Development of an Apparatus Capable of In-Situ Cleaning/Passivation of Silicon Wafers

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For Sergio

You are missed
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Chapter 1

Introduction

1.1 Historical overview

The study of surface interactions, commonly known as “surface science”, has been a topic of interest since as far back as 1774 when noted scientist, inventor, and soon-to-be Founding Father Benjamin Franklin conducted his oil drop experiment after being surprised by how differently oil reacted when placed on a liquid surface; such as water, as compared to a solid surface; such as marble\cite{1}. One of the earliest significant contributions to the field came in 1876 when J.W. Gibbs introduced what the concept of what is now known as “Gibbs Free Energy” that relates the free energy of a system, entropy, enthalpy and temperature \cite{2} by the now famous equation:

\[ \Delta G = \Delta H - T \Delta S_{\text{int}} \]  \hspace{1cm} (1.1)
In the early twentieth century, surface science rose to greater prominence due largely in part to the development of quantum mechanics. Of particular relevance to this thesis is the work done by Irving Langmuir during that time period, in which he introduced the concept of a monolayer [3], and related the adsorption of molecules on a solid surface and pressure by what is known as the Langmuir Equation

\[
\Theta = \frac{\alpha \cdot P}{1 + \alpha \cdot P} \tag{1.2}
\]

where \(\Theta\) is the fractional coverage of the surface, \(P\) is the gas pressure, and \(\alpha\) is the Langmuir adsorption constant [3].

Later, with Katherine Bloedgett, he showed that individual monolayers could be placed, one on top of the other, onto a substrate [4] which paved the way for transistors, integrated circuits, and modern semiconductor manufacturing.

1.2 Monolayers

1.2.1 Surface Energy

If we were to cleave a crystal in an environment that contained no impurities, we would create two new surfaces from parts of the crystal which were formerly in the bulk. The atoms that exist on these newly created surfaces would have an energy higher than they had previously due to the dangling
bonds created by the cleaving. This energy is the surface energy and can be calculated as:

\[ \gamma = \left( \frac{1}{2} \right) N_b \epsilon \rho_a \]  

(1.3)

where \( N_b \) is the number of broken bonds, \( \epsilon \) is the bond strength, and \( \rho_a \) is the surface atomic density of the new surface [5].

A freshly cleaned silicon wafer is exactly analogous to a freshly cleaved crystal. The surface of the wafer is covered in dangling bonds, making the system unstable since it is no longer in the state with the lowest Gibbs free energy. Under normal circumstances, the crystal will not be in an entirely pure environment. Since the rigid nature of the lattice in a crystal at normal temperature prevents it from reducing its surface area to reduce energy, the most likely way it will reduce energy through adsorption of molecules in contact with the surface.

### 1.2.2 Adsorption

Adsorption is the adhesion of atoms or molecules to a surface. In the same year that Langmuir published his paper that related the adsorption of molecules on a surface to pressure, he also published a paper where he looked at adsorption as a density of states problem [6]. From this the adsorption rate can be seen to be
CHAPTER 1. INTRODUCTION

Figure 1.1: Impinging atoms/molecules absorb on areas not already occupied, and reflect off of already occupied areas. This figure assumes that the adsorbed atom/molecule has no dangling bonds of its own.

\[
\frac{d\Theta}{dt} = S \frac{P}{\sqrt{2\pi M kT}} \tag{1.4}
\]

where \( S \) is a sticking coefficient, and \( M \) is the mass of the impinging atom/molecule.

1.2.3 Desorption

Once a monolayer is present on the substrate it may be of interest to remove it. To desorb an atom from the surface of the substrate it must overcome the activation energy for desorption. This can be done via bombardment by energetic particles, heating of the substrate, or if it is energetically favorable, two neighboring atoms may form a bond between themselves and desorb from the surface of the substrate.
Chapter 2

Apparatus

The device consists of four main components: the chamber assembly, the atomic cracker, the sample holder assembly, and the supersonic valve.

Figure 2.1: CAD image of the apparatus, not yet fully constructed
2.1 The Chamber Assembly

The chamber assembly consists of the main body, the vacuum system, the pressure measurement device, and the detection system. The assembly also houses the atomic cracker, the sample holder assembly, and allows entry of molecular beams via the supersonic valve.

2.1.1 Main Body

The main body of the chamber is an 8-inch nipple. Welded to the center of the tubing of the 8-inch nipple are four, 2.75-inch half nipples, placed 90 degrees apart from one another, and one, 4.5-inch half nipple centered 45 degrees from one of the 2.75-inch half nipples. All pieces are constructed from 304 stainless steel, and at the time of this writing can be purchased from MDC (www.mdcvacuum.com). The turbo-molecular pump and the sample holder assembly connect to the 8-inch openings at the top and bottom of the main body respectively. All other connections are annotated in Fig 2.

2.1.2 Vacuum System

To effectively clean and passivate a silicon wafer while in-situ we must be in the ultra-high vacuum (UHV) regime (pressures $\leq 10^{-9}$ torr). When we
Figure 2.2: Top view of the main chamber. Connections are labeled as 1) RGA (reflected beam) 2) RGA (direct beam) 3) Vacuum gauge 4) Beam entry 5) Atomic cracker

invoke kinetic gas theory we can show that the mean free path ($\lambda$) of our particles is

$$\lambda = \frac{kT}{\sqrt{2\pi\xi^2P}}$$

(2.1)

where $k$ is the Boltzmann constant, $\xi$ is the diameter of the particle, $P$ is the pressure of the gas, and $T$ is the temperature. Assuming deuterium to have a diameter of approximately $3 \times 10^{-10}$ m and that we are at room temperature (300K), pressures in the $10^{-9}$ range yield a mean free path of $1 \times 10^6$ m, which is about seven orders of magnitude greater than the size of our chamber. If this were the only thing we had to consider then we would
be able to get away with pressures far greater than $10^{-9}$ torr due to the relatively small size of our chamber. We also have to consider how much time we have between the cleaning and passivation of our wafer. Assuming a sticking probability of unity, a molecular diameter of approximately $3 \times 10^{-8}$ cm and room temperature, kinetic gas theory also tells us that the time it takes for a monolayer to form on a clean surface is

$$
\tau = \frac{2.5 \times 10^{-6}}{P}
$$

(2.2)

where the time $\tau$ is given in seconds and the pressure $P$ is in torr[7]. This means that from the time we clean the wafer we only have approximately 42 minutes before our surface is completely contaminated. The importance of being in the UHV regime becomes obvious now, since pressures at $10^{-8}$ will only allow us about 4 minutes before the surface is contaminated.

To achieve UHV we use a two stage setup: a turbomolecular pump backed by a rotary vane vacuum pump. The rotary vane pump works in the usual gas flow regime (approximately $10^{-2}$ torr) and from there our turbomolecular pump brings us down to UHV. For our roughing pump we selected the Alcatel 2012A, and for our turbomolecular pump we selected the Edwards EXT255H. It should be noted that due to the mounting requirements of the Alcatel 2012A, we were forced to make an adjustment to the chamber. Rather than have the turbopump secured vertically to the 8-inch opening on the main chamber, we installed a t-shaped fitting to the 8-inch opening and
then connected the turbopump to the horizontal opening on the t-fitting. This added chamber volume does not significantly effect the function of the apparatus and so we ignored it for purposes of our calculations.

2.1.3 Pressure Measurement System

For our pressure measurement system we use the HPS Series 943 Cold Cathode Vacuum Gauge System from MKS Instruments.
2.1.4 Detection System

For our detection system we use the SRS Residual Gas Analyzer (RGA). The analyzer works by ionizing incoming atoms then accelerating those ions via a biased focusing ring towards a quadropole [8]. Ions are detected using an integrated electron multiplier. From this we are able to filter out atoms by their charge to mass ratio, which will allow us verify the operation of the atomic cracker as well as test reflected molecular beams from our silicon surface.

2.2 The Atomic Cracker

In order to passivate the surface of our silicon wafer, we must first have a source atomic hydrogen/deuterium/tritium. Atomic crackers are available commercially, but can be prohibitively expensive, so we elected to build our own.

2.2.1 Design

The design of the cracker is simple; it consists of a 4.5 inch blank with two high current feedthroughs (MDC part #9421010 rated for 30A), and a .250 inch diameter gas tube. Molecular hydrogen is introduced into the chamber via a small hole in the bottom of the gas tube. Positioned directly beneath the gas tube is a thin tungsten ribbon tied with tungsten wire to
two tungsten bars which connect back to the electrical feedthroughs. The cracker is powered by the BK Precision 1900 which is capable of 60A at 16V.

2.2.2 Operation

Dissociation of hydrogen occurs by the following reaction:

$$H_2 \rightarrow 2H - 4.5eV \quad (2.3)$$

To achieve a good probability of atomic cracking we need the temperature of our tungsten ribbon to be approximately 2000°C [9]. For a ribbon of thickness $a$ and width $b$ the temperature $T$ results from the equilibrium between the input power due to the Joule effect and radiative losses [10]

$$I = \sqrt{\frac{abc(T)\sigma T^4[2(a + b)]}{\rho(T)}} \quad (2.4)$$
where $\sigma$ is the Stefan-Boltzmann constant, $\epsilon$ is the total emittance which is temperature dependent, and $\rho$ is the electrical resistivity which is also temperature dependent.

Our tungsten ribbon is approximately .002 inches in thickness, .150 inches in width, and .3 inches in length. Using measured values of $\rho$ and $\epsilon$ at different temperatures [11] we were able to extrapolate a value of 60 $\Omega \cdot cm$ for the resistivity and .28 for the emittance. Using these values in formula 8, we were able to determine the current required to maintain our tungsten ribbon at a temperature of 2000$^\circ$C to be 25A.

2.3 The Sample Holder Assembly

2.3.1 Description

The sample holder assembly is the heart of the apparatus. The sample itself is secured to the “tophat” which is broken into two parts, the top and the base, which are both constructed from molybdenum. Initially we were going to have the top screw onto the base, but since the tophat will be heated to high temperatures during operation we elected to go with a different method of attachment to reduce the chance of galling. Two narrow channels were machined into the top that correspond with two posts on the base, to attach one piece to the other you simply align the channels with the posts, then rotate the top 90°.
The tophat attaches to the main body via three screws. To maintain electrical and thermal isolation between the tophat and the main body, the screws are shielded from physical contact with the tophat by alumina washers. A sapphire o-ring prevents direct contact between the tophat and the main body. See Fig 2.7.

The heating element for the sample consists of two tungsten rods that slide through the hole on the main body of the sample holder. To ensure thermal isolation of the main body from the tungsten rods, the rods are encased in an alumina plug. At the tophat end of the tungsten rods a tungsten filament is tied between the two rods. To keep the rods from sliding back and forth they are secured by alumina retaining bars at the front and back of the main body.

The main body itself is composed of 304 stainless steel. Although care has been taken to ensure that the main body is thermally isolated from the rest of the system, a reservoir has been machined into the bottom of the main body to allow for water cooling should the main body become hotter than we desire during operation. The body itself is mounted on a translation stage to allow us to move the sample holder out of the path of the beam so that we can take direct beam measurements.

The sample holder assembly connects to the main chamber through an 8-inch blank with 3, 1 1/3 inch half nipples for feedthroughs (current in/out for the heating element, grounding, and thermocouple) and a 2 3/4 inch half nipple to attach the sample holder to the translation stage.
CHAPTER 2. APPARATUS

Figure 2.6: Close up view of the tophat. Grooves in the top (left) align with posts on the base (right)

Figure 2.7: Exploded view of the sample holder. A) Sample retaining bars B) Silicon wafer C) Tophat top D) Alumina washers E) Tophat base F) Sapphire O-ring G) Main Body
Figure 2.8: The fully assembled sample holder assembly
2.3.2 Cleaning the Sample

Before we can passivate the crystal we must remove surface contaminants. This apparatus was designed to be able to clean a silicon wafer in-situ, however before we put the sample in for the first time we will clean and passivate the wafer ex-situ to allow us to start from an ideal initial surface as well as reduce the amount of contaminants we introduce into our vacuum.

2.3.2.1 Ex-Situ

Prior to putting the silicon wafer into our vacuum chamber we will clean the surface using a wet chemical process commonly known as “RCA cleaning” [14]. The RCA cleaning process is well detailed in [8, 12, 14] so I will not repeat the process here. After the RCA cleaning, the wafer is submerged in a 40% ammonium fluoride solution for 15 minutes to remove the oxide surface left on the wafer from the RCA cleaning, this process is also explained in greater detail in [12].

2.3.2.2 In-Situ

Once the silicon wafer has been placed in vacuum, all cleaning will be done by heating the sample via electron bombardment of the tophat. Because we want an atomically flat surface, care must be taken when heating the sample. The reconstruction of a \((1 \times 1)\) surface to a \((7 \times 7)\) surface in Si \((111)\) occurs at approximately 600°C under UHV conditions [14]. Heating
the sample to approximately 850°C restores the unreconstructed (1 × 1) surface [15]. Since the maximum density of dangling bonds occurs on the unreconstructed surface we want to ensure that it is this and not the (7 × 7) surface that we are passivating, which we do by heating the wafer to 860°C. To calculate the emission current needed to maintain the tophat at 860°C we made a few assumptions: we assumed that all heat loss would be through radiation, we assumed the tophat to be a perfect blackbody radiator, and we assumed that the thermal contact between the top of the tophat and the base of the tophat was poor such that the radiative losses from the surface area of the base of the tophat could be neglected. From there we invoked the Stefan-Boltzmann law

\[ P = A\sigma T^4 \]  
\[ (2.5) \]
(where \( A \) is the surface area of the heated element) to determine that we need an emission current of at least \( 149mA \) given a bias of \( 1000V \) between the filament and the tophat.

To generate the \( 149mA \) emission current that we need to heat the sample, we will build a controller designed for a similar purpose by Dr. Greg Sitz that can provide up to \( 150mA \) emission current at \( 4A \) and \( 3.5V \) [16].
Figure 2.10: Controller circuit provided by [16]
2.3.3 Passivation

Once the silicon wafer has been cleaned, it can be passivated. To start the passivation process, we will first turn on the atomic cracker and introduce atomic and molecular hydrogen to the chamber. We then turn off the heating element in the sample holder so that the silicon wafer can begin to cool. The time it takes for the sample to get back to room temperature is modeled by

\[ Nk \frac{1}{2e\sigma A} \left( \frac{1}{T_{\text{final}}^3} - \frac{1}{T_{\text{hot}}^3} \right) \]  

(2.6)

where \( N \) is the number of particles, \( \epsilon \) is the emissivity (assumed to be 1 as we’re assuming a perfect blackbody radiator), and \( A \) is the surface area of the heated element (which for is the surface area of the top portion of the tophat). We calculated the cooling time to be on the order of five minutes.

While the sample is cooling, particles in the chamber will begin adsorbing on the surface of the wafer. To increase the probability that the adsorbed particle is one we want (hydrogen/deuterium/tritium) and not something else in the chamber, we will adjust the flow of gas into our chamber until the steady state pressure is about two to three orders of magnitude higher than our initial pressure. We expect to be running at \( 10^{-9} \) torr most of the time. At \( 10^{-7} \) torr a monolayer will form on the wafer in approximately 25 seconds by formula 6. Given this, and the time it takes for the sample to get to room temperature, we anticipate a passivated surface within six minutes from the time we turn off the heating element.
2.4 The Supersonic Valve

We expect a fully passivated silicon wafer to have an atomically smooth surface. To test the smoothness of our surface we will measure the intensity of molecular beams reflected off of the surface of the wafer. We use the Even-Lavie valve as our molecular beam source. The beam was developed by Prof. Uzi Even from the Tel Aviv University. The valve can operate with backing pressures of up to 100 atmospheres, producing a very directional (half-angle of $7^\circ$) supersonic beam that is also very monochromatic (less than 1% relative spread in velocity). The valve is capable of pulse durations as short as $10\mu s$ FWHM at a repetition rate of up to 40 Hz. A more detailed description of the valve can be found in [12].

To maximize the detection of specular reflection off of the silicon wafer the chamber was designed such that the incoming beam and the reflected beam detector are separated by $90^\circ$, and our sample holder is oriented such that
the molecular beam will be incident on the sample at 45°.

To get an idea about the quality of our reflected signal, we will compare it to a direct beam signal which will be taken before measurements by lowering the sample holder, allowing the molecular beam an unobstructed path to the direct beam detector.
Chapter 3

Cracker Testing

3.1 Characterizing the Filament

Given that cracking of molecular hydrogen/deuterium does not occur at temperatures lower than $1600^\circ C$ we first had to ensure that our filament was reaching the desired temperatures. Temperature measurements were taken using an optical pyrometer (Land Cyclops 100) pointed at the filament through a view port on the chamber as we scaled the current from 0 to 30 amps. We then annealed the ribbon at $2300^\circ C$ until its resistance stabilized at its minimum value.
CHAPTER 3. CRACKER TESTING

Figure 3.1: The tungsten filament at a) 1700°C b) 2000°C c) 2300°C

Figure 3.2: Temperature vs Current fit for cracker filament
3.2 Cracking Deuterium

In theory our RGA should be able to detect particles as low in mass as 1 amu, however common knowledge has it that in practice the RGA has a hard time detecting mass 1 particles, so we decided to test the cracker with deuterium. The first thing we did was measure our background signal.

As can be seen, the two major contaminants in the chamber are hydrogen and water. Due to time constraints we were unable to bake out our chamber, but a bake out in the future should reduce these signals significantly. One problem of having such a high molecular hydrogen signal is that it shares
Figure 3.4: CAD image of the detector angle with respect to the cracker.

the same charge to mass ratio as the atomic deuterium that we are testing for. We mitigated this by subtracting the background signal from our data, however due to the position of the RGA in relation to the filament, we didn’t expect to see a large direct increase in mass 2 particles anyway. Most of the atomic deuterium is going to fly elsewhere and collide with the chamber walls.

Starting from a pressure of $1.6 \times 10^{-8}$ torr, we then leaked molecular deuterium into the chamber with the cracker turned off until we reached a pressure of $1.1 \times 10^{-6}$ torr, and took a snapshot of our signal. Next, we turned on the cracker and heated our filament to approximately $2000^\circ C$, then took another snapshot of our signal. Figure 3.5 shows much what we expected: with the cracker on we see a decrease in molecular deuterium (mass 4) and a slight increase in atomic deuterium (mass 3). Something important to note
Figure 3.5: Deuterium signal with the cracker on and the cracker off

is the bulge in the signal at mass 3. This corresponds to H-D molecules. The presence of H-D in the chamber can happen via two mechanisms: molecular deuterium colliding with molecular hydrogen in the chamber, or atomic deuterium colliding with the atomic hydrogen that is adsorbed on the chamber walls. Considering the mean free path of the particles in the chamber vs the size of the chamber, collisions between particles in the chamber is extremely unlikely while collision with the chamber wall is almost a certainty. Given this and the fact that we only see the bump when the cracker is on, the existence of a mass 3 signal supports the presence of atomic deuterium in the chamber.
3.3 Conclusion

Having measured our filament temperature to be approximately 2000°C, we introduced molecular deuterium into our chamber. Given the reduction in the mass 4 signal, the increase in the mass 2 signal, and the presence of a mass 3 signal while the cracker is on, it is evident that the cracker is successful as a source of atomic deuterium.
Chapter 4

Future Works

4.1 Assembly and Testing of the Sample Holder

At this point we have built and tested the atomic cracker, but it still remains to build and test the rest of the apparatus. All of the parts required to build the sample holder assembly have either been constructed or can be ordered off the shelf with the exception of the controller for the heating element, whose schematic can be found in Fig 2.10. The supersonic valve is already constructed and only needs to be connected to the main chamber once it is time to test reflections off of the passivated surface.
CHAPTER 4. FUTURE WORKS

4.2 Design Improvements

4.2.1 Main Body of the Chamber Assembly

When we first designed the main body, we neglected the importance of being able to actually look inside the chamber. While testing the cracker, we used blanks with viewports to close off unused sections of our chamber. These viewports were crucial in determining the actual temperature of our tungsten ribbon, and also allowed us to make sure that no parts had detached themselves from the cracker during the heating/cooling process.

Once the entire apparatus is assembled there will be no way to view the interior of the chamber, yet the importance of being able to see inside the chamber will be even greater at this point because we must be able to ensure that parts are aligned properly and that nothing is blocking the path of the beam, and so we suggest either adding viewports to the existing chamber or doing a total redesign on the main body.

4.2.2 The Atomic Cracker

An alternate design for a source of atomic hydrogen has been proposed in [17]. This design differs from ours in that the hydrogen is cracked in the capillary tube that introduces the gas to the chamber, rather than leaking the gas onto a tungsten ribbon, the design also includes a horizontal translation stage that will allow the nozzle of the capillary tube to get very close to the
sample during passivation, but remain out of the way otherwise. It also has an aperture that both collimates the atomic hydrogen and acts as a radiation shield to prevent radiative heating of the wafer. By the time we discovered this article, we had already constructed our cracker and felt confident that what we made would suit our needs. We present this alternate design in the event that our cracker proves insufficient while testing the sample holder assembly.
Bibliography


[16] Per conversation with Greg Sitz