated here. Collisions of highly energetic ions and electrons with the particle are able to cause the particle to lose electrons by secondary electron emission. The absorption of photons by the particle can lead to the release of electrons and is normally referred to as photoemission. The last process which will be treated here is thermionic emission: the release of electrons and ions due to extreme heating. In low pressure plasmas is charge carrier collection the most dominant process, however photoemission can no longer be neglected when a high power laser is used [17].

Orbit-Motion Limited (OML) theory [11] can be used to estimate the charge of a particle $Q_p$. Several assumption are made in this model including: the particle radius $r_p$,
is much smaller than the Debye length, the energy distribution of the ions is Maxwellian and the energy of the electrons follow the Boltzmann distribution. Based on these assumptions the electron and ion currents are calculated. In steady state, these currents should cancel each other out and from this assumption the floating potential of the particle $V_p$ can be determined. This potential only depends on the ratios of the ion-electron temperature and mass. By considering the particle as a capacitor with a capacity of $4\pi\epsilon_0 r_p$, the charge of the particle is given by

$$Q_p = 4\pi\epsilon_0 r_p V_p(r_p),$$  \hfill (2.10)

where $\epsilon_0$ is the vacuum permittivity.

The typical charge of a particle with a radius of 10 $\mu$m is several ten thousand electron charges when confined in a plasma with typical parameters of a $T_e$ of 3 eV and $T_i$ of 300 K. \[15\]

**2.3.2 Forces on Dust Particles**

Several forces act on a dust particle \[20\] and a non-exhaustive list of these forces is treated below. Under specific conditions it is possible that the net force on the particle is zero. Besides the trivial case where the particle is supported by a surface, the net force can be zero while the particle is levitated. A schematic overview of a dust particle that is levitated in the sheath near the bottom electrode and three important forces are shown in Figure 2.3.

**Electric Force**

The interaction of the charged particle with the electric field in the sheath is one of the most dominant interactions, because the fields are up to $10^4$ V/m \[6\] and the particle...
has a non-zero electric charge. Assuming the particle is small with respect to the Debye length, the vector of the electric force $\vec{F}_E$ is given by

$$\vec{F}_E = Q_P \vec{E},$$

(2.11)

where $Q_P$ is the total electric charge of the dust particle and $\vec{E}$ the electric field vector. It must be noted that both $E$ and $Q_P$ vary within the RF cycle. However, the inertia of the particle allows the forces working to be treated as time-averaged.

**Ion Drag Force**

In the sheath’s electric field positively charged ions are accelerated towards the wall. Due to the negative charge of the particle the positive ions are attracted by the particle increasing the chances to collide with the particle. During these collisions with the particle the ions transfer momentum. The force related to this phenomenon is referred to as the collection ion drag force $\vec{F}_{i\text{coll}}$ and is given by

$$\vec{F}_{i\text{coll}} = n_i m_i u_i \sigma_{\text{coll}} ,$$

(2.12)

where $u_i$ the directed velocity of the ions and $\sigma_{\text{coll}}$ the ion collection cross section. Note that this cross section is significantly larger than the geometrical cross section due to the infinite range of Coulomb interactions.

Not all ions which are deflected by the Coulomb field of the dust particle are collected, but due to the deflection a volume with positive space charge beneath the particle will form. This positive charge will pull on the particle. The orbit ion drag force $F_{i\text{orb}}$ is given by

$$\vec{F}_{i\text{orb}} = m_i v_{i,\text{tot}} n_i \vec{u}_i \sigma_{\text{orbit}},$$

(2.13)

where $v_{i,\text{tot}}$ is the total velocity of the ions and $\sigma_{\text{orbit}}$ is the related cross section.

**Gravitational Force**

For micrometer-sized particles gravity plays an important role. The gravitational force $\vec{F}_g$ for a spherical particle is given by

$$\vec{F}_g = \frac{4}{3} \pi r_p^3 \rho_p \vec{g},$$

(2.14)

where $r_p$ is the particle radius, $\rho_p$ the particle mass density and $\vec{g}$ the gravitational acceleration. Changing the gravity conditions in a laboratory requires special equipment like a centrifuge or an airplane for parabolic flights [15].

**Thermophoretic Force**

The position of the particle can also be controlled by applying a temperature gradient, however the disadvantage of this method is that it influences the plasma parameters. If the temperature of the neutral gas has a gradient, the thermal velocity of the neutrals is higher on the warmer side of the dust particle than on the colder side. Therefore more momentum is transferred during collisions at the warmer side. The net force which pushes the particle towards the colder side is called the thermophoretic force $\vec{F}_{th}$ and is given by

$$\vec{F}_{th} = -\frac{32}{15} \frac{r_p^2}{v_{th,N}} \left[ 1 + \frac{5\pi}{32} (1 - \alpha) \right] k_T \nabla T_N,$$

(2.15)
where $v_{th,N}$ the thermal velocity of the neutrals, $k_T$ the thermal conductivity of the gas, $T_N$ the neutral gas temperature and $\alpha$ an accommodation coefficient.
Chapter 3

Ellipsometry on spherical Particles

In this research project spherical particles are studied by means of ellipsometry: a measuring technique based on the change of polarization state of light by a sample. A brief introduction to light and the polarization of light is given in the first paragraph. In the second paragraph an extensive description of a mathematical model which describes light scattering by spherical particles is given. This model is used to calculate physical parameters of the spheres from the results obtained by ellipsometry. This method is treated in paragraph 3.4.

3.1 General Introduction to Light

The term light is used for electromagnetic (EM) radiation with a wavelength visible by the human eye. These transverse waves consist of an electric and a magnetic field, which are mutually perpendicular and perpendicular to the direction of propagation. Figure 3.1 shows a representation of an EM wave propagating in the z direction. EM waves behave in agreement with the Maxwell equations.

The magnetic field strength $H$ and the electric field $E$ of an EM wave at a position

![Figure 3.1: Representation of an electromagnetic wave propagating with a velocity $c$ in the $z$ direction. The magnetic field strength $H$ and the electric field $E$ are perpendicular to each other. (Adapted from [21])](image-url)
\[ H = H_0 \exp \left( i [\omega t - K z + \delta] \right), \]  
\[ E = E_0 \exp \left( i [\omega t - K z + \delta] \right), \]

where \( \omega \) is the angular frequency, \( K \) the wavenumber and \( \delta \) the phase. From these equations can be concluded that the two waves are always in phase. The amplitude of the electric field \( E_0 \) and the magnetic field strength \( H_0 \) are coupled by the following relation

\[ E_0 = c \mu H_0, \] (3.2)

where \( c \) is the speed of light in vacuum and \( \mu \) the magnetic permeability of the medium.

In the interest of simplicity, only the electric field is considered for the description of light. The direction of the oscillation of the electric field is referred to as the polarization. For example EM radiation where the \( E \) fields only oscillates along the \( x \) axis is referred to as \( x \)-polarized light. Linear combinations of electromagnetic waves with phase differences might make it more complex. Therefore methods, to describe light in an optical system, like Jones calculus, are used. In Jones calculus the polarization state of light is indicated by a Jones vector

\[ \vec{E} = \left| E_x \right| \]  
\[ \left| E_y \right| \] .

The polarization of light after an optical element can be calculated by taking the product of the Jones matrix of the optical element and the Jones vector which describes the incoming light. Note that Jones vectors describe the polarization state as a superposition of \( x \)- and \( y \)-polarized light and is therefore not able to represent unpolarized light.

The intensity \( I \) is given by the product of a Jones vector with its complex conjugate

\[ I = \vec{E} \cdot \vec{E}^\dagger. \] (3.4)

The \( xyz \)-coordinate system can be inconvenient for calculating the effects of an optical element, for example reflection or refraction by a surface, and therefore another reference system is introduced. Oscillations of the electric field parallel to the plane spanned by the propagation vector and the normal vector of the surface are referred to as \( p \)-polarized and oscillations perpendicular to the plane as \( s \)-polarized. The designations \( p \) and \( s \) originate from the German words \textit{parallel} and \textit{senkrecht}, meaning parallel and perpendicular respectively. The plane spanned by the propagating vector of the incoming ray and the normal vector of the surface is called the plane of incidence. In this investigation the \( x \) axis is defined parallel to this plane and the polarization states are defined from the point of view of the receiver (ray propagating towards observer).

Reflection of light is an event which changes the amplitude of the fields. The amplitude reflection coefficients \( r_p \) and \( r_s \) for respectively \( p \)- and \( s \)-polarization are given by

\[ r_p = \frac{E_{rp}}{E_{ip}}, \quad r_s = \frac{E_{rs}}{E_{is}}, \] (3.5)

where the incoming beam is indicated by a subscripted \( i \) and the reflected beam by \( r \).
3.2 Mueller Calculus

Contrary to Jones calculus, Mueller calculus is able to treat partially polarized, randomly polarized and incoherent light. The state of polarization in Mueller calculus is given by a 4-dimensional vector which is called a Stokes vector \( \vec{S} \)

\[
\vec{S} = \begin{bmatrix}
I \\
Q \\
U \\
V
\end{bmatrix} = \begin{bmatrix}
I_x + I_y \\
I_x - I_y \\
I_{+45} - I_{-45} \\
I_{RHC} - I_{LHC}
\end{bmatrix},
\]

(3.6)

where \( I_x \) is the light intensity measured in the \( x \) direction, \( I_y \) in the \( y \) direction, \( I_{+45} \) and \( I_{-45} \) under an angle of 45° and -45° in respect to the positive \( x \) axis, \( I_{LHC} \) the intensity of left hand circular polarized light and \( I_{RHC} \) the intensity of right hand circular polarized light. The total intensity is given by the first element \( I \) of the vector \( \vec{S} \). An overview of normalized Stokes vectors (\( I = 1 \)) for common polarizations can be found in Table 3.1.

Most polarization states given in this table do not need additional explanation, however the last mentioned state, elliptically, is less trivial. Elliptically polarized light is a superposition of circularly and linearly polarized light. This polarization state is shown in Figure 3.2 and can be described by the following parameters \( \Delta \) and \( \Psi \) which are given by respectively

\[
\Delta = \delta_x - \delta_y, \tag{3.7a}
\]

\[
\tan (\Psi) = \frac{E_{x0}}{E_{y0}}, \tag{3.7b}
\]

where \( \delta_i \) is the phase of \( i \)-polarized light and \( E_{i0} \) the maximum electric field in the \( i \) direction. For linearly polarized light \( \Delta \) is 0°, while \( \Delta \) is 90° for circularly polarized light.

Effects of optical elements on the polarization can be calculated by applying Mueller calculus on an initial Stokes vector \( \vec{S}_{in} \) to obtain the outgoing Stokes vector \( \vec{S}_{out} \).

\[
\vec{S}_{out} = \mathcal{M} \cdot \vec{S}_{in}, \tag{3.8}
\]

where \( \mathcal{M} \) is a Mueller matrix corresponding to a specific optical element.

Mueller matrices which are needed to describe a rotating compensator ellipsometer are given in Table 3.2. The two optical elements which are often used for ellipsometry are described in more detail below.

Polarizer

An ideal polarizer is an optical element that blocks all light except for one specific polarization. The Mueller matrix \( \mathcal{P} \) describes the behavior of a polarizer with a transmission axis parallel to the \( x \) axis.

Compensator

A compensator, also called retarder, has a refractive index that depends on the polarization and can therefore generate a phase difference \( \Delta_c \) between the two electric field vectors.
Table 3.1: Stokes vectors for common polarization states as defined from the point of view of the receiver.

<table>
<thead>
<tr>
<th>polarization</th>
<th>polarization state</th>
<th>Stokes vector</th>
</tr>
</thead>
<tbody>
<tr>
<td>unpolarized</td>
<td></td>
<td>$\begin{bmatrix} 1 \ 0 \ 0 \ 0 \end{bmatrix}$</td>
</tr>
<tr>
<td>linear at $0^\circ$</td>
<td></td>
<td>$\begin{bmatrix} 1 \ 1 \ 0 \ 0 \end{bmatrix}$</td>
</tr>
<tr>
<td>linear at $90^\circ$</td>
<td></td>
<td>$\begin{bmatrix} 1 \ -1 \ 0 \ 0 \end{bmatrix}$</td>
</tr>
<tr>
<td>linear at $45^\circ$</td>
<td></td>
<td>$\begin{bmatrix} 1 \ 0 \ 1 \ 0 \end{bmatrix}$</td>
</tr>
<tr>
<td>right circular</td>
<td></td>
<td>$\begin{bmatrix} 1 \ 0 \ 0 \ 1 \end{bmatrix}$</td>
</tr>
<tr>
<td>left circular</td>
<td></td>
<td>$\begin{bmatrix} 1 \ 0 \ 0 \ -1 \end{bmatrix}$</td>
</tr>
<tr>
<td>elliptically</td>
<td></td>
<td>$\begin{bmatrix} 1 \ -\cos(2\Psi) \ \sin(2\Psi)\cos(\Delta) \ -\sin(2\Psi)\sin(\Delta) \end{bmatrix}$</td>
</tr>
</tbody>
</table>
\[ \Delta_c = \delta_{x,c} - \delta_{y,c} \]  

where \( \delta_{i,c} \) is the phase difference caused by the compensator in the \( i \) direction.

The azimuth with the lowest refractive index is called the fast axis and is per definition referred to as the \( x \) axis. The phase difference can be controlled by changing the orientation of the compensator. A compensator with a phase difference of \( \delta_c = \frac{\pi}{2} \) is called a quarter-wave plate because the phase difference is in this case exactly a quarter of the wavelength. Incoming \( x \)-polarized light becomes circularly polarized light by passing a quarter-wave plate positioned at an angle of 45°. Note that the phase difference depends on the wavelength of the light as well.

The Mueller matrix of an imperfect compensator \( C \) is given in Table 3.2. During the calibration process, which is described in Appendix 5, the properties \( \Delta_c \) and \( \Psi_c \) of the imperfect compensator are determined.
Table 3.2: Mueller matrices for optical elements which are used in rotating compensator ellipsometry. The transmission axis of the polarizer and analyzer is parallel to the x axis. The properties of the compensator are given by $\Delta_c$ and $\Psi_c$. The fast axis of this optical element is parallel to the x axis. The rotation matrix rotates the frame of reference by an angle $\alpha_i$. The by reflection caused amplitude ratio of p- and s-polarized light is given by $\Psi$ and the phase between the two polarizations by $\Delta$. $r_p$ and $r_s$ are the reflection coefficients for respectively p- and s-polarized light.

<table>
<thead>
<tr>
<th>element</th>
<th>notation</th>
<th>Mueller matrix</th>
</tr>
</thead>
<tbody>
<tr>
<td>polarizer</td>
<td>$\mathcal{P}$</td>
<td>$\begin{pmatrix} 1 &amp; 1 &amp; 0 &amp; 0 \ 1 &amp; 1 &amp; 0 &amp; 0 \ 0 &amp; 0 &amp; 0 &amp; 0 \ 0 &amp; 0 &amp; 0 &amp; 0 \end{pmatrix}$</td>
</tr>
<tr>
<td>analyzer</td>
<td>$\mathcal{A}$</td>
<td>$\begin{pmatrix} 1 &amp; \cos(2\Psi_c) &amp; 0 &amp; 0 \ \cos(2\Psi_c) &amp; 1 &amp; 0 &amp; 0 \ 0 &amp; 0 &amp; \sin(2\Psi_c)\cos(\Delta_c) &amp; \sin(2\Psi_c)\sin(\Delta_c) \ 0 &amp; 0 &amp; -\sin(2\Psi_c)\sin(\Delta_c) &amp; \sin(2\Psi_c)\cos(\Delta_c) \end{pmatrix}$</td>
</tr>
<tr>
<td>imperfect compensator</td>
<td>$\mathcal{C}$</td>
<td>$\begin{pmatrix} 1 &amp; 0 &amp; 0 &amp; 0 \ 0 &amp; \cos(2\alpha_i) &amp; \sin(2\alpha_i) &amp; 0 \ 0 &amp; -\sin(2\alpha_i) &amp; \cos(2\alpha_i) &amp; 0 \ 0 &amp; 0 &amp; 0 &amp; 1 \end{pmatrix}$</td>
</tr>
<tr>
<td>rotation</td>
<td>$\mathcal{R}(\alpha_i)$</td>
<td>$\begin{pmatrix} 1 &amp; -\cos(2\Psi) &amp; 0 &amp; 0 \ -\cos(2\Psi) &amp; 1 &amp; 0 &amp; 0 \ 0 &amp; 0 &amp; \sin(2\Psi)\cos(\Delta) &amp; \sin(2\Psi)\sin(\Delta) \ 0 &amp; 0 &amp; -\sin(2\Psi)\sin(\Delta) &amp; \sin(2\Psi)\cos(\Delta) \end{pmatrix}$</td>
</tr>
<tr>
<td>sample</td>
<td>$\mathcal{S}$</td>
<td>$\frac{1}{\sqrt{r_p^2 + r_s^2}}\begin{pmatrix} 1 &amp; -\cos(2\Psi) &amp; 0 &amp; 0 \ -\cos(2\Psi) &amp; 1 &amp; 0 &amp; 0 \ 0 &amp; 0 &amp; \sin(2\Psi)\cos(\Delta) &amp; \sin(2\Psi)\sin(\Delta) \ 0 &amp; 0 &amp; -\sin(2\Psi)\sin(\Delta) &amp; \sin(2\Psi)\cos(\Delta) \end{pmatrix}$</td>
</tr>
</tbody>
</table>
3.3 Light Scattering by a Sphere: Mie Theory

In 1908 Gustav Mie posited a theory which describes scattering of light with a wavelength in the same order as the radius of a homogeneous spherical particle on which it scatters. This model is applicable for this study as the radius of the particles is several \( \mu \)m and the wavelength emitted by the light source is 532 nm. Only a brief introduction into Mie theory is given here and more complete derivations can be found in literature [23–25]. In this study, the model is used to calculate physical parameters of spheres from the ellipsometric angles obtained by ellipsometry measurements.

A schematic of an incident light wave propagating towards a spherical particle is shown in Figure 3.3. The boundary conditions and the scattered light field are specified in spherical coordinates while the incident wave is planar and favorably described in Cartesian coordinates. In order to express the whole system in the same coordinate system, the incident wave is expanded into spherical harmonics. This transformation is cumbersome, but after this exercise the derivation will become more intuitive and straightforward. A more pragmatic reader might want to skip the transformation and pick up reading at equations 3.24 and 3.25 where the result of the transformation is presented. The starting point of this derivation is the set of Maxwell equations

\[
\nabla \cdot \vec{E} = 0, \tag{3.10a}
\n\nabla \cdot \vec{H} = 0, \tag{3.10b}
\n\nabla \times \vec{E} = \mu \frac{\partial \vec{H}}{\partial t}, \tag{3.10c}
\n\nabla \times \vec{H} = -i \epsilon \frac{\partial \vec{E}}{\partial t}, \tag{3.10d}
\]

where \( \epsilon \) is the electric permittivity. Assuming a time dependency of \( \exp(-i \omega t) \), the latter two equations become

\[
\nabla \times \vec{E} = i \omega \mu \vec{H}, \tag{3.11a}
\n\nabla \times \vec{H} = -i \omega \epsilon \vec{E}. \tag{3.11b}
\]

As can be seen from each of these two equations \( \vec{E} \) and \( \vec{H} \) are not independent on one another. The following equation is obtained by taking the curl of equation 3.11a

\[
\nabla \times \left( \nabla \times \vec{E} \right) = i \omega \mu \nabla \times \vec{H} = \omega^2 \epsilon \mu \vec{E}. \tag{3.12}
\]

After some rewriting and the substitution of \( \omega^2 \epsilon \mu = k^2 \), the following vector wave equation is obtained

\[
\nabla^2 \vec{E} + k^2 \vec{E} = 0. \tag{3.13}
\]

Two vectors, \( \vec{M} \) and \( \vec{N} \), are introduced to find spherical harmonics for the transformation from Cartesian to spherical coordinates. These vectors need to have the same properties as an EM field to describe the field, i.e. they must:

- satisfy the vector wave equation,
Figure 3.3: Spherical coordinate system in which the origin coincides with the center of a spherical particle. A planar light front is incident from the bottom.
• be divergence free (eq. 3.10a and 3.10b),
• have their curls being proportional to another (eq. 3.11).

The vectors \( \vec{M} \) and \( \vec{N} \) will be defined according to these criteria. It is assumed that
\( \vec{M} \) is constructed by the scalar function \( \psi \) and a constant vector \( \vec{c} \)
\[
\vec{M} = \nabla \times (\vec{c}\psi),
\] (3.14)
where \( \vec{M} \) is divergence free as the divergence of every curl is zero.

After taking twice the curl of this vector function \( \vec{M} \) and some rewriting, the following equation is obtained
\[
\nabla^2 \vec{M} + k^2 \vec{M} = \nabla \times (\vec{c}(\nabla^2\psi + k^2\psi)).
\] (3.15)

From this expression it can be concluded that \( \vec{M} \) satisfies the vector wave equation if \( \psi \) is a solution of the scalar wave equation
\[
\nabla^2 \psi + k^2 \psi = 0.
\] (3.16)

\( \vec{M} \) can be used to construct the vector \( \vec{N} \) which scales proportionally with the curl of \( \vec{M} \) by its definition
\[
\vec{N} = \frac{\nabla \times \vec{M}}{k}.
\] (3.17)

By taking the divergence of \( \vec{N} \), it can be seen that the divergence is zero. Subsequently, after some careful rewriting it can be seen that \( \vec{N} \) also satisfies the vector wave equation.

The curl of \( \vec{N} \) resolves the following relation
\[
\nabla \times \vec{N} = k\vec{M},
\] (3.18)
which satisfies the last criterion for the curl of \( \vec{N} \).

It is shown that under the condition that \( \psi \) is a solution of the scalar wave equation, the vectors \( \vec{M} \) and \( \vec{N} \) have the same properties as an EM field and thus these two vectors can be used for the transformation of the coordinate system of the incoming wave. The well-known solutions to the scalar wave equation (equation 3.16) in spherical coordinates are presented below
\[
\psi_{enm} = \cos (m\phi) P_m^n(\cos (\theta)) j_n(kr), \tag{3.19a}
\]
\[
\psi_{omn} = \sin (m\phi) P_m^n(\cos (\theta)) j_n(kr), \tag{3.19b}
\]
where the parity of the function \( \psi \) is indicated by the subscripts \( e \) for even and \( o \) for odd. \( P_m^n \) is an associated Legendre polynomial of the order \( m^{th} \) and the \( n^{th} \) degree. These parameters of \( P_m^n \) must be integers and fulfill the following criterion \( n \geq m \geq 0 \). \( j_n (kr) \) is a spherical Bessel function. Normal Bessel functions can be rewritten to spherical Bessel functions of the first kind according to the following relation
\[
j_n (kr) = \sqrt{\frac{\pi}{2k}} J_{n+\frac{1}{2}} (x). \tag{3.20}
\]
The required mathematical building blocks to express a planar wave in spherical coordinates are derived now. Let one assume an x-polarized incoming light wave:

\[
\vec{E}_{\text{in}} = E_0 e^{ikr \cos(\theta)} \hat{e}_x, \\
= E_0 e^{ikr \cos(\theta)} \left( \sin(\theta) \cos(\phi) \hat{e}_r + \cos(\theta) \cos(\phi) \hat{e}_\theta - \sin(\phi) \hat{e}_\phi \right). 
\]

Expanding this equation into the spherical harmonics \(\vec{M}\) and \(\vec{N}\) results in

\[
\vec{E}_{\text{in}} = \sum_{m=0}^{\infty} \sum_{n=0}^{\infty} \left( B_{emn} \vec{M}_{emn} + B_{omn} \vec{M}_{omn} + A_{emn} \vec{N}_{emn} + A_{omn} \vec{N}_{omn} \right), 
\]

where the subscripts of the vectors \(\vec{M}\) and \(\vec{N}\) indicate the parity and the parameters of the associated Legendre polynomial as explained below equation 3.19. \(A\) and \(B\) are the expansion coefficients, which all vanish due to orthogonality except for \(m = 1\). In this case two expansion coefficients remain and from that the following equation is found

\[
\vec{E}_{\text{in}} = E_0 \sum_{n=1}^{\infty} \left( B_{o1n} \vec{M}_{o1n}^{(1)} - A_{a1n} \vec{N}_{e1n}^{(1)} \right), 
\]

where the use of the spherical Bessel function \(j_n(kr)\) is indicated by the superscripted (1). Linear combinations of spherical Bessel functions are called Hankel functions.

After some more algebra to calculate \(A_{o1n}\) and \(B_{o1n}\) it is possible to express the electric field of the incoming plane wave in the spherical harmonics \(\vec{M}\) and \(\vec{N}\)

\[
\vec{E}_{\text{in}} = E_0 \sum_{n=1}^{\infty} \frac{i^n}{n(n+1)} \left( -b_n \vec{M}_{o1n}^{(1)} + ia_n \vec{N}_{e1n}^{(1)} \right), 
\]

where \(a_n\) and \(b_n\) are newly introduced expansion coefficients.

This expression and equation 3.11a are used to calculate the magnetic field \(\vec{H}_{\text{in}}\)

\[
\vec{H}_{\text{in}} = -\frac{k}{\omega \mu} E_0 \sum_{n=1}^{\infty} n i^n \frac{2n + 1}{n(n+1)} \left( -b_n \vec{M}_{o1n}^{(1)} + ia_n \vec{N}_{e1n}^{(1)} \right). 
\]

Now that the transformation of the incoming EM field into spherical coordinates is finished, it is possible to look at scattering. The sum of the incident fields and the scattered fields should be equal to the internal fields at the edge of the particle

\[
\left( \vec{H}_{\text{in}} + \vec{H}_{\text{scat}} - \vec{H}_{\text{int}} \right) \times \hat{e}_r = 0, \\
\left( \vec{E}_{\text{in}} + \vec{E}_{\text{scat}} - \vec{E}_{\text{int}} \right) \times \hat{e}_r = 0. 
\]

The scattered field \(\vec{E}_{\text{scat}}\) can be written in its turn as an expansion of spherical harmonics

\[
\vec{E}_{\text{scat}} = E_0 \sum_{n=1}^{\infty} i^n \frac{2n + 1}{n(n+1)} \left( -b_n \vec{M}_{o1n}^{(3)} + ia_n \vec{N}_{e1n}^{(3)} \right). 
\]
The expansion of the internal electric field $\vec{E}_{\text{int}}$ is given by

$$\vec{E}_{\text{int}} = E_0 \sum_{n=1}^{\infty} i^n \frac{2n+1}{n(n+1)} \left( c_n \vec{M}_{o1n}^{(1)} - id_n \vec{N}_{e1n}^{(1)} \right),$$  \hspace{1cm} (3.28)

where $c_n$ and $d_n$ are expansion coefficients as well.

The spherical Hankel function can be approximated for the far field ($kr \gg n^2$) by the following relation

$$h_{n}^{1}(kr) \sim (-i)^{n} e^{ikr} \frac{e^{ikr}}{ikr},$$ \hspace{1cm} (3.29)

where $r$ is the distance from the center of the particle to the observer.

The same steps can be performed to find the simplified expressions for $\vec{H}_{\text{scat}}$ and $\vec{H}_{\text{int}}$.

The expansion coefficients $a_n$, $b_n$, $c_n$ and $d_n$ are determined by evaluating the boundary condition (equation 3.26) which is translated in the following independent equations

$$
\begin{align*}
E_{\text{int},\theta} + E_{\text{scat},\theta} &= E_{\text{in},\theta}, \\
E_{\text{int},\varphi} + E_{\text{scat},\varphi} &= E_{\text{in},\varphi}, \\
H_{\text{int},\theta} + H_{\text{scat},\theta} &= H_{\text{in},\theta}, \\
H_{\text{int},\varphi} + H_{\text{scat},\varphi} &= H_{\text{in},\varphi},
\end{align*}
$$

(3.30) \hspace{1cm} (3.30)

for the radial distance $r$ equal to the radius of the particle where $\theta$ is the polar angle and $\varphi$ the azimuthal angle.

Further simplification of the scattering coefficients is achieved by introducing the Ricatti-Bessel functions

$$
\begin{align*}
\psi_n (kr) &= \rho j_n (kr), \\
\zeta_n (kr) &= \rho h_{n}^{(1)} (kr).
\end{align*}
$$

(3.31) \hspace{1cm} (3.31)

After the simplification, the scatter coefficients $a_n$ and $b_n$ are given below

$$
\begin{align*}
a_n &= \frac{\psi_n' (mx) \psi_n (x) - m\psi_n (mx) \psi_n' (x)}{\psi_n' (mx) \zeta_n (x) - m\psi_n (mx) \zeta_n' (x)}, \\
b_n &= \frac{m\psi_n' (mx) \psi_n (x) - \psi_n (mx) \psi_n' (x)}{m\psi_n (mx) \zeta_n (x) - \psi_n (mx) \zeta_n' (x)},
\end{align*}
$$

(3.32) \hspace{1cm} (3.32)

where the size parameter $x$ is given by

$$x = \frac{2\pi r_p}{\lambda},$$ \hspace{1cm} (3.33)

where $r_p$ is the radius of the particle.

These coefficients can be used to express the electric field of the scattered wave $E_{\text{scat}}$ in spherical coordinates for the far field.
\[ E_{\text{scat},\theta} = -iE_0 \cos(\varphi) \frac{e^{ikr}}{k r} \sum_{n=1}^{\infty} \frac{2n + 1}{n(n+1)} \left[ a_n \tau_n(\cos(\theta)) + b_n \pi_n(\cos(\theta)) \right], \quad (3.34a) \]

\[ E_{\text{scat},\varphi} = iE_0 \sin(\varphi) \frac{e^{ikr}}{kr} \sum_{n=1}^{\infty} \frac{2n + 1}{n(n+1)} \left[ a_n \tau_n(\cos(\theta)) + b_n \pi_n(\cos(\theta)) \right], \quad (3.34b) \]

\[ E_{\text{scat},r} = 0, \quad (3.34c) \]

where \( \pi_n \) and \( \tau_n \) are given by

\[ \pi_n(\cos(\theta)) = \frac{P_n^l(\cos(\theta))}{\sin(\theta)}, \quad (3.35a) \]

\[ \tau_n(\cos(\theta)) = \frac{d}{d\theta} P_n^l(\cos(\theta)). \quad (3.35b) \]

Equations (3.34) can be rewritten as a product of a Jones matrix and the Jones vector of the incoming light beam

\[ \begin{vmatrix} E_{\text{scat},p} \\ E_{\text{scat},s} \end{vmatrix} = \frac{e^{-ikr + ikz}}{ikr} \begin{vmatrix} S_2 & S_3 \\ S_4 & S_1 \end{vmatrix} \begin{vmatrix} E_{\text{in},p} \\ E_{\text{in},s} \end{vmatrix}, \quad (3.36) \]

where \( S_1, S_2, S_3 \) and \( S_4 \) are given by

\[ S_1 = \sum_{n=1}^{\infty} \frac{2n + 1}{n(n+1)} \left[ a_n \tau_n(\cos(\theta)) + b_n \pi_n(\cos(\theta)) \right], \quad (3.37a) \]

\[ S_2 = \sum_{n=1}^{\infty} \frac{2n + 1}{n(n+1)} \left[ a_n \tau_n(\cos(\theta)) + b_n \pi_n(\cos(\theta)) \right], \quad (3.37b) \]

\[ S_3 = 0, \quad (3.37c) \]

\[ S_4 = 0. \quad (3.37d) \]

This Jones matrix can be transformed into a Mueller matrix by the procedure described by Azzam and Bashara [27]. This method for Jones-to-Mueller matrix conversion is based on a Kronecker product of the Jones matrix, as given above, and a 2x2 conversion matrix. The Mueller matrix \( \mathcal{M}_{\text{Mie}} \) for light scattering on a single, spherical, homogeneous particle with a radius in the same order as the wavelength of the incoming light is for the far field given by

\[ \mathcal{M}_{\text{Mie}} = \frac{1}{2k^2 r^2} \begin{vmatrix} |S_1|^2 + |S_2|^2 & |S_2|^2 - |S_1|^2 & 0 & 0 \\ |S_2|^2 - |S_1|^2 & |S_1|^2 + |S_2|^2 & 0 & 0 \\ 0 & 0 & 2\Re\{S_1 S_2^*\} & -2\Im\{S_1 S_2^*\} \\ 0 & 0 & 2\Im\{S_1 S_2^*\} & 2\Re\{S_1 S_2^*\} \end{vmatrix}. \quad (3.38) \]
Figure 3.4: Basic principle of ellipsometry. On the left well defined light is propagating towards a sample and on the right the reflected light. The change in polarization state caused by reflection is used to characterize the sample. [21]

3.4 Rotating Compensator Mueller Matrix Ellipsometry

Ellipsometry exists since late 19th century [28], however the technique started to gain popularity since the 1970s. Due to the development of computers, it was no longer necessary to control the setup and do the data analysis by hand.

This technique is based on measuring the change of the amplitude ratio $\Psi$ and the phase difference $\Delta$ between the p- and s-polarized waves due to reflection of EM radiation by a sample. This basic principle of ellipsometry is illustrated in Figure 3.4.

The central equation in ellipsometry gives the relation between the complex reflectance ratio $\rho$ and the ellipsometric angles $\Psi$ and $\Delta$:

$$\tan(\Psi) \exp(i\Delta) \equiv \rho,$$

where $\rho$ is given by the ratio of the amplitude reflection coefficients $r_p$ and $r_s$ (eq. 3.5)

$$\rho \equiv \frac{r_p}{r_s}.$$  

The quantities which are determined by an ellipsometer, $\Psi$ and $\Delta$, are not physical quantities, so a model is needed to obtain the information of interest. In this study the sample is a sphere with a radius in the order of the wavelength of the light emitted by the light source. Therefore Mie theory is used to calculate the radius of the sphere $r$. A short introduction into Mie theory is given earlier in paragraph 3.3.

In ellipsometry the angle between the normal vector of the surface and the incoming ray is defined as the angle of incidence, however this definition is not applicable to spherical particles. Therefore the term scattering angle is used which denotes the angle between the incoming and the detected ray, as is indicated in Figure 3.3.

There are different optical configurations of ellipsometric instruments. Only the configuration which is built as a part of this project and used to perform the measurements will be discussed in this paragraph. Advantages of Rotating-Compensator Ellipsometry (RCE) over other configurations are full determination of Stokes parameters in a single measurement and the uniform sensitivity of $\Psi$ and $\Delta$. There are also some drawbacks to this configuration: the calibration process becomes more complicated due to the compensator and RCE is slow in comparison with phase modulated ellipsometry.

Two configurations of setups of RCE exist: PC$_{R}$SA and PSC$_{R}$A. The latter will be treated here, as this is the variant which is built. The letters in the abbreviation
Figure 3.5: Photograph of the built rotating compensator ellipsometer. Light is emitted by the laser (L) on the right side and is reflected by two mirrors (M). The mirrors are placed for alignment and are not mandatory elements. The ray is polarized by a polarizer (P). The incoming light is split based on its polarization by a polarizer (P); the transmitted beam is linearly polarized and the rejected beam is used to measure the intensity of the beam by a photodiode (PD). The transmitted beam is subsequently reflected by the sample (S) into the detection axis. At the bottom the light passes the rotating retarder ($C_R$), the analyzer (A) and is detected by a photomultiplier (PM).

indicate the order in which the light from the source passes the optical elements and the subscript $R$ denotes the rotating component; polarizer $P$, sample $S$, compensator $C$ and analyzer $A$. A photograph of the built rotating compensator ellipsometer is shown in Figure 3.5. The used compensator has a retardation $\Delta_c$ of a quarter wavelength.

The Stokes vector of the light which enters the detector $\vec{S}_{out}$ can be calculated by multiplying the initial Stokes vector $\vec{S}_{in}$ by the Mueller matrices of the optical elements in the chronological order of passing the optical elements

$$\vec{S}_{out} = R(-\alpha_P) \cdot A \cdot R(\alpha_A - \alpha_C) \cdot C \cdot R(\alpha_C) \cdot S \cdot R(-\alpha_P) \cdot P \cdot R(\alpha_P) \cdot \vec{S}_{in}, \quad (3.41)$$

where $\alpha_P$, $\alpha_C$ and $\alpha_A$ are the angles of respectively the polarizer, the compensator and the analyzer. This equation can be simplified when it is assumed that the light from the light source is invariant under rotation and the detector is assumed not to be sensitive to the polarization state

$$\vec{S}_{out} = A \cdot R(\alpha_A - \alpha_C) \cdot C \cdot R(\alpha_C) \cdot S \cdot R(-\alpha_P) \cdot P \cdot \vec{S}_{in}. \quad (3.42)$$

Earlier it was explained that the total intensity is given by the first element of the
Stokes vector. The light intensity measured by the detector $I$ as a function of the angle of the compensator $\alpha_C$ solved from $\vec{S}_{out}$

$$I(\alpha_C) = [A_0 + A_2 \cos(2\alpha_C) + B_2 \sin(2\alpha_C) + A_4 \cos(4\alpha_C) + B_4 \sin(4\alpha_C)] I_{laser}$$

(3.43)

where $A_0$, $A_2$, $B_2$, $A_4$ and $B_4$ are Fourier coefficients. The Fourier coefficients are given by the following equations

$$A_0 = \frac{1}{2} \left[ 1 + y_c \right] \cos(2\alpha_A) \left[ \cos(2\alpha_P) - \cos(2\Psi) \right] - \cos(2\alpha_P) \cos(2\Psi) +$$

$$\frac{1}{2} \left[ 1 + y_c \right] \sin(2\alpha_A) \sin(2\alpha_P) \sin(2\Psi) \cos(\Delta) + 1,$$

(3.44a)

$$A_2 = x_c \cos(2\alpha_P) - \cos(2\Psi) + x_c \cos(2\alpha_A) \left[ 1 - \cos(2\alpha_P) \cos(2\Psi) \right] -$$

$$z_c \sin(2\alpha_A) \sin(2\alpha_P) \sin(2\Psi) \sin(\Delta),$$

(3.44b)

$$B_2 = x_c \sin(2\alpha_A) \left[ 1 - \cos(2\alpha_P) \cos(2\Psi) \right] + x_c \sin(2\alpha_P) \sin(2\Psi) \cos(\Delta) +$$

$$z_c \cos(2\alpha_A) \sin(2\alpha_P) \sin(2\Psi) \sin(\Delta),$$

(3.44c)

$$A_4 = \frac{1}{2} \left[ 1 - y_c \right] \cos(2\alpha_A) \left[ \cos(2\alpha_P) - \cos(2\Psi) \right] -$$

$$\frac{1}{2} \left[ 1 - y_c \right] \sin(2\alpha_A) \sin(2\alpha_P) \sin(2\Psi) \cos(\Delta) +$$

(3.44d)

$$B_4 = \frac{1}{2} \left[ 1 - y_c \right] \sin(2\alpha_A) \cos(2\alpha_P) \cos(2\Psi) \cos(\Delta).$$

(3.44e)

where $x_c$, $y_c$ and $z_c$ represent the non-ideal behavior of the compensator. These properties can be determined by using the procedure proposed by den Boer [29]. More information concerning the calibration can be found in Chapter 5. The non-idealities $x_c$, $y_c$ and $z_c$ as a function of the properties of the retarder $\Psi_c$ and $\Delta_c$ are given by

$$x_c \equiv \cos(2\Psi_c) \approx 0,$$

(3.45a)

$$y_c \equiv \sin(2\Psi_c) \cos(\Delta_c) \approx 0,$$

(3.45b)

$$z_c \equiv \sin(2\Psi_c) \sin(\Delta_c) \approx 1.$$

(3.45c)

By a clever choice of the angles of the polarizer ($\alpha_P = 45^\circ$) and the analyzer ($\alpha_A = 0^\circ$) equations 3.44a - 3.44e can be simplified to

$$A_0 = 1 - \frac{1}{2} \left[ 1 + y_c \right] \cos(2\Psi),$$

(3.46a)

$$A_2 = x_c \left[ 1 - \cos(2\Psi) \right],$$

(3.46b)

$$B_2 = \sin(2\Psi) \left[ x_c \cos(\Delta) + z_c \sin(\Delta) \right],$$

(3.46c)

$$A_4 = \frac{1}{2} \left[ y_c - 1 \right] \cos(2\Psi),$$

(3.46d)

$$B_4 = \frac{1}{2} \left[ 1 - y_c \right] \sin(2\Psi) \cos(\Delta).$$

(3.46e)

Resulting in a system of five equations with two unknown parameters. This is an over-determined system and this allows being selective in which equations are used for
solving $\Psi$ and $\Delta$. $A_2$ is not used, because this coefficient should be very small as it equals zero for an ideal compensator. $A_0$ may contain background emission and is for this reason not used. No absolute intensities are known and therefore it is necessary to normalize the coefficients, which is done by dividing by $B_2$ and $B_4$ by $A_4$. $B_2$ and $B_4$, normalized by $A_4$, as a function $\Psi$ and $\Delta$ are given by

$$\frac{B_2}{A_4} = -\frac{2x_c}{1-y_c} \tan (2\Psi) \cos (\Delta) \frac{2z_c}{1-y_c} \tan (2\Psi) \sin (\Delta), \quad (3.47)$$

$$\frac{B_4}{A_4} = -\tan (2\Psi) \cos (\Delta). \quad (3.48)$$

With the following intermediate step,

$$X_1 \equiv \tan (2\Psi) \sin (\Delta) = \frac{B_4 x_c}{A_4 z_c} - \frac{B_2 (1-y_c)}{2A_4 z_c}, \quad (3.49)$$

$$X_2 \equiv \tan (2\Psi) \cos (\Delta) = -\frac{B_4}{A_4}. \quad (3.50)$$

Expressions for the amplitude component $\Psi$ and the phase difference $\Delta$ can be found as given below

$$\tan (2\Psi) = \sqrt{X_1^2 + X_2^2}, \quad (3.51)$$

$$\tan (\Delta) = \frac{X_1}{X_2}. \quad (3.52)$$

During the process of rewriting expressions 3.51 and 3.52 to respectively $\Psi$ and $\Delta$, information will be lost. The procedure to regain the lost information is stated in equations 3.53, 3.54 and 3.55.

$\Psi$ can be corrected following the criterion

$$A_4 > 0 \rightarrow \Psi_1 := 90^\circ - \Psi, \quad (3.53a)$$

$$A_4 < 0 \rightarrow \Psi_1 := \Psi. \quad (3.53b)$$

The first correction of $\Delta$ based on the sign of $B_4$ and is given by

$$B_4 > 0 \rightarrow \Delta_1 := \Delta, \quad (3.54a)$$

$$B_4 < 0 \rightarrow \Delta_1 := \Delta + 180^\circ. \quad (3.54b)$$

The phase difference $\Delta$ is by definition positive

$$\Delta_1 > 0 \rightarrow \Delta_2 := \Delta_1 + 360^\circ, \quad (3.55a)$$

$$\Delta_1 < 0 \rightarrow \Delta_2 := \Delta_1. \quad (3.55b)$$

The subscripts of the ellipsometric angles can now be removed and the ellipsometric angles used in a model to obtain the quantities of interest. The Mueller matrix which describes the change of polarization due to the sample $S$ (page 16), given in $\Psi$ and $\Delta$, should be equal to the Mie Mueller matrix $M_{Mie}$ (page 22) multiplied by a constant $w$.
\[ S = wM_{Mie}. \] (3.56)

The physical parameters, which are the quantities of interest, in the Mie Mueller matrix \( M_{Mie} \) can be determined by solving a set of 4 nonlinear equations. However finding the physical quantities for a set of determined ellipsometric angles is challenging. Therefore a lookup table is created for \( \Psi \) and \( \Delta \) for different particle radii by calculations. More information concerning these calculations can be found in Paragraph 7.2.

Now all theory needed for this investigation is treated, more information on the setup is described in the next chapter.
Chapter 4

Experimental Details

In this investigation plasma levitated dust particles are studied with imaging and ellipsometry. The required vacuum system, the components to drive the plasma, the camera and the ellipsometer are described below. A schematic overview of the setup can be found in Figure 4.1.

4.1 Plasma Setup

The dust particles which are investigated in this research project levitate in an argon or oxygen plasma. The pressure of the background gas is 0.1 - 10 Pa while the plasma is typically driven at 10 W.

The vacuum vessel which is used for the experiments is an aluminum cube with ribs of 20 cm. The gas handling is performed from the bottom. A Brooks 5850E mass flow controller is used to measure and control the incoming gas flow, a Pfeiffer TSH 071E pump is used to create low pressure and a manually controlled valve is placed between the chamber and the pump. This valve can be closed during experiments to prevent gas flows in the chamber.

An Agilent 33120A function generator is used to generate the 13.56 MHz sinusoidal signal, which is amplified by a Kalmus 150c RF power amplifier. A matching network is placed between the amplifier and the electrodes. Two metal plates of 7x7 cm$^2$ at a distance of 4.0 cm to each other are used as electrodes. The lower electrode is powered and specially designed to horizontally confine the dust particle(s) in a potential well. The top electrode is grounded and contains a hole through which a dust particle can be injected.

A dust injector is mounted on top of the vessel, above the hole in the electrode, and can best be compared with a saltshaker which is driven by a DC motor. At each of the sides of the vessel a window is mounted. These are used for the optical axes for the imaging and ellipsometry setup.

A photograph of the vessel with inside a plasma in Argon is shown in Figure 4.2.

4.2 Imaging Setup

The levitated dust particles are illuminated by a CNI V-H532 DC diode laser which emits light at 532 nm and has a maximum power output of 137 mW. An Imagingsource GigE Mono - DMK 23GM021 CCD camera with an optical system consisting of three lenses and a narrow band filter (Thorlabs FL532-3) is used to record videos of the position of
the dust particles in the sheath. The magnification of the optical system is 1.4 which is determined with a micrometer screw, therefore each pixel (3.75 µm * 3.75 µm) of the CCD chip corresponds to an area of 2.7 µm by 2.7 µm in the object plane. The position of the dust particle in the video is determined by a MATLAB script.

4.3 Ellipsometry Setup

A rotating compensator ellipsometer is developed, built and characterized during this project to study dust particles. A detailed description of the apparatus is given below while the calibration of the setup is described in Chapter 5. A photograph of the ellipsometry setup is shown in Figures 4.3 and 4.4.

A CNI V-H532 DC diode laser which emits light at 532 nm is used to illuminate the levitating dust particle(s). The characterization of the laser is described in Appendix C. Two mirrors in kinematic mounts are used to allow alignment of the laser beam. A Glan-Laser polarizer (Thorlabs GL10), mounted on an adjusted kinematic mount attached to an Elmekanic stepper motor controlled rotational stage, is used to polarize the light at an angle of 45° with respect to the scattering plane. This type of polarizer has a high extinction ratio and can be used for a broad wavelength range. The intensity of the light rejected by the polarizer is measured by a photodiode to monitor the laser intensity.

The polarized light is scattered by the in the plasma levitated dust particle(s). Light which scatters towards the detector first passes a zero-order quarter-wave plate (Thorlabs WPQ10M-532) which is optimized for 532 nm. This compensator is rotated by a Elmekanic stepper motor with a step size of 0.01°. The compensator does not rotate during the determination of the light intensity.

Subsequently the light becomes linearly polarized at an angle of 0° by a reflection
Figure 4.2: Photograph of the vacuum vessel with inside a radiofrequency-driven Ar plasma and a dust injector mounted on top. The sheath, the dark space, surrounding the bottom electrode is clearly visible. In this region, just above the specially designed electrode, a micro-particle is trapped.
polarizer (Thorlabs PBSW-532) mounted on a manual controlled rotational stage in a kinematic mount. The polarizer is designed specifically for the wavelength of 532 nm and ideally transmits the p-polarized light while the s-polarized light is reflected. This type of polarizer is relatively inexpensive and has a high extinction coefficient of for more than 40000:1 for the transmitted light.

The intensity of the by the analyzer transmitted light is measured by a Hamamatsu H10720-110 photomultiplier. Small apertures are placed between the sample and the compensator to reduce the opening angle which can be detected. Measures are taken to reduce the amount of background light to fall on the detector by placing a narrow band filter (Thorlabs FL532-3) just before the detector and by building a light-tight enclosure around the complete setup which is further divided in compartments. A lightproof fan is mounted on the enclosure to create an airflow to remove heat from the setup. The temperature at different positions in this enclosure is monitored by two temperature sensors (Microchip MPC9808).

Electronics and Data Processing

A microcontroller system is developed in the framework of this project to read out all sensors, control the stepper motors and send the light intensity of the photomultiplier and photodiode, and all other measurement data to a computer. More information on the electronics, including a schematic diagram, can be found in Appendix A. The designed source code for the microcontroller can be found in Appendix B. The measurement data is transmitted by an emulated serial connection to a computer, where the data is gathered and saved by a MATLAB script. This script also evaluates the stability of the laser intensity during each measurement and indicates possible unreliable measurement data due to mode hopping of the laser. The data analysis is performed by a MATLAB script after the experiment. A Fluke 45 multimeter is connected to each light detector to give the operator a fast and on the flow method to monitor the setup. The mode, the azimuth of the compensator and the measurement number are indicated by the LCD of the microcontroller system.

Definitions

Some definitions concerning the processes of collecting the light intensity are posited here for clarity. A single determination of the light intensity for a certain orientation of the compensator is called a read. The light intensity is the obtained value by one read or the averaged value over multiple reads at the same angle. The term measurement is used for a closed series of reads at evenly distributed positions which cover a single but complete rotation within a limited time. A set of subsequent measurements is referred to as a measurement series.

4.4 Dust Particles

The dust particles used in this investigation are made of melamine formaldehyde by Microparticles GmbH and have a diameter of 9.78±0.17 µm. Melamine formaldehyde has a mass density of 1574 kg/m³, a refractive index of 1.68 and is reactive with an oxygen plasma. It is expected that the mass, and therefore the position of these particles, changes while being trapped in oxygen plasma. To verify experiments on particle position as function of time measurements have been carried out (Figure 6.1).
Figure 4.3: Photograph of the developed ellipsometer in scatter configuration to study plasma levitated dust particles. The black box which is further divided in compartments is built to reduce background light being detected. The box is covered by a lid and the light-tight fan during experiments.

Figure 4.4: Photograph of the ellipsometer in the light-tight black box. The developed microcontroller system is shown in the front.
Chapter 5

Calibration of the Ellipsometer

The calibration of the setup is a crucial step in the process of the construction of a rotat-
ing compensator ellipsometer and therefore the calibration is explained in this chapter. The procedure starts with the alignment of the laser beam, polarizer and analyzer. The compensator’s ellipsometric angles ($\Psi_c$ and $\Delta_c$) and fast axis are determined last.

Laser Beam

The first step of the calibration procedure is to position the two mirrors in kinematic mounts in a way that the laser beam is propagating in the center of an optical rail at a constant height. This is verified by looking at the location of the laser spot by sliding an aperture mounted on a rail carrier over the optical rail. A schematic of the used configuration is shown in Figure 5.1a.

Polarizer and Analyzer

The transmission axes of the polarizing elements need to be aligned with the scattering plane of the ellipsometer. First, the analyzer (Thorlabs PBSW-532) is placed on the optical rail and a second optical rail is mounted on the table perpendicular to the first optical rail (Figure 5.1b). The reflected laser beam can now be used to position the polarizer in the kinematic mount by turning the screws till the laser spot does not change when the aperture is slid over the second optical rail. The light transmitted by the optical element is linearly polarized at an angle of 0° with respect to the future scattering plane. After that the polarizer (Thorlabs GL10) is placed between the second mirror and the analyzer. Light back scattered by the entry window is used as a reference to place the normal vector of the window of the Glan-Laser polarizer parallel to the incoming light beam. The polarizer is rotated until the escape ray is propagating at a constant height over the table for which it is assumed that the output ray is linearly polarized at an angle of 0° in respect to the scattering plane.

According to Aspnes [30] the measured light intensity should not differ more than 0.1% in case the angle of a polarizer is changed by 180°. To check whether this condition is fulfilled, the light intensity measured of the light which passed subsequently the polarizer and the analyzer for different angles of the polarizer (Figure 5.1d). Significant differences in intensity for opposite angles are found. Moreover, the beam of the back reflected light no longer coincides with the laser beam which means that the motor is not mounted perfectly perpendicular to the scattering plane. This means that the polarizer should
Figure 5.1: Schematics of the configurations used to calibrate the developed ellipsometer with the following optical elements: mirror (M), aperture (a), polarizer (P), rotating compensator \(C_r\), analyzer (A), neutral density filter (ND) and photomultiplier (PM).
be realigned for every position without influencing the azimuth angle in the case the mounting of the motor is not improved.

**Fast Axis of the Compensator**

The polarizer and analyzer are aligned now, however the non-idealities of the compensator still need to be determined and the compensator is positioned at an arbitrary angle $\alpha_C$. For this part of the calibration the procedure described by den Boer [29] is followed. The compensator is placed between the polarizer and analyzer to characterize the retarder as is shown in Figure [5.1d]. The optical elements are now positioned in straight through mode and a neutral density filter is placed just in front of photomultiplier to prevent damaging the detector. The light intensity $I$ as a function of the angle of the compensator $\alpha_C$ is given by

$$I(\alpha_C) = [A_0 + A_2 \cos (2\alpha_C) + B_2 \sin (2\alpha_C) + A_4 \cos (4\alpha_C) + B_4 \sin (4\alpha_C)] I_{\text{laser}}, \quad (5.1)$$

where $A_0, A_2, B_2, A_4$ and $B_4$ are the Fourier coefficients. These coefficients can be determined with a Fourier transform method and are used to obtain the parameters of the compensator.

First, the angle of the compensator’s fast axis $\alpha_{C,0}$ is determined which is given by

$$\alpha_{C,0} = \alpha_C + \alpha_{dC}, \quad (5.2)$$

where $\alpha_{dC}$ is the compensator azimuth angle. The initial position of the compensator $\alpha_C$ is $0^\circ$ and both the polarizer and analyzer are positioned parallel to the scattering ($0^\circ$). For these angles the Fourier coefficients are given by

$$A_0 = \frac{1}{2} [1 - y_c] [1 - \cos (2\Psi)], \quad (5.3a)$$

$$A_2 = 0, \quad (5.3b)$$

$$B_2 = 0, \quad (5.3c)$$

$$A_4 = -\frac{1}{2} [1 - y_c] [1 - \cos (2\Psi)] \cos (4\alpha_{dC}), \quad (5.3d)$$

$$B_4 = \frac{1}{2} [1 - y_c] [1 - \cos (2\Psi)] \sin (4\alpha_{dC}). \quad (5.3e)$$

Figure [5.2a] shows the average intensity signal of the successful measurements out of a 250 measurement series. This signal is used to obtain the following coefficients

$$A_0 = 8849 \pm 4, \quad (5.4a)$$

$$A_2 = -62 \pm 4, \quad (5.4b)$$

$$B_2 = -14 \pm 4, \quad (5.4c)$$

$$A_4 = 2991 \pm 4, \quad (5.4d)$$

$$B_4 = -518 \pm 4. \quad (5.4e)$$

The error angle $\alpha_{dC}$ is given by

$$\tan (4\alpha_{dC}) = -\frac{B_4}{A_4}, \quad (5.5)$$
(a) Polarizer angle $0^\circ$, analyzer angle $0^\circ$, averaged over 248 measurements.

(b) Polarizer angle $45^\circ$, analyzer angle $0^\circ$, averaged over 236 measurements.

Figure 5.2: Average light intensity as a function of the angle of the compensator. During these measurements the laser is driven at a constant voltage of 1.7 V, a current of 0.306 A and the gain of the photomultiplier is 830. For each measurement the intensity is determined at 200 angles, each with 20 reads.
resulting in an $\alpha_{dC}$ of $(2.45 \pm 0.02 + f \cdot 45)^\circ$ where $f$ is an integer. $f$ is 0, because the compensator was placed in the motorized rotation stage with the engraved lines to denote the fast axis at roughly $0^\circ$ of the analog indication. As the fast axis of the compensator is determined in respect to the position of the polarizer and the error in $\alpha_P$ is $\pm 0.02^\circ$, the compensator’s fast axis $\alpha_{C,0}$ is finally determined to be $357.55 \pm 0.04^\circ$.

**Non-idealities of the Compensator**

The non-idealities of the compensator $x_c$, $y_c$ and $z_c$ are given by

\begin{align}
  x_c &\equiv \cos (2\Psi_c) \approx 0, \quad (5.6a) \\
  y_c &\equiv \sin (2\Psi_c) \cos (\Delta_c) \approx 0, \quad (5.6b) \\
  z_c &\equiv \sin (2\Psi_c) \sin (\Delta_c) \approx 1. \quad (5.6c)
\end{align}

where $\Delta_c$ and $\Psi_c$ are the ellipsometric angles of the compensator which need to be determined. For an ideal quarter-wave plate these are $90^\circ$ and $45^\circ$ respectively.

A similar method is used to obtain the non-idealities, however the light from the source is now polarized at an angle of $45^\circ$ and the analyzer is still positioned at $0^\circ$. The Fourier coefficients for this configuration are given by

\begin{align}
  A_0 &= 1, \quad (5.7a) \\
  A_2 &= x_c \left[ \cos (2\alpha_C) + \sin (2\alpha_C) \right], \quad (5.7b) \\
  B_2 &= x_c \left[ \cos (2\alpha_C) - \sin (2\alpha_C) \right], \quad (5.7c) \\
  A_4 &= -\frac{1}{2} \left[ 1 - y_c \right] \sin (4\alpha_C), \quad (5.7d) \\
  B_4 &= \frac{1}{2} \left[ 1 - y_c \right] \cos (4\alpha_C). \quad (5.7e)
\end{align}

The light intensity as a function of the compensator azimuth averaged over 236 measurements can be seen in Figure 5.2b and is used to determine the following Fourier coefficients

\begin{align}
  A_0 &= 3393 \pm 4, \quad (5.8a) \\
  A_2 &= 1 \pm 4, \quad (5.8b) \\
  B_2 &= -3 \pm 4, \quad (5.8c) \\
  A_4 &= 340 \pm 4, \quad (5.8d) \\
  B_4 &= 1970 \pm 4. \quad (5.8e)
\end{align}

The DC component of the signal needs to be increased by $284 \pm 9$ to correct for an offset of the voltage in the dark. This value is obtained by measuring the voltage over the resistor of the photomultiplier with the ADC in a dark environment. The obtained Fourier coefficients will be normalized ($A_0 = 1$) later on.

$x_c$ and $y_c$ can be obtained by solving the equations above

\begin{align}
  x_c &= \pm \sqrt{\frac{2A_2^2 + 2B_2^2}{2A_0}}, \quad (5.9a) \\
  y_c &= 1 - 2 \sqrt{\frac{A_4^2 + B_4^2}{A_0}}. \quad (5.9b)
\end{align}
Table 5.1: Obtained properties of the rotating compensator ellipsometer.

<table>
<thead>
<tr>
<th>parameter</th>
<th>value</th>
</tr>
</thead>
<tbody>
<tr>
<td>$\Psi_c$</td>
<td>45.00±0.03°</td>
</tr>
<tr>
<td>$\Delta_c$</td>
<td>93.08±0.24°</td>
</tr>
<tr>
<td>$x_c$</td>
<td>(0.0 ± 1.6) · 10^{-3}</td>
</tr>
<tr>
<td>$y_c$</td>
<td>(-5.3 ± 0.4) · 10^{-2}</td>
</tr>
<tr>
<td>$z_c$</td>
<td>0.9985±0.0002</td>
</tr>
<tr>
<td>$\alpha_{C,0}$</td>
<td>357.55 ± 0.04°</td>
</tr>
<tr>
<td>$\alpha_{P,0}$</td>
<td>1.88 ± 0.02°</td>
</tr>
</tbody>
</table>

The obtained values for $x_c$ and $y_c$ are ± (0.6 ± 1.1) · 10^{-3} and (-5.3 ± 0.4) · 10^{-2} respectively. Without further knowledge of the compensator it is not possible to determine the sign of $x_c$. As the relative error in $x_c$ is larger than 1, it is decided to assume $x_c$ is zero and increase the error in order to cover the whole domain.

From equations 5.6a - 5.6c are the following equations for the parameters of the compensator derived

\[
\cos(2\Psi_c) = x_c, \quad (5.10a)
\]
\[
\cos(\Delta_c) = \frac{y_c}{1-x_c^2}. \quad (5.10b)
\]

The values obtained for $\Psi_c$ and $\Delta_c$ are 44.98±0.03° and 93.08±0.24° respectively. The accuracy of the retardance is specified by Thorlabs as smaller than $\lambda/300$ (1.2°) and therefore the obtained $\Delta_c$ is not in agreement with the specifications. The deviating value might be explained by the fact that $\Delta_c$ strongly depends on $A_0$ and an increase of only 5.4% of the DC signal is enough to decrease the retardance to 90.00°. Overall it is likely that the determined value for $\Delta_c$ is not correct and for this reason the results in this work will be presented for $\Psi_c$ and $\Delta_c$ as determined during the calibration and for an ideal compensator. The DC signal is used as well to determine $\Psi_c$, however $\Psi_c$ is less sensitive for an error in $A_0$ for this set of Fourier coefficients.

Now $\Psi_c$ and $\Delta_c$ are known, $z_c$ can be determined by equation 5.6c

**Calibration completed**

All properties of the ellipsometer determined during the calibration process described above are presented in Table 6.1. After the compensator is positioned in the position $\alpha_{C,0}$, the ellipsometer is fully calibrated and can be rebuild to the scatter configuration as is shown in Figure 3.5 to investigate surfaces. After the working of the ellipsometer is verified the sample can be replaced by the vacuum vessel as shown in Figure 4.3 to start investigating plasma levitated dust particles.
Chapter 6

Results

First, the investigation of a ‘burning’ dust particle i.e. a confined polymer particle being etched in O$_2$ plasma by the imaging setup is described. In the second paragraph the ellipsometry results are described. The working of the developed ellipsometer is demonstrated by investigating Si wafers and comparing the results to those from a commercial ellipsometer. The results of calculations and experiments on spherical particles are treated in the last part of this chapter.

6.1 Imaging

Single dust particles are successfully trapped in the sheath, near the powered electrode, of a 10 W oxygen plasma at a pressure of 2 $\cdot$ 10$^1$ Pa. The position of the dust particle during two identical measurements is shown in Figure 6.1. Over a period of 30 minutes the height of both particles increases approximately 0.25 mm.

6.2 Ellipsometry

6.2.1 Wafers as a Reference

Insight in the working and accuracy of the self-built ellipsometer is obtained by comparing results of the apparatus with the ellipsometric angles obtained by a commercial ellipsometer. A J.A. Woollam M-2000 spectroscopic ellipsometer on a gonio stage is used to characterize two wafers at three spots as reference samples. Measurement data obtained at an angle of incidence of 70° is used to model the properties of the wafers while the ellipsometric angles determined at an angle of incidence of 45° act as a reference. An overview of the obtained ellipsometric angles is given in Table 6.1.

Native Oxide Si

The investigation by the M-2000 at an angle of incidence of 70° shows that the with phosphorus n-type doped Si (100) wafer has a native oxide layer of 1.5±0.1 nm.

The sample is investigated by the self-built ellipsoid at an angle of incidence of 45° and to overcome problems with the stability of the laser intensity the data of 230

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$^{1}$The used J.A. Woollam M-2000 is owned by the Plasma and Materials Processing group of the Eindhoven University of Technology. I am grateful to the group for making it available to me for these measurements and I would like to thank Vincent Vandalon for the refresher course to operate the apparatus he gave me.
Figure 6.1: The evolution of the position of a dust particle in an oxygen plasma at a pressure of $2 \cdot 10^1$ Pa during two separate measurements.

Table 6.1: Ellipsometric angles of a native oxide and a thermally oxidized Si wafer obtained by measurements by the self-built ellipsometer and the Woollam M-2000. Calibration is performed to determine the non-idealities of the setup, these parameters are used in the analysis to obtain $\Psi$ and $\Delta$ given in the columns ‘non-ideal’. The values presented in the last columns are obtained when the compensator is assumed to be ideal.

<table>
<thead>
<tr>
<th>sample</th>
<th>Woollam</th>
<th>non-ideal</th>
<th>ideal</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>$\Psi$ ($^\circ$)</td>
<td>$\Delta$ ($^\circ$)</td>
<td>$\Psi$ ($^\circ$)</td>
</tr>
<tr>
<td>native</td>
<td>35.16</td>
<td>178.97</td>
<td>34.75±0.32</td>
</tr>
<tr>
<td>thermally</td>
<td>38.34</td>
<td>151.42</td>
<td>38.03±0.29</td>
</tr>
</tbody>
</table>
measurements is averaged to obtain the intensity as a function of the compensator angle as shown in Figure 6.2a. The average light intensity is used to determine the following Fourier coefficients

\[
\begin{align*}
A_0 &= 2132 \pm 4, \\
A_2 &= 2 \pm 4, \\
B_2 &= 59 \pm 4, \\
A_4 &= -633 \pm 4, \\
B_4 &= -1694 \pm 4.
\end{align*}
\] (6.1a-d)

From these coefficients are the ellipsometric angles \(\Psi\) of \(34.75 \pm 0.32^\circ\) and \(\Delta\) of \(178.90 \pm 0.15^\circ\) determined, which are indicated by squares in Figure 6.3a. Assuming that the compensator behaves ideally the ellipsometric angles \(\Psi\) and \(\Delta\) become \(34.75 \pm 0.32^\circ\) and \(178.90 \pm 0.08^\circ\) respectively.

As a reference the same sample is investigated by the M-2000 at the following angles \(44^\circ, 44.5^\circ, 45^\circ, 45.5^\circ\) and \(46^\circ\). Figure 6.3a shows \(\Psi\) in red and \(\Delta\) in green of the native oxide wafer for an angle of incidence of \(45^\circ\) (thick) and neighboring angles (thin) over a wavelength range of 400-660 nm. The determined \(\Psi\) and \(\Delta\) for the wavelength in our setup (532 nm) nearest to the laser wavelength are \(35.16^\circ\) and \(178.97^\circ\) respectively.

The \(\Psi\)'s obtained from the self-built ellipsometer are the same and deviate \(0.4^\circ\) from the reference measurements. The difference in the obtained \(\Delta\) for the non-ideal as well as the ideal compensator is only several hundredth degree.

**Thermally Oxidized Si**

The second sample, a p-type Si:B (100) wafer, has an oxide layer of \(268 \pm 1\) nm according to the model which is used to analyze the data of the measurement at an angle of incidence of \(70^\circ\).

In Figure 6.2b is the averaged light intensity of 233 measurements of the self-built rotating compensator ellipsometer shown for an angle of incidence of \(45^\circ\). The Fourier coefficients of this signal are

\[
\begin{align*}
A_0 &= 3460 \pm 4, \\
A_2 &= 24 \pm 4, \\
B_2 &= -2057 \pm 4, \\
A_4 &= -535 \pm 4, \\
B_4 &= 1864 \pm 4.
\end{align*}
\] (6.2a-e)

Resulting in \(\Psi\) of \(38.03 \pm 0.29^\circ\) and \(\Delta\) of \(149.76 \pm 0.62^\circ\) for the non-ideal compensator. These ellipsometric angles are indicated in Figure 6.3b by squares. Assuming the compensator behaves ideally, the obtained \(\Psi\) and \(\Delta\) are \(37.94 \pm 0.26^\circ\) and \(151.11 \pm 0.46^\circ\) respectively.

The same sample is investigated by the commercial ellipsometer at the 5 angles of incidence evenly distributed from \(44^\circ\) up to \(46^\circ\). The ellipsometric angles for an angle of incidence of \(45^\circ\) (thick) and neighboring angles (thin) over a wavelength range of 400 up to 660 nm are shown in Figure 6.3b. The obtained \(\Psi\) and \(\Delta\) for the wavelength closest to the laser wavelength are \(38.34^\circ\) and \(151.42^\circ\) respectively.
Figure 6.2: Average light intensity as a function of the angle of the compensator. During these measurements the laser is driven at a constant voltage of 1.7 V, a current of 0.306 A and the gain of the photomultiplier is 830. For each measurement the intensity is determined at 200 angles, each with 20 reads.

(a) n-type native oxide Si:Ph (100), averaged over 230 measurements.

(b) p-type thermally oxidized Si:B (100), averaged over 233 measurements.
Figure 6.3: The $\Psi$ (red) and $\Delta$ (green) of wafers measured by a J.A. Woollam M-2000 over the wavelength range of 400-660 nm at an angle of incidence of 45° are indicated by the thick lines while the thin lines represent the angles 44°, 44.5°, 45.5° and 46°. The ellipsometric angles obtained by the non-ideal ellipsometer are indicated by the squares.
The difference in the determined Ψ’s in respect to the reference measurements is similar to the difference for the native oxide Si wafer. For the non-ideal compensator, the obtained ∆ deviates significantly from the result of the Woollam. However, when the compensator is assumed to behave ideally the ∆ is in agreement with the reference ellipsometric angles. Which strengthens the suspicion that the ∆c determined during the calibration is incorrect.

6.2.2 Plasma Levitated Particles

The working of the self-built is ellipsometer is verified in the previous section and therefore it is possible to start investigating plasma levitated dust particles. As the ellipsometric angles, Ψ and ∆, determined by means of ellipsometry are not the physical quantities nor the quantities of interest a model is required. The first part of this paragraph treats the calculations performed to create a lookup table for the analysis of the experiments. Results of the first measurements on a dust particles are described in the last part of the paragraph.

Calculations of Ellipsometric Angles of a Particle

For the analysis of experimental results on spherical particles it is necessary to convert the obtained Ψ and ∆ into physical parameters of the particle. Optimizing the parameters of the particle for a set of experimentally determined ellipsometric angles is complex. For that reason it is decided to create a lookup table for Ψ and ∆ as function of the radius of the particle.

The Mueller matrix describing the sample \( S \) (Table 3.2) equals the Mueller matrix of the model \( \mathcal{M}_{Mie} \) (equation 3.38). After removing the redundant equations, the following four coupled nonlinear equations remain

\[
\begin{align*}
|S_1|^2 + |S_2|^2 - a_{fit} &= 0, \quad (6.3a) \\
|S_1|^2 - |S_2|^2 + \cos(2\Psi) a_{fit} &= 0, \quad (6.3b) \\
2R(S_1 S_2^*) - \sin(2\Psi) \cos(\Delta) a_{fit} &= 0, \quad (6.3c) \\
23(S_1 S_2^*) + \sin(2\Psi) \sin(\Delta) a_{fit} &= 0, \quad (6.3d)
\end{align*}
\]

where \( a_{fit} \) is a fitting parameter which represent the fractions in front of the matrices.

A MATLAB script is used to calculate Ψ and ∆ analytically for particle radii in the range of 1 - 20 \( \mu \)m in steps of 0.01 nm, refractive index of 1.68, scattering angle \( \theta \) of 90\( ^\circ \) and a wavelength of 532 nm. The results of these calculations for particle radii 4.8 - 5.0 \( \mu \)m are shown in two type of graphs in Figure 6.4a and 6.4b.

The ellipsometric angles as presented by Swinkels (\( \lambda = 488 \) nm, \( \theta = 90^\circ \), \( \varphi = 45^\circ \), \( r_p = 4.7-4.9 \mu m \) \( m = 1.68 \)) are compared with the Ψ and ∆ obtained by the MATLAB script for the same conditions. Although the LHS of the equations 6.3 are in order of \( 10^{-14} \) with an \( a_{fit} \) of typically \( 10^2 \), the ellipsometric angles are not in agreement with the work of Swinkels. Further investigation is needed to explain the different results.

\[\text{I would like to thank Leroy Schepers for making his Mie script available to me so I was able to use and modify parts of his code and Bart van Lith for our discussion concerning the best way to solve this set of equations.}\]
and what is intended by Swinkels by the azimuth angle $\varphi$ of 45° as rotating an isotropic sample, a homogenous sphere, should not make a difference for the end result.

The ellipsometric angles are calculated for a single spherical particle, however these can be used as well for a dust cloud of monodisperse particles when the cloud can be considered as a point source and the light scattered by the particles does not interact again with other particles in the cloud. Although this is theoretically permitted, in practice there will be a distribution in the size of the particles which needs to be taken in account as small changes in radius can results in large changes of the ellipsometric angles.

Experiments on Dust Particles

The laser used to verify the working of the built ellipsometer broke down and needed to be replaced. At that time only a modified laser pointer which, according the specification, emits light at 532 nm was available and to impel the project it was decided to use the light source despite its expected non-ideal behavior. An Ocean Optics THR1000 is used to characterize the light emitted by the pointer and the central wavelength is 533 nm and the FWHM is roughly 1 nm. Two driving modes can be distinguished based on the amount of light emitted by the laser (approximately double) while the same voltage and current are supplied and only measurements in the mode with the high light intensity will be used. This might be caused by mode hopping and the laser operates generally in the same mode for several hours. The laser is on shorter time scales very instable as the changes in intensity are easy detectable by eye. Furthermore, the pointing stability is very poor which will affect the alignment of the setup and therefore the results.

A dust cloud is trapped in a potential well in an Ar plasma with a background gas pressure of 4.3 Pa and an RF power of approximately 10 W. Due to instabilities of the laser intensity a lot of averaging is needed and therefore a flow of 8 scc/m is used to maintain the position of the particle during the experiment. The gain of the photomultiplier is set to maximum to keep sufficient signal while decreasing the apertures as much as possible.

The average light intensity of the dust cloud, the plasma and stray light of the laser over 488 measurements as a function of the angle of the compensator is indicated by the red line in Figure [5.5]. The solid lines represent the intensity of the dust cloud, which is calculated by subtracting the background (dashed black line) of the intensity of the plasma, laser and dust particles. The background signal is obtained by averaging over 303 measurements with plasma and the laser on however without dust particles. The Fourier coefficients of the signal after background subtraction are

\begin{align*}
A_0 &= 872 \pm 4, \\
A_2 &= -16 \pm 4, \\
B_2 &= -8 \pm 4, \\
A_4 &= 155 \pm 4, \\
B_4 &= 294 \pm 4.
\end{align*}

(6.4a)  
(6.4b)  
(6.4c)  
(6.4d)  
(6.4e)

These Fourier coefficients are used to calculate the ellipsometric angles $\Psi$ of 58.89±1.87° and $\Delta$ of 359.18±0.53° for this dust cloud and the non-ideal compensator. For the ideal compensator, $\Psi$ and $\Delta$ become 58.89±1.87° and 359.21±0.44° respectively. The errors in the obtained angles are relatively large due to the low light intensity.
Figure 6.4: The ellipsometric angles calculated according to Mie theory for a sphere with a radius of 4.8 up to 5.0 µm, a scattering angle of 90°, a refractive of the particle of 1.68 and a wavelength of 532 nm.
Figure 6.5: Average light intensity as function of the angle of the compensator for a dust cloud (red) and a bigger dust cloud (blue). The solid lines indicate the intensity of the particle, meaning after subtraction of the light emitted by the plasma and stray light of the laser indicated by the black line.

More particles are injected to check whether the dust cloud can be treated as a single dust particle. The average signal of the bigger dust cloud over 718 measurements is indicated by the dashed blue line in Figure 6.5. The signal from the particles after background subtraction (solid blue line) is used to determine the following Fourier coefficients

\[
\begin{align*}
A_0 &= 4647 \pm 4, \\
A_2 &= -24 \pm 4, \\
B_2 &= -499 \pm 4, \\
A_4 &= 653 \pm 4, \\
B_4 &= 735 \pm 4.
\end{align*}
\]

(6.5a) (6.5b) (6.5c) (6.5d) (6.5e)

For these Fourier coefficients and a non-ideal compensator, \( \Psi \) is 64.96±0.71° and \( \Delta \) is 340.27±0.60°. Assuming the compensator is ideal leads to a \( \Psi \) of 65.04±0.67° and 341.23±0.45° respectively. These ellipsometric angles deviate significantly from the angles obtained from the smaller cloud and for that reason it is not possible that both clouds can be considered as a single particle. Possible causes which contribute to the change in the ellipsometric angles are: change in distribution of scattering angle \( \phi \) due to the increased volume of particles and a different particle radius distribution in the illuminated volume due to crystallographic organization.

Although single particles cannot be investigated due to insufficient signal, the developed ellipsometry setup demonstrates full feasibility from the fundamental point of view.
The development towards diagnostics on one single particle is a matter of technological implementations rather than physical hold back. Increasing the signal and improving the light source is the way towards measurements on a single particle.
Chapter 7

Discussion

The work which is performed in the framework of this project will be discussed in the following chapter. Furthermore ideas to improve the ellipsometry setup and other ideas related to research are presented here as well.

7.1 Imaging

The imaging setup is fully functional as it is able to determine the position of a single plasma levitated dust particle. This functionality will be used during the electric field measurements to determine the position of the probe. The first test measurements already showed that the position of the dust particles increases approximately 250 µm in an oxygen plasma over a period of 30 minutes. This strongly indicates that the particles indeed react in the O₂ plasma.

Future Plans to improve the Imaging Setup

More information can be gained from the experiments with the imaging setup by using two cameras, one for p- and s-polarized light, which monitor the plasma levitated particles from the same angle. This can be done by for instance a polarizing beam splitter. Note that the rejected beam commonly consist out of a mixture of polarization states and an additional polarizer is needed to remove other polarization states. Greiner et al. [22] used a similar setup to study the growth of nanodust clouds in an argon-acetylene plasma. This setup would become even more versatile by including a rotating compensator in front of the beam splitter or by placing an additional rotating compensator directly after the polarizer of the developed ellipsometer.

7.2 Ellipsometry

The discussion of the ellipsometry results is divided into three sections. The calibration and the investigation of the Si wafers will be discussed first, followed by the calculations performed for the lookup table and the experiments on dust particles. This paragraph concludes with a list of changes to be made on the setup.

Calibration and Si wafers

The working of the developed ellipsometer is verified by the investigation of oxide layers on two different Si wafers by the self-built ellipsometer and a commercial ellipsometer.
The obtained ellipsometric angles are in good agreement when it is assumed that the compensator of the built setup behaves ideally. The largest difference between the angles is $0.41^\circ$ while the smallest difference is $0.02^\circ$. For the non-ideal compensator, the obtained $\Delta$ for the thermally oxidized Si wafer deviates significantly from the value obtained by the commercial ellipsometer, which is most likely caused by an overestimation of the retardance of the compensator $\Delta_c$. It is expected that the error in the measured $\Psi$ and $\Delta$ will decrease further when the properties of the compensator are determined more precisely. As a comparison the accuracy of the ellipsometer used by Swinkels is approximately $2^\circ$ in both ellipsometric angles [31].

The retardance $\Delta_c$ is determined during the calibration of the ellipsometer and as described earlier is the obtained value is not in agreement with the specifications of the manufacturer. Another strong indication that the determined values are incorrect is that the results of the non-ideal compensator have a larger discrepancy with results of the commercial ellipsometer than for the ideal compensator. Possible causes of the inaccurate values obtained during calibration are non-perfect alignment, nonlinear light detection and laser instabilities. The graphs of the light intensity show some unnatural curvature, especially for the lower light intensities it is clearly observable, which might be an indication that the measured light intensity does not scale linearly with the real intensity.

The signals of more than 230 measurements are averaged during the calibration to overcome the problems with laser instability, but it might be that more measurements are needed for convergence. This might be another cause of inaccurate values for $\Psi_c$ and $\Delta_c$.

Several improvements of the setup are proposed below, however only three are crucial and will be implemented before the setup will be recalibrated.

**Calculations on Dust Particles**

Calculations are performed to create a lookup table which will be used to convert the measured ellipsometric angles into the parameters of the particle of interest. As a test for the MATLAB script are the $\Psi$ and $\Delta$ calculated for the same conditions as presented by Swinkels [31]. Although the results do not coincide and further investigation is needed to explain the differences, the plots do show a similar type of behavior: curls and spikes.

These spikes are caused by resonances in the scattering frequencies $a_n$ and $b_n$ [31]. The advantage of these spikes is that the change in ellipsometric angles is very large for small changes in radius. This would result in an increased resolution for the electric field measurements if the probes have a size near a resonance spike.

**Experiments on Dust Particles**

Attempts to study a single dust particle by means of ellipsometry have not been successful so far as the light intensity of a single dust particle has not been sufficient to differentiate it from the background. It is remarkable that the light intensity scattered by a single particle is sufficient for the CCD in the imaging setup and not for the photomultiplier used for the ellipsometry measurements. Several settings of the apertures and the height of the detector have been tried to solve the problem.

For dust clouds, sufficient light has been detected to determine the ellipsometric angles $\Psi$ and $\Delta$. However, the obtained ellipsometric angles changed after injecting more dust in the plasma. Based on that change, one can conclude that not both clouds
can be considered as a single particle. However, it is very unlikely that either of the clouds can be considered as a single particle due to the spread in the radius is 0.17 \( \mu m \) according to the manufacturer and the ellipsometric angles depend strongly on the radius. The model must be expanded to take into account this size distribution.

Studying time dependent processes of dust clouds, like ‘burning’ the particles in an oxygen plasma, is not possible due to the very unstable light source.

**Future Plans to improve the Ellipsometry Setup**

There are strong indications that the parameters which represent the non-ideal behavior of the compensator are inaccurate. For that reason the steps of the calibration will be executed again, however this will be done after the following improvements have been made:

- Both light sources used in this investigation are far from ideal, therefore an order is placed for a laser with a high power stability, low noise amplitude and which emits light with polarization at a constant azimuth. It is expected that the new laser will reduce the need to average measurements and reject measurements due to instabilities. If necessary, the output of the laser can be further stabilized by an in-house custom-built power supply with feedback system[32]. The laser will be temperature stabilized by a Peltier cooler.

- Aligning the sample and the optical axes of the ellipsometer close to perfect is crucial to obtain good results. A systems needs to be developed to verify the alignment without the need to demount the complete setup. This can be done by for example monitoring the beam spot of the incoming axis by placing a CCD chip behind the mirror which reflects the light into the beam dump. As a start the new laser has a good pointing stability (< 50 \( \mu \text{rad} \)).

- There are indications that the detected light intensity does not scale linearly with the actual light intensity. Although photomultipliers are known for their linearity over several orders of magnitudes, the linearity can be improved as described by Aspnes[33]. However the ADC is the most probable cause of the nonlinear behavior. For that reason the current ADC (Texas Instruments ADS1115) will be replaced by an Agilent U2541A.

The following improvements of the setup may be performed over the following years:

- Currently one measurement (200 angles, 20 reads for photomultiplier and photodiode) takes roughly 100 seconds and to study time dependent processes it is necessary to do many measurements within the typical time period of the experiment. The duration of one measurement can be shortened by reducing the number of reads and angles, but these might lead to an increased error. The following changes can be made to reduce the time it takes to do a single measurement without affecting the accuracy: replacing the current microcontroller by for example an AT91SAM3X8E Cortex-M3 which has a higher clock speed and supports higher baud rates, reducing the commands to transfer data by combining messages, ramping the speed of the stepper motors and increasing the voltage on the coils of the stepper motors which will lead to more torque which allows shorter delays between the pulses to the stepper motor driver. A small reduction
of the duration can be achieved by reducing the changes on the display of the microcontroller system.

- The calculations of $\Psi$ and $\Delta$ for the lookup table show that a combination does not point to a unique radius. For that reason an additional measuring method might be implemented to do a first order estimation of the size of the particle(s).

- The fitting of the model will give better results if the ellipsometric angles for multiple angles and/or wavelengths are known. Especially when the scattering signal is low under a scattering angle for the used wavelength and particle size. Multiple light sources and detection axes, with a detector for each light source, can be added to the setup to obtain these additional ellipsometric angles.

- As strain free windows influence the properties of the light less, more accurate and constant results are expected by replacing the current conventional viewports of the incoming and detection axes with strain free windows.

- Replacing the window of the viewport used for the beam dump by a brewster window would result in less backscattered light on the particle. Therefore the light which scatters on the particle is better defined.

- Mounting the vacuum vessel or the electrodes on a linear z translation stage would allow to keep the sample in the perfect position for the ellipsometer as the position in the sheath of dust particles depends on the properties of the plasma and the particle. For the case that the light detector and laser beam are large in comparison to the particles’ displacement, it might be sufficient to change the height of the apertures.

7.3 Figments

There are many materials with special properties which are interesting to investigate as probes for ellipsometry. A non-exhaustive list is given here: magnetostrictive materials, pH-sensitive polymers, electrochromic materials,形状-memory alloys and materials which extremely expand by a change in pressure or temperature.

The potential applications of these probes can be expanded by developing other ways to support these probes. For instance placement in a very rubbery encapsulation which barely interacts with the light, fluids, gas flows or optical tweezers.

Imaging Mueller matrix ellipsometry on dust clouds can be an interesting method to investigate light scattering by diffusers of for example LED’s. The advantage of Mueller matrix ellipsometry is that the off diagonal elements of the Mueller matrix indicate the anisotropic behavior of the diffusor.

Paeva [17] investigated the mechanics of orbiting dust particles in a potential trap. Further elucidation of the mechanics can be achieved by studying the motion of ‘propeller’ shaped particles of photomechanical [34] material. The shape of these materials can be controlled by the wavelength of the light which is used to illuminate the particle.
During this project a novel diagnostics is developed to measure the electric field strength in the plasma sheath spatially resolved and nonintrusively. This method elucidates the electric field profile which will improve the understanding of fundamental plasma physics. Despite that the measuring technique has not been fully implemented yet, good progress has been made towards the realization of this idea. The most crucial step, the construction and calibration of a rotating compensator ellipsometer, has been realized successfully. A microcontroller system has been developed to control the ellipsometer and the required source code has been written. The working of the built apparatus is demonstrated by investigating oxide layers on wafers from which the results are compared with results obtained by a commercial ellipsometer. There are strong indications that the determined non-ideal properties of the compensator are not sufficiently accurate yet and therefore the setup will be recalibrated. The largest difference in the obtained ellipsometric angles for the assumed ideal compensator in respect to results of the Woollam is $0.41^\circ$. As a comparison, Swinkels reported an accuracy of $2^\circ$ for his ellipsometer [31].

Calculations are performed to create a lookup table for ellipsometric angles for different radii. This table will be used for the analysis of the measurement data obtained by experiments on spherical particles. The validation of the lookup table is still pending.

Ellipsometric angles of dust clouds have been determined with the developed ellipsometer. The current model, which is used to determine physical parameters, does not take in account the required particle size distribution. Three improvements to the setup are proposed in Paragraph 7.2 which will help to study a single dust particle and to overcome the necessity to average over hundreds of measurements due to laser instabilities. It is expected that the radius of a single particle can be determined when these improvements are realized.

The imaging setup is fully functional and is able to determine the position of a single plasma levitated dust particle. This functionality will be used to determine the position of the plasma levitated probes during the electric field measurements. This setup is already used to perform the first experiments in which the position of a dust particle which reacts with an oxygen plasma over time is determined. Due to the oxygen plasma the mass of the particle decreases and during two identical measurements is shown that the height of each particle increases approximately 250 $\mu$m in 30 minutes.

Although determining physical parameters of the plasma levitated dust particles has not been successfully performed yet, a solid foundation is built for unraveling the principle physical processes in the sheath of a low pressure plasma by the development
of a novel measuring method and the accompanying setup.
Dankwoord


Met deze opmerking is de trend gezet voor een jaar met flauwe grappen en woordspelingen. Leroy, ik ga je natuurlijk bedanken voor je grappen en het lachen om mijn grappen (als enige), maar bovendien voor de zeer goede begeleiding die je me hebt gegeven. Bedankt voor je grappen, het lachen om mijn grappen en de zeer goede begeleiding!

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Appendix A

Developed Microcontroller System

The complete setup is driven by a 16 MHz ATmega2560 microcontroller with 8 KB SRAM and 256 KB programmable non-volatile memory. The source code which is used to operate this microcontroller is stored on this programmable memory and is presented in Appendix B. The microcontroller is mounted on a board which has 54 digital input/output pins, 16 analog inputs pins and an USB connector. The USB connector is used to transfer data to and from a computer by an emulated serial connection. A schematic representation of the electrical circuit can be found in Figure A.1.

To operate this setup the user has two momentary buttons and two toggle switches at his disposal. A 16x2 character LCD display gives the user information concerning the measurement number, the current angle of the compensator and the intensity of the light measured by the detector. This display can be controlled by a serial connection, but to reduce the amount of required pins on the board a serial-to-I^2C converter is used.

The stepper motor which is used to rotate the compensator is driven by a modified EasyDriver V4.4 stepper motor driver board. Two additional stepper motor drivers are placed for future expansions of the setup. These boards require 8 pulses from the microcontroller to make the stepper motors do one step. The polarity of the voltage on each coil of the stepper motor is indicated by two LEDs which are reversely connected.

Dust is injected in the vessel by a DC motor driven 'saltshaker'. An optically isolated relay is used to open and close the circuit of this dust injector.

The voltage of the pressure meter output is first halved by a voltage divider and subsequently measured by an analog-digital converter (ADC) of the microcontroller. A stabilized voltage source of 5 V is used as reference for this internal ADC.

A photodiode is used to measure the intensity of the light which is rejected by the polarizer and a photomultiplier is used to measure the light transmitted by the analyzer. The voltages over the resistors caused by the current generated by the photodiode and the photomultiplier are measured by an external ADC based on a Texas Instruments ADS1115. The advantages of this IC over the in the microcontroller integrated ADC are the 5 bit higher resolution, the integrated programmable gain amplifier and the possibility to use differential inputs instead of single ended. The two temperature sensors are based on MCP9808 IC’s. The I^2C protocol is used for the communication between the microcontroller and the IC’s.
Figure A.1: Schematic diagram of the electrical circuit which is designed and constructed by the author to control the stepper motor and read out the sensors by the microcontroller.
Appendix B

Code for the Microcontroller

The source code for the microcontroller is written in C and can be found on pages 62-68. Compiling of the code and transferring the compiled code to the microcontroller is done in the arduino integrated development environment (IDE).

Five header files are needed for the interaction of the microcontroller with the external components. The Wire.h file is used to support the I²C protocol and is standard included in the IDE. The required code for the ADS1115 and MPC9808 IC’s are provided by several parties, the Adafruit_ADS1015.h and Adafruit_MCP9808.h files which are used here are provided by Adafruit. The libraries for the LCD display (LCD.h and LiquidCrystal_I2C.h) are written by Malpartida.

The functions which are available for the user to operate the setup are discussed below.

- The calibration mode is selected by placing the left and the right toggle in the low and high position respectively or by sending c via a command line interface (CLI). The measurement series starts by pressing the right push button or by the command s.

- Dust can be injected by pressing the left button if both toggle switches are in the downward position or by sending i.

- Selecting the RCE mode is done by placing both toggles in the downward position or via the CLI by sending r. The experiment starts when the right push button is pressed or when s is sent.

- The position of the compensator can be changed by pressing the left button when the left toggle is pointing down and the right toggle up. This functionality is standard deactivated in the code and cannot be used via the CLI.

Measurement series can be interrupted by placing the right toggle in the upward position or by sending a. The settings which control the operation of the setup can be changed by changing the values of the variables in the preamble in the settings sections.
// = = = = = = = = = = = = = = = header files = = = = = = = = = = = = = = =
#include <Wire.h> // needed for I2C
#include <LCD.h> // needed for LCD
#include <LiquidCrystal_I2C.h> // needed for LCD
#include <Adafruit_ADS1015.h> // needed for external ADC
#include <Adafruit_MCP9808.h> // needed for temperature sensors

// = = = = = = = = = = = = = = = LCD = = = = = = = = = = = = = = =
#define I2C_ADDR 0x27 // address I2C-to-serial
#define Rs_pin 0
#define Rw_pin 1
#define En_pin 2
#define BACKLIGHT_PIN 3
#define D4_pin 4
#define D5_pin 5
#define D6_pin 6
#define D7_pin 7
LiquidCrystal_I2C lcd(I2C_ADDR, En_pin, Rw_pin, Rs_pin, D4_pin, D5_pin, D6_pin, D7_pin);

// = = = = = = = = = = = = = = = stepper motors = = = = = = = = = = = = = = =
int PinStepperHorizontalStep = 7; // pin stepper motor on table
int PinStepperHorizontalDir = 6; // pin direction stepper motor on table
int PinStepperVerticalStep = 5; // pin stepper motor vertical
int PinStepperVerticalDir = 4; // pin direction stepper motor vertical
int PinStepperMotorStep = 3; // pin extra stepper motor
int PinStepperMotorDir = 2; // pin direction extra stepper motor

// = = = = = = = = = = = = = = = dust injector relay = = = = = = = = = = = = = = =
#define DustInjectorRelay 12 // pin of relay for dust injector

// = = = = = = = = = = = = = = = buttons and toggles = = = = = = = = = = = = =
int PinButtonLeft = 11; // pin dust injector button
int ValueButtonLeft = 0; // variable for reading the pin status
int PinButtonRight = 10; // pin start measurement button
int ValueButtonRight = 0; // variable for reading the pin status
int PinToggleLeft = 8; // pin toggle left
int ValueToggleLeft = 0; // value for toggle left
int PinToggleRight = 9; // pin toggle right
int ValueToggleRight = 0; // value for toggle right
// = = = = = = = = = = = = = = = ADC = = = = = = = = = = = = = = =
int PinPressure = A1; // input pin for the analog output of the pressure meter
int ValuePressure = 0; // value of pressure meter
int TotalValuePressure; // total value pressure reads
int AverageValuePressure; // average value pressure one measurement

// = = = = = = = = = = = = = = = external ADC = = = = = = = = = = = = = = =
Adafruit_ADS1115 ads;
int16_t ValueMultiplier;
signed long int TotalValueMultiplier;
int AverageValueMultiplier;
int16_t ValueDiode;
signed long int TotalValueDiode;
int AverageValueDiode;

// = = = = = = = = = = = = = = = temperature sensors = = = = = = = = = = = = = = =
Adafruit_MCP9808 tempsensor = Adafruit_MCP9808();
float TemperatureLaser; // temperature near laser
float TemperatureDetector; // temperature near detector

// = = = = = = = = = = = = = = = general variables = = = = = = = = = = = = = = =
int mmax; // maximum measurements in selected mode
int m = 0; // measurement
int readsmultiplier; // number of reads multiplier in selected mode
int readsdiode; // number of reads diode in selected mode
int r; // read on this position multiplier
diode
int q; // read on this position
long int s; // number of steps per angle difference
long int anglecompensatorh; // current position compensator
int deltaangle; // delta angle compensator
signed long int pulses; // pulses required for deltaangle
int select = 0; // indication for mode
int angles; // number of angles in each measurement
char mode; // selected mode
char remote; // command line interface
// settings: general

int delayoverheating = 0; // delay to prevent overheating drivers
int timebeforestart = 5000; // time before start is started in ms
int pulselength = 50; // pulse length motor driver in micros, fails <40 us for 12V

// settings: rce

int mmaxrce = 250; // maximum amount of measurements in rce mode
int readsmultiplierrce = 30; // number of reads of multiplier in rce mode
int readsdioderce = 20; // number of reads of diode in rce mode
int delt Panglerrce = 180; // change of angle in rce mode (100 = 1s)

// settings: cal

int mmaxcal = 100; // maximum amount of measurements in cal mode
int readsmultipliercal = 20; // number of reads of multiplier in cal mode
int readsdiodecal = 20; // number of reads of diode in cal mode
int deltanglecal = 50; // change of angle in cal mode (100 = 1s)

// settings: dust injection

int timedust = 200; // duration dust injection in ms

// settings: move to position

int manualmove = 0; // manual move to position on (1) off (0)

unsigned long int CurrentPosition = 35755; // current position (183.20s => 18320)
unsigned long int NewPosition = 1016; // new position (183.20s => 18320)
long int ChangePosition; // change of position

void setup()
{
  pinMode(PinStepperHorizontalDir , OUTPUT);
  pinMode(PinStepperHorizontalStep , OUTPUT);
  pinMode(PinStepperVerticalStep , OUTPUT);
  pinMode(PinStepperVerticalalDir , OUTPUT);
  pinMode(PinStepperMotorStep , OUTPUT);
  pinMode(PinStepperMotorDir , OUTPUT);
}
pinMode(DustInjectorRelay, OUTPUT);
digitalWrite(PinStepperHorizontalStep, LOW);
digitalWrite(PinStepperHorizontalDir, HIGH);
digitalWrite(PinStepperVerticalStep, LOW);
digitalWrite(PinStepperVerticalDir, LOW);
digitalWrite(PinStepperMotorStep, LOW);
digitalWrite(PinStepperMotorDir, LOW);
digitalWrite(DustInjectorRelay, LOW);
Serial.begin(9600); // speed of the serial communication

while (!Serial) { ; } // wait for serial port to connect

ads.setGain(GAIN_SIXTEEN); // 16x gain ext ADC
    limit = +/- 0.256V
    // ads.setGain(GAIN_EIGHT); // 8x gain ext ADC
    limit = +/- 0.512V

cdc.begin (16, 2); // dimensions display
cdc.setBacklightPin(BACKLIGHT_PIN, POSITIVE);
cdc.setBacklight(HIGH);
cdc.home(); cdc.print(“mode:”);

void loop() {
    ValueToggleLeft = digitalRead(PinToggleLeft); // reading left toggle
    ValueToggleRight = digitalRead(PinToggleRight); // reading right toggle
    ValueButtonLeft = digitalRead(PinButtonLeft); // reading left button
    ValueButtonRight = digitalRead(PinButtonRight); // reading right button
    if (Serial.available()) {
        remote = Serial.read(); // reading serial data
    } else {
        remote = ’0’;
    }

    // ============== select mode ==============
    if (ValueToggleLeft == HIGH && ValueToggleRight == HIGH && select != 0) { // deselect
        select = 0;
        cdc.setCursor(5, 0); cdc.print(”des”);
    } else if (ValueToggleLeft == LOW && ValueToggleRight == LOW && select != 1 || remote == ’r’) { // select rce
        select = 1;
        mmax = mmaxrce;
        readsmultiplier = readsmultiplierrce;
readsdiode = readsdioderce;
deltaangle = deltaanglerc;
mode = 'rce';
lcd.setCursor(5,0); lcd.print("rce");
}
else if (ValueToggleLeft == LOW && ValueToggleRight == HIGH
&& select != 2) // select single
    select = 2;
    mode = 'mov';
lcd.setCursor(5,0); lcd.print("mov");
}
else if (ValueToggleLeft == HIGH && ValueToggleRight == LOW
&& select != 3 || remote == 'n') // select cal
    select = 3;
    mmax = mmaxcal;
    readsmultiplier = readsmultipliercal;
    readsdiode = readsdiodecal;
    deltaangle = deltaanglecal;
    mode = 'cal';
lcd.setCursor(5,0); lcd.print("cal");
}
// ============ inject dust ============
else if (ValueButtonLeft == LOW && select == 1 || remote == '
    i') {
    lcd.setCursor(0,1); lcd.print("injecting dust");
    delay(500);
    digitalWrite(DustInjectorRelay,HIGH); // dust injector
    relay on
    delay(timedust);
    digitalWrite(DustInjectorRelay,LOW); // dust injector relay
    off
    delay(1000);
    lcd.setCursor(0,1); lcd.print(" ");
}
// ============= start measuring =============
else if (ValueButtonRight == LOW && select == 1 ||
    ValueButtonRight == LOW && select == 3 ||
    remote == 's' &&
    select == 1 ||
    remote == 's' &&
    select == 3) {
    Serial.print(mode); Serial.print("\n");
    angles = 36000/deltaangle;
    Serial.print(mmax); Serial.print("\n");
    Serial.print(angles); Serial.print("\n");
    Serial.print(readsdiode); Serial.print("\n");
    Serial.print(readsmultiplier); Serial.print("\n");
    lcd.setCursor(0,1); lcd.print("m=0__________________0________");
    delay(timebeforestart);
    pulses = deltaangle * 8; // 8 pulses per step
}
m = 0;

while (m < mmax && ValueToggleRight == LOW && select < 1000) { // till mmax, abort via CLI or right toggle is up
    lcd.setCursor(2,1); lcd.print(m);
    ValuePressure = analogRead(PinPressure); // measure and store voltage of pressure meter
    Serial.print(ValuePressure); Serial.print("
");
    if (!tempsensor.begin(0x19)) { // select T−sensor near laser
        Serial.println("cannot_find_T−sensor_laser");
        while (1);
    }
    TemperatureLaser = tempsensor.readTempC();
    Serial.print(TemperatureLaser); Serial.print("
");
    delay(250);
    if (!tempsensor.begin(0x18)) { // select T−sensor near detector
        Serial.println("cannot_find_T−sensor_detector");
        while (1);
    }
    TemperatureDetector = tempsensor.readTempC();
    Serial.print(TemperatureDetector); Serial.print("
");
    anglecompensatorh = 0; // reset value
    while (anglecompensatorh < 36000) {
        TotalValueDiode = 0;
        q = 0;
        while (q < readsdiode) {
            ValueDiode = − ads.readADC_Differential_0_1();
            TotalValueDiode = TotalValueDiode + ValueDiode;
            q++;
        }
        Serial.print(TotalValueDiode); Serial.print("
");
        TotalValueMultiplier = 0;
        r = 0;
        while (r < readsmultiplier) {
            ValueMultiplier = − ads.readADC_Differential_2_3();
            TotalValueMultiplier = TotalValueMultiplier + ValueMultiplier;
            r++;
        }
        Serial.print(TotalValueMultiplier); Serial.print("
");
        AverageValueMultiplier = TotalValueMultiplier / readsmultiplier;
        lcd.setCursor(12,0); lcd.print("...");
        lcd.setCursor(11,0); lcd.print(AverageValueMultiplier);
        lcd.setCursor(11,1); lcd.print(anglecompensatorh);
        s = 0;
    }
while (s < pulses) { // rotate compensator to next angle
digitalWrite(PinStepperHorizontalStep, HIGH);
delayMicroseconds(pulseLength);
digitalWrite(PinStepperHorizontalStep, LOW);
delayMicroseconds(pulseLength);
s++;
}
anglecompensatorh = anglecompensatorh + deltaAngle;
delay(delayoverheating);
}
lcd.setCursor(0, 1); lcd.print("\n");
ValueToggleRight = digitalRead(PinToggleRight); // reading right toggle
if (Serial.available()) {
    remote = Serial.read(); // reading serial data
}
if (ValueToggleRight == HIGH || remote == 'a') {
    select = 1000; // stopping criteria
}
Serial.print(select); Serial.print("\n");
m++;
}

// = = = = = = = = = = = = = = = move to position = = = = = = = = = = = = = = =
else if (ValueButtonRight == LOW && select == 2 &&
    manualmove == 1) {
    ChangePosition = CurrentPosition - NewPosition;
    if (ChangePosition < 0) {
        ChangePosition = ChangePosition + 36000;
    }
pulses = ChangePosition * 8; // 8 pulses per step
s = 0;
while (s < pulses) { // rotate to new position
digitalWrite(PinStepperHorizontalStep, HIGH);
delayMicroseconds(pulseLength);
digitalWrite(PinStepperHorizontalStep, LOW);
delayMicroseconds(pulseLength);
s++;
}
delay(1000);
}

// = = = = = = = = = = = = = = = nothing = = = = = = = = = = = = = = =
else { ; }
Appendix C

Characterization of the Laser

Light emitted by the CNI V-H-532 laser is used in this investigation. As small changes in the polarization of the scattered light are measured and one measurement takes up to several minutes it is important to know the stability of the emitted intensity in this time scale. The spectral properties of the laser are investigated as the scattering profile and the properties of the optical elements depend on the wavelength.

The measured light intensity over a period of 16 hours is shown in Figure C.1a. Directly after turning the laser on (t=0), the light intensity drops rapidly and stabilizes after roughly 10 minutes. From that moment on the intensity increases gradually except from several short peaks. The gradual increase might not be a problem as the change is small during a measurement, but the short peaks can severely affect the results. After 10 hours the intensity is really instable. Figure C.1b shows the intensity emitted by the laser measured with a higher time resolution as before and several peaks in the intensity are shown. These peaks might be caused by mode hopping. Due to the instable intensity within the time of one measurement it is necessary to monitor the emitted light intensity and possibly reject measurements. In order to do this a photodiode is placed which measures the intensity of the by the polarizer rejected light. A MATLAB script is made to assess the stability and indicates potentially unreliable measurements due to mode hopping.

For low light intensities, the laser is powered by a current source with a constant voltage of 1.7 V and a current of 2.35 A. The original non-adjustable power supply is used for high intensities. According to the specifications of the manufacturer the wavelength of the emitted light is 532 nm and this is verified with an Ocean Optics spectrometer. Because of the limited resolution of the spectrometer, a Jobin-Yvon THR1000 monochromator with a Stanford Computer Optics 4Picos iCCD camera is used to determine the width in the spectral domain of the light. For low light intensities the laser is stable in one domain which has a width of 0.1 nm. For high light intensities, multiple modes are clearly identifiable (Figure C.2) with a total width of 0.4 nm. The properties of the optical elements can be considered similar over this wavelength range. Further broadening of the spectrum might occur due to mode hopping.

Finally, the azimuth of the dominant polarization changes over time which makes it difficult to improve the stability by a custom made power supply with feedback loop.

Concluding, the laser is far from ideal and for that reason a new laser has been ordered.
(a) Intensity of the laser over 15 hours from the moment of turning on the laser (t=0). First fast decay, followed by gradual increase and after 10 hours the intensity becomes unstable.

(b) Laser intensity over a period of 3 minutes with several peaks in the laser intensity possibly caused by mode hopping. These fast changes of the intensity are problematic for the measurements.

Figure C.1: The intensity of the light emitted by the laser over time obtained by two separate measurements.

Figure C.2: Photo (exposure time 5 µs, 800 V and 50 frames/exposure) made by an iCCD camera connected to a monochromator. The wavelength is on the horizontal axis and the spatial information can be found on the vertical axis. For high power it is clear that the light contains at least three modes. The width of each pixel coincides roughly with 14 pm in wavelength. The total spectral width of the emitted light is 0.4 nm.
Bibliography


[32] Eddie van Veldhuizen and Ab Schraber. personal communication.


The white house on the corner, Wokrenter Strasse 35 in Rostock, is the birth house of Gustav Mie. In 1981 - 1982 the house was rebuilt after being destroyed during the second world war.