Självständigt arbete på avancerad nivå

Independent degree project – second cycle

Electronics Design

Hall Measurement for Characterization of Doping Profile in Semiconductor

Habib Ullah Khan Jadoon
Abstract

The aim of project is to characterize a doping profile in relation to the development of detectors. Future generations of CMOS devices will require source and drain contacts, which have a depth of less than 10 to 20nm. The sample preparation and anodic oxidation are the two primary and foremost tasks of the project. The stripping Hall machine HL5900+, which is a fully automatic measuring system (AMS), measured the profiles of semiconductor materials automatically, by repeating anodic oxidation, etching and measurement using the Hall Effect. The concentration profiles of n-type and p-type were measured by means of Hall-effect sheet-resistivity measurements combined with a layer removal by using anodic oxidation and etching. The oxide layer was removed by using an electrochemical process through anodic oxidation.
Acknowledgements

Allah’s Blessing and mercifulness has enabled me to accomplish the goal.

Apart from the individual efforts, the success of any project depends largely on guidance and encouragement. I would like to thank my supervisor Goran Thungstrom for his support, co-operation and encouragement with regards to the successful completion of this project. I am also thankful to the department of Mid Sweden University, where I have been given an environment of innovation and research.

I am grateful to my parents and other family members who have constantly motivated me towards the completion of this task. I also wish to thank my friends and seniors in the Mid Sweden University for their encouragement, which has enabled me to accomplish the task.
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**Terminology**

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<th>Symbol</th>
<th>Definition</th>
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<tbody>
<tr>
<td>$V_H$</td>
<td>Hall Voltage</td>
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<tr>
<td>$R_H$</td>
<td>Hall Coefficient</td>
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<tr>
<td>$R_s/\rho_s$</td>
<td>Sheet resistance</td>
</tr>
<tr>
<td>$R_{SH}$</td>
<td>Sheet Hall Coefficient</td>
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<td>Bath Resistance</td>
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<td>$\mu$</td>
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<tr>
<td>$\rho$</td>
<td>Resistivity</td>
</tr>
<tr>
<td>$\sigma$</td>
<td>Conductivity (reciprocal of resistivity)</td>
</tr>
<tr>
<td>AMS</td>
<td>Automatic Measurement System</td>
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<td>AO</td>
<td>Anodic Oxidation</td>
</tr>
<tr>
<td>SRP</td>
<td>Spreading Resistance Profiling</td>
</tr>
<tr>
<td>SIMS</td>
<td>Secondary Ion Mass Spectrometry</td>
</tr>
<tr>
<td>$B$</td>
<td>Magnetic field</td>
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<tr>
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</tr>
<tr>
<td>$\text{KNO}_3$</td>
<td>Potassium Nitride</td>
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<tr>
<td>$\text{H}_2\text{SO}_4$</td>
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</tr>
<tr>
<td>HF</td>
<td>Hydrofluoric</td>
</tr>
<tr>
<td>$\text{Si}$</td>
<td>Silicon</td>
</tr>
<tr>
<td>$\text{SiO}_2$</td>
<td>Silicon Dioxide</td>
</tr>
<tr>
<td>$\Omega$</td>
<td>Ohm unit of resistance</td>
</tr>
<tr>
<td>nm</td>
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1 Introduction

A semiconductor is a material in which the electrical conductivity occurs between a conductor and an insulator. There are two types of semiconductor materials, namely, intrinsic semiconductor and extrinsic semiconductors. The pure semiconductor material is composed of only one kind of material called intrinsic semiconductor. The intrinsic semiconductors are also known as un-doped semiconductors. The examples of common semiconductors are Silicon, Germanium etc. To form an extrinsic semiconductor, impurities are added, which in turns changes the conductivity of the semiconductor by, the process called “doping”. Conductivity and electrical properties also depends on the number of impurity atoms.

Semiconductor materials, which are doped with elements having spare electrons in their outermost shell, or which are doped with donor impurities, are called n-type, while those semiconductors doped with elements containing fewer electrons in their outmost shell or which are doped with the acceptor impurities, are called p-type. In the p-type, the majority carriers are holes and the minority carriers are electrons, while in the n-type, the majority carriers are electrons and the minority carriers are holes.

In the proposed project, the characterization of the p-type doping profile, diffused with Boron at 950°C and, the n-type doped with phosphorous at 900°C, was conducted using the Hall measurement technique combined with layer removal by means of anodic oxidation. The advantage of using anodic oxidation is that it provides good electrolyte solubility; good control over the thickness of the oxide layer removed and, at each step, provides high purity with low cast, strain free removal of oxide layer, independent crystal orientation and a dopand.

The aim of project is to characterize a doping profile in relation to the development of detectors. Future generations of CMOS devices will require source and drain contacts which have depth of less than 10 to 20nm. In order to define the electrical properties and to enhance the conductivity of the active region for detectors and other electronic devices, it is important to have knowledge of the doping profiles of the semiconductor.
This is particularly the case in a radiation detector application for which a shallow doping layer is necessary in order to avoid any significant recombination of the generated carriers. Another example is the small feature size of a MOSFET, where the control of the source and drain doping is of importance. The downscaling of the dimensions of a MOS transistor requires a reduction of the source/drain junction depth to compensate for the influence of the shorter channel length on the threshold voltage [22].

1.1 Semiconductors Diffusion Techniques and Processes

Diffusion is the basic step for the fabrication of all microelectronics and discrete devices. In order to alter the electrical properties of a semiconductor without there being any crystal damage, impurities are introduced with a minimum leakage current and, in addition, quality junctions are introduced by means of a diffusion process. Diffusion takes place by a series of random jumps in all dimensions; these jumps can take place by means of different techniques which include:

- Interstitial Diffusion
- Substitutional Diffusion
- Interstitial –Substitutional diffusion
- Interstitialcy Diffusion
- Interchange Diffusion
- Grain Body Diffusion

An interstitialcy diffusion method is used for the diffusion of boron and phosphorus in silicon. In this method, vacant sites are required into which the impurity can move. For this purpose interstitials host atoms (self-interstitials) are annihilated by pushing substitutionally located impurity atoms into the interstitial sites, after which the impurities are diffused into adjacent substitutional sites and new self-interstitials are created as shown in figure 1.1.

Therefore, the interstitial position of diffusing an impurity atom is purely a transition state, in moving from one substitutional site to another which increases the concentration of vacant substitutional sites over its equilibrium [16], thus the interstitialcy diffusion technique is faster than others.
The jump rate depends on the temperature, activation energy and Boltzmann constant shown below:

\[ \nu = 4v_o e^{-E_d/kT} \]

\[ E_d = E_s + E_n \]

where \( E_s \) is the activation energy for silicon and its value is 1eV, which must be lower than 2.6eV which is value of the Schottky defects and \( E_n \) is thermal energy ranging from 0.6eV → 1.2eV.

Similarly, for diffusivity

\[ D = D_o e^{-E_d/kT} \]

where \( D_o \) is the diffusion constant in cm²s⁻¹ and it which must be few hundred degrees over the range of the diffusion temperature.

The value of \( E_s \) can be even lower if diffusion takes place in an oxidizing ambient, because the oxidation of silicon promotes vacancies in the near surface region by the creation of self-interstitials [16].

1.1.1 Silicon

Silicon is an intrinsic semiconductor. Its chemical symbol is Si and it has 2, 8, 4 electrons in the K, L and M shells, respectively, and has an atomic number of 14. It is a tetravalent metalloid, with four valence electrons in its outermost shell either for donating or for sharing with other elements.
Silicon is less reactive than carbon but more reactive than germanium. It very rarely occurs in its pure state; however, it is the second most abundant element in the form of silicates and minerals in the earth’s crust and constitutes nearly 28% by mass after oxygen [1]. At present silicon is the most essential element used commercially in electronic devices and modern technology depends on it because of the widespread use of silicon in integrated circuits [2].

1.1.2 Boron

Boron is an intrinsic semiconductor, as is silicon. The chemical symbol used for boron is B. It is group a 5 element. It is a trivalent metalloid which mostly occurs in compounds of borate minerals such as borax and kernite. Its most common isotope is $^{11}$B which has 5 electrons and 6 neutrons. It loses or shares 3 electrons with other elements from its outermost shell and therefore possesses a charge of +3 [4].
Chemically, boron is very similar to silicon and crystalline boron is chemically inert and cannot react with hydrofluoric and hydrochloric acids, but it can be doped with a silicon lattice by means of covalent bonding.

### 1.1.2.1 Doping with silicon and Impurities Profile Behavior

Boron has 3 electrons in its outermost shell and silicon has 4 electrons on its valence shell (see Fig. 1.4)

![Figure 1.4 Outer Shell of Boron and Silicon](image)

Boron is forced to bond covalently with the four silicon atoms around it. Boron has a deficiency of one electron, while making its bond with silicon, therefore the boron atom causes there to be an empty space where an electron should be [3, 21]. This is called an electron hole and the semiconductor is called p-type semiconductor, which is mostly used in solar cells.

![Figure 1.5 Doping of Boron with Silicon or p-type Semiconductor](image)

To observe the concentration profile, diffusion is conducted from an unlimited source. In this case, a silicon wafer is exposed to an infinite...
amount of impurity during the period of diffusion. The difference between the acceptor and donor concentrations provides the effective doping concentration at any point from the following relation, see the appendix.

\[ N(x,t) = N_0 \text{erfc} \frac{x}{2\sqrt{Dt}} \]

where \( N_0 \) is the impurity surface concentration, \( t \) is the diffusion time and \( x \) is the depth. The electrically active concentration of boron varies from \( 10^{18} \rightarrow 10^{20} \text{cm}^{-3} \) with respect to temperature, see the appendix. The behavior of a theoretically computer aided normalized diffusion profile of boron into silicon at different surface concentrations, is shown in figure 1.6. If the impurity level or surface concentration increases, initially, the profile will be flatter and a further increase of surface concentration will leads to a step profile rather than an anomalous curve, shown in the figure below.

Figure 1.6 Normalized Diffusion Profile of Boron into silicon at Different Surface Concentration [16]

1.1.3 Phosphorous

Phosphorous is a chemical element with an atomic number of 15. The chemical symbol used for phosphorous is P. It is a pentavalent non-metal and it mostly occurs as a mineral in its oxidized state. It is never found as a free element because of its high reactivity. There are twenty three isotopes of phosphorous \(^{24}\text{P} \) to \(^{46}\text{P} \) and the most stable isotope is \(^{31}\text{P} \). It has 2, 8, 5 electrons in the K, L and M shells, respectively. It will gain or share 3 electrons with other elements from its outermost shell and therefore possesses a charge of -3 [5][20].
1.1.3.1 Doping with silicon and Impurities Profile Behavior

Phosphorous has 5 electrons in its outermost shell and silicon has 4 electrons on its valence shell (see Fig. 1.7, 1.2)

Phosphorous is bonded covalently with the four silicon atoms around it. It has one spare electron while making a bond with silicon, see fig 1.8, and this electron is free to move. The extra negative charge provided by the electron makes the phosphorous doped silicon grid to be negatively charged [21].

For the concentration profile of phosphorus, the diffusion of phosphorus is also conducted from an unlimited source. In this case, the silicon wafer is exposed to an infinite amount of impurity during the period of diffusion. Phosphorus has a high solid solubility around $10^{21} \text{cm}^{-3}$ with an active carrier concentration of $3 \times 10^{20}$ and it is extensively used for the fabrication of MOS and bipolar micro-electronics. The electrically active concentration of phosphorus ranges from $10^{19} \rightarrow 10^{21} \text{cm}^{-3}$ with
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respect to temperature, see the appendix. The behavior of a shallow diffused/doped profile of an unlimited source of phosphorous into silicon is illustrated in fig 1.9a and fig 1.9b shows the behavior in the tail region.

Figure 1.9 (a) Behavior of Phosphorous Diffused in Silicon (b) Diffusivity of Phosphorous in Tail Region [16]

1.2 Hall Effect in Semiconductors

When a current-carrying semiconductor is retained in a magnetic field, the charge carriers of the semiconductor experience a force in a direction perpendicular to both the magnetic field and the current. At equilibrium, a voltage appears at the semiconductor edges [6].

For a simple metal, where there is only one type of charge carrier (electrons), the Hall voltage \( V_H \) is given by

\[
V_H = -\frac{IB}{ned}
\]

where \( I \) is the current across the plate length, \( B \) is the magnetic field, \( d \) is the depth (thickness) of the plate, \( e \) is the electron charge, and \( n \) is the charge carrier density of the carrier electrons.

The Hall coefficient is defined as
The simple formula for the Hall coefficient, given above, becomes more complex in semiconductors where the carriers are generally both electrons and holes, which may be present in different concentrations and have different mobilities. For moderate magnetic fields the Hall coefficient is [6].

\[ R_H = \frac{\rho \mu_p^2 - n \mu_n^2}{q(p\mu_p + n\mu_n)^2} \]

where \( p \) is the hole concentration, \( \mu_p \) is the hole mobility, \( n \) is the electron concentration (density), \( \mu_n \) is the electron mobility and \( q \) is the absolute value of the electronic charge.

There are various methods to prepare the samples of semiconductor materials for the stripping Hall measurement. There are many semiconductor materials but, in proposed project, silicon wafers of n-type and p-type with a sheet resistivity of 11.7 ohm/square and 67 ohm/square, respectively, have been used. The silicon P-type wafer is doped with boron at 950 °C and the n-type is doped with phosphorous at 900 °C.
The stripping Hall machine HL5900+, is a fully automatic measuring system (AMS) and was used to measure the profiles of the semiconductor materials, automatically, by repeated anodic oxidation, etching and measurement using the Hall Effect. The AMS provides the carrier concentration and mobility profiles of the semiconductor materials as a function of depth. The software for the equipment contains graphics for converting the resistivity into concentration and mobility. [7]

1.3 Outline

The thesis report is presented in the following manner.

Chapter 1 provides the introduction

Chapter 2 describe the theory related work, different methods for doping profile measurement and the process of anodic oxidation.

Chapter 3 describes the experimental setup, the working of the Hall profiler, silicon sample wafer processing and preparation and the different parameters used in the measurement cycle.

Chapter 4 includes the results and the discussions which took during the measurement phase.

Chapter 5 includes the conclusion and future work.
2 Theory

The research and development of semiconductor devices requires a reliable determination of the doping profiles in the device layer structures. For the doping profile measurements, the methods used are the “four point probe method”, “secondary ion mass spectrometry” “spreading resistance”, “differential conductance”, “stripping Hall effect method”, Rutherford back scattering and the C-V method. Some of these techniques are explained below.

In this project, the measuring of the doping profile for the n-type and p-type samples was performed, and the second step includes the measurement of the carrier concentration and the mobility of the carrier as a function of depth.

The resistivity $\rho$ is the important factor for the characterization of the semiconductor material and devices because it affects the device threshold voltage, capacitance and its series resistances. It depends on the densities of the electrons and holes and, additionally, on the mobilities of the electrons and holes.

\[ \rho = \frac{1}{q(n\mu_n + p\mu_p)} \]  

where $\rho$ is the resistivity, n and p are the electron and hole densities (concentrations), respectively. $\mu_n$ and $\mu_p$ are the electron and hole mobilities, respectively, and q is the unity charge.

For the n-type material

\[ \rho = \frac{1}{q(n\mu_n)} \]

and for the p-type material

\[ \rho = \frac{1}{q(p\mu_p)} \]

The conductivity is the reciprocal of the resistivity.

\[ \sigma = \frac{1}{\rho} \]
2.1 Four Point Probe Method

The four point probe method is a very common technique for measuring the resistivity of semiconductor materials.

![Four Point Probe Method](image)

Figure 2.1 illustration of Four Point Probe Method [28]

Current I from the constant current source flow through the outermost probes and the voltage drop V is measured between the inner two probes. However, the contact resistance $R_c$ is negligible and is thus ignored. The four point probe method has equal probe spacing and, the measurement of resistivity for a relatively thick sample ($t >> s$) as compared to that for the probe spacing is defined as;

$$\rho = 2\pi s \frac{V}{I}$$  \hspace{1cm} 2.2

$\rho$ is the resistivity expressed in ohm-cm, s is the probe spacing, which is normally in the range of 0.5mm to 1mm, V is the measured voltage and I is the current in amperes.

For arbitrary shape samples, the resistivity becomes;

$$\rho = 2\pi s \left(\frac{V}{I}\right) F$$  \hspace{1cm} 2.3

F is the correction factor, which corrects for probe location, probe placement sample thickness and the nature of the bottom wafer surface boundary.

Where $F = \frac{t/s}{2\ln(2)}$ for $t << S/2$

Therefore the resistivity becomes;

$$\rho = 2\pi s \left(\frac{V}{I}\right) \frac{t/s}{2\ln(2)}$$
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\[
\rho = \pi \frac{V}{I} \frac{t}{\ln(2)}
\]

\[
\rho = 4.532 \ t \left(\frac{V}{I}\right)
\]

where \( t \) is the sample thickness.

The sheet resistance is the measure of the average resistivity over the sample thickness [8]. The sheet resistance \( R_s \) for a homogenously doped sample is given as;

\[
R_s = \frac{\rho}{t} = 4.532 \left(\frac{V}{I}\right)
\]

The sheet resistance \( R_s \) for a non-homogenously doped sample is given as;

\[
R_s = \frac{1}{q \int_0^t \left[ n(x) \mu_n(x) + p(x) \mu_p(x) \right] dx}
\]

Advantages

- The four point probe method provides accurate results for equally spaced thick and flat samples.

Drawbacks

- It is difficult to calibrate all four probes with the same spacing.
- It is difficult to calibrate a force of nano Newtons on the tips of the probes in order to measure the resistivity of the sample under consideration.
- The four point probe method is unable to measure the mobility independently.

2.2 Spreading Resistance Profiling Method

The four point probe method provides the sheet resistance. The spreading resistance technique is used to generate the resistivity and dopant density depth profiles on a wide dynamic range from \(10^{12}\) to \(10^{21}\) cm\(^3\). It has the capability of profiling shallow junctions.
The two aligned probes of a tungsten-osmium alloy, having the probe resistance of $R_p$, are placed on a bevelled surface of a semiconductor as shown in figure 2.2.

![Image of spreading resistance profiling](image)

**Figure 2.2 Principle of Spreading Resistance Profiling [10]**

The electrical resistance between the probes is measured by applying a bias dc voltage of 10mV. The measured resistance is given as:

$$ R = 2R_p + 2R_c + 2R_{sp} $$

where $R_p$ is the probe resistance, $R_c$ is the contact resistance and $R_{sp}$ is the spreading resistance under each probe. The range used for the probe spacing is 20 to 40um. The voltage between the probes during the measurement is maintained at a constant level within the range 5 to 10mV dc so as to reduce the effect of the contact resistance. The probe-semiconductor contact is a metal semiconductor contact that generally has a nonlinear current-voltage characteristic [9].

$$ I = I_0(e^{qV/kT} - 1) $$

However this current voltage is characteristically linear for voltages less than

$$ kT/q \approx 25mV. $$

The displacement of the probes $\Delta x$, having a slight bevelled angle $\alpha$, as shown in figure 2.2, enables the spreading resistance values detected on the bevelled surface. The detected values are converted into the corresponding depth resistance by means of the relationship:

$$ \Delta h = \Delta x \cdot \sin \alpha $$
where Δh is the corresponding vertical step for the successive removal of the thin layer;

Consider the current I which flows from the tip of diameter 2r of the probe into a semiconductor with resistivity ρ. The current is concentrated at the tip of the probe and spreads out radially from the tip, hence is called the “spreading resistance”.

The spreading resistance for a semi-infinite homogenous sample having a cylindrical contact with the planer and the non-indenting probe tip is given as;

$$R_{sp} = \frac{\rho}{4r} \text{ ohms}$$  \hspace{1cm} 2.7

The spreading resistance for a hemispherical sample having an indenting probe tip of radius r is given as;

$$R_{sp} = \frac{\rho}{2\pi r} \text{ ohms}$$  \hspace{1cm} 2.8

For successful SRP measurements, a uniform low angle bevels is required. The bevelled surfaces are obtained by lapping a small piece of semiconductor in a diamond paste and thus avoid the photo conductance effect for silicon. The photo conductance effect is avoided by taking the measurement in the dark.

Advantages

- SRP provides dopant density depth profiles for a wide dynamic range of $10^{12}$ to $10^{21}$ cm$^{-3}$.
- A complete profile on a small area can be achieved by using SRP.
- Shallow junction profiles can be measured.

Drawbacks

- The equipment is very expensive.
- The equipment requires both skill and expertise in order to calibrate the probes on a bevelled surface so as to set an accurate angle for the measurement.
- The minimum requirement of the step size for measurement is larger than 500 Å.
2.3 **Stripping Hall Effect Method**

The principle of the Stripping Hall Effect constitutes both sheet resistance $R_S$ and the Hall sheet coefficient $R_H$ measurements versus depth. The active layer is being thinned down by stripping off a thin sub-layer after each measurement to ensure the depth monitoring [10]. According to the Hall Effect principle, current $J_x$ is fed to the uniform sample and a constant magnetic field of 0.31T is applied perpendicular to the sample and thus the potential difference is measured.

![Figure 2.3 Principle of Hall Effect](image)

The force on a charge in an electric field is

$$ F = qE $$

where the electric field is proportional to both the current and magnetic field, therefore

$$ E_y = R_H J_x B_Z $$

$R_H$ is the Hall coefficient and can be calculated through the Hall voltage

$$ R_H = \frac{t V_H}{J_x B_Z} $$

where $t$ is the thickness of the active layer,

$R_H$ can also be calculated from the carrier concentration and mobilities as shown below

$$ R_H = \frac{r p \mu_p^2 - n \mu_n^2}{q \left( p \mu_p + n \mu_n \right)^2} $$

2.9
where \( P \) is the hole concentration, \( \mu_P \) is the hole mobility, \( n \) is the electron concentration (density), \( \mu_n \) is the electron mobility and \( q \) is the absolute value of the electronic charge and \( r \) is the correction factor, also called the Hall scattering factor. The Hall scattering factor is assumed to be equal to 1.

The Hall sheet coefficient for an average value of \( V_H \) is defined as

\[
R_{SH} = \frac{V_H}{J_x B_Z}
\]

or

\[
R_{SH} = \frac{R_H}{t}
\]

The sheet carrier concentration (density) \( N_s \) is given as

\[
N_s = \frac{1}{q R_{SH}}
\]

The Hall mobility is defined as the product of the Hall coefficient and the conductivity [10]

\[
\mu_H = |R_H \sigma|
\]

The relation for the drift mobility with the Hall mobility is given as

\[
\mu_H = r \mu
\]

The sheet conductance is expressed as

\[
G_s = \frac{1}{R_s}
\]

The sheet resistance \( R_s \) is measured when the remaining thickness of the removed thin layer is equal to the \( W \) shown in Fig 2.3.

\[
R_s = [\int_0^w \sigma(x)dx]^{-1}
\]

The sheet Hall coefficient is given as

\[
R_{SH} \sigma^2 = \int_0^w R_H(x) \sigma^2(x) dx
\]
During the stripping Hall measurement the thickness of the removed layer must be controlled and uniform.

\[
n_i = -\frac{\Delta G_{s,i}}{\Delta h_i} q \mu_i
\]

where \( \Delta h_i \) is the thickness of the \( i \)-th removed layer and \( \mu_i \) is the drift mobility and is given as

\[
\mu_i = \frac{1}{r} \frac{\Delta (G_{s,i}^2 R_{SH,i})}{\Delta G_{s,i}}
\]

Electrochemical is an effective method for the growth of a thin anodic oxide layer because it allows for a good uniform control regarding the thickness of the grown oxide, based on the electrolytic cell calibration. Further, the process of anodic oxidation and the thickness of the grown oxide is explained in section 2.3.3.

2.3.1 Van Der Pauw (VDP) Method

In order to improve the Hall Effect measurement technique for a flat, homogeneous sample, the Van Der Pauw method is used, particularly for depth profiling. There are two VDP patterns, namely, clover leaf and a Greek cross. However, clover leaf is recommended for reducing the errors in the Hall voltage \( V_H \).

![Figure 2.4 Clover leaf VDP Pattern](image)
2.3.1.1 Resistivity Measurement

For resistivity ($\rho$) measurement using Van Der Pauw, the current ($I_{12}$) is applied to contacts 1 and 2 and, the voltage drop ($V_{43}$) is measured across contacts 4 and 3 as shown in figure 2.5. After this the pair of contacts is changed for both the current and voltage i.e. 1 and 4 and 3 and 2, respectively.

![Figure 2.5 Resistivity Measurement Arrangement](image)

\[
\rho = \frac{mt}{2\ln(2)} \left[ \frac{V_{43}}{I_{12}} + \frac{V_{32}}{I_{41}} \right] \times F(Q) \quad (\Omega \cdot cm) \quad 2.13
\]

where $t$ is the thickness of the active layer, $F$ is the correction factor and $Q$ is the symmetry factor for the sample defined as

\[
Q = \frac{V_{43}I_{41}}{V_{32}I_{12}} \quad 2.14
\]

The correction factor $F$ is a function of the symmetry factor and is defined as

\[
F = 1 - 0.34657A - 0.09326A^2 \quad 2.15
\]

where

\[
A = \begin{bmatrix}
\frac{V_{43}}{I_{12}} & \frac{V_{32}}{I_{41}} \\
\frac{V_{43}}{I_{12}} & \frac{V_{32}}{I_{41}}
\end{bmatrix}^2 = \left[ \frac{Q-1}{Q+1} \right]^2 \quad 2.16
\]
A sheet resistance is defined when the specimen thickness is unknown

\[ \rho_s = \frac{\rho}{t} \quad (\Omega/\text{square}) \quad 2.17 \]

The resistivity measurement in HL5900+ is based on a constant current value for all six possible combinations therefore, the equations 2.13 and 2.14 for \( \rho_s \) and \( Q \) are reduced respectively.

\[ \rho_s = 2.2662 \frac{V_{43} + V_{32}}{I} \times F(Q) \quad (\Omega/\text{square}) \quad 2.18 \]

\[ Q = \frac{V_{43}}{V_{32}} \quad 2.19 \]

### 2.3.1.2 Hall Coefficient

After the resistivity measurement based on equations 2.15, 2.18, and 2.19, it is possible to measure the Hall coefficient by using Van Der Pauw. A constant current is applied to two adjacent contacts of the sample while the potential difference is measured across the remaining adjacent contacts, as shown in fig 2.6 and a constant magnetic field of 0.31 Tesla is applied perpendicular to the sample.

\[ q(v \times B) = qE \quad 2.20 \]

The equation shows the balancing of both the magnetic and electric fields on a mobile charge.

The current is given as

\[ I = qtvNW \quad 2.21 \]

where \( t \) is the thickness of the layer, \( q \) is the elementary charge on either the electron or the hole, \( v \) is the carrier velocity, \( N \) is the carrier density and \( W \) is the separation between the two contacts. The above equation is only valid for a constant carrier distribution.
The Hall coefficient is calculated by replacing the vector quantities with scalar quantities in equations 2.20 and 2.21

\[
\frac{I}{qN} = \frac{V_H t}{I_B} = R_H \quad \text{cm}^3/\text{C} \tag{2.22}
\]

where \(V_H\) is the measured Hall voltage \((V_H = E.W)\) and \(R_H\) is the Hall coefficient. The sheet Hall coefficient is defined in eq 2.23 when the layer thickness is unknown for an average value of \(V_H\)

\[
R_{HS} = \frac{R_H}{\ell} \quad \text{cm}^2/\text{C} \tag{2.23}
\]

The sheet carrier concentration (density) \(N_s\) is given as

\[
N_s = \frac{1}{qR_{HS}} \tag{2.24}
\]
The effective Hall mobility is defined as

\[ \mu_H = \frac{R_{HS}}{\rho_s} \]  

**2.3.2 Depth Profiling**

The depth profile of the n-type and p-type samples is achieved by a well-controlled and repeated removal of thin homogenous layer by anodic oxidation using the stripping Hall machine HL5900+. AMS measures the sheet resistance \( \rho_s \) and the Hall sheet coefficient \( R_{HS} \) of the remaining sample after each removal of a layer.

**2.3.3 Anodic Oxidation**

This is the most appropriate method for Hall profiling as it provides good control in relation to the thickness of the layers removed at each step. Anodic oxidation is also known as anodization. The electrolytic bath is used in the method which involves a plunged semiconductor acting as the anode whereas; the carbon electrode behaves as a cathode during the process. The oxide layer is formed on the surface of a semiconductor when a current flows through the electrolyte leads.

The thickness of an oxide layer, formed on the surface of a semiconductor, is a function of a voltage difference between the electrolyte and the semiconductor. The oxide is then selectively etched away without there being any loss of the underlying semiconductor material. A strong illumination is required to photo generated holes in the n-type material and also important in the p-type material to ensure the anodization current is independent of the carrier concentration.

There are two anodization methods, namely constant voltage and constant current.

**Constant Voltage Method:**

In the constant voltage method, the anodization current is allowed to drop from an initial value to a fixed preset value.
**Constant Current Method:**

In the constant current method, the voltage is allowed to increase to the predetermined value, while the current remains constant until voltage has reached the preset voltage, when the anodization voltage is reached, the current decays to 30% of the initial current value, as shown in fig 2.7. The decay time for the current is set after the first measurement by the user and is known as the hold time.

![Figure 2.7 IV Curve Demonstrating Constant Current Method](image)

The crucial aspect, throughout this process, is the removal of uniform thin layers from a sample in a controlled and repeatable manner. However, in anodic oxidation, this is achieved by means of constant current method. To improve the resolution of the thickness of the oxide layer, the offset voltage i.e. the voltage drop due to the finite resistance of the electrolyte and the semiconductor, should be taken into account. When the anodized current is applied, the offset voltage is recorded and the voltage across the anodizing cell is allowed to increase until a stabilized voltage, known as V anodize, is reached.
The control circuitry switches to a constant voltage mode when $V_{anodize}$ is reached and the current is then allowed to decay so as to homogenize the thickness of the oxide across the whole area of the sample. The homogeneous oxide thickness is also directly dependent on the net forming voltage.

$$V_{net} = V_{anod} - V_{offset}$$

The thickness of the semiconductor material converted into an oxide is calculated from the following relationship.

$$T_{0x} = V_{anod} - V_{offset}(1 - I_{hold}/I_{anod})$$

where $(I_{anod})$ is the initial anodizing current and $I_{hold}$ is the final anodizing current, $V_{offset}$ is the initial offset voltage and $V_{anod}$ is the final value of the anodizing voltage.

To reveal the carrier concentration $n(x)$ profile, it is necessary to calculate the parameters of the anodic oxidation (AO), the thickness $\Delta h$ ($\Delta x$) of Si to be removed, the number of removed layers $\Delta h_n$ necessary to determine the whole profile and the voltage required to create an oxide layer of thickness $\Delta h$, which is subsequently removed by electrochemical etching. The removed $\Delta h$ thickness of silicon (Si) can be calculated as [15]; for a p-type semiconductor (boron-implanted silicon).

$$\Delta h = 12.8662e^{(6.8022\times10^{-3}\Delta V)}$$

and for the n-type (phosphorus-implanted silicon)

$$\Delta h = 11.5162e^{(7.91435\times10^{-3}\Delta V)}$$

$\Delta V$ is the voltage drop across the anodic oxide.

$$\Delta V = V_{anod} - V_{offset}$$

$V_{anod}$ is the applied voltage and $V_{offset}$ is the offset, or, the rest potential of the anodization bath.
2.3.3.1 Chemical Process in Anodic Oxidation

The impurity profiles are not altered during anodic oxidation because AO is conducted at room temperature and thus; it is important for the controlled layer removal of the silicon. In relation to the anodization process, the semiconductor is connected to the positive terminal of the power supply, which acts as the anode in the electrolytic cell, whereas, platinum or carbon is connected to the negative terminal of the supply and acts as the cathode. The source impedance of the supply should be kept low so that the anodization occurs at constant voltage, alternatively, high source impedance allows for the anodization to proceed under constant current conditions [16].

The primary oxidizing component in the anodization process is water, which dissociate into \( H^+ \) and \( (OH)^- \). The dissociation reaction of water is,

\[
H_2O \leftrightarrow H^+ + (OH)^- 
\]

The equilibrium constant is \( 10^{-14} \) for de-ionized water at 24°C. At equilibrium \( [H^+] = [(OH)^-] \), the concentration of \( [H^+] \) is \( 10^{-7} \), results in a pH of 7. The dissolution rate and the resistance of the electrolyte can be changed by adding pH modifiers.

The anodization process is conducted in a non-aqueous solution, where the pH is set before the addition of the non-aqueous medium. As a non-aqueous medium, ethylene glycol is used and another component, KNO\(_3\) is used in the electrolyte to control the viscosity and dielectric constant of the process. It also improves the sensitivity and the reproducibility to ambient conditions and with regards to any contaminants of the process. Porous oxide is formed as a result of the anodization, contains water, which can be used as an advantage for removing a controlled layer of a semiconductor at room temperature in the anodic oxidation process followed by either chemical or electrochemical dissolution [17].
Anodic oxides of silicon are generally poorer than those which are thermally grown, so that their use is restricted to singular applications. However, the anodic oxides of polycrystalline silicon have higher breakdown electric field strengths as compared to those for thermal processes [18].

There are four main steps involved in the reaction for the anodic oxidation of silicon which are as follows:

1) The primary oxidizing component in the anodization process is water, which dissociate into $\text{H}^+$ and $(\text{OH})^-$

The dissociation reaction of water is

$$2\text{H}_2\text{O} \leftrightarrow 2\text{H}^+ + 2(\text{OH})^-$$

2) The charge transfer from the silicon until equilibrium is reached is due to the electrochemical potential difference between the silicon and electrolyte and surface layer being partially depleted of electrons.

$$\text{Si} + 2\text{h}^+ \rightarrow \text{Si}^{2+}$$

The holes are supplied from the bulk of the semiconductor to the semiconductor-electrolyte interface and this promotes the silicon surface atom to the higher oxidation state during anodization process.

3) Hydro-oxide is formed when $\text{Si}^{2+}$ reacts with $(\text{OH})^-$. 

$$\text{Si}^{2+} + 2(\text{OH})^- \rightarrow \text{Si}(\text{OH})_2$$

4) Silicon dioxide is subsequently formed from silicon hydro-oxide and, the reaction is given as:

$$\text{Si}(\text{OH})_2 \rightarrow \text{SiO}_2 + \text{H}_2$$

The overall reaction can be written as:

$$\text{Si} + 2\text{h}^+ + 2\text{H}_2\text{O} \rightarrow \text{SiO}_2 + \text{H}_2 + 2\text{H}^+$$
This delivers holes to the semiconductor surface by means of a battery and this is relatively complex in the n-type material as compared to the p-type during anodization. However, the initial charges transfer (to establish equilibrium) in the n-type material creates:

- The depletion layer in the semiconductor
- A barrier to the flow of holes.

The anodization cell that is operated at a voltage exceeding the junction breakdown voltage and, additionally, avalanche-generated holes allow for the oxidation to proceed [16]. When the oxide grows, the available voltage reduces across the depletion layer and the anodization process will not stop until the voltage drop is less than the breakdown voltage of the electrolyte-semiconductor system.

2.4 Oxide Growth Rate

2.4.1 Faraday’s Law of Electrolysis

The consumption of semiconductor material into its oxide, during electrolysis, is directly proportional to the quantity of electrical charge transferred. The equivalent weight or mass ($m$) in grams of a semiconductor consumed per coulomb of the charge is expressed as:

$$m = \left(\frac{Q \times M}{F \times z}\right)$$

where

- $m$ is the mass of the substance liberated at an electrode in grams
- $Q$ is the total electric charge passed through the substance
- $F$ is the Faraday constant = 96,485 C mol$^{-1}$
- $M$ is the molar mass of the substance
- $z$ is the valence number of ions of the substance (electrons transferred per ion).

$$if \quad \frac{Q \times M}{z} = W$$
Then the Faraday’s equation can be written as

\[ m = \left( \frac{W}{F} \right) \]

where \( W \) is the electrochemical equivalent weight in grams of the material involved and \( F \) is the Faraday constant, 96,485 C mol\(^{-1}\).

The important factor during anodization is the oxide dissolution rate and it depends on the concentration and nature of the electrolyte. When the bath resistance \( R_B \) and circuit resistance \( R_C \) are considered the initial current flows through the sample and can be written as:

\[ I_0 = \frac{\Delta V}{R_B + R_C} \]

For p-type semiconductor

\[ \Delta V = V_A - V_R \]

\( V_A \) is the applied voltage and \( V_R \) is the offset or, the rest potential of the anodization bath (reverse electromotive force generated when the cell is used as a battery).

For an n-type semiconductor, \( V_R \) is the sum of the rest potential and the breakdown voltage associated with its depletion layer [19].

Now, the relationship of the dissolution rate \( (f_d) \) of the film and the dissolution current \( (I_d) \) is expressed as:

\[ f_d = I_d \times \alpha \]

where \( \alpha \) is the oxide formation rate and is defined as the increase in film thickness per unit charge, passing through the unit area, related to the current efficiency.

The current efficiency is the ratio of the experimental values to the theoretical values expressed in terms of percentage and the current efficiency for silicon is 1-3%. 

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The current flow during anodic oxidation through the oxide is initially ionic and is expressed as \( J = e^{K E_{ox}} \) where \( E_{ox} \) for silicon is \( 1 \times 10^7 \) to \( 1 \times 10^8 \) V/cm.

The final equation for the thickness of oxide growth is:

\[
Net \, oxide \, thickness = \frac{\Delta V}{E_{ox}} (1 - \frac{I_d}{I_o})
\]

An input of the value of \( \Delta V \) means that the equation becomes

\[
Net \, oxide \, thickness = \frac{V_A - V_R}{E_{ox}} (1 - \frac{I_d}{I_o})
\]

The net oxide thickness for silicon is about 2.2 Å/V to 3 Å/V.

By using the above relationship for the oxide thickness and voltage, the maximum oxide growth has been calculated under ideal conditions in ideal conditions for different voltages levels:

**Mathematical calculations:**

\[
1\, \text{Å} = 0.1 \text{nm} \\
3\, \text{Å/V} = 0.3 \text{nm/V}
\]

At 20 volts the oxide thickness is

\[
20(3\, \text{Å/V}) = 20(0.3 \text{nm/V}) \\
60\, \text{Å}/20\, \text{V} = 6 \text{nm}/20\, \text{V}
\]

At 40 volts the oxide thickness is

\[
120\, \text{Å}/40\, \text{V} = 12 \text{nm}/40\, \text{V}
\]

At 60 volts the oxide thickness is

\[
180\, \text{Å}/60\, \text{V} = 18 \text{nm}/20\, \text{V}
\]
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2 Theory

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Similarly, for 80 volts the oxide thickness is

\[ 240\text{Å}/80V = 24\text{nm}/80V \]

Theoretically it is proven that the oxide thickness grown on silicon is linear with respect to the voltage. The time constant for the growth is calculated as:

\[ \tau = \frac{R_B + R_C}{\alpha E_{ox}} \]

In a similar manner to that for the voltage, the oxide thickness should vary linearly with time. The electric field is constant during the anodization process; therefore, the voltage drop should also increase linearly with time.

Advantages

- The AMS for Hall measurement is cheaper as compared to that for SRP and others.
- Equipment is easier to use.
- Both shallow junction profiles and implanted profiles can be measured.
- Hall Effect method is the best for measuring the concentration and mobility of the profile.

Drawbacks

- Sample preparation is difficult to achieve in relation to providing a good resolution of reliable results.
- Ohmic contacts and all conducting paths require preventative measures with regards to a leakage current, to ensure the isolation of contacts during AO and oxide stripping.
- A well-controlled uniform growth and repeated removal of the oxide layer is necessary to attain good depth resolution.
2.5 Secondary ion Mass Spectrometry (SIMS)

Secondary ion mass spectrometry is a very powerful technique for the characterization of a chemical profile of a semiconductor. This technique is also known as “ion microprobe” and “ion microscope”. The technique depends on the sputtering of the material from the solid. The material, which is removed slowly by this technique, generally has neutral atoms that cannot be analyzed but, a few atoms are ionized atoms that are either positively or negatively charged and can be analyzed by passing them through the energy filter and mass spectrometer.

This method has the ability to detect many elements because of its good detection sensitivity; however, it is less sensitive as compared to the electrical or optical methods. SIMS has a depth resolution of 1 to 5nm and the simultaneous detection of different elements can also be achieved by means of this method. During SIMS analysis, the slow sputtering mode is called the static SIMS and is used for depth profiles, while the second mode is called the dynamic SIMS and it yields quantitative information.

![Figure 2.8 Block Diagram of Secondary ion Mass Spectrometry Measurement](image)

The following steps are involved for SIMS measurement.
2.5.1 Sputtering

Sputtering is a process in which monatomic and polyatomic particles of the sample material are produced by the bombardment of an incident (primary) ion which loses its energy as a result of momentum transfer, and which; comes to rest below the sample surface, up to tens of nm. The process ejects atoms within the sample and it takes place when atoms near the sample surface receive the sufficient energy from the primary ion to be injected from the sample.

The primary energy of 1 to 20KeV is required to implant and mix the primary ion of species such as $C_6^+$, $O_2^+$, $Ar^+$, $Ga^+$ and $O$ with the monolayers of sample atoms; the range of the depth is 1 to 10nm in relation to ion beam sputtering in SIMS measurement.

The sputtering rate varies from 0.5 to 5nm/sec, depending upon the incidence angle and the nature and energy of the primary ion; it also depends on the target sample material and its crystal orientation and introduces roughness of the surface in the sputter craters. The lateral resolution range is from 0.5μm to 100μm with depth resolution of 5 to 10nm.

The average number of atoms sputtered per primary ion or. the ratio of the number of atoms sputtered to the number of primary ions. is called the “Sputtering rate”.

2.5.2 Secondary Ion Yield

Yield is the ionization efficiency in SIMS measurements. Only ions can be detected, therefore, the secondary ion yield or the yield of ionized ejected atoms is very important however, the secondary ion yield is very low as compared to the total yield. For electropositive elements such as boron in silicon and electronegative oxygen $O_2^+$ species, produces predominantly positive secondary ions and yields vary for different elements.
There is also a variation in terms of the secondary ion yield in the same element in different samples which is known as the “matrix effect”. The yield of Si in SiO2 is about 100 times higher than the yield of Si from an Si substrate [11].

2.5.3 Approaches for SIMS

There are two approaches used in the SIMS measurement technique.

I. Ion microprobe
II. Ion microscope

2.5.3.1 Ion Microprobe

The output of the mass spectrometry and secondary ion mass analyzer is synchronized with the primary beam in order to create a map of the secondary ion intensity across the surface and is displayed on the CRT in “ion microprobe instrumentation. The mass spectrometer has both magnetic and electrostatic sector analyzers in tandem. In the electrostatic analyzer, the ion travels between two plates, which are separated by a distance d with a radius of curvature, rc. The energy E of an ion in an electrostatic analyzer is given as

\[ E = \frac{qVr_c}{2d} \]  

where V is the voltage between two plates, which allows an ion having an energy E to be transmitted without being struck by either plate. In the magnetic sector analyzer, the relationship of mass per charge with other parameters is given as

\[ \frac{m}{q} = \frac{qB^2r^2}{2E} \]

where B is the magnetic field around the ion of mass m, r is the radius of the path through which the ion travels. By substituting E in the above equation, the mass per charge or mass resolution equation becomes

\[ \frac{m}{q} = \frac{dB^2r^2}{Vr_c} \]
The mass resolution can be used to detect two masses differing by only 0.003%.

### 2.5.3.2 Ion Microscope

This instrumentation approach of the SIMS measurement involves a direct imaging system in which a secondary ion is captured over the entire image area of the sample and is illuminated by a primary ion beam flow with a resolution of 1μm. In order to preserve the spatial distribution of the secondary image, the system includes both electrostatic and magnetic sector analyzer and the image is then displayed on a fluorescent screen, after amplification through a micro channel plate. For analysis of a selected area a small aperture insertion is feasible. The lateral resolution can be as small as 50nm, depending on the beam size of the primary ion.

Another technique for obtaining an ion image is the raster scanning of an ion beam across a sample of square area. The secondary ion intensity of the squared area sample is measured and displayed as a function of the lateral position of the scanning ion beam’s small area. It is important to analyze the uniform bottom area of the sputtered crater so as to obtain a high depth resolution rather than to analyze the crater from the edges, side walls or top surface because of the higher doping density as compared to that for the bottom.

### 2.5.4 Time of Flight SIMS

The pulsed ions of a primary beam from a liquid Ga⁺ gun with a beam diameter of 0.3μm are used instead of continuous sputtering by an ion beam. The ions are sputtered in brief burst for a pulse width of an order of nanoseconds. The time taken to reach the detector for these ions is measured and is linked with the kinetic and potential energies and is given as:

\[ qV = \frac{mv^2}{2} \quad 2.29 \]

where \( v \) is the ion velocity and is equal to
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\[ v = \frac{L}{t} \]  \hspace{1cm} 2.30

where \( L \) is the distance from the sample to the detector and \( t \) is the transit time for the ion beam.

Substituting the velocity, the equation then becomes

\[ \frac{m}{q} = \frac{2t^2}{L^2} \]  \hspace{1cm} 2.31

The sensitivity of the system can be enhanced by ionizing the neutral atoms either by laser or by using an electron gas and then the ions are analyzed in a time-of-flight mass spectrometer.

**Advantages**

- The method is very sensitive and has the ability to detect many elements simultaneously.
- SIMS measurement provides a dopant distribution.

**Drawbacks**

- A reference sample with known profile properties is required to verify the measurement.
- SIMS measurement is not suitable for an electrically active profile.
- Equipment is very expensive.
3 Experimental Setup

The stripping Hall machine HL5900+, which is fully automatic measuring system, measured the profiles of semiconductor material. The profiler system has different pots inside it. These pots have different chemical solutions used to test the sample.

3.1 Working of HL5900+

The automatic measuring system shown in figure 3.1 consists of a PC, special measuring card, mechanical part, electronics of the measuring unit, power electronics, magnet poles and 4-point probe equipment.

Figure 3.1 Drawing of Automatic measuring system (AMS) [14]

The menu driven software for the automatic measuring system (AMS) ensures communication with the measuring equipment, commands its single parts and provides the measurement of the required parameters and communication with the operating personnel. The operating personnel, by means of the driven software, enters the necessary data for the measurement process and, at the same time, the results of the measurements form a listing on the display or are placed in a file [14].
Figure 3.2 Physical view of Automatic measuring system (AMS)

In figure 3.2 shown above, from the left side in the red pot is used for etching, the next is the de-ionized water, which is circulated by means of the water pump to wash the sample, the next pot contains Potassium nitrate (KNO₃) for oxide growth and, the last pot contains iso-propanol for drying purposes. To measure a sample, the sequence of single operation for a one cycle is as follow.

Firstly the sample holder is connected to the socket set in the carousel head of the Hall profiler machine, the Hall measurement is initiated for the anodic oxidation - washing - drying - etching- washing - drying - measurement out of the magnetic field (Vander-Pauw) and the measurement in the magnetic field (Hall).

Hall measurement → Etch → Wash → Anodizing → Wash → Dry

This measurement cycle is repeated until the results are obtained.
3.2 Silicon sample wafer processing

Sample preparation processing is performed in the clean room of Mid Sweden University. The process required the diffusion of the n-type and p-type layer onto the silicon wafer. The n-type and p-type silicon wafer with sheet resistivity 11.7ohm/square and 67ohm/square have been selected and they are diffused at 900°C and 950°C, respectively.

3.2.1 Impurities removal process

It is very important to remove the unnecessary particles from the wafer before diffusing boron and phosphorous onto silicon wafer, because impurities have a large impact on the electrical properties of the semiconductor. This process is also known as the standard cleaning process. In this process, the silicon wafer is washed for 10 minutes in a chemical solution, called 7-up solution. The 7-up solution contains 400ml H$_2$SO$_4$ +100ml H$_2$O. The sample is then washed for 3 min in a bubbler pot containing de-ionized water.

In the next step, the wafer is placed into an Imec solution of 3000ml H$_2$O+30ml HF (49% Conc.)+ 3milliliter iso-propanol, after this step the sample is washed again in de-ionised water for 3 minutes. Finally, the wafer is dried in a standard spin-rinse dryer for 400 sec.

3.2.2 Doping

The process of adding impurities to a semiconductor material to activate their electrical conductive properties is called doping. The silicon wafer is doped with phosphorous to make it into an n type and the wafer is doped with boron to make it into a p-type. The process is performed in the furnace for 30 minutes at a temperature of 950 °C for the p-type and at a temperature of 900°C for the n-type, as shown in figure 3.3.
3.2.3 Oxidation

Oxidation is the process in which oxygen molecules interact with different substances, which they may be in contact with, from metal to living tissue and which is more precisely defined as the loss of at least one electron when two or more substances interact [12]. In oxidation, the silicon reacts with oxygen to form silicon dioxide.

In this step, an N₂ +O₂ combustion method is adopted. In this method, pure oxygen is added to the silicon which reacts and thus makes silicon dioxide. The oxidation process for the p-type is performed at 950 °C for 30 minutes, as shown in figure 3.3.

![Figure 3.3](image)

Figure 3.3 (a) shows Doping of n-type, (b) shows Doping and Oxidation of p-type.

3.3 Sample preparation

Good sample preparation is a pivotal factor in order to obtain reproducible and reliable results by using the automated Hall profiler system HL5900. Sample preparation is achieved by following the steps given below.

3.3.1 Sample selection

As thin layers are removed from the sample surface, they are translated into a profile of carrier density $N_S$ and mobility $\mu$ for the sample under observation, therefore, according to the differential Hall technique requirement; the selected sample is 5μm in thickness.
3.3.2 Sample geometry

There are two VDP patterns, namely a clover leaf and Greek cross. However, the clover leaf is recommended for reducing the errors in Hall voltage $V_H$ and thus the decision was taken to use the clover leaf sample pattern for these measurements. The crucial task is to transfer the pattern onto a wafer. A VDP clover leaf pattern of $9 \times 9 \text{mm}^2$ in Corel draw, with the assistance of laser printing, will print the pattern onto a plastic sheet which has glue on one side that can easily stick to the wafer. The printed sheet is fixed onto a wafer and the pattern is filled with a black wax.

When the wax has dried, the plastic sheet is ripped from the wafer and the etching process initiated. The liquid chemicals or etchants are used to remove materials from the wafer instead of the patterns defined by the black wax mask on the silicon wafer. Buffered hydrofluoric acid (BHF) is used to etch away the silicon dioxide layer, which is over the silicon surface. The solution used to etch a silicon wafer area is made up of

$$10 \text{mlHF} + 10 \text{ml water} + 70 \text{mliter } \text{HNO}_3$$

The wafer was placed into this solution for three minute for deep etching after which, it is rinsed and the wafer is then dried in the bubbler spinning-rinse dryer. The deep etching was on the VDP pattern, to isolate the bulk sample from the active layer or to isolate the p'n junction.

![Clover leaf VDP Pattern](image)

Figure 3.4 Clover leaf VDP Pattern
The size of a sample should be a square with dimensions 10 X10mm². Any sample larger than these dimensions is unable to fit into a sampler holder and a sample with a smaller specimen complicates the sample mounting procedure. Therefore, the selected sample with dimensions 10 X10mm² for the Vander Pauw defined pattern of a clover leaf and which can see in figures 3.4, 3.5 is used for this project so as to reduce the errors in $\rho_S$ and $R_{SH}$ due to contact size.

![Silicon Wafer Sample with VDP Pattern](image.png)

Figure 3.5 Silicon Wafer Sample with VDP Pattern

By using the cutter pencil, these samples were cut away and thus large number of samples was achieved. The sample symmetry must remain constant throughout the depth profiling.

### 3.3.3 Sample Mounting

The sample is mounted on the sample holder strip shown in Figure 3.6. The cyano-acrylate glue is used for mounting the sample on the sample holder.

### 3.3.4 Ohmic contacts

The ohmic contacts are made on each corner/edge of the 10 X10mm² sample. The contact size is maintained at as small as a size as possible and is kept away from the centre in order to achieve reproducible results. Gal/In eutectic alloy (25% in the weight) makes a very good ohmic contact for the most common semiconductors. The advantage of Gal/In is that it is liquid at room temperature and can be painted onto the surface without any heat treatment. Two points should also be considered when making ohmic contacts. Firstly, both standard surface cleaning and the oxide removal procedures must be conducted before alloying the contacts. Secondly, spreading of Gal/In on the whole surface of the sample should also be avoided [13].
A thin wire of 0.2mm in diameter, with a length less than 6mm, is soldered so as to make the connection of the sample to the sample holder contact pads. The length should be adjusted so that the wire is as flat as possible but which reaches the Gal/In dot but, does not exceed these Gal/In dots. After this the wires are cut to the correct length, the tip of a wire is bent so that it makes contacts with the Gal/In. A small extra amount of Gal/In is applied in order to cover the whole tips of the wire, as shown fig 3.6.

![Mounted Sample on a Sample Holder along with connected wires and Ohmic Contacts](image)

After making contacts it is necessary to confirm whether or not the contacts are working by means of a multimeter. All contacts were found to be working and also the resistivity of the sample was also checked.

![IV Plot for Good, Bad and non ohmic contacts](image)
3.3.5 Sample Insulation

To profile the sample by means of anodic oxidation and with an oxide strip or electrochemical etching, all conductive paths on the sample holder, apart from the sample top surface, must be electrically isolated from the electrolyte. For this purpose, silicon rubber was used to cover the conductive paths, as shown in fig. 3.8, because this rubber is easy to apply and is, additionally, transparent. The underside of the sample holder was also covered with silicone rubber.

![Figure 3.8 Coated Sample with Silicone Rubber](image)

This completes the sample preparation and it is thus ready for further processing.

3.4 Electrolytes for anodic oxidation of Si

The choice with regards to the anodizing and oxide strip solution depends on the semiconductor to be profiled. The following electrolyte is used to test the sample of silicon in the automatic Hall measurement machine.

<table>
<thead>
<tr>
<th>Material</th>
<th>Anodizing Solution</th>
<th>K-anodize(nm/v)</th>
<th>Strip solution</th>
</tr>
</thead>
<tbody>
<tr>
<td>Si</td>
<td>0.05M KNO$_3$+90%Ethylene Glycol+10% water</td>
<td>0.26</td>
<td>5% HF</td>
</tr>
</tbody>
</table>

Table [3.1] Electrolytes for anodic oxidation and oxide strip

0.05M KNO$_3$ in a mixture of 10% H$_2$O with 90% Ethylene Glycol HOCH$_3$CH$_2$OH
M is molarity and is defined as

**Definition:**
A concentration unit, defined to be the number of moles of solute divided by the number of litres of solution.

To determine the molarity of a solution the relation used.

\[
\text{Molarity} = \frac{\text{Grams}}{(\text{Molecular Weight} \times \text{Volume})}
\]

\[
\text{Grams needed} = \frac{\text{Moles/litre} \times (\text{Grams/mole} \times \text{Litres})}{\text{Volume}}
\]

Grams needed = 0.05 X 101

Therefore, dissolve 5.05g of KNO₃ (MW 101.1) in 100ml of de-ionized water. Pour the 900ml of ethylene glycol (MW 62.07)

The advantage of the solution listed above is that it produces excellent results for a wide range of structures and doping levels and also produces uniform and compositionally reproducible oxides. The other advantages of this electrolyte include its low cost, high purity and good electrolyte solubility. It is recommended that after the test of each sample or, at least every day, all the solutions should be replaced and pots used for these solutions should be cleaned.

### 3.5 Parameters for Anodic Oxidation

There are different parameters required to be set by a user for a controlled removal of a layer at each cycle for the Hall measurement.

#### 3.5.1 Auto-decrement

There are two settings, namely, “YES” and “NO” in the HL5900+ for a change in the resistivity value from one step to another. “YES” will automatically perform the decrement in step size on the basis of the resistivity value, monitored by Hall profiler. However, it is important to mention that the initial value of the step size, while selecting “NO”, will remain constant throughout the measurement.
The step size is the thickness of the layer removed at each step.

### 3.5.2 K-anodize

The voltage drop during the anodization process is due to the conversion of Si into oxide, therefore the K-anodize are the thickness of the oxide, in nanometres, to support a voltage drop of 1 volt. The value of the K-anode for silicon is 0.26nm/volt see fig 3.9.

### 3.5.3 I-anodize

A constant current value is required to support the process of anodic oxidation. The standard value of I-anodize for Si is 3mA for a sample area of 10mm². The value for the anodizing current used during the measurement is 4.8mA for a sample area near to 16mm².

### 3.5.4 V-anodize

It is the required voltage value for the step size and the K-anodize. HL5900+ supports a maximum value of 127.5 volts. The value of the V-anodize used is 80 volts, as shown in fig 3.9.

### 3.5.5 Illumination

The illumination is required for n-type semiconductors to ensure that the anodization process is independent of the carrier type and concentration. It has two settings “ON” and “OFF”. “ON” should be selected for the n-type during the process of anodic oxidation.

### 3.5.6 Contacts

There are different current and voltage paths on both sides of the sample holder, which make contacts with the sample on both sides. In AMS, there are two settings, “EPI ONLY” and “EPI+SUBS”. During this process “EPI ONLY” is used because the substrate is not of a conductive type, whereas “EPI+SUBS” is used when the substrate is conductive.
3.5.7 Hold time

This is the time required for the anodization voltage to be reached and for the current to decays to 30% of the initial current value so as to provide homogenous oxide thickness. The decay time for the current is set after the first measurement by the user and is known as hold time. The hold time set for the measurement is 5 seconds, see fig 3.9.

3.5.8 Repetition

This is the number of repetitions of the complete process of anodizing and oxide strip before the electrical measurement. The default value of repletion of oxidation and oxide striping is 1; it can be change by the user when a large step size is required.

3.5.9 Save Graph

There are two modes “YES” and “NO”. The IV curves for the anodization process will be saved when “YES” mode is selected and vice versa.

![Image of Anodizing Etch parameters]

Figure 3.9 Parameters for AO during Actual Measurement
3.6  **Time Parameters during Measurement Cycle**

The time for each process in the complete anodization process measurement cycle is set by the user.

### 3.6.1 Wash Time

This is the time required to wash away the anodization solution from the sample during measurement in a wash tank. The range for the wash time is 20 to 30 seconds but, in this case, 40 seconds was used in order to provide good results.

### 3.6.2 Strip Time

The time required for removing or stripping the oxide layer after anodization is known as the strip time. It depends on the nature of the chemical used; for 5% diluted HF 20 to 30 seconds are sufficient to strip the oxide layer up to 30 nm. 40 seconds was used in this case for the chemical etching of the oxide shown in fig 3.10.

### 3.6.3 Wash Time

The time spent to wash away the HF solution from the sample after etching. The range for the wash time is 20 to 30 seconds but 40 seconds was used here so as to ensure that complete etching solution is washed, see fig 3.10.

### 3.6.4 Dry Time

Before the electrical measurements are made it is important to dry the sample after the last wash. The time for the drying process is dependent on the nitrogen pressure. At a nitrogen pressure of 2psi or 50 bar, the minimum time required to dry a sample is 30 seconds. However, in this case, 50 seconds was used for the complete drying of a sample before proceeding to the Hall measurement, see fig 3.10.
3.7 Error Consideration

It is important to consider a few errors in relation to any profile measurement. The most important, which must be considered, are explained below.

- The Hall scattering factor “r” or symmetry factor “q” is not always unity. The value for the Hall scattering factor varies with the carrier concentration, the precise relationship depending on the semiconductor material being measured.
- The errors in the sheet Hall coefficient and sheet conductivity give rise to a scatter in the profiles.
- There is a depletion of carriers at the surface of the sample. This effect will clearly depend on the material under consideration and the doping level.
- The depth resolution is directly limited by the accuracy to which the sheet values can be measured.
- The main problem faced during measurement is the failure to reach the anodizing voltage and leakage current as shown in fig 3.11. The problem was due to an incomplete insulation of the copper/metalized tracks and to areas of the sample or sample
holder being exposed in the anodization solution. A condensed coating of the silicone rubber was used to overcome this problem.

![Graph](image)

**Figure 3.11 IV Error faced during Measurement**

- An excessively long time (>3 minutes) required in order to attain the anodizing voltage is due to the very low value of the anodizing current as shown in fig 3.11. The value of the anodizing current increased before proceeding with further measurements.

- A very small step size caused excessive scatter in the carrier concentration and the mobility profile, as shown in fig 3.12, which leads to small differences in successive sheet resistivity $\rho_S$ and Hall coefficients $R_{HS}$. The reason was the electrical noise in the measurement and subsequent numerical differentiation of values introduces large noise into the concentration $N_S$ and mobility $\mu_H$. This problem was removed by initially choosing a relatively large step. Another method is to continue the measurements using the initial setting of the step size until the end of the measurement is reached and then to use the Hall process utility to recalculate the
concentration and mobility values by means of a moving average so as to obtain the smooth depth profile.

![Graph showing scattered concentration error during measurement.](image)

**Figure 3.12 Scattered Concentration Error faced during Measurement**
4 Results

The n-type wafer was phosphorous doped and had an orientation $<100>$ with a substrate concentration of $3 \times 10^{14}$. The doping was conducted at 900°C for 30 minutes, while the p-type sample was boron doped, with an orientation $<100>$ and with a substrate concentration of $8 \times 10^{14}$. The wafer was annealed and oxidized at 950°C for 30 minutes. The bulk resistivity is 17-25Ωcm for both the n and p types. However, the complete sample thickness is 5.25μm.

The measurement is conducted in four steps. The first step is to measure the thickness of the oxide layer by means of an optical method; the second step includes the step measurement of the removed silicon by the AFM, while in the third step the characterization of the n-type and p-type doping profile is performed. The last step involves a comparison being made between the simulated values and the theoretical calculations with actual measurements.

![Figure 4.1 Flow Chart for Measurement Steps](image-url)
4.1 Oxide Thickness Measurement

The measurement of the thickness of the oxide layer is performed on the prepared samples by using an optical reflector-meter. The software Ava-soft translates the white light pattern through mathematical functions into an optical thickness calculation with respect to the reference sample. The software has the ability to measure the layers of 10 nm to 50um with a resolution of 1 nm. The oxide thickness grown on silicon is linear and is accurate with respect to the voltage based on the theoretical calculation in chapter 2. The oxide thickness measured at 40 volts is shown in fig 4.2.

In fig 4.3 the oxide thickness is measured at 80 volts. The thickness of the oxide layer is double that grown at 40 volts and is 4 times that generated at 20 volts. From the theoretical calculation it is found that the oxide thickness at 80 volts is 24 nanometres, while during the real measurement the oxide thickness is found to be 23.2nm. See fig 4.3
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Figure 4.3 Thickness Measurement of SiO$_2$ at 80 volts

4.2 Step Measurement through Atomic Force Microscopy (AFM)

Atomic force microscopy or scanning force microscopy (SFM) is a very high-resolution type of scanning probe microscopy. The step measurement is carried out after 20 repetitions of anodization and etching at 80 volts. It is extremely difficult and it is crucial to see the exact step, however, the step size or oxide growth after 20 cycles should be 480nm and smooth, whereas the step measured is 408nm in depth and slightly sharp but is satisfactory with regards to the number of anodization cycles for the measurement. The y-axis in fig 4.4 shows the step while the x-axis describes the area scanned by a microscopic camera.
Figure 4.4 Step Measurement using AFM

Figure 4.5 describes the view of the semiconductor at the nano scale during the step measurement. The area of 70μm² of the sample is scanned, therefore, it is crucial to see the exact step and oxide, and however, the white spots are clearly visible at the centre, which describes a clear change from the rest of the sample. The black area in fig 4.5 shows the concentration of silicon in the sample.

Figure 4.5 Scanned Semiconductor at nano-scale through AFM during the step measurement
4.3 Measurements for n-type Sample

The concentration profiles, $N_s$ were measured. The measurement was carried out on the sample resistivity $\rho=11.7\,\Omega/\text{sq}$ and the sample is phosphorous doped in silicon at $900^\circ\text{C}$ for 30 minutes, see appendix.

4.4 Simulation Curve

The simulated/ theoretical concentration profile as a function of depth is shown in fig 4.6. The concentration starts from $10^{20}$, see appendix, and drop after each anodization step. The y-axis shows the concentration in per centimetre cube and the x-axis describes the depth of the measuring profile at each anodization process. Initially, the concentration is very high and at the tail it is low, therefore, the step size during the actual measurement should be in order small to analyze and measure the exact change in the profile. Fig 4.6 is the complete concentration profile $N_s$ for the n-type with respect to the depth of a sample.

![Complete Theoretical Concentration Profile for n-type](image)

Figure 4.6 Complete Theoretical Concentration Profile for n-type

The fig 4.7 describes the clear drop of concentration as a function of depth. It starts from $10^{20}$ and drops after each measurement. At $0.4\mu\text{m}$ the concentration drops to $10^{19}$ from $10^{20}$, at a depth of $0.8\mu\text{m}$ the value of the concentration is $2\times10^{18}$ and at $1\mu\text{m}$ the value is $2\times10^{17}$ and so on, see fig 4.7.
4.4.1 Actual Measurement

It is important to verify the symmetry factor, correction factor, ohmic contacts and sheet resistance of the real sample for the actual measurements. The symmetry factor should not be greater than 1.2 for good ohmic contacts and the correction factor should be 1 for good samples. It was found in actual the measurement that the value of the symmetry factor is 1.02 and the correction factor is 1.00.

<table>
<thead>
<tr>
<th>Meas</th>
<th>+++</th>
<th>Ym</th>
<th>Sym Factor</th>
<th>Sheet R</th>
</tr>
</thead>
<tbody>
<tr>
<td>43</td>
<td>1.01e-05</td>
<td>-6.73e-06</td>
<td>1.02</td>
<td>10.9</td>
</tr>
<tr>
<td>41</td>
<td>9.25e-06</td>
<td>-7.25e-06</td>
<td>1.02</td>
<td>18.9</td>
</tr>
<tr>
<td>21</td>
<td>1.02e-05</td>
<td>-6.70e-06</td>
<td>1.03</td>
<td>18.9</td>
</tr>
<tr>
<td>23</td>
<td>1.06e-05</td>
<td>-5.84e-06</td>
<td>1.02</td>
<td>18.8</td>
</tr>
</tbody>
</table>

Figure 4.8 Verification of factors for actual sample before measurement
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The y-axis on the left hand side describes the concentration \( N_s \), whereas the y-axis on the right hand side shows the mobility \( \mu \) and the x-axis shows the depth in micrometres in fig 4.9.

The actual measured concentration \( N_s \) of the n-type is \( 8 \times 10^{20} \) and the mobility is \( 1.1 \times 10^1 \) before any anodization process. When the anodization process is initiated along with the etching the concentration is \( 2 \times 10^{20} \) and the mobility is \( 6 \times 10^1 \) at its first step. The concentration drops continuously at each step, while the mobility is the reciprocal of it, in differential Hall measurement. At the depth of 1.1 \( \mu m \) the concentration is almost \( 4 \times 10^{17} \) whereas the mobility is \( 5 \times 10^2 \). The sample was deeply etched, therefore, in result of the bulk sample, a depth of 1.11 \( \mu m \) shows the complete removal of the active layer. The value of the concentration \( N_s \) or \( N \) is \( 1.17 \times 10^{17} \) and mobility is \( \mu \) is \( 1.91 \times 10^3 \), shown in fig 4.9.

![Figure 4.9 Actual Concentration and Mobility Profile](image)

The yellow dot and red mark in fig 4.9 shows that the measured n-type sample is completely etched and has reached the bulk sample or non-active layer, at this point, Hall profiler stopped the measurement of the profile under consideration.
In fig 4.10 the y-axis on the left hand side represents the resistivity $\rho$, whereas the y-axis on the right hand side shows the Hall coefficient $R_H$ and the x-axis shows the depth in micrometres.

The resistivity starts from $8 \times 10^{-4}$ approximately and is increased gradually with each measurement step and reached to $2.97 \times 10^{-2}$ at a depth of $1.1 \mu m$ and this is represented in $\Omega$-cm. However, the Hall coefficient starts initially at approximately, $8 \times 10^{-2}$ and reached $5.66 \times 10^{-1}$ at same the depth and is represented in $Cm^3/C$, see fig 4.10.

Figure 4.10 Representation of Resistivity and Hall coefficient in Differential Measurement for n-type

AMS also measured the sheet resistance ($R_s$ or $\rho_S$) and the Hall sheet coefficient ($R_{SH}$) automatically and the representation of the sheet resistance and the Hall sheet coefficient is shown in fig 4.11. The y-axis on the left hand side represents the sheet resistance, whereas, the y-axis on the right hand side shows the Hall sheet coefficient and the x-axis shows the depth in micrometres in fig 4.11.

The sheet resistance started from $2 \times 10^{-1}$ and increases up to $4.71 \times 10^{-3}$ while the Hall sheet coefficient starts from approximately $7 \times 10^{-2}$ and reached $4.29 \times 10^{-6}$ at the depth of $1.121 \mu m$ at the depth of 1.121 micrometres see fig 4.11.
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Figure 4.11 Representation of Sheet Resistance and Hall Sheet Coefficient Measurement for n-type

The change in concentration/cm² and mobility/cm² of the profiles under observation with respect to the sheet resistance is shown in fig 4.12. The concentration/cm² is represented on the left side of the y-axis, mobility/cm² is represented on right side whereas the depth is on the x-axis.

The concentration is $10^{16}$ initially and drops according to the parameters defined for the anodization process. At a depth of 1.121μm, the concentration/cm² is $1.45 \times 10^{12}$ the mobility/cm² starts from approximately $7 \times 10^3$ and is increased to $9.11 \times 10^2$ at a depth of 1.121μm, see fig 4.12.

Figure 4.12 Representation of Concentration and Mobility per cm²
When the active layer is completely removed all the factors remain same, the exception being the symmetry factor. Symmetry remains constant i-e 1.02 throughout the whole anodization process but is changed to 52.21 when the measurement reached the bulk sample as shown in fig 4.13.

![Figure 4.13 Comparison of Symmetry Factor before and after Measurement](image)

### 4.5 Measurements for P-type Sample

The concentration profiles $N_s$ were measured. The measurement is conducted for sample resistivity $\rho=67\Omega/sq$ and the sample is boron doped in silicon at 950°C for 30 minutes, see appendix.

### 4.6 Simulation Curve for P-type

The simulated/ theoretical concentration profile, as a function of depth, of the p-type is shown in fig 4.14. The concentration starts from $10^{19}$ and drops after each anodization step, however, the small ripple at the first step in the simulation is due to the presence of oxide on the sample. The y-axis shows the concentration per centimetre cube and the x-axis describes the depth of the measuring profile at each anodization process. Initially, the concentration is very high and at the tail it is low therefore step size during the actual measurement should be small in order to analyse and measure the exact change in the profile. Fig 4.14 is the complete concentration profile $N_s$ for the p-type with respect to the depth of a sample.
The fig 4.15 describes the clear drop of concentration as a function of depth. It starts from $10^{19}$ and drops after each measurement. At 0.4μm the concentration drops to $10^{18}$ from $10^{19}$, at the depth of 0.6μm the value of the concentration is $8 \times 10^{16}$ and at 0.8μm the value is $4 \times 10^{14}$, see fig 4.15.
4.6.1 Actual Measurement

It is important to verify the symmetry factor, correction factor, ohmic contacts and sheet resistance of the real sample for the actual measurement. The symmetry factor should not be greater than 1.2 for good ohmic contacts and the correction factor should be 1 for good samples. It was found that in the actual measurement the value of the symmetry factor is 1.06 and the correction factor is 1.00, see fig 4.16

<table>
<thead>
<tr>
<th>Meas</th>
<th>Sym</th>
<th>Vm</th>
<th>Resistivity</th>
</tr>
</thead>
<tbody>
<tr>
<td>43</td>
<td>1.04e-04</td>
<td>-1.33e-04</td>
<td>73.8</td>
</tr>
<tr>
<td>41</td>
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<td>-1.60e-04</td>
<td>73.8</td>
</tr>
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<td>21</td>
<td>1.49e-04</td>
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<tr>
<td>23</td>
<td>1.76e-04</td>
<td>-1.58e-04</td>
<td>73.8</td>
</tr>
</tbody>
</table>

Figure 4.16 Verification of factors for actual sample before measurement

The y-axis on the left hand side describes the concentration Ns, whereas the y-axis on the right hand side shows the mobility \( \mu_X \) and the x-axis shows the depth in micrometres in fig 4.17.

The actual measured concentration Ns of the p-type is \( 4 \times 10^{19} \) and the mobility \( 10 \times 10^1 \) before any anodization process. When the anodization process initiated, together with etching, the concentration was increased to \( 6 \times 10^{19} \) and the mobility to \( 500 \times 10^1 \) because of the presence of some oxide on the sample. The concentration drops continuously after the depth of 0.5\( \mu \)m while the mobility is increased during differential Hall measurement. At the depth of 0.8\( \mu \)m the concentration is almost \( 4 \times 10^{18} \) whereas the mobility is \( 9 \times 10^1 \). The sample was deeply etched, therefore, in result of the bulk sample, a depth of 0.815\( \mu \)m shows the complete removal of the active layer, where the value of the concentration Ns drops to \( 1.85 \times 10^{17} \) and the mobility \( \mu_X \) is increased to \( 1.13 \times 10^2 \), as shown in fig 4.17.
The blue dot and red mark at the depth of 0.815μm in fig 4.17 shows that the measured p-type sample is completely etched and has reached the bulk sample or non-active layer, at this point the Hall profiler stopped the measurement of the profile under consideration.

In fig 4.18 the y-axis on the left hand side represents the resistivity, whereas the y-axis on right hand side shows the Hall coefficient $R_H$ and the x-axis shows the depth in micrometres.

The resistivity starts from approximately $2 \times 10^2$, drops gradually for a few measurements step and reaches $5 \times 10^{-3}$ at a depth of 0.5μm due to the oxide presence. The resistivity is represented in Ω-cm and after a depth of 0.5μm the resistivity starts increasing and reaches $2.99 \times 10^{-1}$. However, the Hall coefficient is initially, approximately $6 \times 10^{-1}$ and drops to $1 \times 10^1$ at a depth of 0.5μm, while after 0.5μm, the value of the Hall coefficient increases and reaches $3.36 \times 10^4$ at 0.815 μm see fig 4.18. The Hall coefficient is represented in Cm$^3$/C.
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Figure 4.18 Representation of Resistivity and Hall coefficient in Differential Measurement for p-type

The AMS also measured the sheet resistance ($R_s$ or $\rho_s$) and the Hall sheet coefficient ($R_{sh}$) automatically. The representation of sheet resistance and hall sheet coefficient is shown in fig 4.19. The y-axis on the left hand side represents the sheet resistance whereas the y-axis on the right hand side shows the Hall sheet coefficient and the x-axis shows the depth in micrometre in fig 4.19.

The sheet resistance started from $9 \times 10^{-1}$ and increases up to $3.97 \times 10^{-5}$ while the Hall sheet coefficient starts from approximately, $5 \times 10^{-3}$ and reaches $1.75 \times 10^{-7}$ at the depth of 0.816 micrometres, see fig 4.19.

Figure 4.19 Representation of Sheet Resistance and Hall Sheet Coefficient Measurement for p-type
The change in concentration/cm² and mobility/cm² of the profiles under observation with respect to the sheet resistance is shown in fig 4.20. The concentration/cm² is represented on the left side of the y-axis, mobility/cm² is represented on the right side whereas the depth is on the x-axis.

The concentration is $10^{15}$, initially and drops according to the parameters defined for the anodization process, and at the depth of 0.6μm the concentration/cm² is $3 \times 10^{14}$, the mobility/cm² starts from approximately $5 \times 10^1$ and increases to $1.47 \times 10^2$ at the depth of 0.812μm, whereas, at the same depth, the concentration dropped rapidly from $3 \times 10^{12}$ to $1.24 \times 10^{11}$ with respect to the sheet resistance and the defined area, see fig 4.20.

Figure 4.20 Representation of Concentration and Mobility per cm²
At the depth of 0.815μm, the active p-type layer is removed from the sample and the measurement reached the bulk level, the blue dot and mark indicates the bulk sample during the measurement. At the depth of 0.816μm it is confirmed that the active layer is completely removed due to the deep etching of the sample during the sample preparation by using the Vander Pauw pattern transfer. At the depth of 0.816μm the concentration shown in fig 4.21 represents the measurement of the bulk sample or material, the value of concentration is 3.56X10^{11} and the value for the mobility is 4.41X10^{1} see fig 4.21. In AMS there are two setting “EPI ONLY” and “EPI+SUBS”. “EPI ONLY” was used during the measurement because the substrate is not of a conductive type. “EPI+SUBS” is used when the substrate is conductive and therefore the Hall profiler ceases any further measurement on the bulk sample as the p-type layer is completely removed.

![Figure 4.21 Representation of Concentration and Mobility per cm²](image)

When the active layer is completely removed, all the factors remain the same with the exception of the symmetry factor. Symmetry remains constant i-e 1.06 throughout the whole anodization process, but, changed suddenly to 6.72 when the measurement reached the bulk sample, as shown in fig 4.22.
4.7 Comparison of Actual Measurement with Simulated Values

The simulation shows a typical doping dependence, characteristic for n and p-profiles in silicon. A good agreement is achieved comparing simulation and measured doping profiles. The curve of concentration with respect to depth, in the case of the n-type, as shown in figure 4.7, obtained from simulation is the same as that for the practical experiment as shown in figure 4.9. While in the case of the p-type, the depth obtained from the simulation, as shown in figure 4.15, is nearly the same as the practical results shown in figure 4.17.
5 Conclusions/ Future Work

Theoretically and practically it has been proven that the oxide thickness grown on silicon is accurate with respect to the voltage. The oxide growth is 240Å or 24nm for the anodization voltage of 80 volts independent of the anodization current of 2~8mA. The simulation shows a typical doping dependence, characteristic for n and p-profiles in silicon. The doping profiles as a function of depth, achieved during the actual measurements are accurate and are in accordance with the simulation results.

It is also concluded from the measurements that the concentration of the electrolyte has an influence on the depth profiling and step measurement. Time parameters for the measurement cycle and the parameters of anodic oxidation also affect the results of the sample under measurement. A good resolution and smooth curve is widely dependent on good sample preparation, especially with regards to the ohmic contacts and sample insulation.

The presented work is a part of on-going research for the development of detectors. In the future, applications such as thermal, position sensitive detectors and solar cells can be manufactured on the basis of this work.

Gaussian, linear and step profiles at different temperatures for different semiconductors including silicon-arsenic, gallium-arsenide and silicon-antimony are required to be measured in order to compare and verify the value of the K-anodize for the AMS system.
Hall Measurement for Characterization of Doping Profile in Semiconductor
Habib Ullah Khan Jadoon

References


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References


Solid Solubility

Solid Solubility of Impurities in Silicon with Respect to temperature

Solid Solubility of Impurities in Silicon with Respect to temperature [16]
Solubility Limit and Electrically Active Region over Temperature

![Graph showing solubility limit and electrically active region](image)

Solubility Limit and Electrically Active Region of Boron and Phosphorous as a function of Temperature [16]
ERFC and Gaussian Profile

Gaussian and erfc profile Impurities concentrations/surface concentrations with respect to depth [16]