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Student Signature: Nikhil Jain

This work and its defense approved by:
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Process development for ICP patterning of through-wafer periodic micro-pores in silicon wafers

Nikhil Jain

Delhi College of Engineering, University of Delhi, New Delhi, India

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Cincinnati, OH

Thesis Committee –

Dr. Joseph Boyd – Committee Chair

Dr. Robert A. Jones – Thesis Advisor

Dr. Mark Schulz – Committee member
Abstract

Silicon has been the material of choice in the semiconductor industry since the last 50 years. Silicon etching forms an essential step in the fabrication of micro-electro-mechanical-systems, more commonly known as MEMS devices. Advancements in silicon etching have made possible the era of integrated on-chip sensors. An increase in the feature density is mirrored in the processing power, memory capacity and sensing ability of devices. Through-wafer etching has applications in forming vias for circuits as well as for functioning as interconnects. There is a pressing need for anisotropy in these features. Having a high aspect-ratio is the single most-important requirement for these applications. This prevents us from opting for conventional methods of wet etching which are dependent on the crystal orientation of the wafer and give a big taper. This thesis introduces a technique of fabricating a patterned array of through-wafer micro-holes in silicon wafers using the dry etching technology. Inductively coupled plasma (ICP) etching has emerged as the preferred way of making deep/through-wafer features in silicon wafers as it provides high aspect ratios and much better control over the etch profile as compared to the conventional Reactive ion etching method. A time-multiplexed switched plasma approach is explored for creating vertical sidewall features in the wafers by developing an etch-deposition process. The thesis delves into the intricacies of ICP etching. In the end, possible applications are explored and a particular application involving growth of carbon nanotubes (CNTs) in these holes for making an e-beam device is explained.
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Introduction ......................................................................................................................... 1

1.1 The importance of deep silicon etching ................................................................. 1

1.2 Methods of etching ............................................................................................... 3

1.2.1 Wet etching ......................................................................................................... 3

1.2.2 Reactive Ion Etching (RIE) ............................................................................. 4

1.2.3 Deep Reactive Ion Etching (DRIE) ................................................................. 5

1.3.2 A brief history of plasma ..................................................................................... 9

1.3.3 Plasma physics ....................................................................................................... 11

1.3.3.1 Condition of quasi-neutrality......................................................................... 12

1.3.3.2 Debye Length and plasma sheath ................................................................. 13

1.3.3.3 Sheath formation .............................................................................................. 14

1.3.3.4 Strongly and weakly coupled plasmas and plasma parameter ..................... 15

1.3.3.5 Gas Discharge Theory .................................................................................. 17

1.3.3.6 Plasma discharge balance .............................................................................. 19

1.3.4 Sources of plasma ............................................................................................... 20

1.3.4.1 RIE discharge reactors .................................................................................. 20

1.3.4.2 Electron cyclotron resonance (ECR) Sources ............................................. 23

1.3.4.3 Inductively Coupled Plasma (ICP) Sources ............................................... 25

1.3.5 Plasma chemistry ................................................................................................. 26

1.3.5.1 Plasma etching mechanism .......................................................................... 27

1.3.5.2 Spontaneous surface etching and deposition .............................................. 28

1.3.5.3 Ion bombardment and sputtering ............................................................... 30

1.3.5.4 Ion-enhanced chemical etching .................................................................... 30

1.4 The BOSCH Process and this project ................................................................. 31
Equipments used in ICP etching

2.1 Process flow diagram
2.2 The Versaline ICP etch system
2.3 Salient features of ICP etching
   2.3.1 Uniformity over the critical dimension
   2.3.2 Anisotropy
   2.3.3 Selectivity
   2.3.4 Etch rate
   2.3.5 Control of etch profile
   2.3.6 Wafer and mask damage
   2.3.7 Sidewall passivation
   2.3.8 Residue removal
   2.3.9 Unwanted features and corrosion
2.4 Characterization tools
   2.4.1 Profilometry
   2.4.2 Scanning electron microscopy (SEM)
2.5 Summary

Process Development

3.1 Overview
3.2 Wafer preparation
   3.2.1 Material Selection
   3.2.2 Wafer Cleaning
   3.2.3 Metal (Aluminum) coating
   3.2.4 Lithography
   3.2.4.1 Resist coating
   3.2.4.2 Patterning and Exposure
   3.2.4.3 Developing the resist and hard bake
3.2.5 Metal removal (metal etch) ........................................................................... 49
3.2.6 Resist Stripping ............................................................................................... 50
3.3 Plasma Ignition .................................................................................................. 50
  3.3.1 Effects of pressure variation on plasma ignition ............................................. 51
  3.3.2 Effects of ICP and bias power on plasma ignition ......................................... 53
3.4 Effects of parameter variations on the plasma .................................................. 58
  3.4.1 Effects of variation in gas flow rates on plasma ............................................. 58
    3.4.1.1 Effects of variation in SF\textsubscript{6} flow rate on plasma intensity .............. 58
    3.4.1.2 Effects of variation in SF\textsubscript{6} flow rate on etch rate .......................... 59
    3.4.1.3 Effects of variation in argon flow rate on plasma intensity ....................... 59
    3.4.1.4 Effects of variation in argon flow rate on etch rate ................................. 60
    3.4.1.5 Effects of variation in O\textsubscript{2} flow rate on plasma intensity .................. 61
    3.4.1.6 Effects of variation in oxygen flow rate on etch rate ............................... 61
  3.4.2 Effects of base holding wafer on etch rate .................................................. 63
    3.4.2.1 Loading effect .......................................................................................... 63
  3.4.3 Effects of temperature variations on the plasma .......................................... 64
3.5 Plasma with various gases ................................................................................. 64
3.6 Time-multiplexed switched-plasma approach .................................................. 66
  3.6.1 CF\textsubscript{4} and SF\textsubscript{6} switching ................................................................. 66
  3.6.2 SF\textsubscript{6} and O\textsubscript{2} switching ................................................................. 68
  3.6.3 CF\textsubscript{4} and CHF\textsubscript{3} switching ............................................................ 69
  3.6.4 SF\textsubscript{6} and CHF\textsubscript{3} switching ............................................................ 70

Conclusions ........................................................................................................... 74

4.1 Conclusions ....................................................................................................... 74
4.2 Suggestions for future work and possible applications ..................................... 76

References ............................................................................................................ 77
List of Figures

Figure 1 (a) Design illustration; (b) physical dimensions and; (c) SEM image of planar piezoresistive accelerometer [10] ..................................................................................................................2

Figure 2 Profile and surface of features obtained with wet etching [24] .........................................................................................3

Figure 3 Optical micrographs of RIE profiles 50 µm deep; (a) large sidewall taper; (b) Controlled taper with process optimization [30] ........................................................................................................5

Figure 4 Micro gas turbine showing use of ICP etching [33] ........................................................................................................6

Figure 5 Phase Fresnel Lens [34] ............................................................................................................................7

Figure 6 Plasma sheath formation ..........................................................................................................................12

Figure 7 Maxwell distribution ..........................................................................................................................13

Figure 8 Sheath potential in plasma ..................................................................................................................14

Figure 9 Parallel plate RIE discharge reactor [37] .................................................................................................21

Figure 10 Illustrating the change in plasma potential when electrode B is biased; (a) No bias; (b) Positive bias; (c) Negative bias ........................................................................................................22

Figure 11 ECR source with a large area [37] ........................................................................................................24

Figure 12 ICP chamber cross section .................................................................................................................26

Figure 13 Boundary between polymer growth and substrate etching [46] .................................................................29

Figure 14 Combined ion and neutral bombardment gives a high etch rate [46] .......................................................31

Figure 15 Process flow diagram describing the experimental steps ........................................................................33

Figure 16 Versaline ICP system at ERC Cleanroom .................................................................................................35

Figure 17 ICP Graphic User Interface ................................................................................................................36

Figure 18 Profile defects; (a) Bowing, (b) Faceting, (c) Notching, (d) Trenching ................................................39
Figure 19 Stylus of a profilometer, tip angle 60 degrees, tip curvature 5 µm, axes units are in µm
........................................................................................................................................42

Figure 20 Figure showing SEM imaging principles ..................................................................43

Figure 21 Karl Suss MJB3 Aligner ...........................................................................................48

Figure 22 SEM images explaining the effect of pressure variations on anisotropy; (a) 11.2 mTorr; (b) 18.2 mTorr; (c) 50.4mTorr [35] .................................................................51

Figure 23 Plasma intensity vs. pressure ....................................................................................52

Figure 24 Effects of pressure variation in plasma [55] ............................................................53

Figure 25 Plasma intensity vs SF₆ flow rate ...........................................................................58

Figure 26 SF₆ flow rate vs. Etch rate ......................................................................................59

Figure 27 Plasma intensity vs Argon flow rate .....................................................................60

Figure 28 Argon flow rate vs. etch rate ................................................................................60

Figure 29 Oxygen flow rate vs. plasma intensity ..................................................................61

Figure 30 Reactions in the plasma involving oxygen .................................................................62

Figure 31 Oxygen flow rate vs etch rate ..............................................................................62

Figure 32 Plot of etch rate of CF₄ and SF₆ ............................................................................65

Figure 33 Effects of varying O₂ flow rate on etch rate in the SF₆-O₂ switching experiment ....69

Figure 34 SEM image of the final etch features showing near vertical etch profile ...............72

Figure 35 Backside addressable patterned CNT arrays ..........................................................76
List of Tables

Table 1 Characteristic values for typical plasmas [42] .......................................................... 17
Table 2 Etch Parameters ............................................................................................................. 37
Table 3 Parameter effects on process ......................................................................................... 37
Table 4 Properties of silicon wafers used in the experiments ...................................................... 46
Table 5 Bakerclean Process ....................................................................................................... 47
Table 6 Metal Deposition ........................................................................................................... 47
Table 7 Photoresist coating ...................................................................................................... 48
Table 8 Resist developing process ........................................................................................... 49
Table 9 Aluminum etching ........................................................................................................ 49
Table 10 ICP etch basic recipe .................................................................................................. 50
Table 11 Effect of reflected power on plasma ignition ............................................................... 54
Table 12 Process conditions for basic ICP etch ....................................................................... 55
Table 13 Gas stabilization step ................................................................................................ 56
Table 14 Plasma ignition steps. The bias and ICP power were incremented in each step by 50W and 200W respectively after Step 3 ................................................................. 56
Table 15 Standard etch recipe .................................................................................................. 57
Table 16 Loading effect in ICP ................................................................................................ 63
Table 17 Switching between CF₄ and SF₆ plasma ................................................................. 67
Table 18 Modified recipe .......................................................................................................... 68
Table 19 SF₆ and O₂ plasma switching ...................................................................................... 69
Table 20 CF₄ - CHF₃ plasma switching; Etch-Deposition process ............................................ 70
Table 21 SF₆-CHF₃ switching; Etch - Dep process .................................................................71
Table 22 Final etch recipe ..................................................................................................72
Chapter 1

Introduction

1.1 The importance of deep silicon etching

Deep etching is a very significant process in silicon processing for fabrication of microelectronic and MEMS-based devices. It offers the advantage of creating vertical sidewall and through-wafer features which can be used for making various devices or device components. The fields of photonics [1], chemical sensors [2], electrowetting [3], and high-frequency electronic devices [4] have taken a major leap since the development of deep etch technology [5]. While the semiconductor industry works with structuring layers of semiconductor to fabricate micrometer scale structures, mechanical structures often require processing of thicker layers and at times etching through the whole 500 µm thick wafer. Deep etching to make high aspect-ratio structures is important for front-end as well as back-end applications. Front end applications involve MEMS structures where deep trenches can be used as optical fiber alignment devices in micro mirrors. Such devices include optical cross-connects, variable optical attenuators and fourier transform spectrometers [6]. Another front-end application is the use of trenches as microfluidic channels in lab-on-a-chip (LoC) devices [7]. Back-end applications involve use of through-wafer interconnects (TWIs) for chip scale packaging to ensure more device density and better electrical performance [8]. The first commercial application which provided the impetus to the field of MEMS was the accelerometer; it used deep silicon etching for fabrication [9]. A typical
accelerometer device is shown in Figure 1. The design illustrates the need for deep etching in the most basic MEMS devices.

![Figure 1](image)

Figure 1 (a) Design illustration; (b) physical dimensions and; (c) SEM image of planar piezoresistive accelerometer [10]

The accelerometer detects a rapid deceleration and deploys an airbag when necessary. The proof mass is attached to a slender flexural element. This flexural element gets deflected whenever there is acceleration in the plane of the sensor. It has a piezoresistive material that changes resistance with change in strain due to deflection. The physical dimensions of the accelerometer determine the performance specifications of the sensor. Minimizing the flexural width $w$ and maximizing the radial length $r$, results in high sensor sensitivity (voltage per unit acceleration). The length of the flexure $l$, determines the bandwidth of the accelerometer [10].

The field of MEMS has also grown to include various areas of research with applications in biotechnology (lab-on-a-chip) [11], optoelectronics (waveguides and switches) [12], power generation (micro-engines) [13], and many more. The requirement of through-wafer features in these devices is pushing the research in the field of deep silicon etching. The goal of this project is to develop a standardized process for ICP patterning of through-wafer (~160 µm) periodic
pores in silicon wafers. The following review section illustrates how various ways of deep etching were adopted based on application requirements.

1.2 Methods of etching

Etching is a process which involves chemical or mechanical removal of layers of a substrate from the surface. This section describes the main methods of etching which have been developed to make features in a substrate.

1.2.1 Wet etching

Etching of silicon wafers with liquid chemicals like EDP, TMAH, NaOH and KOH has been an important silicon processing step for a number of years [14]-[23]. Deep sloped structures in silicon can be made using wet KOH as the etchant. The taper angle obtained using such an approach is 54.7 degrees. This becomes a severe limitation because etching at 54.7 degrees makes one side of the feature too wide to be of any use.

Hence, aspect-ratio is very limited with wet KOH etching. Also, silicon 1-0-0 etches very differently compared to silicon 1-1-0. This crystal orientation dependency of wet etching is a disadvantage. Wet etching rate is governed by the principle of diffusion which requires etchant species to diffuse to the surface and the product species to diffuse away from the surface. As the

Figure 2 Profile and surface of features obtained with wet etching [24]
etch keeps getting deeper and deeper, the diffusion rate keeps diminishing resulting in reduction in etch rate. Low selectivity is a problem with wet etching. Wet etchants like KOH are very aggressive and attack the hard mask material as well as the substrate, the former at a slower rate. This puts a limitation on the minimum thickness of the hard mask material. Another disadvantage with wet etching is chemical waste disposal as its liquid by-products are hazardous. Special waste bottles are kept with the wet benches which store these etch by-products until they are specially disposed off. Also, when the application is semiconductor industry based, the use of wet etching on the production line is very limited. This confines the use of wet etching to research laboratories [25]. All these limitations of the wet etching technique have forced researchers to explore other modes of anisotropic etching.

### 1.2.2 Reactive Ion Etching (RIE)

Use of plasma for dry etching has been a subject of study not only for silicon devices but also for other materials [26]. RIE gets its name from the formation of reactive species in plasma by dissociation of etchant gas molecules. These reactive species then adsorb to the substrate surface and react to form volatile compounds which get desorbed and are pumped away subsequently. Vias through GaAs have been reported using chlorine based chemistries [27]. While such processes work very well for GaAs, getting a substantial etch rate in silicon has always been a problem with chlorine even though HCl has been shown to create deep trenches in silicon substrates [28]. Methods have also shown that a mix of etchant gas consisting of a carbon-fluoride and a secondary gas containing hydrogen can give high etch rate and high etch selectivity [29].
The drawback of using RIE is that the bias and the plasma power are interconnected. So if the bias power is increased for a more directional etch, the ion energy becomes too high causing wafer damage. The sidewall taper is large and hence limits the etch depth. Taper angle optimization is therefore very important as shown in Figure 3. Eventually it proves as a major limitation observed with RIE. Scalloping is another phenomenon observed with RIE samples which limits the use of this technique.

1.2.3 Deep Reactive Ion Etching (DRIE)

The traditional methods of the semiconductor industry, namely, wet etching, RIE, laser cutting, drilling [31] or milling [32] were employed for bulk etching. However it was clear that improvement in the components of several mechanical structures can be brought about by fabrication of thin and deep trenches/holes or tall walls (high aspect-ratio structures). The DRIE tools were introduced to tackle this problem using halogen plasma for silicon etching. The plasma source in the DRIE tools could be Electron Cyclotron Resonance (ECR) or Inductively
Coupled Plasma (ICP). Various plasma source configurations will be addressed with the discussion will mainly revolve around the ICP source.

Since its inception, the ICP technology has been used to fabricate various structures and structural components that help make a multitude of devices. The major advantage of ICP enabled DRIE tools is the possibility to fabricate high-quality, high-aspect ratio features with excellent profile-control. Researchers at Massachusetts Institute of Technology showed the fabrication of a micro gas turbine which used liquid gels to produce electrical power [33]. The design shown in Figure 4 is made possible by the use of DRIE along with wafer bonding techniques.

Another research group at University of Maryland [34] showed the fabrication of a Phase Fresnel Lens using gray scale lithography and DRIE. This device shown in Figure 5 produces a phase difference between certain parts of the beam.

Figure 4 Micro gas turbine showing use of ICP etching [33]
It would be incorrect to say that research in the field of DRIE has only been driven by the MEMS industry. DRIE finds application in the VLSI industry as well. Advances in VLSI technology have produced IC’s with large number of transistors and long bus lines resulting in longer delay. The transistors continue to scale in size reducing the capacitive drive capability but the capacitance of interconnect lines is not known to scale with line dimension. Through-wafer interconnects made using DRIE offer a solution as they use the vertical dimension in enabling the chip stacking applications [35]. Deep silicon etching is also used for the fabrication of deep trench capacitors and deep trench isolation for high power semiconductor devices [36]. When the application requires vertical sidewalls in through-wafer features, DRIE leads the way with ICP being the most widely accepted plasma source. The next section describes the basics of plasma before delving into the dynamics of plasma ignition and etching. The discussion is mainly limited to the effects observed during ICP etching.
1.3 Plasma Etching

Plasma is a state of matter in which the particles are stripped of at least one electron from their outer shells resulting in positively charged ions floating around in the sea of free electrons. The percentage of ionization in plasma depends on the plasma temperature. Hot plasmas, like those involved in nuclear fusion research may be 99% ionized. Cold plasmas, on the other hand have 1-10% ionization.

1.3.1 The world of plasma

It has been noted that plasma forms approximately 99% of the matter in this world [37]. This means that most of the matter in this universe exists in an electrified state where atoms are dissociated into electrons (negatively charged) and positive ions. The composition and atmosphere of stars as well as interstellar hydrogen are all examples of plasma existing naturally. The fact that sky is luminous is because the plasma emits light. In addition to plasmas available in space, we come across plasmas in our daily life as well with the fluorescent tube and neon signs being good examples of man-made plasma. Controlled fusion and high-temperature plasmas have been studied extensively over the years to replicate the process which provides energy to the sun. At the same time, low-temperature plasma physics has been studied extensively for its application in etching of substrates. Further, low temperature plasmas also find application in space science and astrophysics. Plasma processing is a very important process for fabrication of various parts of a semiconductor circuit like interconnects and vias along with various other components which require deep etching. Plasma processing is needed for accessing a parameter space in material processing which is unachievable by chemical methods. It involves using plasmas to provide the activation energy for initiation of surface chemical reactions on the
substrate [38]. Plasma processing is the commercial method for etching away from or deposition of materials on a substrate surface. With feature size becoming smaller and the feature density increasing, the etch profile is increasingly expected to be vertical. Plasma processing provides this control on the sidewall taper. IC manufacturing and sensor production are fields which require a high degree of anisotropy. This has been a major force in the development of the plasma processing technology [38].

1.3.2 A brief history of plasma

The term plasma was introduced by the great Czech medical scientist, Johannes Purkinje, who gave this name to the fluid which remains when the blood is cleared of its various corpuscles in early nineteenth century. In 1927, Irving Langmuir, an American chemist, used the term plasma to describe ionized gases. Langmuir was working on enhancing the lifetime of tungsten filaments at that time. During that research, he put forward the theory of plasma sheaths. These sheaths are the boundary layers which occur between the plasma and solid surfaces [39]. Langmuir studied plasma waves which are a periodic distribution of electron density in a plasma tube. Plasma studies have evolved in various directions [39] - [44], namely:

i. Radio communication studies – E.V. Appleton and K.G. Budden developed the theory of propagation of electromagnetic waves in magnetized non-uniform plasma. This work was done to explain and correct the distortion occurring in radio transmissions depending on the earth’s ionosphere.

ii. Astrophysics – Astrophysicists discovered that the universe was mainly made up of plasma. Hannes Alfven put forward the theory of magnetohydrodynamics (MHD) where plasma is essentially treated as a conducting fluid. This work was done to better
understand the astrophysical phenomena. Applications of this theory have helped understand sunspots, solar winds, star formation and solar flares amongst other topics of astrophysics.

iii. Controlled thermonuclear fusion – Thermonuclear fusion has many a times been touted as the permanent fix for energy issues on the planet earth and has been heavily invested in by the research community. Physicists working on fusion are very interested in understanding the mathematics of plasma and also about the ways of confining plasma.

iv. Space plasma physics – Van Allen radiation belts were discovered in 1958 which led to the development of space plasma physics. The research in all the three previous fields was applied and further results were found.

v. Laser plasma physics – Laser ablation leads to the formation of plasma at the boundary between the beam and the target. This finds application in inertial confinement fusion to focus laser beams on solid matter to create densities and temperatures that are characteristic of nuclear fusion. Laser plasma physics also finds application in particle acceleration as very strong electric fields are produced when a wave passes through an ionized gas plasma. These fields can then be used for particle acceleration.

Plasmas based devices today find application in various other fields apart from particle accelerators and lasers. Pulsed power technology is being used to simulate nuclear weapon effects. Femtosecond lasers make possible the study of very short biological and chemical events. Industrially, plasmas are being used for hardening metal parts in automobiles and airplane turbines. Other industrial applications include making diamond coatings and nitriding to prevent corrosion and abrasion in metal surfaces. The most direct impact of plasmas on our life is through their application in the semiconductor fabrication industry.
1.3.3 Plasma physics

The presence of neutral atoms and lack of complete ionization makes the study of plasmas challenging. Plasma can be considered as a charged fluid with particles undergoing collisions with each other and interacting by long-range electric and magnetic fields. Firstly the fields are modified by the plasma itself so the charged particles in plasma cannot be treated one at a time. Secondly most plasmas cannot be considered as continuous fluids. The particle densities in a typical plasma ($10^9$ cm$^{-3}$) are several orders of magnitude smaller than those found in normal continuous fluids like air ($\sim 3 \times 10^{22}$ cm$^{-3}$) or water ($\sim 3 \times 10^{19}$ cm$^{-3}$).

The exception is low-temperature plasmas which can be easily described by the classical fluid theory of plasmas and the quantum mechanical effects of solid state semiconductors need not be considered. Typically, the temperature of lab plasma would be around 20,000K. The involvement of such high numbers for describing plasma temperatures makes the eV system a better choice. If an electron gains energy equal to KT when it falls through a potential of 1 V, then that temperature is called 1 eV (where K is the Boltzmann’s constant).

The conversion is given by,

$$1 \text{ eV} = 11,600 \degree K$$

It should be noted that the plasma consists of three entities; ions, electrons and neutrals. Each of these has a different temperature ($T_i$, $T_e$ and $T_n$) and density ($n_i$, $n_e$ and $n_n$). So, if the electron density is less when compared to the overall particle density, the temperature of the plasma will be much smaller than the electron temperature, $T_e$. 
1.3.3.1 Condition of quasi-neutrality

Most usable plasmas are only partially ionized. If $n_i$ is the density of ions in the plasma and $n_e$ is the density of electrons, then the ion density and electron density in a plasma are equal and the condition of quasi-neutrality states that,

$$n_i = n_e$$

It should be noted that this condition holds only inside the plasma. In the sheath region, this condition does not hold true.

![Figure 6 Plasma sheath formation](image)

The sheath region exists at the boundary of the plasma with walls, probes or other objects. At these interfaces, a layer of positively charged ions gets created. This layer is called the plasma sheath in which the ions outnumber the electrons. This creates a strong electric field. The walls are negatively charged with respect to the plasma creating a coulomb repulsion for the electrons and keeping them confined to the plasma. If this was not the case, the electrons would escape
from the plasma leaving it positively charged instead of being quasi-neutral. The particle orbits are controlled in the plasma by the electric and magnetic fields. Simultaneously, charge bunches can be formed due to the motion of the charged particles creating electric fields and currents which in turn create magnetic fields.

1.3.3.2 Debye Length and plasma sheath

As mentioned before, the walls of the plasma reactor as well as the substrate are at a negative potential with respect to the plasma. The plasma shields this potential $V_0$ in a few debye lengths. This shielding region is called the sheath. The Debye length is defined as the distance over which the potential gets reduced by a factor of $e$. This “e-folding” distance is given by,

$$\lambda_D = \left( \frac{\varepsilon_0 kT_e}{n_e e^2} \right)^{1/2}$$

$K$ – Boltzmann’s constant

Typical values of $\lambda_D$ in a lab plasma are in the range of 10-80 µm [37].

For partially ionized plasmas, the velocity distribution of the molecules is assumed to be a Maxwell distribution as shown in Figure 7. So,

$$f(v) = A e^{-\frac{\varepsilon_0 m v^2}{2 kT}}$$

A – Normalization factor
m – Mass of the particle
v – Velocity of the particle
T – Temperature

![Figure 7 Maxwell distribution](image)
1.3.3.3 Sheath formation

The sheaths get formed close to the reactor walls inside the plasma chamber and around the substrate. The walls are at a positive potential with respect to the plasma. Therefore electrons have a tendency to escape from the walls. This results in a large number of ions around the walls as compared to electrons. This sheath of positive ions now repels the electrons back in to the plasma helping maintain the quasi-neutrality in the plasma.

In the sheath region, the ions outnumber the electrons and the condition of quasi-neutrality does not hold. As shown in Figure 8, if the wall is assumed to be at \( x = 0 \) then the plasma is extending to the right \( (x > 0) \). The potential drop is shielded within 5 debye lengths \((5\lambda_D)\) shown in the figure as sheath thickness \( s \). This is called Debye shielding and after this distance, \( n_i = n_e \) holds in the plasma. The electron density in the sheath region space is given by:

\[
\frac{n_e}{n_s} = \exp(eV/KT_c)
\]

\( n_s \) – Density of ions and electrons at the sheath edge

The ions enter the sheath at the sheath edge with velocity \( v_s \) and get accelerated due to the E-field. The equation of continuity gives:

\[
n_i v_i = n_e v_s
\]

\( n_i \) – Ion density in the sheath

\( v_i \) – Ion velocity in the sheath
Applying the laws of conservation of energy, we get:

\[ \frac{1}{2} Mv_i^2 + eV = \frac{1}{2} Mv_s^2 \]

\( M \) – Ion mass

Solving the above two equations:

\[ \frac{n_i}{n_s} = \frac{1}{\left[ 1 - 2eV / Mv_s^2 \right]^{1/2}} \]

As ion density in the sheath is greater than the electron density, we can say that:

\[ n_i > n_e \]

This helps us establish that for a sheath:

\[ v_i > (KT_e / M)^{1/2} \]

The above condition is known as the Bohm sheath condition and the velocity in this condition is called the Bohm speed. It states that the ions must enter into the sheath with speed \( v_s \). The sheath drop is such that the ion flux and electron flux leaving the plasma are equal. Therefore the condition of quasi-neutrality is maintained [38].

1.3.3.4 Strongly and weakly coupled plasmas and plasma parameter

If \( n \) is the density of the plasma then the average distance between particles in the plasma is defined by:

\[ r_d \equiv n^{-1/3} \]

The energy of a charged particle is made up of the kinetic energy due to its velocity and the electrostatic energy due to the presence of other charged particles. This energy is given by:
The distance at which the Coulomb energy of one particle vanishes in the electrostatic field of the other, in other terms, the distance of closest approach is given by the following relation:

\[ U(r, v) = \frac{1}{2} m v^2 - \frac{e^2}{4\pi \varepsilon_0 r} \]

The ratio \( r_d / r_c \) determines whether the plasma is strongly related or weakly related. When this ratio is small, the particles are close to each other and their kinetic energies are small compared to their interactive potential energies as they are dominated by each other’s electrostatic influence. Such plasmas are called strongly coupled plasmas. When the ratio \( r_d / r_c \) is large, the particles are far apart and the electrostatic influences are rare because a particle is influenced only by particles in its Debye sphere. Such plasmas are weakly coupled.

The number of particles in a Debye sphere is defined as the plasma parameter given by the following relation:

\[ \Lambda = 4\pi n \lambda_D^3 \]

For weakly coupled plasmas, the plasma parameter is much greater than 1 while for strongly coupled plasmas, it is much smaller than 1. The strongly coupled plasmas are very dense and cold with prime examples being the laser ablation plasma, high-pressure arc discharges, atmospheres of white dwarfs and neutron stars. The weakly coupled plasmas on the other hand are hot and diffused. Examples of these weakly coupled plasmas are encountered in astrophysics, plasma physics and ionosphere. This discussion will consider weakly coupled plasmas with application in plasma physics. Table 1 gives a description of a few plasmas encountered.
1.3.3.5 Gas Discharge Theory

For all practical purposes, only collisions between ions and electrons with neutrals are of interest when gas discharge is studied. A neutral atom has no electric field of its own. The electrons feel the presence of neutrals only when they come within its atomic radius. This momentum transfer cross section that an electron sees depends on its energy (temperature). At very high energies, there is no time for the outer electrons to modify the momentum of the passing electron/ion and hence the effective cross-section is small. At lower energies though, the cross section can be considered constant or can even increase with energy [37].

The frequency of electron-neutral collisions is given by [37]:

\[ \nu_{en} \approx (3.3 \times 10^{13}) p \cdot (10^{-16}) \cdot 6 \times 10^7 T_{eV}^{1/2} \]
\[ \approx 2 \times 10^5 p_{\text{Torr}} T_{eV}^{1/2} \]

\( p \) – Pressure

In the case of ion-neutral collisions, the effective cross section is much larger due to comparable mass of the ions and neutrals. When ions collide with neutrals of the same species, a charge exchange collision takes place. A fast ion colliding with a neutral ionizes the neutral to make it a slow ion and in the process the fast ion becomes a fast moving neutral. So, while the actual momentum exchange in the system is very small, it appears there is a huge change in momentum.
due to the neutral/ion identity change. Hence, charge exchange cross sections are very big. There
is also a possibility of ionization (single or double) as well as excitation when a collision takes
place. This leads to terms such as ionization cross section and excitation cross section. Some ions
have an affinity for electrons giving rise to an attachment cross section. In the case of an
electron-ion collision, the statistical frequency is:

\[
\nu_{ei} \approx 2.9 \times 10^{-5} \frac{n}{T_{eV}^{3/2}}
\]

The ratio of electron-ion and electron-neutral collisions is:

\[
\frac{\nu_{ei}}{\nu_{en}} \approx 1.5 \times 10^{-10} \frac{n}{P} \frac{T_{eV}^{-2}}{n^{2}}
\]

These collisions are equal when:

\[
n_{crit} \approx 6.9 \times 10^{9} p_{\text{mTorr}} \frac{T_{eV}^{2}}{n} \text{ cm}^{-3}
\]

\[n_{crit} \quad \text{Density at which the electron-ion and electron neutral collisions are equal}
\]

\[P_{\text{mTorr}} \quad \text{Plasma pressure}
\]

Electron–ion collisions dominate in high density plasma sources (HDP) whereas low-density
sources like the RIE work with electron-neutral collisions.

When we apply an electric field to plasma, the field has an effect on the motion of electrons as
well ions. Both drift and diffusion happen due to this electric field. The ion and electron flux are
given by:

\[
\Gamma_{e} = -n \mu_{e} E - D_{e} \nabla n
\]

\[
\Gamma_{i} = +n \mu_{i} E - D_{i} \nabla n
\]

\[n \quad \text{Density of the plasma}
\]

\[\mu_{e} \quad \text{Electron mobility}
\]
$\mu_i$ – Ion mobility

D – Diffusion Coefficient

As the mobility and diffusion coefficient of electrons is greater than the corresponding value for ions, the electron flux is much larger than ion flux. To maintain quasi-neutrality, an electric field called an ambipolar field gets generated in the plasma which speeds up the diffusion of ions and retards the diffusion of electrons.

If an electric field is applied to the plasma walls, it gets shielded by the movement of electrons. There is also a small field in the main plasma body which causes the ions to gain the Bohm speed by virtue of collisions or ionization. Hence, some part of the applied field always leaks out to the main plasma. This field is directly proportional to the applied pressure. But the reactors do not work in this high pressure regime, the reasons for this are explained later. Large electric fields are applied by using an inductive coupling. External coils generate a time-varying magnetic field in the plasma. According to the Faraday’s Law an electric field is exerted due to this magnetic field. Electron currents are formed to shield this out as well although in a different manner as explained later in the section discussing plasma sources.

1.3.3.6 Plasma discharge balance

Factors like plasma density, density of neutrals and electron temperature in the bulk determine the reactor performance and need to be considered to design an etch/deposition recipe. These are the factors which have a direct bearing on the ion energy and ion flux incident on the wafer. Equilibrium conditions are required for etching and deposition processes for which the plasma reactors are used.
While electrons are confined by sheaths, the ions keep striking the walls of the chamber and the substrate. This leads to a loss of ions from the plasma. For plasma to remain neutral, these ions need to be replenished. This happens through ionization. Equating the rate of loss of ions and the ionization rate, a relation is obtained between $T_e$ (electron temperature) and $n_n$. Hence, electron temperature is related only to the neutral density and not to the plasma density.

The absorbed energy from the RF antenna by the plasma gives an indication of the equilibrium density of plasma. At the same time, particle loss and radiation of spectral lines contribute to the loss of energy by the plasma. Equating these energies we can calculate the density.

The electron temperature is calculated by balancing the energy loss by heat radiation and the heat gain by conduction (if there the downstream region has no energy input). The ions pick up energy from their collisions with the electrons while losing energy to collisions with neutrals. The ions and neutrals are similar in their mass so the ion-neutral collisions dominate over the ion-electron ones and the ion temperature is close to the neutral temperature.

### 1.3.4 Sources of plasma

Various plasma sources have been explored over the years starting from parallel plate RIE discharge sources to the current high density plasma sources (ECR, ICP). These sources are described with important characteristics and parameters which govern their application. The focus of the discussion is to determine the optimum source for deep silicon etching.

#### 1.3.4.1 RIE discharge reactors

A schematic of RIE discharge reactors is shown in Figure 9. The bottom plate holds the process substrate. There is typically a provision for applying bias to the wafer as well.
Plasma is obtained by applying radio frequency (RF) power to either one or both the plates. The chamber is made up of an inert material like aluminum oxide or steel and is grounded. In a general configuration, the wafer holding plate is grounded and the top plate is biased with a 13.56 MHz RF power supply. The field causes a few electrons to start ionizing the atoms. The electrons resulting from this process cause further ionization and result in an avalanche effect. The wafer gets shielded from the plasma due to the presence of the sheath and ions bombard the wafer due to a potential drop along the sheath. These ions moving towards the wafer cause the etching of the substrate.

These ions can be accelerated by applying a DC bias or a RF bias. In Figure 10 if both plate A and B are set at zero potential, the sheaths developing at both the plates are identical. If the plate B is now made positive, sheath voltage must rise because the sheath drop needs to remain constant. This happens because as soon as the sheath drop becomes smaller, more electrons flow
to plate B than ions and hence the sheath drop rises again to the same level. On the other hand, if plate B is made negative, the extra negative voltage is absorbed by an ion-heavy sheath called as the Child-Langmuir sheath which adds up to the Debye sheath. Hence, the most positive electrode determines the plasma potential.

![Diagram](image)

Figure 10 Illustrating the change in plasma potential when electrode B is biased; (a) No bias; (b) Positive bias; (c) Negative bias

If RF power is given to the plate B, sheath potential follows the potential of plate B during the positive cycle and remains constant during the negative cycle. Hence, the sheath drop will be bigger in the positive cycle of the voltage being applied to plate B as compared to the sheath drop during the negative cycle. The time-averaged sheath drop is going to be larger in the presence of RF as opposed to DC bias. Hence, more energy for anisotropic etching is available in case of RF bias.

RIE etching has its drawbacks as well. Mostly DC bias cannot be applied to the wafer as the wafer has some insulating layers on it. The flip-side is that the RF supply controls the plasma density as well as the ion current. As the ion current is required to be high, we cannot reduce the ion energy impinging the wafer. However we can increase it by having another RF bias oscillator applied to electrode A. Another disadvantage of RIE etching is that a high pressure (~ 100 mTorr) is needed to obtain a reasonable plasma density. However at high pressures, the collision
mean free path is very small and this leads to a very large number of collisions in the sheath. The ions lose their energy and direction in these collisions and this is very harmful for anisotropic etching. The plasma formed in a RIE reactor is not totally conductive and has appreciable resistivity especially at low temperature. This leads to the formation of displacement currents which can end up heating the wafer and the chamber. If the biasing is too high, it can also end up heating the plasma itself and causing changes in plasma density. These factors have resulted in plasma sources which have separate power supply for bias and plasma namely electron cyclotron resonance (ECR) reactors, high density plasma (HDP) reactors and the inductively coupled plasma (ICP) reactors. These sources are discussed in the next few sections.

1.3.4.2 Electron cyclotron resonance (ECR) Sources

ECR sources require a magnetic field such that the electron cyclotron frequency and the applied microwave frequency (2.45 GHz) are in resonance. In order for the microwaves to penetrate through the plasma, the microwave frequency must be greater than the plasma frequency. At microwave frequency of 2.45 GHz, the maximum possible plasma density through these sources is on order of $10^{10}$ cm$^{-3}$. This may seem like a limiting factor because at such a low density of plasma there would not be enough etchants. However plasma densities on order of $10^{12}$ cm$^{-3}$ have been reported with ECR reactors. The 2.45 GHz field has a free space wavelength of 12.2 cm. Therefore the plasma is within the near field where the above limiting condition does not exist.
Major drawback of the ECR system is that due to the physical principle of ECR, the electrons gain enormous amounts of energy and can even generate harmful X-rays on striking the walls of the reactor. The thermal velocities of the electrons ensure that they do not stay in the resonance zone long enough and prevent them from reaching dangerously high levels of energy. ECR sources are not very common in research environment. They have very specific uses like diamond deposition and oxide etching.

Figure 11 ECR source with a large area [37]
1.3.4.3 Inductively Coupled Plasma (ICP) Sources

The name ICP originates from the fact that an external antenna induces the RF field in the plasma. No internal electrodes are needed as is the case with capacitive coupled systems and no DC magnetic field is required like ECR reactors. This RF field can be induced by various configurations thereby giving different names for the ICP sources. TCP refers to Transformer Coupled Plasma while DPS signifies Detached Plasma Source. Typically the ICP source is made up of a spiral coil going around a ceramic cylinder acting as an electromagnet. This coil creates the RF magnetic field inside the chamber which results in an RF electric field according to Faraday’s Law. While the magnetic field is perpendicular to the antenna current, the electric field is in line with the electric current in the coil but in the opposite direction. Just as plasma shields an applied voltage, it also generates currents to shield the applied magnetic field. This is described by the skin depth effect. Skin depth is the region in which the RF field is observed before it decays to become inconsequential. If we assume no collisions to be taking place, the skin depth would be in the order of 0.5 cm for a $10^{12}$ cm$^{-3}$ plasma. The reason we get a uniform distribution of RF power even in the center of the plasma (about 10 cm away) is because ICP systems are not collisionless and collisions increase the skin depth. Once the plasma is formed in the vicinity of the coils, it diffuses in the chamber to become even and uniform.

There are several advantages that ICP possesses over RIE. The wafer bias is separately controlled from the RF potential in the plasma. This allows ion energy to be well controlled. Also these tools have a higher ionization efficiency and more uniform distribution of plasma over the wafer. ICP does not involve the use of any microwave power systems making it a simpler construction.
1.3.5 Plasma chemistry

The plasma is made up of ionized gases which provide the ions and neutrals for etching. This sections details the chemical aspects of the plasma ignition and etching illustrating the chemical reactions involved.
1.3.5.1 Plasma etching mechanism

The following steps explain the mechanism of plasma etching. These steps remain the same even when different reactant species are used which provide the F radicals.

i) Creation of reactive species in the plasma by electron-neutral collisions and chemical reactions. For example:

\[ e^- + CF_4 \rightarrow CF_2 + 2F^* + e^- \]

ii) Transportation/Diffusion of reactive species to the substrate.

\[ F^* \text{(plasma)} \rightarrow F^* \text{(surface)} \]

iii) Physisorption/Chemisorption of reactive species on the substrate surface.

\[ F^* \text{(surface)} \rightarrow F^* \text{adsorbed} \]

iv) Reactant dissociation, formation of surface bonds and formation of desorbing species.

\[ F^* \text{adsorbed} + Si \rightarrow SiF_x \]

\[ SiF_x + F^* \text{adsorbed} \rightarrow SiF_4 \]

v) Desorption of the product.

\[ SiF_4 (s) \rightarrow SiF_4 (g) \]

vi) Transportation/Diffusion of product species back to the plasma.

The following reactions explain the etching mechanism with SF₆.

\[ SF_6 + e^- \rightarrow S_xF_y^+ + S_xF_y^* + F^* + e^- \]

\[ Si + F^* \rightarrow Si-nF \]
1.3.5.2 Spontaneous surface etching and deposition

Fluorine reacts with silicon spontaneously and so does chlorine with aluminum. This reaction takes place even in the absence of any energetic radiation. Of all the steps listed in the previous section, anyone could be the rate-determining step for the etching process. This way some reactions are surface reaction kinetics limited, some are electron impact reaction limited while some are ion-bombardment induced surface kinetics limited. The rate of reactions that are surface reaction kinetics limited is strongly dependent on the surface temperature. These reactions generally give rise to isotropic profiles.

The chemical barrier for chemisorption at room temperature is removed due to the formation of free radicals in the plasma phase. These free radicals react with the substrate after getting adsorbed to the surface. Halogens are the elements of choice for silicon etching. Fluorine is the best followed by chlorine and bromine. The order is due to the size of radicals and steric hindrance that bigger atoms face.

Deposition happens when the reactive radicals have low volatility and high sticking coefficient. This results in the formation of thin films which can be useful as well as harmful in an etching process. The deposition can be conformal as well as non-conformal depending on the sticking coefficient. A low sticking coefficient results in a conformal deposition. When etching silicon, deposition is a necessary process to get good anisotropy [45]. Too much deposition however can be detrimental to the process. The deposition rate and thickness can be controlled by plasma parameters and process time. This is covered more in detail in the next chapter.
Polymerization and etching can happen in very similar conditions and developing a process where one takes place and the other is hindered takes a good knowledge of conditions suitable for etching and deposition. The following graph indicates the effect of C/F ratio, ion bombardment energy, loading and H₂ & O₂ addition on deciding whether the plasma is going to deposit a polymer or etch away at the substrate [46].

The polymer gets deposited when free radicals keep adding up to the polymer chain. CF₄ discharge produces four types of radicals as seen, CF₃, CF₂, CF and F. CF and CF₂ radicals attach to the polymer chain without affecting the probability of attachment of further radicals whereas CF₃ and F radicals diminish the probability of the polymer chain getting elongated.

Figure 13 Boundary between polymer growth and substrate etching [46]
further. Relative concentration of these radicals in the plasma determines the polymerization rate. A high F/C ratio will increase etching and reduce deposition whereas adding H₂ will create HF on reaction with F, reducing the amount of available fluorine radicals and hence increasing polymerization. Addition of O₂ will combine with the carbon atoms and create more free fluorine radicals increasing the etching. However at very high concentrations of O₂, the plasma gets diluted and hence the etching becomes slow again. A high plasma power also increases the formation of F radicals. Ion bombardment results in the breaking of polymer bonds and thus opposes deposition. Hence deposition is more likely to take place on the sidewalls rather than the bottom of the profile. This effect leads to creation of anisotropic features when etching and deposition are done cyclically.

1.3.5.3 Ion bombardment and sputtering

Ion bombardment can result in etching if sufficient momentum is transferred to the surface atoms for them to break loose from the surface potential. For this to happen, the incident particle needs to be within 40 to 70 degrees from normal. If the incident angle is lesser then the momentum transfer does not take place in the proper direction. If the incident angle is more, then the incident particles reflect from the substrate surface.

1.3.5.4 Ion-enhanced chemical etching

Coburn studied the combined effects of ion bombardment and neutral bombardment on substrates [46]. A substrate was bombarded with XeF₂ and Ar⁺ beams separately as well as together. The result shows that a combination of fluorine source and ions resulted in an etch rate an order of magnitude higher than physical sputtering. Ion enhanced etching of surfaces depends
on the flux of incoming particles. The presence of carbon in a precursor gas inhibits the etching of silicon and instead helps in etching of SiO$_2$.

![Graph showing etch rate over time](image)

Figure 14 Combined ion and neutral bombardment gives a high etch rate [46]

### 1.4 The BOSCH Process and this project

Laermer and Schilp developed a process for deep etching of silicon using etch-passivation plasma which is patented by Robert Bosch GmbH [47]. This process was developed to fabricate features with vertical sidewalls and high-aspect ratios. The salient feature of this process was the use of high-speed mass flow controllers (MFCs) to have alternate sequential etching and passivation steps. While Bosch process is a patented technology and the Versaline ICP system is not equipped with the high-speed mass flow controllers required for Bosch etching, this project is aimed at modeling the process development on the basics of Bosch etching method in order to achieve similar etch features with the Versaline system. The apparatus was bought from
PlasmaTherm Systems (formerly Oerlikon systems) in 2008 and the installation of this system was finished in October 2008. Most research groups have published results which have been obtained by working on the Alcatel ICP-RIE system [48] or the STS Multiplex ICP system [49]-[52]. These results unfortunately do not work for this new ICP system due to inherent differences in the way these machines are built. While the basics of plasma etching discussed here remain the same, the performance and working windows permissible for various ICP machines from different vendors are different. The relationship of each controllable ICP system parameter with the plasma is explored in the next chapter. The studies of these relationships paved the way for the development of a time-multiplexed etch-deposition process for deep silicon etching to create periodic patterned micro-pores.

The following chapter describes the equipments used for the experimental work undertaken for developing the etching recipe. Chapter 3 describes the experimental methods and the results. The aim is to use the Versaline ICP system to replicate the kind of vertical sidewall deep features which are a characteristic of Bosch etching.
Chapter 2

Equipments used in ICP etching

2.1 Process flow diagram

A substrate goes through a number of basic processing steps before it is placed in the ICP chamber for etching. The process flow is described in the following figure:

Figure 15 Process flow diagram describing the experimental steps
As can be seen in the process flow diagram, various equipments used during the experiments are the photolithography apparatus (Karl Suss MJB3 Aligner, Wet Bench, Spin coater), e-beam evaporator (metal coating), ICP system (etching), characterization tools (Profilometer, SEM).

This chapter discusses the concepts and the process design considerations involved in working with these equipments.

### 2.2 The Versaline ICP etch system

The Versaline system is a result of years of technical evolution with previous versions being 790, Shuttlelock and Versalock from PlasmaTherm. The Versaline system is equipped with the following features which make it an attractive tool for plasma etching:

1. 2 MHz ICP RF for efficient plasma coupling with the power
2. ICP source with temperature control capable of high power
3. Substrate temperature control via mechanical clamp
4. Efficient thermal management with monoblock electrode construction and an active backside helium circuit
5. Thermally managed chamber liner and pump train
6. Digital MFCs with filtered and bypassed gas lines
7. Available substrate electrode temperature ranges –40°C to 40°C
8. Chambers with corrosion resistant surfaces
9. Mag-Lev turbo pump
10. Bulkhead or ballroom installation
The system comes with easy-to-use software with the following features,

i. User-friendly interface

ii. Comprehensive data logging

iii. Automated cleaning program

iv. Real-time process data display

v. Fully integrated endpoint system

vi. Option to edit recipes during runs

vii. Multiple user access levels

viii. Alarm history
The system makes process development simpler for the user with the following features:

i. Optimized etch rates  
ii. High uniformity  
iii. Low particulates  
iv. Tunable selectivity  
v. Multilayer etch detection  
vi. Profile Control  
vii. Extensive process library
The system offers control over various parameters which have a direct effect on the plasma and the feature geometry that is obtained with the etch process. Figure 16 and Figure 17 show the system available in the Engineering Research Center’s Cleanroom at University of Cincinnati. The variable process parameters are listed in Table 2 and their effect is illustrated in Table 3.

<table>
<thead>
<tr>
<th>Gas Selection</th>
<th>Gas flow rate</th>
</tr>
</thead>
<tbody>
<tr>
<td>Time of etch</td>
<td>Pressure</td>
</tr>
<tr>
<td>Helium Pressure</td>
<td>Electrode Temperature</td>
</tr>
<tr>
<td>Bias Power</td>
<td>ICP Power</td>
</tr>
<tr>
<td>Lid Temperature</td>
<td>Spool Temperature</td>
</tr>
</tbody>
</table>

Table 2 Etch Parameters

<table>
<thead>
<tr>
<th>Result of the process</th>
<th>Independent variable which causes this effect</th>
</tr>
</thead>
<tbody>
<tr>
<td>High Etch Rate</td>
<td>Power levels</td>
</tr>
<tr>
<td>Good Selectivity</td>
<td>Gas Composition</td>
</tr>
<tr>
<td>Profile Control</td>
<td>Etch/Dep Process Ratio</td>
</tr>
<tr>
<td>Anisotropy</td>
<td>Pressure and Temperature</td>
</tr>
<tr>
<td>Notching</td>
<td>Initial Step Parameters</td>
</tr>
</tbody>
</table>

Table 3 Parameter effects on process

### 2.3 Salient features of ICP etching

This section details some of the most significant features of plasma etching which are very important in process design. Most times the process optimization demands balancing one of these parameters at the expense of the other.
2.3.1 Uniformity over the critical dimension

A good wafer design has uniform feature density throughout the wafer. In some cases though, it is not possible to have features distributed all across the wafer. A wafer typically has features of different depths. The etch rate is known to differ with aspect ratio. This effect is better known as Aspect Ratio Dependent Etching (ARDE). Having uniformity is critical for a consistent device performance. The uniformity in etch rate is generally considered in three different aspects. Etch rate should be uniform over a wafer, within an etcher and from batch to batch. Processes are considered good if their uniformities are within ± 3%.

2.3.2 Anisotropy

This feature is generally necessitated by the ever-increasing demands of a higher feature density on the chip. Etched feature sidewalls are expected to be nearly vertical for a high packing density. A perfectly anisotropic process has the anisotropy of infinity with no sideways etching. On the other hand, a perfectly isotropic process has anisotropy of 1.

2.3.3 Selectivity

Selectivity is defined as the ratio of etch rate of the substrate to etch rate of the hard mask. In an etch process its mandatory to have a high selectivity to the material that needs to be etched (Si) over the material that acts as the hard mask (photo-resist or metal, Aluminum in our case).

2.3.4 Etch rate

The etch rate is expected to be high for good system throughput. One of the disadvantages of RIE is that the etch rate is typically very slow (in the range of 0.5 µm/min). Etch rate generally
gets compromised with selectivity and wafer damage. This is because highly energetic radicals are helpful in high etch rates but they also etch the hard mask very aggressively and create wafer damage.

2.3.5 Etch profile control

Perfectly anisotropic etch profiles are not obtainable currently. There is always a slight taper which needs to be controlled and the process conditions have to be controlled in a way that this taper is kept to a minimum. There are other feature profile defects as shown in Figure 18 like bowing, faceting, notching and trenching.

Figure 18 Profile defects; (a) Bowing, (b) Faceting, (c) Notching, (d) Trenching
2.3.6 Wafer and mask damage

The reactive species in the plasma are accelerated by the sheath potential and hit the wafer surface with some energy. When this energy is very high, the incoming ions damage the wafer lattice and create defects that can be harmful to the device. The hard mask can get damaged beyond its tolerance limit and result in the silicon getting etched at places where the mask was supposed to protect it.

2.3.7 Sidewall passivation

Passivation is important for getting anisotropy and smooth sidewalls. Carbon from the etchant halocarbons recombines with halogen radicals and other by-products to form a polymer like substance inside the features protecting the sidewalls from further etching. The directional nature of the incoming ions ensures that there is no polymer formation on the horizontal surface as it gets etched at a much faster rate than its deposition rate. On the other hand, the sideways etching is much slower and can be balanced with the deposition rate thus protecting the sidewalls from etching and giving highly anisotropic features.

2.3.8 Residue removal

By-products of the etch step can either be volatile or non-volatile. Non-volatile by-products like the polymer molecules get deposited in the features while volatile products like SiH$_4$ are pumped out of the chamber. In any case, by-products cover the chamber walls and need to be cleaned up periodically. These residues can be controlled by carefully controlling the temperature, RF bias, backside He cooling and process pressure.
2.3.9 Unwanted features and corrosion

The occurrence of stringers, fences, veils, and crowns is observed during etching at times and these are almost always undesirable. Corrosion is mainly a problem with metal etching. Water vapor (by-product of reactions) reacts with the metal to form corrosive substances.

2.4 Characterization tools

For studying the high aspect-ratio features, characterization tools are required which can measure the etch topology ranging from the etch depth measurement to the surface morphology of the sidewalls and the base of the features. This section talks about the main characterization tools used over the course of this project.

2.4.1 Profilometry

Profilometer works on the electronic measurement of the stylus deflection when it scans over a surface. It gives an indication of the surface morphology/depth of the etch features. Profilometer scans are based on actual contact of the stylus with the surface and give accurate and reliable results on hard surfaces and thin films. These scans can be used to find information about the surface roughness of horizontal (or slightly tapering) features although anything more slanting than the tip angle cannot be characterized.

Depending on the table movement accuracy and tip dimensions, the profilometer horizontal resolution is in the range of 1 µm while the vertical resolution, depending on the sensitivity of the electronic system to external vibrations, is in the range of 2-10 nm.
Profiler systems allow the users to write sequences so that wafer characterization can be automated. Tip angle and the radius of curvature of the tip present the limiting case for the use of profiler system for scanning high aspect-ratio structures. In a typical case, the tip angle is 60 degrees and the tip curvature is 5-10 µm. This limits the use of these systems to features with aspect ratios in the range of 1:1.

Newer systems have a mode known as the dipping mode. In this mode a long and thin stylus is dropped over the feature for scanning and these systems can scan aspect ratios of up to 4:1. The scan area diameter is limited to 10 µm (approx.) and hence presents a limiting case. It is for these reasons that scanning electron microscopy (SEM) or atomic force microscopy (AFM) is used for deeper features.

Even though the stylus presents a limit on the use of profilometer for deeper features, the system still provides a cheap and quick means to identify the depth achieved when test runs of 100-300 seconds are done on the wafers to optimize the etch rate.
2.4.2 Scanning electron microscopy (SEM)

Scanning electron microscopy is a powerful characterization tool with application in various fields of science. It is based on the detection of secondary electrons emitted from the sample surface when it is bombarded with high energy electrons. The principle of SEM therefore limits its use to conductive samples. The insulating and non-conductive samples tend trap forward scattered energetic electrons. The trapped electrons cannot flow to the ground and cause a negative potential on the surface. This surface potential can deflect the high-energy electrons from the primary electron beam to the SEM detector.

![SEM imaging principles](image)

Figure 20 Figure showing SEM imaging principles

SEM scans give excellent information about the surface morphology. The resolution of the SEM scanner in ERC Cleanroom available for this project is 50 nm.
The drawback of SEM is that it is a destructive method of analysis. The samples need to be cleaved to get a good cross-sectional view of the features. The measurements need to be made perpendicular to the surface, as off-perpendicular measurements tend to be inaccurate. This not only destroys the sample but also makes SEM analysis a very time consuming process.

2.5 Summary

Chapter 1 has described the plasma and plasma parameters. Chapter 2 has explained the plasma etching and characterization tools which were used over the course of this project. The next chapter deals with the discussion of ways to develop etch recipes using an ICP system. Plasma processing finds widespread application in the microelectronics industry for etching and thin film deposition. ICP systems have gained a lot of popularity in the recent times. Various ICP systems differ from each other in the way they are designed. Recipes depend on the system construction and hence need to be developed for each type of system. There are various brands of ICP systems available in the market. Basic principles of etching remain the same. The next chapter explores a development of a recipe for making high aspect-ratio deep circular etched features in a silicon wafer using PlasmaTherm’s Versaline ICP etch system. The following experiments have helped characterize this system to benefit other researchers in the university as well as the local research community.
Chapter 3

Process Development

3.1 Overview

This chapter discusses the experiments conducted to obtain a feasible window for plasma etching of silicon wafers. The first few experiments were aimed at finding a set of parameters which can be used to ignite the plasma. Once a set of parameters was discovered, further experiments were conducted to improve the etch rate and plasma intensity. This chapter illustrates the effects of modifying various plasma parameters to get a better etch rate and surface quality. Further, plasmas from different gases were obtained and the etch rates were compared to determine the best working chemistry for etching. Time-multiplexed switched-plasma method of etching through the substrate was explored using various combinations of gases.

3.2 Wafer preparation

3.2.1 Material Selection

Silicon was the material of choice as we aimed at exploring new methods of making patterned deep vertical sidewall features. MEMS industry is a derivative of the IC industry and started working with silicon. Most processing methods for silicon had already been developed for IC fabrication. Silicon possesses good mechanical and peizo-resistive properties making it an ideal material for MEMS devices with control circuitry [36]. Most primitive MEMS structures very rarely cared as to which material was being used for fabrication. Some structures had electronic circuits on them making silicon a perfect substrate material. This project involves making a
patterned array of through-wafer features in silicon using DRIE technology with an ICP source. Table 4 indicates the basic properties of the wafers used in all the subsequent experiments.

<table>
<thead>
<tr>
<th>Property</th>
<th>Value</th>
</tr>
</thead>
<tbody>
<tr>
<td>Resistivity</td>
<td>0.01-0.02</td>
</tr>
<tr>
<td>Orientation</td>
<td>1-0-0</td>
</tr>
<tr>
<td>Diameter</td>
<td>2.25”</td>
</tr>
<tr>
<td>Dopant Type</td>
<td>BN</td>
</tr>
<tr>
<td>Type</td>
<td>p-type</td>
</tr>
</tbody>
</table>

Table 4 Properties of silicon wafers used in the experiments

3.2.2 Wafer Cleaning

Bare silicon wafers can have a lot of scum and impurities present on the surface, resulting in non-uniform etch current. This non-uniform etch current, in turn, results in a non-uniform etch rate across the wafer. Wafer cleaning prevents these undesirable effects and reduces the occurrence of pinholes and other defects in the photoresist layer. It also helps in getting a good adhesion for the photoresist/hard mask layer to silicon. There are various processes available to chemically clean the wafers, viz. acetone cleaning, RCA clean, Bakerclean process [53]. In these experiments, Bakerclean process was used as compared to the RCA for the sole benefit of the ease of procedure which is explained in Table 5.

**Step 1**  HF Dip - 288 ml of H₂O mixed with 12 ml of HF (49%). Dip wafer for 30 seconds. Rinse in DI water for 30 seconds.

**Step 2**  JTB 100 cleaning – 80 ml of JTB 100 mixed with 16 ml of H₂O₂ (30%) and 400 ml of H₂O. Heat solvent to 70 degree Celsius and dip for 10 minutes.
Step 3  Rinse in DI water.

Step 4  Blow dry with N₂ gas.

Table 5 Bakerclean Process

3.2.3 Metal (Aluminum) coating

The clean wafer is coated with aluminum using the e-beam evaporator as shown in Table 6. The aluminum deposition is followed by rapid thermal annealing for 5 minutes at 450 degree Celsius to form a good bond with the silicon. Aluminum acts as an etch stop during etching as the selectivity of Si to Al is very high with fluorine plasma.

<table>
<thead>
<tr>
<th>Metal</th>
<th>Aluminum</th>
</tr>
</thead>
<tbody>
<tr>
<td><strong>Emission Current</strong></td>
<td>200mA</td>
</tr>
<tr>
<td><strong>Deposition Rate</strong></td>
<td>10 Angstroms/sec</td>
</tr>
<tr>
<td><strong>Time</strong></td>
<td>20 seconds</td>
</tr>
<tr>
<td><strong>Total deposition</strong></td>
<td>200 Angstroms</td>
</tr>
</tbody>
</table>

Table 6 Metal Deposition

3.2.4 Lithography

Lithography consists of resist coating, patterning, exposure and developing to transfer the mask patterns on to the wafer.

3.2.4.1 Resist coating

Shipley 1818 (S1818), which is a positive photoresist, was chosen for this application. The choice was simply based on excellent characteristics of Shipley 1818 and the availability in the
Spin coating of S1818 on the clean wafer surface was achieved using the process illustrated in Table 7.

<table>
<thead>
<tr>
<th>Step 1</th>
<th>Place wafer on the resist spinner chuck and secure it with vacuum.</th>
</tr>
</thead>
<tbody>
<tr>
<td>Step 2</td>
<td>Cover three-fourths of the wafer surface with S1818.</td>
</tr>
<tr>
<td>Step 3</td>
<td>Spread cycle is 500rpm for 10 seconds spin cycle is 4000 rpm for 30 seconds.</td>
</tr>
<tr>
<td>Step 4</td>
<td>Put for soft bake i.e. 90-95 degree Celsius for 20 minutes.</td>
</tr>
</tbody>
</table>

Table 7 Photoresist coating

3.2.4.2 Patterning and Exposure

The wafer is placed in Karl Suss MJB3 Aligner and is patterned using a chrome mask. The exposure time is set at 10 seconds.
3.2.4.3 Developing the resist and hard bake

The exposed resist is developed using the Microposit 351 developer. The solution is made as per Table 8.

<table>
<thead>
<tr>
<th>Step 1</th>
<th>Mix 50 ml of Microposit 351 with 250 ml of DI water.</th>
</tr>
</thead>
<tbody>
<tr>
<td>Step 2</td>
<td>Dip wafer in solution for 2 minutes followed by rinsing in DI water.</td>
</tr>
<tr>
<td>Step 3</td>
<td>Dry with N$_2$ gas.</td>
</tr>
</tbody>
</table>

Table 8 Resist developing process

The developed wafers are put into the hard bake oven at a temperature of about 120 degree Celsius for 30 minutes. This makes the cross-linking chains strong enough in the resist so that it withstands the effects of etchants.

3.2.5 Metal removal (metal etch)

The patterns that have been opened up in the resist after development need to be further opened up in the aluminum layer. This is done by the Al etch process explained in Table 9.

<table>
<thead>
<tr>
<th>Step 1</th>
<th>Mix 240 ml of Phosphoric Acid (85%) with 15 ml of HNO$_3$ (70%), 15 ml of Acetic acid (99%) and 30 ml of water.</th>
</tr>
</thead>
<tbody>
<tr>
<td>Step 2</td>
<td>Heat this etchant mixture to 40 degree Celsius.</td>
</tr>
<tr>
<td>Step 3</td>
<td>Dip the wafer for 2 minutes.</td>
</tr>
<tr>
<td>Step 4</td>
<td>Rinse with DI water for 2 minutes.</td>
</tr>
<tr>
<td>Step 5</td>
<td>Dry with N$_2$.</td>
</tr>
</tbody>
</table>

Table 9 Aluminum etching
3.2.6 Resist Stripping

The remaining photoresist should be removed to keep the plasma chamber clean and free from Carbon contamination. The chlorine present in photoresist is known to attack aluminum which is the hard mask in this process. Resist stripping is done by dipping the wafer in another Microposit 351 solution which is much more concentrated (3 parts of Microposit 351 with 2 parts of DI water) than the developer.

3.3 Plasma Ignition

Plasma ignition is based on the combined effect of all the ICP parameters. Getting the plasma to ignite on an uncharacterized machine is the first challenge. The first experiment was based on values provided in Table 10.

<table>
<thead>
<tr>
<th>Process Parameter</th>
<th>Value (Units)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Time</td>
<td>300</td>
</tr>
<tr>
<td>Pressure</td>
<td>70mTorr</td>
</tr>
<tr>
<td>SF$_6$ flow rate</td>
<td>125 sccm</td>
</tr>
<tr>
<td>O$_2$ flow rate</td>
<td>0 sccm</td>
</tr>
<tr>
<td>CHF$_3$ flow rate</td>
<td>0 sccm</td>
</tr>
<tr>
<td>CF$_4$ flow rate</td>
<td>0 sccm</td>
</tr>
<tr>
<td>Ar flow rate</td>
<td>0 sccm</td>
</tr>
<tr>
<td>Bias Power</td>
<td>600W</td>
</tr>
<tr>
<td>Source Power (ICP)</td>
<td>2000W</td>
</tr>
<tr>
<td>Electrode Temperature</td>
<td>10 °C</td>
</tr>
</tbody>
</table>

Table 10 ICP etch basic recipe
Plasma ignition also depends on the chamber characteristics and the distance of the wafer from the plasma source. This is how most chambers differ from each other and to balance the effect of this factor, changes in various parameters were tried out to find a working window.

3.3.1 Effects of pressure variation on plasma ignition

Various parameters were modified to obtain a stable sustained plasma. A high pressure of 70 mTorr was not helping to ignite the plasma so lower values of pressure were tried. As the pressure was reduced below 15 mTorr, faint plasma was obtained which became stronger as pressure was further reduced. Eventually the system’s lower limit of pressure was reached at a value of 7 mTorr. This value provided the best possible plasma intensity. The results are shown in Figure 23.

The chamber pressure has a direct impact on the plasma as shown in Figure 24. Pressure determines the collisionality of the sheath. High pressure means more collisions and hence the ions lose their energy and directionality in the sheath resulting in failure to ignite plasma. These ions will not have enough energy to travel to the substrate. At low pressure the ions are directional and energetic to reach the substrate for etching [35].

Figure 22 SEM images explaining the effect of pressure variations on anisotropy; (a) 11.2 mTorr; (b) 18.2 mTorr; (c) 50.4 mTorr [35]
In Figure 24, it can be seen that as pressure in the chamber is made lower, the ions can attack the substrate with more energy. This has a positive effect in the form of a higher etch rate and better anisotropy (the ions remain directional) and a negative effect in the form of high surface damage (high energy ions strike the surface and remove the surface atoms).
3.3.2 Effects of ICP and bias power on plasma ignition

The plasma was ignited using lower pressure values but it became unstable with the values of ICP and bias power being so high. The system indicated that the values of reflected power were too high. This was causing the system to shut down. To compensate for this effect, the values of ICP and bias power were reduced in steps to identify the allowed upper limit. This experiment is shown in Table 11. High values of ICP and bias power are desirable. The addition of ICP power creates a high energy ion bombardment. This helps in getting the ions through to the substrate. These ions still have enough energy to cause substrate etching. The bias power pulls these charged particles down to the substrate. The purpose of bias power is to create anisotropy. This gives directionality to the ions when they are approaching the substrate [56]. Hence bias power
helps in reducing sideways etching and undercutting. The drawback of having a high value of bias power is that it results in very heavy bombardment of the substrate which may cause surface damage, lattice damage and scalloping [35]. Hence the requirement is to get the plasma to sustain itself with the highest values of ICP and bias power possible and at the same time keeping the surface and lattice damage to a minimum.

<table>
<thead>
<tr>
<th>Bias Power (W)</th>
<th>ICP Power (W)</th>
<th>Result</th>
</tr>
</thead>
<tbody>
<tr>
<td>550</td>
<td>1900</td>
<td>Too high reflected power. System shuts down</td>
</tr>
<tr>
<td>500</td>
<td>1900</td>
<td>Too high reflected power. System shuts down</td>
</tr>
<tr>
<td>400</td>
<td>1600</td>
<td>Too high reflected power. System shuts down</td>
</tr>
<tr>
<td>300</td>
<td>1200</td>
<td>Too high reflected power. System shuts down</td>
</tr>
<tr>
<td>300</td>
<td>900</td>
<td>Too high reflected power. System shuts down</td>
</tr>
<tr>
<td>250</td>
<td>600</td>
<td>Plasma sustains</td>
</tr>
</tbody>
</table>

Table 11 Effect of reflected power on plasma ignition

It was also observed that reducing the amount of SF₆ in the chamber helped in obtaining bright and more stable plasma. Hence, the SF₆ flow rate was reduced to 100 sccm while Ar and O₂ gas were added to the plasma. The addition of Ar stabilizes the plasma and O₂ helps in removing the sulfur deposits from the chamber which can otherwise coat the wafer with a thin film of sulfur and hinder the etching process.

This experiment aimed at getting a working etch step with SF₆ plasma. After various ICP runs and modification in various parameter values, a working recipe was found for the Versaline ICP etcher. This is given in Table 12.
<table>
<thead>
<tr>
<th>Process Parameter</th>
<th>Value (Units)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Time</td>
<td>300 seconds</td>
</tr>
<tr>
<td>Pressure</td>
<td>10 mTorr</td>
</tr>
<tr>
<td>SF&lt;sub&gt;6&lt;/sub&gt; flow rate</td>
<td>100 sccm</td>
</tr>
<tr>
<td>O&lt;sub&gt;2&lt;/sub&gt; flow rate</td>
<td>20 sccm</td>
</tr>
<tr>
<td>CHF&lt;sub&gt;3&lt;/sub&gt; flow rate</td>
<td>0 sccm</td>
</tr>
<tr>
<td>CF&lt;sub&gt;4&lt;/sub&gt; flow rate</td>
<td>0 sccm</td>
</tr>
<tr>
<td>Ar flow rate</td>
<td>20 sccm</td>
</tr>
<tr>
<td>Bias Power</td>
<td>250 W</td>
</tr>
<tr>
<td>Source Power (ICP)</td>
<td>600 W</td>
</tr>
<tr>
<td>Electrode Temperature</td>
<td>10 °C</td>
</tr>
<tr>
<td>Helium Pressure</td>
<td>4000 mTorr</td>
</tr>
</tbody>
</table>

Table 12 Process conditions for basic ICP etch

An etch depth of 1.87 µms was obtained with this recipe giving us an etch rate of 0.37 µm/min.

It can be seen that a higher value of ICP and bias power will be needed to get higher plasma intensity and subsequently a higher etch rate. As we see in Table 11 that the plasma gets extinguished due to reflected power when the wafer is biased above 250 W and provided ICP power above 600 W, a step-wise approach to increase the power rating was explored. Also, a new step is introduced in the beginning to stabilize the gas flow rates before starting the plasma. This helps in getting rid of any residual gases already present in the chamber before the process is started. This gas stabilization step is shown in Table 13.
Time (sec) | Press. (mTorr) | SF\textsubscript{6} (sccm) | O\textsubscript{2} (sccm) | CHF\textsubscript{3} (sccm) | CF\textsubscript{4} (sccm) | Ar (sccm) | Bias (W) | ICP (W) | Temp (°C) | He (mTorr) 
--- | --- | --- | --- | --- | --- | --- | --- | --- | --- | --- 
10 | 10 | 100 | 20 | 0 | 0 | 20 | 0 | 0 | 10 | 4000 

Table 13 Gas stabilization step

After the gas stabilization step, plasma ignition steps are introduced. The bias power is increased in steps of 50 W each while the ICP power was increased in bigger steps of 200 W each. This experiment is shown in Table 14.

<table>
<thead>
<tr>
<th>Process Parameter</th>
<th>Gas Stabilization</th>
<th>Plasma Ignition 1</th>
<th>Plasma Ignition 2</th>
</tr>
</thead>
<tbody>
<tr>
<td>Time</td>
<td>10 sec</td>
<td>5 sec</td>
<td>5 sec</td>
</tr>
<tr>
<td>Pressure</td>
<td>10 mTorr</td>
<td>10 mTorr</td>
<td>10 mTorr</td>
</tr>
<tr>
<td>SF\textsubscript{6} flow rate</td>
<td>100 sccm</td>
<td>100 sccm</td>
<td>100 sccm</td>
</tr>
<tr>
<td>O\textsubscript{2} flow rate</td>
<td>20 sccm</td>
<td>20 sccm</td>
<td>20 sccm</td>
</tr>
<tr>
<td>CHF\textsubscript{3} flow rate</td>
<td>0 sccm</td>
<td>0 sccm</td>
<td>0 sccm</td>
</tr>
<tr>
<td>CF\textsubscript{4} flow rate</td>
<td>0 sccm</td>
<td>0 sccm</td>
<td>0 sccm</td>
</tr>
<tr>
<td>Ar flow rate</td>
<td>20 sccm</td>
<td>20 sccm</td>
<td>20 sccm</td>
</tr>
<tr>
<td>Bias Power</td>
<td>0 W</td>
<td>200 W</td>
<td>250 W</td>
</tr>
<tr>
<td>Source Power (ICP)</td>
<td>0 W</td>
<td>600 W</td>
<td>800 W</td>
</tr>
<tr>
<td>Electrode Temp</td>
<td>10 °C</td>
<td>10 °C</td>
<td>10 °C</td>
</tr>
</tbody>
</table>
| Helium Pressure  | 4000 mTorr        | 4000 mTorr        | 4000 mTorr        

Table 14 Plasma ignition steps. The bias and ICP power were incremented in each step by 50 W and 200 W respectively after Step 3.
This way we reach up to 550W of bias power and 1900W of ICP power. Although this experiment produces a lot of surface damage on the wafer due to high power values, it shows that impedance matching with the load is done in a much easier way with small increments in power as opposed to directly providing a high power input. The effective recipe which becomes the standard recipe for testing the effects of other parameters is shown in Table 15. All the experiments conducted from here on have gas stabilization and plasma ignition steps as explained in Table 14 before the etch step. These steps are not listed in the recipe every time. The input power to the system need not be as high as its upper limit and a moderate value is selected.

<table>
<thead>
<tr>
<th>Process Parameter</th>
<th>Value (Units)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Time</td>
<td>300 seconds</td>
</tr>
<tr>
<td>Pressure</td>
<td>10 mTorr</td>
</tr>
<tr>
<td>SF₆ flow rate</td>
<td>100 sccm</td>
</tr>
<tr>
<td>O₂ flow rate</td>
<td>20 sccm</td>
</tr>
<tr>
<td>CHF₃ flow rate</td>
<td>0 sccm</td>
</tr>
<tr>
<td>CF₄ flow rate</td>
<td>0 sccm</td>
</tr>
<tr>
<td>Ar flow rate</td>
<td>20 sccm</td>
</tr>
<tr>
<td>Bias Power</td>
<td>400 W</td>
</tr>
<tr>
<td>Source Power (ICP)</td>
<td>1400 W</td>
</tr>
<tr>
<td>Electrode Temperature</td>
<td>10 °C</td>
</tr>
<tr>
<td>Helium Pressure</td>
<td>4000 mTorr</td>
</tr>
</tbody>
</table>

Table 15 Standard etch recipe
3.4 Effects of parameter variations on the plasma

The basic working recipe was used as a base to explore the effects of variations of parameters on the plasma intensity and etch rate. The following few sections explain the effects observed in plasma intensity and etch rate when we vary one parameter keeping the others constant. In all the tables below, the basic recipe is same as the one shown in Table 15.

3.4.1 Effects of variation in gas flow rates on plasma

This section illustrates the effects that were observed in the plasma intensity and etch rate when the gas flow rates were altered. A high etch rate is desirable for making high aspect-ratio features. These experiments improved upon the etch rate already obtained while also helped to determine the conditions in which the wafer damage would be reduced and the aluminum layer would hold up better.

3.4.1.1 Effects of variation in SF$_6$ flow rate on plasma intensity

The SF$_6$ flow rate is inversely related to the plasma intensity. This is shown with experimental results in Figure 25.

![Figure 25 Plasma intensity vs SF$_6$ flow rate](image)
3.4.1.2 Effects of variation in SF$_6$ flow rate on etch rate

Over the range of values taken, the etch rate shows an increase with the increase in SF$_6$ content in the plasma. The effect of SF$_6$ on the plasma is described by the chemical equations given below. So, we can see that as the amount of SF$_6$ increases, more etching takes place. Hence we observe that the etch rate shows an increase.

$$\text{SF}_6 + e^- \rightarrow S_x F_y^+ + S_x F_y^* + F^* + e^-$$

$$\text{Si} + F^* \rightarrow \text{Si-nF}$$

$$\text{Si-nF} \xrightarrow{\text{ion energy}} \text{Si-F}_x(\text{ads})$$

$$\text{Si-F}_x(\text{ads}) \rightarrow \text{Si-F}_x(\text{gas})$$

3.4.1.3 Effects of variation in argon flow rate on plasma intensity

Etching through sputtering is obtained by the Ar$^+$ ions in the plasma. These ions gain energy in the sheath and strike the substrate with enough force to knock out an atom.
The effects are shown in Figure 27.

**Figure 27** Plasma intensity vs Argon flow rate

### 3.4.1.4 Effects of variation in argon flow rate on etch rate

Noble gases (usually Ar or He) bring stability and homogeneity to the plasma. This happens primarily due to two main reasons, one being the thermal properties of the discharge gas getting affected and the other being a shift in the electron energy and density whose results are a direct consequence of altered electron balance processes. Addition of argon enhances anisotropic etching because it provides the non-reactive ion bombardment. This helps in gasification of the surface which helps in etching [57]. The experiment and its results are shown in Figure 28.

**Figure 28** Argon flow rate vs. etch rate
Argon provides the neutral molecules for etching but there is a counter-effect on the wafer surface. The wafer surface becomes very rough and damaged. This puts a limit on the amount of argon that can be used to improving the etch rate.

### 3.4.1.5 Effects of variation in $O_2$ flow rate on plasma intensity

$O_2$ is an important gas in the plasma. Its main function is to help in getting an anisotropic etch. This is accomplished by reacting with the silicon present in the craters to form a complex compound. The preferential etching on the bottom of the crater means that the etch rate becomes directional. Addition of oxygen doesn’t change the way plasma gets formed or its density. Oxygen has no role in plasma formation. This is observed experimentally in Figure 29 where plasma intensity is constant with increased oxygen flow.

![Figure 29 Oxygen flow rate vs. plasma intensity](image)

3.4.1.6 Effects of variation in oxygen flow rate on etch rate

Addition of oxygen helps in getting carbon removed from the chamber when using halocarbons for etching. Hence oxygen helps improve the F/C ratio.
The increase in F/C ratio should help improve etch rate but as seen in Figure 31, the etch rate reduces with addition of O\textsubscript{2}. Oxygen aids the formation of a Teflon-type polymer on the walls of the etch features. This layer helps in getting an anisotropic etch but reduces the effective etch rate. Also, at high values of oxygen concentration, the O radicals compete with F radicals in the plasma and result in lowering of the etch rate. In case of SF\textsubscript{6}, this effect is observed even at very small concentrations of oxygen and hence etch rate goes down with more and more oxygen being pumped into the chamber.

![Figure 30 Reactions in the plasma involving oxygen](image)

![Figure 31 Oxygen flow rate vs etch rate](image)
It should also be noted that less oxygen leads to lesser damage on the masking layer of aluminum.

### 3.4.2 Effects of base holding wafer on etch rate

The ICP system takes only 6” wafers. The samples are pasted on base wafers with the help of a silicon/carbon based adhesive. The wafers are primarily silicon wafers.

#### 3.4.2.1 Loading effect

When the surface area exposed to the plasma increases, a drop is observed in the etch rate. This is defined as the loading effect. This is particularly true in cases where major portions of the reactive plasma species are consumed in the reaction. In the case of silicon etching with fluorine plasma, most F radicals are used for etching of silicon. Hence the surface of the carrier wafer affects the etch rate. This effect can be balanced by increasing the flow rate of the reactant species. Although this helps counter the loading effect to an extent, the increased number of unreacted species will need to be removed which may result in an even lower etch rate.

Silicon wafers were coated with Ni and Al to provide surfaces that don’t etch with fluorine plasma. The etch rate is dependent on the selectivity of silicon over the material of the base wafer. The experimental results are shown in Table 16.

<table>
<thead>
<tr>
<th>Base wafer</th>
<th>Etch rate (µm/min)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Nickel</td>
<td>0.63</td>
</tr>
<tr>
<td>Aluminum</td>
<td>0.58</td>
</tr>
<tr>
<td>Silicon</td>
<td>0.41</td>
</tr>
</tbody>
</table>

Table 16 Loading effect in ICP
It can be seen that when the base wafer is silicon, the etch rate is low. When using nickel coated base wafers the etch rate is the highest while aluminum coated wafers also provide a reasonable etch rate improvement over silicon. The selectivity of Ni over Si is better than the selectivity of Al over Si which is mirrored in the results of this experiment. The experiments were done with base wafers coated with aluminum where aluminum was deposited using the e-beam evaporator. As it is also the masking material, the only factor needing consideration was the Al to Si selectivity with fluorine plasma.

3.4.3 Effects of temperature variations on the plasma

Temperature directly affects the spontaneous etching process. High temperature is needed for more spontaneous etching. This leads to an isotropic etch. In features where anisotropy is needed, the temperature is kept at a minimum so that the spontaneous etching is stopped and only the bottom of the feature gets etched owing to bombardment. Cryogenic processes run at very low temperatures and give excellent anisotropy. PlasmaTherm ICP at University of Cincinnati can run at as low as -40 degrees and can give appreciable anisotropy.

3.5 Plasma with various gases

This experiment aimed at comparing the etch rates with different plasma chemistries. CF$_4$ etching and SF$_6$ etching are compared. The reason behind conducting this experiment was to determine the best etchant gas for fluorine plasma etching. CF$_4$ was found to ignite very easily and plasma intensities of even 100% were found. SF$_6$ plasma is comparatively difficult to ignite and plasma intensity obtained with SF$_6$ etch steps was always low.

The results are shown in Figure 32.
Etching plasma can be formed by various different gases. For silicon, gases which give a strong supply of F radicals are used for dry etching. SF₆, CHF₃, CF₄ are all examples of gases which can be broken into F radicals. The F/C ratio is an important criterion in determining which gas can be used for etching. A high F/C ratio makes a gas more suitable for etching purposes. A low F/C ratio in turn makes the gas more suitable for plasma deposition. This happens due to formation of non-volatile polymers on the feature walls when the chamber parameters are favoring such reactions.

Plasma deposition is not an undesirable phenomenon. It can be used to help achieve anisotropy [47]. It can also be used for thin film deposition which is becoming a very big industry. A time-multiplexed etching and deposition plasma approach can be used to create features having very high aspect ratios. In such a process, an etch step is followed by a deposition step which coats the whole feature with a polymer. When we switch back to the etch gas, the directional nature of the incoming ions on to the substrate ensures that the bottom of the feature gets etched at a very rapid rate, much quicker than the sidewalls. So, the polymer at the sidewalls stays for a much

![Figure 32 Plot of etch rate of CF₄ and SF₆](image.png)
longer time than the polymer at the base. This means that while the sidewalls are protected, the base of the feature keeps getting etched. The etching step is stopped when the sidewalls lose their polymer coating. At this stage, the feature is again completely coated with the polymer. A sequence of such steps helps in getting a feature with nearly vertical sidewalls and appreciable depth. This is a concept that was first used by Robert Bosch and the process is patented as BOSCH process [45]. This project aims at developing an indigenous process without using the Bosch equipment and Bosch recipe. The PlasmaTherm Versaline ICP etch system does not provide the luxury of hi-speed switches for switching between etching and passivation plasmas. The etch steps and polymerization steps are consequently longer. Hence the feature evolves in a different manner.

3.6 Time-multiplexed switched-plasma approach

Etch rate is not the only parameter that determines which gas gives a better etch. There is consideration for etch profile, surface roughness and mask damage as well. The following section describes various plasma combinations and the corresponding conclusions which helped develop the final etch recipe.

3.6.1 CF₄ and SF₆ switching

This process was aimed at getting an etch step which has a good etch rate as well as low wafer damage. While SF₆ proved to be the gas which gave the highest etch rate, it also gave the most wafer damage and surface roughness. CF₄ which was simple to ignite and easier to work with was much softer on wafers and the mask. As a result, it was believed that switching the plasma from SF₆ to CF₄ and back may provide an ideal situation where positives from both the etch chemistries can be utilized. The experiment is shown in Table 17.
<table>
<thead>
<tr>
<th></th>
<th>Step 1</th>
<th>Step 2</th>
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</tr>
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</tr>
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<td>60</td>
<td>60</td>
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</tr>
</tbody>
</table>

Table 17 Switching between CF₄ and SF₆ plasma

This experiment gave an etch depth of 2.2 μm in 5 minutes. This means an etch rate of 0.44 μm/min was achieved. A very important observation was that if the process is started with the SF₆ step, the plasma did not ignite but if the CF₄ step is there in the beginning, the process worked well. SF₆ plasma is known to be very hard to ignite. CF₄ on the other hand is very easy to ignite and gives a plasma intensity of close to 100% when provided with enough power. A healthy etch rate with this process provided the inspiration to develop this process to further improve the etch rate. Modified parameters, as shown in Table 18 provided an etch rate of 1.28 μm/min. This etch was obtained by reducing the power and SF₆ flow rate. The improvement in etch rate by doing so indicates that excess SF₆ (excess F radicals) actually impedes the etch process. This happens because all the SF₆ molecules do not get dissociated and hence they need to be removed from the chamber. This in turn reduces the etch rate.
<table>
<thead>
<tr>
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<th>Step 3</th>
<th>Step 4</th>
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</tr>
</tbody>
</table>

Table 18 Modified recipe

### 3.6.2 SF₆ and O₂ switching

SF₆ is the gas with best etch properties and highest etch rates. O₂ is known to help in creating passivation layers in silicon. Switching between etching and passivating plasmas can help create deep etches with high aspect-ratios (vertical sidewall etch). The recipe for this etch process is shown in Table 19.

An attempt was made to explore if reducing the amount of oxygen is going to have an effect on the etch rate. The results of this experiment are shown in Figure 33.

The etch rate obtained in this experiment was lower than the etch rate in CF₄ – SF₆ switching experiment but the wafers obtained with SF₆-O₂ had more vertical sidewall features, as observed in SEM images of the scans.
<table>
<thead>
<tr>
<th></th>
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<th>Step 2</th>
<th>Step 3</th>
<th>Step 4</th>
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</tr>
</tbody>
</table>

Table 19 SF₆ and O₂ plasma switching

![Graph](image)

Figure 33 Effects of varying O₂ flow rate on etch rate in the SF₆-O₂ switching experiment

### 3.6.3 CF₄ and CHF₃ switching

While CF₄ is an etching gas, CHF₃ under certain conditions can become a deposition gas which can cover the feature with a polymer. The switching between two chemistries can lead to anisotropic deep silicon etch features. This prospect was examined and the experiment recipe is listed in Table 20. As the sidewall etching is much slower than the base etching, polymer holds up for a long enough time on sidewalls. This enables us to have a long etch step followed by a
short polymerization step. In this case etch step was 300 seconds while polymerization was 10 seconds.

<table>
<thead>
<tr>
<th></th>
<th>CF&lt;sub&gt;4&lt;/sub&gt; etch</th>
<th>CHF&lt;sub&gt;3&lt;/sub&gt; step</th>
<th>CF&lt;sub&gt;4&lt;/sub&gt; etch</th>
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</tr>
</tbody>
</table>

Table 20 CF<sub>4</sub> - CHF<sub>3</sub> plasma switching; Etch-Deposition process

This recipe gave an etch rate of 0.31 µm/min. This was followed by an experiment where the etch step was made smaller. This new etch step of 60 seconds (as opposed to 300 seconds) was made necessary because the etch profile observed in the previous experiment showed notches on the sidewalls. The etch rate increased to 0.53 µm/min when the etch step times were reduced to 60 seconds. Various other experiments were conducted and the best etch-deposition time cycle ratio obtained was 5:1 for a 50 second etch with 10 second deposition time.

### 3.6.4 SF<sub>6</sub> and CHF<sub>3</sub> switching

Success in the previous experiment led to the exploration of SF<sub>6</sub>-CHF<sub>3</sub> plasma switching. The profiles obtained were neat and undamaged to a large extent. The mask erosion was minimal, giving high selectivity. The sidewalls were smoother than most previous experiments and to top it all, the etch rate was around 1 µm/min, which by now has been accepted as a benchmark for calling a process feasible. Through-etching of wafers cannot be done using processes which are
slower than 1 µm/min. It will take inconsiderable amount of time to get the depth needed. The process will not be cost effective. The development of this recipe took a long while as favorable conditions were examined with the goal to achieve a balance between all the factors listed in this paragraph. The recipe details are given in Table 21.

<table>
<thead>
<tr>
<th></th>
<th>CHF₃ step</th>
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</table>

Table 21 SF₆-CHF₃ switching; Etch - Dep process

A healthy etch rate of 1.16 µm/min was obtained.

The etch profile was further improved by the addition of an O₂ step after every CHF₃ step. The final recipe developed is shown in Table 22. This recipe gave an etch rate of 1.10 µm/min with better sidewalls and very little wafer damage as observed in Figure 34.
<table>
<thead>
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<th></th>
<th>CHF&lt;sub&gt;3&lt;/sub&gt; step</th>
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</table>

Table 22 Final etch recipe

Figure 34 SEM image of the final etch features showing near vertical etch profile
3.7 Summary

The etching of silicon wafers with ICP has been achieved with appreciable results. The differences in the PlasmaTherm system from the different systems in the literature made this project a challenge as results obtained from other reactors could not be put to use for developing the recipe. The results have opened the window for further process improvement while working with this recipe as the base. Effects of variation in various parameters was observed and documented and used to develop a recipe which gives a healthy etch rate and decent surface topology after the etch process. The absence of hi-speed mass flow controllers in the system limited the length of etch and passivation steps providing another hurdle which was crossed by modifying the parameters.
Chapter 4

Conclusions

4.1 Conclusions

This project was successful in developing an etch recipe with a high etch rate for fabricating anisotropic features in silicon wafers with good surface topology and near vertical sidewalls. The project also succeeded in characterizing the newly acquired Versaline ICP Etch System and to develop a process for high aspect-ratio deep silicon etching. The formation of micro-pores has been shown to be possible using the ICP etch technology.

The factors which affect the etch rate are gas flow rates, process pressure, bias power, ICP power, temperature and the material of the carrier wafer. The shape of the features also affects the etch rate. Smaller features are known to etch slower. This is because of the difficulty in removing material from the feature in smaller features. The relationships of these parameters with respect to the plasma and etch rate of the process were studied.

The etch rate of the developed process is 1.10 µm/min which means that ideally, to etch through a wafer of 160 µm thickness, it would take about three hours. The etch process is aspect-ratio dependent and hence as the feature becomes deeper, the rate keeps dropping, suggesting that this process will in fact be longer.

The single most challenge aspect of the fabrication of these features with regards to getting a proper recipe to work was the ignition and sustenance of the plasma. A low value of power
would not ignite the plasma while a high value would give such high reflected power that the plasma tool would shut down. Finding the middle path between the two factors was the first big achievement.

Another challenge was to control the sidewall and surface roughness. The addition of O₂ took care of the sidewall roughness as it forms a Teflon-like polymer at the surfaces to protect them. It was noted that while this deposition took place at all the surfaces, the horizontal surfaces lost this coating much quicker than the vertical surfaces due to the directionality of the incoming ions which cause the etching. The flipside was that addition of O₂ creates oxygen damage on the wafers which increases surface roughness. Once again, experiments were conducted to find the middle path and success was achieved.

The next challenge was to get a high enough etch rate which can help get through-wafer features feasibly. For example, an etch rate of 0.1 µm/min cannot be used for through-wafer etching because it will take a very long time to complete the process and the sidewall/surface morphology will not be as desired.

Recipes being developed were logged in the ICP usage log-book in the Cleanroom and the processes have helped other researchers to develop their own recipes for different types of etching with same/different substrates. Various research groups order wafers from outside which have patterned through-wafer holes in them. With this technology, we can now develop an indigenous recipe for making such wafers and save on precious research time and cost.
4.2 Suggestions for future work and possible applications

The ICP can be used for making features ranging from a few nanometers to features which have feature dimensions in the micron scale. A recipe which works for fabricating features at the small scale is the next logical step in this process. The etch rates are very small when etching nanoscale features and this is a positive phenomenon because it makes the value of etch time very manageable. A process with etch rate of 1 µm/min cannot be used to make a feature which is 20 nm deep.

Once the patterned array of through-wafer features is obtained, it can be used for various applications. One such possible application is a back-side addressable patterned array of carbon nanotubes. For this, the wafer with deep holes is etched from the back side to make nanoscale holes which line up with the microsize holes. This gives such through-wafer features which have the radius of about 50 µm on one side and about 5 nm on the other. The smaller diameter opening can be used to grow vertical CNTs and the larger diameter opening can be used to fill up the hole with a conducting material. This will give control over each CNT for separate programming and the backside of the wafer can still be used for control circuitry allowing smart CNT devices like an e-beam lithography tool or a display device.

Figure 35 Backside addressable patterned CNT arrays
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